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## INTERNATIONAL CONFERENCE ON RAW MATERIALS TO PROCESSED FOODS

## 03 – 04 JUNE 2021

## CONFERENCE PROCEEDINGS

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### **RPFOODS 2021 CONFERENCE PROCEEDINGS**

2<sup>nd</sup> International Conference on Raw Materials to Processed Foods

#### **Editors**

Prof. Dr. Serkan SELLI / Food Engineering Department, Cukurova University Prof. Dr. Hasim KELEBEK / Food Engineering Department, Adana Alparslan Turkes Science and Technology University

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03-04 June 2021, Turkey

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J. A. ALI WALA - SUDAN	SALİH KARASU
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03-04 June 2021, Turkey

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03-04 June 2021, Turkey

## **Conference Programme**

	International Conference on Raw Materials to Processed Foods 2021					
Scientific Program – Oral Sessions						
	Thursday 3 June 2021					
10:00	10:1     Opening Speech       5     Prof. Dr. Serkan SELLI – Prof. Dr. Hasim KELEBEK –					
10:15	10:4 5		Plenary Session: HydroSOStainable almonds, a model to optimize irrigation water in agriculture: HydroSOStainability PROF. DR. ANGEL ANTONIO CARBONELL BARRACHINA The Miguel Hernández University of Elche, SPAIN -			
10:45	10:5 0		BRI	EAK		
10:50	11:0 5		Concurrent Session 1 Session Chair: Prof. Dr. Angel A. Carbonell Barrachina (The Miguel Hernández University of Elche, Spain) Moderation of polyphenol composition in the cranberry extract powders by spray	Concurrent Session 2 Session Chair: Prof. Dr. Gürbüz Güneş (Istanbul Technical University, Turkey) Nutritional values in fresh beans Leyla İdikut, Esra Odabaşioğlu, Duygu		
			drying parameters and carrier addition <b>Michalska-Ciechanowska Anna,</b> Wojdyło Aneta, Kramek Diana, Brzezowska Jessica, Hendrysiak Aleksandra, Majerska Joanna	Uskutoğlu, Gülay Zülkadir		
11:05	11:2 0		Characterization of aroma-aroma active compounds of qvevri white wines produced from ketengömlek grapes with Gas Chromatography–Mass Spectrometry– Olfactometry (GC–MS–O) <b>Müge Canatar,</b> Merve Darıcı, Turgut Cabaroğlu	Change in the antioxidant activity and total phenolics with thermal treatment and incorporation way of <i>Pistacia terebinthus</i> in ice cream <b>Çağım Akbulut Çakır,</b> Metehan Ergenekon		
11:20	11:3 5			polyphenoloxidase from myrtle berries ( <i>Myrtus communis</i> L.) <b>Fırat Çınar</b> , Salih Aksay		
11:35	11:5 0		Effect of pasteurization on interaction of bioactive compounds in pomegranate-sweet orange mix juice <b>Bhoite Anjali Ashokrao</b> , Gaikwad Nilesh Nivruti, Sathe Shivaji Jagannath, Banerjee Kaushik, Dashrath Oulkar, Zarine Khan	Characterization of volatile composition of mint and oregano obtained from different drying methods Nurten Cengiz, Gamze Guclu, Hasim Kelebek, Serkan Selli, Abhijit Tarawade, Shakhnoza Sultanova, <b>Jasur Safarov</b>		
11:50	12:0 5		Sensory lexicon and major volatiles of rakı using descriptive analysis and GC-FID-MS <b>Merve Darıcı,</b> Koray Özcan, Duygu Beypinar, Turgut Cabaroglu	Effect of modified atmosphere packaging on quality parameters of fresh-cut 'deveci' pears <b>Gozde Oguz-Korkut,</b> Sumeyra Kucukmehmetoglu, Gurbuz Gunes		



12:05	12:2 0	Antifungal activities of essential oils against mycotoxigenic fungal agent <i>Fusarium</i> <i>incarnatum</i> , causal disease agent of pepper fruit rot <b>Soner Soylu</b> , Mehmet Atay, Merve Kara, Aysun Uysal, Emine Mine Soylu, Şener Kurt	Effect of industrial freezing process on the bioaccessibility of carotenoids in organic butternut squash <i>(Cucurbita moschata)</i> <b>Senem Kamiloglu,</b> Elif Koç Alibaşoğlu, Büşra Acoğlu
12:20	12:4 0	BRI	EAK
		<b>Concurrent Session 3</b> <b>Session Chair: Prof. Dr. Parmjit S. Panesar</b> (Sant Longowal Institute of Engineering & Technology, India)	<b>Concurrent Session 4</b> <b>Session Chair: Prof. Dr. Abdul Malik</b> (Aligarh Muslim University, India)
12:40	12:55	, ,	Impact of sodium alginate packaging film with seed extract of <i>Syzygium cumini</i> on Multi-drug resistant <i>Escherichia coli</i> isolated from raw buffalo meat <b>Abdul Malik</b>
12:55	13:1 0		Effect of chia ( <i>Salvia hispanica</i> L.) seed mucilage on lipid oxidation of reduced-fat beef patties <b>Özlem Yüncü</b> , Serpil Kavuşan, Meltem Serdaroğlu
13:10	13:2 5		5
13:25	13:4 0	A comparison of the acid gelation properties of nonfat cow, sheep and goat milks with standardized protein contents <b>Çağım Akbulut Çakır,</b> Ergül Teker	A sensory observation at a house for different fish species stored at room temperature Zafer Ceylan, Oktay Tomar, Abdullah Çağlar, <b>Turgay Çetinkaya</b>
13:40	13:5 5	Optimization of extraction parameters to improve cottonseed milk yield and reduce gossypol levels using response surface methodology (RSM) <b>Thirukkumar S,</b> Hemalatha, Vellaikumar, Amutha	Development of special designed meatballs technology <b>Tanase (Butnariu) Luiza-Andreea</b> , Andronoiu Doina-Georgeta, Nistor Oana- Viorela, Mocanu Gabriel-Danut, Barbu Vasilica-Viorica, Botez Elisabeta
13:55	14:1 0	BREAK	
14:10	14:4 0	Plenary Session: Biotechnologically prepared functional agro-based bioproducts as new types of food/feed supplement with required nutritional design PROF. DR. MILAN CERTIK Slovak University of Technology in Bratislava, SLOVAKIA	



1		Concurrent Session 5	Concurrent Session 6
		Session Chair: Prof. Dr. Apostolos	Session Chair: Prof. Dr. Milan Certik
		Kiritsakis	(Slovak University of Technology in
		(International Hellenic University,	Bratislava, Slovakia)
		Thessaloniki, Greece)	
		The role of olive oil polyphenols in oxidativ	e Grain quality characteristics of local popcorn
		stress, telomeres and aging	populations
14:40	14:5	Apostolos Kiritsakis, Eugenio Luigi Iorio	
14.40	5	Dimitrios Gerasopoulos, Charalampo	
		Anousakis, Kostas Kiritsakis	Seçilmiş
		Allousakis, Kustas Kilitsakis	Dioxins as environmental pollutants
		The Effect of maturation status on fatty aci	
14:55	15:1	profile of <i>xanthium strumarium</i> I. oil	<sup>d</sup> Eyad Aoudeh, Emel Oz, <b>Fatih Oz</b>
	0	Mozhgan Zarifikhosroshahi, Zeynep Ergü	n
		Bioavailibility of olive and olive oil phenoli	ic Chemistry of plant waters – Demystifying
	15.0	compounds	hydrolats
15:10	15:2	Turkan Mutlu Keceli, Senem Kamiloglu	
	5	Esra Capanoglu, Apostolos Kritsakis	-
		Oleuropein extraction from leaves of three	DNA degradation from raw material to
	45.	olive varieties ( <i>Olea europaea</i> L.):	canned products
15:25	15:4	Antioxidant and antimicrobial properties of	
	0	purified oleuropein and oleuropein extracts	
		Semra Topuz, Mustafa Bayram	
		Investigation of fatty acid compositio	n UV-C irradiation for inactivation of Listeria
	15:5	including trans fatty acids and erucic acid i	
15:40	5	selected salty snack foods	Ayse H. Baysal
	J	Tugba Ozdal, Perihan Yolci Omeroglu	, ,
			Elucidation of volatiles, anthocyanins,
			antioxidant and sensory properties of cv.
		Reduced salt Spanish style green table olive	Caner ( <i>Punica granatum</i> L.) pomegranate
4	16:1	(cv. Chalkidiki) preserved in flavored olive o	il Juices produced from three squeezing
15:55	0	Maria Papapostolou, Fani Th	methods
		Mantzouridou, Maria Z. Tsimidou	Jurga Budiene, Gamze Guclu, <b>Kouame</b>
			Fulbert Oussou, Hasim Kelebek, Serkan Selli
10.10	16:3		DEAK
16:10	0	В	REAK
			<b>C</b>
	17.0	Plenary Session:	
16:30	17:0	Ileal-digesting starchy foods can be weight reducing	
	0	PROF. DR. BRUCE HAMAKER	
		Purdue University, USA           Concurrent Session 7         Concurrent Session 8	
			Concurrent Session 8 Session Chair: Prof. Dr. Adamo D. Rombola
		-	(Bologna University, Italy)
			Agroecological Strategies for vineyard
17:1		5	management
5	17:30		Adamo D. Rombola
5		Senay Simsek	
		Senay Simsek	



17:3 0	17:45	Differently structured systems as a carrier for the bioactive sea buckthorn pomace extract <b>Viktorija Eisinaite</b> , Greta Svermickaite Daiva Leskauskaite, Rimas Venskutonis	e conventional and microwave vacuum evaporation: Thermal degradation kinetics of
17:4 5	18:00	Effect of different thermal processing on copper and chromium bioaccessibility from various cereals and pulses <b>Meena Kumari</b> , Kalpana Platel	Vibrational spectroscopy, a versatile technique applied to food science <b>Michele Di Foggia</b>
18:0 0	18:15	Investigation of gluten-free cake production from poppy seed ( <i>Papaver</i> <i>somniferum</i> L.) pulp: TOPSIS application <b>Tuğba Dedebaş</b> , Meryem Göksel Saraç	Feasibility of a chromameter and chemometric techniques to discriminate pure and mixed organic and conventional red pepper powders: A pilot study <b>Muharrem Keskin,</b> Aysel Arslan, Yurtsever Soysal, Yunus Emre Sekerli, Nafiz Celiktas
18:1 5	18:30	Nutritional composition of functiona snack grain-based mix <b>Emir Ayşe Özer</b> , Cahide Yağmur	Effect of roasting and drying on phenolic compounds and color properties of domat variety olive seeds <b>Alev Yüksel Aydar</b> , Tuba Aydin, Büşra Baykan, Tuncay Yilmaz
18:3 0	18:45	Functional food formulated with food industry by-product <b>Meric Simsek</b> , Özge Süfer	Assessment of acrylamide in potato chips and french fries consumed by the romanian population Adriana Laura Mihai, <b>Mioara Negoiță,</b> Gabriela Andreea Horneț
18:4 5	19:00	The effect of activated carbon obtained from hazelnut shell by KOH on the removal of aqueous methylene blue solutions <b>Birsen Sarici,</b> Esra Altıntığ, Sukru, Karatas	Comparison of volatile compounds in sesame oil and sesame cake extract <b>Berfu Gelen,</b> Serkan Selli



03-04 June 2021, Turkey

## **Conference Programme**

International Conference on Raw Materials to Processed Foods 2021						
Scientific Program – Oral Sessions						
	Friday 4 June 2021					
10:00	10:15	Opening	-			
10.00	10.15	Prof. Dr. Serkan SELLI – P				
		Plenary S				
10:15	10:45	Different cooking methods are able to aff rice grown	-			
10.15	10.45	DR. MATTEO	2			
		University of Easter				
10:45	10:55	BRE				
		Concurrent Session 1	Concurrent Session 2			
		Session Chair: Dr. Matteo Bordiga	Session Chair: Prof. Dr. Mohamed			
		(University of Eastern Piedmont, Italy)	Bouaziz (University of Sfax, Tunisia)			
		Improving mulberry shelf-life with combined	Olive by products: low-cost, renewable			
10:55	11:10	effect of 1-Methylcyclopropene with Modified	source of high added value phenolic compounds and their biological and			
		Atmosphere Packaging on fresh black mulberries in cold storage	compounds and their biological and functional activities			
		Fatma Hepsağ, <b>Tefide Kızıldeniz</b> , İbrahim				
		Hayoğlu				
		A comparative study on physicochemical	Development of slow melting dietary fiber-			
		properties and in vitro bioaccessibility of	<b>u</b>			
11:10	11:25	bioactive compounds in rosehip ( <i>Rosa canina</i> L.) infusions treated by non-thermal and	bacterial cellulose and inulin Janifer Raj Xavier, Karna Venkata Ramana			
11.10	11.25	thermal treatments	Samer Raj Aavier, Rama venkata Ramana			
		Gulay Ozkan, Tuba Esatbeyoglu, <b>Esra</b>				
		Capanoglu				
		LC-DAD-ESI-MS/MS characterization of	Determination of chemical component of			
		phenolic compounds in wines from Vitis	essential oil of Origanum dubium plants grown at different altitudes and antifungal			
11:25	11:40	vinifera 'Shesh i bardhë' and 'Vlosh' cultivars	activity against Sclerotinia sclerotiorum			
		<b>Dritan Topi</b> , Hasim Kelebek, Gamze Guclu, Serkan Selli	Musa Türkmen, Merve Kara, Hasan Maral,			
			Soner Soylu			
	11.55		Chemical and sensory characterization of	An assessment of using pea and brown rice		
11:40		kalecik karası wines produced from two	proteins to formulate flexitarian and vegan burger patties			
11.40	11:55	different regions in turkey using chemometrics	Burcu Öztürk-Kerimoğlu, Şimal Bakınay,			
		Merve Darıcı, Turgut Cabaroglu	İrem Nur Öget			
			Comparative evaluation of seed size and			
		FTIR based chemometric analysis of bioactive	growing regions on the chemical			
11:55	12:10	compounds of peach juice during thermal treatment	compositions of raw and roasted NC-7 peanut cultivars			
		Hande Baltacıoğlu	Tulin Eker, <b>Merve Darici</b> , Serkan Selli,			
		·········	Turgut Cabaroglu			
		FT-NIRS and chromameter-based estimation	-			
12:10		of applied microwave power of black carrot				
	12:25	powders Muharrem Keskin, Yurtsever Soysal, Nafiz	obtained from different altitudes of Adana- Turkey			
		Celiktas, Yunus Emre Şekerli	Gürkan Türk, Kemal Şen			
			ş			
12:25	12:45	BRE	AK			



		Concurrent Session 3	Concurrent Session 4	
		Session Chair: Prof. Dr. Esra Capanoglu	Session Chair: Prof. Dr. Ayse H. Baysal	
		(Istanbul Technical University, Turkey)	(Izmir Institute of Technology, Turkey)	
12:45	13:00	Antiglycation potential of freeze-dried powders obtained from different fruit fractions Jessica Brzezowska, Anna Michalska- Ciechanowska, Aleksandra Hendrysiak	Antiglycation potential of freeze-dried powders obtained from fructose into allulose <b>Erva Parıldı</b> , Osman Kola, Bahri Özcan, Murat Akkaya, Elif Dikkaya	
13:00	13:15	Production of cocoa powder with low protein content Konul Mehdizade, <b>İnci Cerit</b> , Omca Demirkol	Volatiles of canned tuna fish and the effects of different parameters <b>Mehmet Yetişen</b> , Serkan Selli	
13:15	13:30	Effect of jam and marmalade processing and storage on phytochemical properties of currant cultivars (Ribes Spp.) <b>Esra Esin Yucel,</b> Cemal Kaya	Migration of lead and cadmium from ceramic kitchenware and estimation of sampling uncertainty Ruya Bulut, Perihan Yolci Omeroglu, Busra Acoglu, <b>Elif Koc Alibasoglu</b>	
13:30	13:45	Totalphenolicandantioxidantbioaccessibilities of cookies enriched with beepollenAyse Neslihan Dundar	1 1 1	
13:45	14:00	Chemical composition and functional properties of <i>Cynara cornigera</i> lindley shoot system extract <b>Melis Sumengen Ozdenefe</b> , Hatice Aysun Mercimek Takcı, Fikret Buyukkaya Kayıs	Ayşe Tülin Öz, Hayrünnisa Yurdakul	
14:00	14:15	Optimization of low fat high protein cookies formulation: effects of using butter and composite flour on nutritional, physical and sensory properties Emir Ayşe Özer, Neslihan Şimşek, <b>Beyza</b> <b>Özpalas</b>	with nutritious ingredients: evaluating a new food product <b>Emir Ayşe ÖZER</b>	
14:15	14:20	BRE	EAK	
		Concurrent Session 5 Session Chair: Prof. Dr. Fatih Oz (Atatürk University, Turkey)	Concurrent Session 6 Session Chair: Prof. Dr. Turgut Cabaroğlu (Cukurova University, Turkey)	
14:20	14:35	Current trends in encapsulation: applications in food science Eda Adal	The impact of various sowing applications on nutritional value of quinoa dry herb <b>Gülay Zulkadir</b> , Leyla İdikut	
14:35	14:50	Black garlic in the food industry Eyad Aoudeh, <b>Emel Oz,</b> Fatih Oz	Effects of different cooking methods on quality of bulgur produced from emmer and einkorn varieties Mehmet Tekin, <b>Ümit Babacan</b> , Orhan Batu, Taner Akar, Mehmet Fatih Cengiz	
14:50	15:05	Modeling of hibiscus anthocyanins transport to apple tissue during ultrasound assisted vacuum impregnation <b>Cüneyt Dinçer</b>		



15:05	15:20	_	A sensory observation for cold stored beef steak and norway salmon Zafer Ceylan, Oktay Tomar, Abdullah Çağlar, <b>Turgay Çetinkaya</b>		
15:20	15:35	Investigation of the use of pekmez in orange nectar production Hande Baltacıoğlu, Merve Ceyhan	Some reological characteristics of kefiran biopolymer isolated from kefir grains biomass <b>Eda Ondul Koc</b> , Mahmut Inal		
15:35	15:50	The effects of ultraviolet light application on the quality of kaymak (clotted cream) during storage period <b>Gamze Sonkaya</b> , Müge Urgu-Öztürk, Gülten Tiryaki Gündüz, Duygu Kışla, Sevcan Ünlütürk, Nurcan Koca	The improvement of rheological properties, emulsion and oxidative stability of low-fat salad dressing by cold pressed hot pepper seed oil by-product <b>Salih Karasu</b>		
15:50	16:05	The effect of different drying methods on drying kinetic, bioactive and color properties of cape gooseberry fruit <b>Esra Avci,</b> Zeynep Hazal Tekin-Cakmak, Selma Kayacan, Salih Karasu	Volatiles Compositions of Strawberry Fruit During Shelf Life Using Pre and Postharvest Hexanal Treatment <b>Ayşe Tülin Öz</b> , Ebru Kafkas		
16:05	16:30	BRE	AK		
16:30	17:00	<b>E</b> vidence that freezing does not signification	Plenary Session: Evidence that freezing does not significantly alter nutrient content of produce PROF. DR. RONALD PEGG		
		Concurrent Session 7 Session Chair: Prof. Dr. Abderrahmane Ait- Kaddour (VetAgroSup, France)	Concurrent Session 8 Session Chair: Prof. Dr. Sudip Chakraborty (University of Calabria, Italy)		
17:15	17:30	Development and characterization of a Exopolysaccharide -functionalized acid whe cheese using <i>Lactobacillus delbrueckii</i> ssp <i>bulgaricus</i> Sara Carrero-Puentes, Carlos Fuenmayor, Carlo Jiménez-Pérez, <b>Francisco Guzmán-Rodríguez</b> Lorena Gómez-Ruiz, Gabriela Rodríguez Serrano, Sergio Alatorre-Santamaría, Mariano García-Garibay, Alma E. Cruz-Guerrero	n Membrane- Food and food industrial y waste treatment 5. <b>Sudip Chakraborty</b>		
17:30	17:45	Reduction or partial substitution of NaCI: Wha effects sensory and biochemical properties of semi-hard cheeses Bord, C, Loudiyi, M, <b>Aït-Kaddour A</b>	-		
17:45	18:00	Investigation of sensorial and physicochemica properties of yoghurt colored with phycocyani of <i>Spirulina platensis</i> <b>Rıdvan Arslan,</b> Salih Aksay	n leaves and roots of two species from the genus Plantago: <i>Plantago major</i> L. and <i>Plantago lagopus</i> L. <b>Anwar Bouali</b> , Héla El Ferchichi Ouarda		
18:00	18:15	Effects of different drying methods on th physicochemical and antioxidant content of "cempedak" ( <i>Artocarpus Integer</i> L.) powder Mhoneswari Gopinathan, Yus Aniza Yusof, <b>Liev</b> <b>Phing Pui</b>	f thermodynamic parameters by thermal		



			Arteaga, Luis Guillermo González- Olivares, Javier Añorve-Morga, Nelly Cruz-Cansino
18:15	18:30	The use of <i>Torulaspora delbrueckii</i> yeast for th production of beer <b>Firuze Kayadelen,</b> Bilal Agirman, Huseyin Erte	basilicum L. via drought stress induced
18:30	18:45	The effect of different fermentation temperatures on şalgam quality Mehmet Ali Cirak, <b>Bilal Agirman,</b> Huseyin Erte	MS and Determination of Antioxidant
18:45	19:00	Elucidation of retro-and orthonasal arom differences of biscuits ( <i>Panis biscoctus</i> ) using artificial masticator Ahmet Salih Sonmezdag, Clement Catanec <b>Cécile Rannou</b> , Serkan Selli, Carole Prost	from cannabis species for use as food additives



	Ir	nternational Conference on Raw Materials to Processed Foods 2021
		Scientific Program – Poster Sessions
		Thursday 3 June 2021
		Reduction or partial substitution of NaCl: What effects sensory and biochemical
10:00	10:10	properties of semi-hard cheeses Bord, C., <b>Loudiyi, M</b> ., Aït-Kaddour A.
		Effect of adding muskmelon (Cucumis melo L.) fruit on physico-chemical properties and
10:10	10:20	sensory attributes of yoghurt
		J. A. Ali Wala, Ibtisam E. M. El Zubeir
		How plant bioregulators affect fruit set and quality attributes of sweet cherry ( <i>Prunus avium</i> L.)
10:20	10:30	Zoran Keserović, Biserka Milić, Jelena Kalajdžić*, Nenad Magazin, Maja Miodragović,
		Gordana Popara
10.20	10:40	Determination of Catechol in Water Extract of Tea using CPE Modified with Banana
10:30	10.40	tissue Nevila Broli, Loreta Vallja, <b>Alma Shehu</b> , Majlinda Vasjari
		Optimization of iron-oligofructose formulation on wheat flour of a high extraction rate
10:40	10:50	on dough rheological properties
		Georgiana Gabriela Codină, Adriana Dabija, Silviu Gabriel Stroe, <b>Sorina Ropciuc</b>
10:50	11:00	Investigation of the Bio-Dynamic Commands Use Effect on Mucilage Content and Germination Behavior in 3 Ecotype of Basil (Ocimum Sp.)
		Mozhgan Sabet Teimouri
		The Comparison Effect of Hydro Alcoholic and Hydro Distilation Extract of Melissa
11:00	11:10	officinalis o Acne and Pimple Giti Sabet Teymouri, Mozhgan Sabet Teimori
		Screening of the antioxidant, nutritional, physical and functional properties of bran
11:10	11:20	obtained from six Indian wheat cultivars
		Reshma Saroj, Vinti Singh, Radha Kushwaha, Monika Singh, <b>Devinder Kaur</b>
		The study of elimination potential of sulfur Blankit (Na2S2O4) and recovery of sugar juice specification in sugar factories using membranous
11:20	11:30	Nano-filtration method
		Mozhdeh Sabet Teymouri, Mozhgan Sabet Teimouri
11.20	11:40	Phytochemical analysis of phenolic compounds, antioxidant and anti-
11:30		acetylcholinesterase activities of extracts from eight populations of <i>Jatropha curcas</i> L. <b>Wafa Ghnimi</b> , Amadou Dicko, Mohamed Larbi Khouja, and Héla El Ferchichi Ouarda
		The stability of mayonnaise model system incorporated with black cumin ( <i>Nigella sativa</i> )
11:40	11:50	seed oil
		Intan Nursyakila Zainal Abidin, Anida Yusoff
11:50	12:00	BREAK
12:00	12:10	Nutraceuticals and Functional foods from Agri-horticulture waste of Indo-Argentina
12.00	12.10	Dhan Prakash, <b>Charu Gupta</b> and Monica Azucena Nazareno
		Effect of CMC and guar gum on oil absorption and sensory quality of banana (Musa acuminate) fritters during repeated frying
12:10	12:20	Norizzah Abd Rashid, Maryam 'Afifah Abdul Latif, Zaibunnisa Abdul Haiyee, Anida
		Yusoff, Maimunah Sanny
		Phytochemical evaluation and antimicrobial activity of selected pigmented plants:
12:20	12:30	Garcinia mangostana, Clitoria ternatea, Ardisia colorata var elliptica and Syzygium cumini
		Noriham A., <b>Siti Azima A. M</b> ., Manshoor, N.
		Advances in Extracting and Understanding the Bioactivities of Marine Organism
12:30	12:40	Peptides: A Review
		Qing-Hao Jin, Ding-Xin Peng, Zhou-Jun Zheng



12:40	12:50	Monosaccharide Removal and Effects of Komagataeibacter Xylinus Fermentation on Antioxidant Capacity and Flavor Profile of Chinese Wolfberry Juice Tianzhen Zhang, Yuqing Shen, <b>Senjia Zhang</b> , Zexiong Xie, Xiyu Cheng, Wenchao Li, Cheng Zhong
12:50	13:00	BREAK
13:00	13:10	Effect of storage on fatty acid composition of hazelnut ( <i>Corylus avellana</i> L.) varieties cultivated in Turkey Hasim Kelebek, <b>Zeynep Ergün</b> , Türkan Uzlaşır
13:10	13:20	Cloud point extraction of lutein and $\beta$ -carotene from spinach waste <b>Bahar Er</b> , Gökhan Durmaz
13:20	13:30	Effect of ultrasound-assisted extraction application on total phenolic substance, catechin and caffein amounts of green tea ( <i>Camellie Sinensis</i> ) extract <b>Esra Esin Yücel</b> , Cemal Kaya
13:30	13:40	Optimization of tray dryer drying parameters of Hacıhaliloğlu apricot using response surface methodology Hamza Bozkir, Ahsen Rayman Ergün, <b>Asuman Adali</b> , Mehmet Güldane
13:40	13:50	Influence of the carrier type and drying methods on the physico-chemical properties of sustainable powders gained from chokeberry pomace extracts Aleksandra Hendrysiak, <b>Anna Michalska-Ciechanowska</b> , Aneta Wojdyło, Jessica Brzezowska
13:50	14:00	Comparison of some local banana types and varieties in terms of physical qualities, pomological properties and phytochemical contents <b>Evren Caglar Eroglu</b> , Rıdvan Arslan, Mustafa Unlu, Rasim Arslan
14:00	14:15	BREAK
14:15	14:25	Assessment of hygiene procedures in fresh fishery products retailers of Lisbon's traditional food markets <b>Rafael S Oliveira,</b> Maria José Rodrigues, Ana Rita
14:25	14:35	Determination of honey adulteration with high fructose wheat syrup Tamer Arslan, <b>Gökhan Durmaz</b> , Oktay Yıldız, Durmuş Özdemir
14:35	14:45	Pollen content in raw Spanish rosemary honey: influence of the geographical origin Isabel Escriche, Marisol Juan-Borrás, Mario Visquert, José-Miguel Valiente
14:45	14:55	Antioxidant potential of milk obtained from the most important breeds of dairy cattle in Poland Jolanta Jola Król, Aneta Brodziak, Magdalena Stobiecka
14:55	15:05	Delineation of molecular structure modification during coagulation of mixed camel and cow milk by mid-infrared spectroscopy and parallel factor analysis <b>Abderrahmane Ait Kaddour</b>
15:05	15:20	BREAK
15:20	15:30	Color quality, ascorbic acid and total carotenoid contents of dried orange slices as influenced by packaging methods and storage conditions Süleyman Polat
15:30	15:40	Volatile profile of Spanish raw citrus honey: the best strategy for its correct labelling <b>Isabel Escriche,</b> Marisol Juan-Borrás, Mario Visquert, Eva Domenech, Andrea Asensio, José-Miguel Valiente
15:40	15:50	The development of melon sorbets with acacia or lavender syrup <b>Oana Viorela Nistor,</b> Doina G Andronoiu, Luiza Andreea Tanase, Gabriel Danut Mocanu, Vasilica Viorica Barbu, Elisabeta Botez
15:50	16:00	Application of response surface methodology (RSM) to optimize the concentrations of essential oils in olive oil used as a preservation means for reduced salt green table olives <b>Maria Papapostolou</b> , Fani Mantzouridou, Maria Tsimidou



16:00	16:10	Functional properties of soy and pea protein isolates <b>Beyza Özpalas</b> , Emir Ayşe Özer
16:10	16:15	BREAK
16:15	16:25	Determination of physical quality and phytochemical properties of prickly pear ( <i>Opuntia ficus-indica</i> ) Evren Caglar Eroglu, Rıdvan Arslan, Ayşegül Güleç, Salih Aksay
16:25	16:35	Sensitivity of biofilms formed by <i>Listeria monocytogens</i> and <i>L. innocua</i> to biocides <b>Sara Atek Lezzoum,</b> Leila Bouayad, Taha-Mossadak Hamdi
16:35	16:45	The preservative potential of essential oils in a real food system: A comprehensive review <b>Mohamed Nadjib Boukhatem</b>
16:45	16:55	Development of mucilage powder from <i>Basella rubra</i> and elderly products application <b>Teerawan Suwan,</b> Nopparat Muangma, Benyapa Soponpattarin, Nattakan Jakkranuhwat
16:55	17:05	Texture and color evaluation of dough and tortillas elaborated by adding solids from nixtamalization waste <b>Francisco Guzmán-Rodríguez</b> , Ruth Peña-Reyes, Lorena Gómez-Ruiz, Gerardo Ramírez-Romero, Alma E. Cruz-Guerrero
17:05	17:15	Determination of some physical and chemical properties and antioxidant activity of cranberry pulp produced with the addition of different sweeteners <b>Hacer Ünver</b> , Memnune Şengül
17:15	17:25	<ul> <li>Prediction and Qualitative Analysis of Sensory Perceptions over Temporal Vectors Using Combination of Artificial Neural Networks and Fuzzy Logic: Validation on Indian Cheese (Paneer)</li> <li>Kartikey Chaturvedi, Sucheta Khubber, Siddhartha Singha3, Himanshu Goel, Francisco J. Barba, Kalyan Das</li> </ul>
17:25	17:35	Rheological, textural and digestibility characteristics of chapatti as affected by incorporation of type 4 Resistant starch prepared from sorghum and corn starch Faiza Shaikh, <b>Tahira Mohsin Ali</b> , Saqib Arif, Lubna Raza, Abid Hasnain
17:35	17:45	Development of value-added functional food by fusion of colored potato and buckwheat flour through hot melt extrusion Md Obyedul Kalam Azad, Md. Adnan, In Je Sung, Jung Dae Lim, Jong-Suep Baek, Young Seok Lim, <b>Cheol Ho Park</b>
17:45	17:55	Effect of quinoa and germinated wheat flour in physicochemical, textural and sensory properties of cupcakes Negin Javaheripour, Lida Shahsavani Mojarrad, Shadi Mehdikhani, Yaser Inanloo, <b>Ali</b> <b>Rafe</b>
17:55	18:05	Impact of low pH on the textural and structural characteristic of canned meatballs (rista) Sajad Ahmad Mir, <b>Shoib Mohmad Wani</b>

	International Conference on Raw Materials to Processed Foods 2021			
Scientific Program – Poster Sessions Friday 4 June 2021				
13:00	13:1 0	The effect of mixing milk of different species on chemical, physicochemical, and sensory features of cheeses: A Review Oumayma Boukria, El Mestafa El Hadrami, Sofiane Boudalia, Jasur Safarov, <b>Françoise</b> Leriche, Abderrahmane Aït-Kaddour		
13:10	13:2 0	Investigating the effect of the Maltodextrin gel usage on oil cake formulation <b>M. Zelanvar,</b> B. Ghiasi Tarzi, P. Damanafshan		
13:20	13:3 0	Development of cakes with Almond Baru flour: characteristics and their correlations with the Texture Profile Analysis Ana Flávia Ramos, Gabriela R. Lemos Mendes, Fabiane Neves Silva, Renato Souza Cruz,		



		Geany Peruch Camilloto, Bruna Mara Aparecida de Carvalho, William James, Nogueira Lima, Milton Nobel Cano Chauca, Janaína Teles de Faria, Juliana Pinto de Lima, Sérgio Henrique Sousa Santos, <b>Igor Viana Brandi</b>
13:30	13:4 0	Effect of cultivar and maturity on functional properties, low molecular weight carbohydrate and antioxidant activity of Jackfruit seed flour Radha Kushwaha, Neha Taslim Fatima, Monika Singh, Vinti Singh, Seeratpreet Kaur, Vinita Puranik, Rajendra Kumar, <b>Devinder Kaur</b>
13:40	13:5 0	Next-Gen probiotics for the management of obesity and weight control Charu Gupta, <b>Dhan Prakash</b>
13:50	14:0 0	2D-Cross correlation spectroscopy coupled with molecular fluorescence spectroscopy for analysis of molecular structure modification of camel milk and cow milk mixtures during coagulation <b>Oumayma Boukria</b> , El Mestafa El Hadrami, Shaxnoza Sultanova, Jasur Safarov, Françoise Leriche, Abderrahmane Aït-Kaddour
14:00	14:1 5	BREAK
14:15	14:2 5	Visible and near-infrared multispectral features in conjunction with artificial neural network and partial least squares for predicting biochemical and micro-structural features of beef muscles Abderrahmane Aït-Kaddour, <b>Donato Andueza</b> , Annabelle Dubost, Jean-Michel Roger, Jean-François Hocquette, Anne Listrat
14:25	14:3 5	Experimental studies of the drying process of plant materials
14:35	5 14:4 5	Jasur Safarov, <b>Gani Dadayev</b> First principles study of structural and electronic, properties of ScxGa1-xN alloys <b>Beloufa Nabil</b> , Benazouzi Aicha
14:55	15:0 5	Storage of herbal raw materials Narzullaev M.S., Jumaev B.M., Khudoyberdiev M.A.
15:05	15:2 0	BREAK
15:20	15:3 0	Research solar water heating drying plant for drying medicinal plants Shaxnoza Abduvaxitovna Sultanova, Tojiniso Tursunboyevna Raxmanova
15:30	15:4 0	Drying herbal raw materials Kanaeva R.N., Samandarov D.I., Yulibayev M
15:40	15:5 0	Effect of cultivar and maturity on functional properties, low molecular weight carbohydrate and antioxidant activity of Jackfruit seed flour <b>Radha Kushwaha</b> , Neha Fatima, Monika Singh, Vinti Singh, Seeratpreet Kaur, Vinita Puranik, Rajendra Kumar, Devinder Kaur
15:50	16:0 0	Effect of endogenous lipids and proteins on the antioxidant and pasting properties of Sorghum bicolor flour <b>Emmanuel Anyachukwu Irondi</b> , Adekemi Esther Adewuyi and Tolulope Muktar Aroyehun
16:00	16:1 0	Comparison of the potential abilities of three spectroscopy methods: near-infrared, mid- infrared, and molecular fluorescence, to predict carotenoid, vitamin and fatty acid contents in cow milk <b>Julien Soulat</b> , Donato Andueza, Benoît Graulet, Christiane L. Girard, Cyril Labonne, Abderrahmane Aït-Kaddour, Bruno Martin, Anne Ferlay
16:10	16:2 5	BREAK
16:25	16:3 5	Quality evaluation of pumpkin ( <i>Cucurbita pepo</i> ) powder produced using three different drying methods <b>Munir Dandago</b>
16:35	16:4 5	Chapatti (flat bread) characteristics as affected by onion peel powder Nabia Siddiqui, <b>Tahira Mohsin Ali</b> , Abid Hasnain
16:45	16:5 5	Drying treatments change the composition of aromatic compounds from fresh to dried centennial seedless grapes Hafiz Umer Javed, Dong Wang, Rani Andaleeb, Muhammad Salman Zahid, Ying Shi, Saeed



		Akhtar, Wang Shiping, Chang-Qing Duan
16:55	17:0 5	Phytosterols as nutraceutical for cardio-vascular diseases Charu Gupta, Dhan Prakash
17:05	17:1 5	Monoacylglycerol and diacylglycerol production by hydrolysis of refined vegetable oil by- products using an immobilized lipase from Serratia sp. W3 <b>Zarai Zied</b> , Ahlem Edahech, Francesco Cacciola
17:15	17:2 5	<ul> <li>Phytochemicals, antioxidant attributes and larvicidal activity of <i>Mercurialis annua</i> L.</li> <li>(Euphorbiaceae) leaf extracts against <i>Tribolium confusum</i> (Du Val) larvae (Coleoptera; Tenebrionidae)</li> <li>Rania Ben Nasr, Amadou Dicko, Hela El Ferchichi Ouarda</li> </ul>
17:25	17:3 5	Novel methods for the extraction of bioactive components and essential oils from foods Ishrat Majid, <b>Shafat Khan</b> , Madhuresh Dwevedi, Aamir Hussain Dar
17:35	17:4 5	The effects of hydrocolloids on the physical properties of sponge cakes Noorlaila Ahmad
17:45	17:5 5	Characterization of Citrus Latifolia by-products (peel and pomace) and their incorporation effect on the quality of cookies Ali Tahir, Aliza Zulfiqar, <b>Miral Javed,</b> Nayyar Iqbal, Muhammad Asif Ismail
17:55	18:0 5	Liposomal encapsulation of omega-3 and lipoic acid conjugate for cow milk fortification <b>Pintu Choudhary</b> , Sayantani Dutta, Moses JA, C. Anandharamakrishnan
18:05	18:1 5	Study on the ozone treatment process on the bacteria damaging nutritive fruits Sathya, R., Showkat Ahmed, L., MubarakAli, D., <b>Jung-Wan Kim</b>



### Biotechnologically Prepared Functional Agro-Based Bioproducts as New Types of Food/Feed Supplement with Required Nutritional Design

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#### ABSTRACT

Development of functional diet enriched with biologically active compounds and their applications in nutrition is one of the strategic targets in food and feed industry. Increasing commercial demand for natural improvement of food/feed functionalities has led for the search of economically and industrially accessible biotechnological methods. Attention has been focused on development of solid-state fermentation (SSF) processes where appropriate microorganisms successfully utilize raw agroindustrial materials and enrich them with biologically active metabolites. Zygomycetous filamentous fungi (Thamnidium sp., Cunninghamella sp., Mucor sp., Umbelopsis sp., Mortierella sp.) are able effectively transform agroindustrial substrates to fermented bioproducts containing polyunsaturated fatty acids (PUFAs), sterols (ergosterol, desmosterol), carotenoid pigments, coenzyme Q, glycolipids, dietary fibres, various enzymes (e.g. amylases, proteases, lipases) and amino-polysaccharides (chitin, chitosan). Fungi also improve content of dry matter, proteins and ash in fermented bioproducts. Depends on the fungal strain and cultivation conditions, a range of agro-based bioproducts enriched with PUFAs (up to 2.4% gamma-linolenic acid, 4.2% arachidonic acid, 2.1% dihomogamma linolenic acid, 2.3% eicosapentaenoic acid) and pigments (0.26% betacarotene) have been prepared and successfully employed for making cereal goods (e.g. rolls, bread and pasta) and tested as a feed additive for animal diet (chicken, egg-laying hens, calves, artificial rumen). These tailor-made microbial-derived bioproducts with improved nutritional and functional properties are generally recognized as safe (GRAS) and represent a challenging and potentially rewarding subject for preparation of new types of food/feed with required nutritional design.

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### Monoacylglycerol and Diacylglycerol Production by Hydrolysis of Refined Vegetable Oil By-Products Using an Immobilized Lipase from *Serratia* sp. W3

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#### ABSTRACT

In the present work, the hydrolysis of lipid fraction by-products of refined vegetable oils was performed by Serratia sp. W3 lipase immobilized on CaCO3. This support was selected out of 4 carriers as it exhibited the finest activity support (950 U/g) and the most satisfactory behavior at use. The immobilized lipase was stable and active in the whole range of pH and temperature, yielding a 75% degree of hydrolysis at optimal environmental conditions of pH 8.5 and temperature 55°C. TLC, GC and LC methods were evaluated to determine the analytical characterization of hydrolysis products. For monoacylglycerols, diacylglycerol fractions identified in the samples, a novel approach by LC method was employed. The adopted approach allowed the use of basic instrumentation set-ups, without the need of sophisticated detectors, such as mass spectrometers. Thus, it could be an effective alternative to produce emulsifiers from cheap vegetable oils.

**Keywords:** by-products; refined vegetable oils, monoacylglycerols, diacylglycerols, immobilized microbial lipase



## Sensitivity of Biofilms Formed by *Listeria monocytogens* and *L. innocua* to Biocides

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#### ABSTRACT

The present study was carried out to assess the sensitivity of biofilms formed by *Listeria monocytogenes* and *Listeria innocua* to 04 biocides which contain amine, quaternary ammoniums and sodium hypochlorite. The bacteria were tested alone and in combinations under different conditions by calculating the percentages of reduction of biofilms.

The results revealed that the disinfectants were more or less effective depending on the nature of the biofilm and the incubation conditions. The highest reduction percentages were observed using the amine and the alkaline quaternary ammonium detergent and disinfectant. *L. innocua* showed more sensitivity to the biocides. The reduction rates of biofilms formed under aerobic conditions were greater than those obtained under microaerophilic conditions. Overall, the percentage reduction in biofilms ranged from 100% to 0%. Also, the percentages of reduction of multi-species biofilms were less than those obtained on biofilms formed by a single bacterial species.

More researches are needed for a better understanding of the phenomena involved in the formation of biofilms and their effects on sensitivity to biocides. These informations would allow manufacturers to adapt their cleaning and disinfection protocols in order to prevent contamination problems and resistance to biocides.

Keywords: biocides, biofilm, L. innocua, L. monocytogenes, sensitivity



### Utilizing Agri-Horticultural Wastes for Development of Functional Foods and Nutraceuticals

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#### ABSTRACT

Antioxidants are known to defuse free radicals leading to limited risk of oxidative stress and associated disorders. Phytochemicals with antioxidant capacity naturally present in food are of great interest due to their beneficial effects on human health as they offer protection against oxidative deterioration. Several epidemiological and in vitro studies have supported the idea that plant constituents with antioxidant activity are capable of exerting protective effects against oxidative stress in biological systems which is among the major causative factors in induction of many chronic and degenerative diseases. Three agri-horticultural wastes like left over residuals from Chenopodium album (Bathua; Family: Chenopodiaceae), Coriandrum sativum (Coriander; Family: Apiaceae), and Brassica oleracea (Cauliflower; Family: Brassicaceae) were selected and studied for their antioxidant activity individually and synergistically to determine their nutraceutical potential by CUPRAC assay method. The antioxidant activity of residuals of chenopodium, coriander and cauliflower were 842.4, 632 and 181.6 µmTE/g (TE= Trolox equivalent) respectively whereas the antioxidant activity of their synergistic combinations of chenopodium and coriander; chenopodium and cauliflower; coriander and cauliflower and all three (chenopodium, coriander, and cauliflower) in combination was found to be 540.8, 928.8, 771.2 and 406.4 µmTE/g respectively. The synergy fold (1.89) was highest in combination of coriander and cauliflower. Thus, the present studies showed that agri-horticultural wastes have tremendous potential to be used as antioxidants and for the development of nutraceutical and functional foods for preventing diseases caused by oxidative stress.

Keywords: Agri-horticultural wastes, Functional foods, Antioxidant activity, Oxidative stress, Nutraceutical



#### **Phytosterols as Nutraceutical for Cardio-Vascular Diseases**

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#### ABSTRACT

Phytosterols are defined as plant sterols and plant stanols that the human body cannot synthesize and therefore originate from the diet. Phytosterols lower total and low-density lipoproteins (LDL) blood cholesterol by preventing cholesterol absorption from the intestine, so they are known as blood cholesterol-lowering agents. Phytosterols are naturally present in fruits, vegetables, nuts and principally oils. Dietary phytosterol intakes normally range from 160-400mg/day with variations depending on food culture and major food sources. Studies have shown that maximum cholesterol lowering benefits are achieved at doses of 2-3g per day. Therefore, today's use implies the need for enriched functional foods, which give enough phytosterols intake thereby contributing to lowering LDL cholesterol levels. Dairy foods remain a food of choice for use as delivery vehicle for many functional ingredients including phytosterols. At the current growth rate of cardio-vascular disease (CVD) throughout world, it is expected that the world market demand for phytosterol fortified products would increase in the near future. There is no doubt that phytosterol as a functional food ingredient will be a new approach to reduce LDL cholesterol through dairy foods and hold a great promise for long term health management. The use of phytosterols in commonly consumed dairy products may soon provide an effective tool against CVD and its introduction in world market is worth anticipating in the near future.

Keywords: Phytosterols, Nutraceuticals, Herbal, Cardio-Vascular Diseases (CVD), Cholesterol, functional foods



### Assessment of hygiene procedures in fresh fishery products retailers of Lisbon's traditional food markets

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#### ABSTRACT

Fresh fishery products consumption has a long tradition in Portugal. Fresh fish retailers (FFR) represent an important link in the food chain continuum, as these food business operators have the responsibility of assuring that perishable fresh fish will get to the consumer as fresh as possible, which rely widely on the prevailing hygienic conditions and practices.

This study aimed to assess hygiene procedures compliance by FFR in Lisbon's traditional food markets. For that, 74 FFR were assessed in 18 food markets. Business owners were interviewed for data collection and an audit was performed, using a specifically prepared checklist considering hygiene requirements, based on European hygiene regulations, including practices and food safety management system (FSMS) and premises assessment.

Business owners' interviews revealed that 68% of the participants were aged 50 or plus and 15% were over 70 years old; while 7% were illiterate, the majority (74%) had basic education level. Additionally, most of the participants (80%) had a basic training in food hygiene and safety but were in need of an update. Audit results demonstrated that only 15% of the FFR presented FSMS related documents, such as the ones contemplated in the hygiene program. Most of the operators (73%) lacked hot water for hand-washing purposes. Even though some FFR wore reusable rubber gloves, hand-washing procedures were in most cases incomplete, and gloves were not considered in the regular hygiene practices. Regarding food-contact surfaces, most of the FFR did not use a proper cleaning method, still 64% applied a sanitizer to disinfect fresh fish contact surfaces, such as cutting boards, trays and exposition benches.

Taken together, our results emphasize the need for a thorough training on hygiene practices and food safety management systems of FFR, as well as an enhancement of these retailers' premises in traditional food markets.

**Keywords:** Traditional Food Markets, Fresh Fish Retailers, Hygiene Assessment, Food Safety Management Systems

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## Effect of different thermal processing on copper and chromium bioaccessibility from various cereals and pulses

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#### ABSTRACT

The present investigation was undertaken to study the influence of heat processing on the total content and bioaccessibility of copper and chromium from various cereals and pulses. The bioaccessibility of these minerals was determined by employing an *in vitro* dialysability procedure. Microwave cooking, pressure cooking, and open-pan boiling were the three heat treatment methods used in this study. The copper bioaccessibility in different cereals and pulses was increased as a result of microwave cooking, pressure cooking and open-pan boiling ranging from 34 to 99%, 16 to 131% and 18 to 133%, respectively. Similarly, the chromium bioaccessibility was also increased as a result of these three heat processing methods in cereals and pulses ranging between 23 to 126%, 15 to 98%, and 30 to 58%, respectively. The bioaccessibility of copper and chromium in most of the analyzed grains was enhanced by pressure cooking; hence, it was the most efficient method. A general enhancing effect of heat processing on copper and chromium bioaccessibility from food grains was observed. These methods can be implemented to improve the same from cereals and pulses. This is the first paper to report the effect of domestic processing on the bioaccessibility of trace minerals.

Keywords: Bioaccessibility, Cereals, Heat processing, Pulses



#### Comparative Evaluation of Seed Size and Growing Regions on The Chemical Compositions of Raw and Roasted NC-7 Peanut Cultivars

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#### ABSTRACT

This study examined the impact of seed size, roasting process, growing region and their interactions on chemical composition of NC-7 peanut cultivar from Turkey. Peanut seeds obtained from Osmaniye and Adana regions of Turkey were sized into two market grades as 7 mm and 11 mm and roasted until obtained equal skin color. It was found that the moisture, total sugar, stachyose, raffinose sucrose and amino acid values of small sized samples were generally significantly higher than the larger ones. Region, size and process effects were found significant on oleic (O) and linoleic (L) acid content of samples. Significantly higher O/L ratios were obtained for the large seeds. Significant size x process interactions were indicated for O/L ratio and linoleic acid content of samples. Larger size seed especially in Osmaniye region seems to have a positive effect on the shelf life of peanut seeds in regard to O/L ratio. In Osmaniye region, raw samples showed higher concentration for detected sugars than Adana region. Sucrose and stachyose contents of both seeds decreased after roasting in Osmaniye, while a slight increase was obtained in Adana region. It was determined that roasting process generally caused a decrease in the amount of amino acids. It was elucidated that seed size, growing regions and roasting process were crucial factors for the chemical compositions of peanut seed samples.

Keywords: Peanut, Growing Region, Size, Roasting, Sugar, Amino Acid



### **Chemistry of Plant Waters – Demystifying Hydrolats**

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#### ABSTRACT

Hydrolats or often referred to as aromatic or plant waters are obtained after steam distillation of aromatic plants in the case of essential oil production. They are enriched not only with small amounts of aromatic herbal substances, but also with valuable non-volatile but water-soluble compounds such as: polyphenols, flavonoids, sugars, organic acids, or vitamin C. Most plants with low amount of essential oil are ideal for hydrolat production. Such products are much safer to use for kids, pregnant women, and sensitive people.

In our research volatile organic compounds (VOCs), total phenolic content (TPC) and radical scavenging activity of six different hydrolats of aromatic plants: birch (*Betula pendula* L.), yarrow (*Achillea millefolium* L.), mountain pine (*Pinus mugo* L.), linden (*Tilia cordata* L.), elder (*Sambucus nigra* L.), meadow-sweet (*Filipendula ulmaria* L.) growing wild in Lithuania were investigated.

Plant botanical name	DPPH <sup>-</sup> , Trolox mM/L	TPC, mg/L
Betula pendula	9.4±0.05	18.7±0.2
Pinus mugo	8.2±0.03	17.8±0.1
Tilia cordata	9.7±0.07	1.5±0.05
Sambucus nigro	8.1±0.01	2.3±0.1
Fillipendula ulmaria	8.6±0.02	178.0±0.5
Achillea millefolium	20.2±0.1	2.8±0.05

Table 1. Radical scavenging ability and total phenolic content of six investigated hydrolats

Despite the exceptionally low content of volatile organic compounds in hydrolats, the main ones were those whose therapeutic properties are well known. The TPC for all six investigated hydrolats varied widely however their radical scavenging ability were remarkably similar, except yarrow. It could be assumed that this is related to the amount and composition of volatile organic compounds in the hydrolats.

Keywords: Hydrolats, VOCs, Total Phenolic Content, radical scavenging ability



### Elucidation of Volatiles, Anthocyanins, Antioxidant and Sensory Properties of cv. Caner Pomegranate (*Punica granatum* L.) Juices Produced from Three Juice Extraction Methods

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#### ABSTRACT

This study deals with the characterization of the phytochemical profiles and antioxidant activities of cv. Caner pomegranate (*Punica granatum*) juices obtained from three different juice extraction methods including halved pomegranate (HPJ), arils (AJ), and macerated arils (MAJ) extraction for the first time. It was found that the type of the juice extraction process had substantial effects on the volatiles, anthocyanin compositions, and antioxidant activities of the samples. Results showed that the AJ sample (593 mg L–1) had more anthocyanin compounds followed by HPJ (555 mg L–1) and MAJ (408 mg L–1) samples. GC-MS analysis revealed a total of 34 volatile compounds. The highest number of volatiles was found in the MAJ sample (1872  $\mu$ g L–1); thus, the aril maceration process played an important role in increasing the volatiles as compared to the HPJ (751.8  $\mu$ g L–1) and AJ (710.7  $\mu$ g L–1) samples. Sensory analysis showed that the HPJ sample was the most preferred and its general impression was higher as compared to the AJ and MAJ samples. The findings of this study elucidated that the juice extraction technique had a significant influence on the phytochemical profiles, sensory quality, and antioxidant activity of pomegranate juices.

Keywords: pomegranate; cv. Caner; juice extraction methods; aroma compounds; anthocyanins; antioxidants

#### INTRODUCTION

Pomegranate fruit (*Punica granatum* L.) belonging to the Punicaceae family is primarily considered to be a crucial source of bioactive compounds that are claimed to possess health beneficial properties. Thus, recently, there has been a massive increment in the popularity of pomegranate consumption [1]. This fruit, originating from the Middle East, has been a widely known fruit since ancient times. The total global production of pomegranate is reported to be over 3 million tons, and Turkey is an important producer with a total annual production of 581.189 tons [2]. The interest in pomegranate fruits is growing year by year not only because it is pleasant to eat, but also because it is a fruit with a good source of minerals, acids, sugars, vitamins, polysaccharides, and phenolics such as anthocyanins and phenolic acids [3]. Anthocyanins are well known to function as natural antioxidants and play a role in the protection against oxidative stress, reduction of risks of chronic diseases, as well as prevention of their progression [4,5]. Recently, numerous studies demonstrated that there has been a relation between the intake of fruits and vegetables comprising natural antioxidants and the inhibitions of many diseases including cancers [6,7]. Pomegranate fruits are generally consumed fresh, but lately, there is a substantial demand in industry to obtain pomegranate juice, jams, jelly, vinegar, and wine [8]. The overall quality of pomegranate cultivars depends on its taste components, aroma profiles, and color



properties. Among these, aroma is a vital quality criterion for foods affecting the consumer's acceptance and preference to a higher extent. These compounds, also known as volatile organic compounds, can be chemically classified as aldehydes, alcohols, acids, ketones, esters, lactones, and terpenes. These constituents are also present in very low concentrations in food samples, and due to their different molecular characteristics, every single aroma compound has different contributions to the final aroma of a food sample [9]. Even if exhaustive works are present in the literature concerning the health effects of pomegranates and their juices, only a small portion of them focus on volatile compounds. In one of the most comprehensive papers, it is reported that a mixture of volatiles responsible for the green, fruity, floral, and earthy notes composes the pomegranate juice aroma [10]. In addition, Beaulieu et al. [11] investigated the quality properties and aroma components in sweet, sweet-sour, and sour pomegranate cultivars from around the world that were grown in a collection of California-grown pomegranates from the National Clonal Germplasm Repository. They found that aldehyde and terpene compounds characterize cultivar differences and 3-hexenol and 1-hexanol were the dominant compounds. Another important group of compounds in pomegranates are phenolics. They have great importance because of their role for quality parameters such as color, taste, and their favorable impacts on health [1]. Pomegranates are very rich in terms of anthocyanins, ellagic acid, phytoestrogenic flavonoids, and tannins, which have the ability to act as antioxidant properties [12,13]. Among these, anthocyanins are the key pigments in pomegranate cultivars found in various parts of the pomegranate trees, leaves, flowers, peels, and fruits. In the extant literature, six anthocyanin molecules were determined in different pomegranate fruits, including mono- and di-glucosides of cyanidin, delphinidin, and pelargonidin [14]. In addition to their contribution to sensory profiles, they are also important for health with their antitumor, antioxidant, antimicrobial, and anti-inflammatory effects. As pomegranates and their products are rich in phenolics, specifically anthocyanins, they display these health beneficial properties, and [14,15]. Kostka et al. [15] extracted and separated pomegranate polyphenolics into an anthocyanin and copigment fraction with the aid of the adsorptive membrane technique. They elucidated that the total phenolics and free radical scavenging activity were considerably higher in the XAD-7 extract compared to the juice and that anthocyanins and copigments act together in decreasing oxidative stress. Pomegranate fruit, being rich in these mentioned healthbeneficial and bioactive compounds, requires handling with care. The processes like juice extraction to be applied on this fruit may result a loss in desired compounds, so the conditions of the procedures should be decided carefully to get the highest benefit from the compounds. There has been no comprehensive work on the color quality, volatiles, anthocyanins, sugar, and organic acid constituents of cv. Caner pomegranate juice produced from different juice extraction methods in the extant literature. So, this work was established to assess the influence of three juice extraction methods (halved fruit, arils, and macerated arils extraction) on the amounts and types of the volatiles, anthocyanins, antioxidant properties, sugars, and organic acids of cv. Caner pomegranate. Besides, in the work that was conducted in this comprehensive manner for the first time, a sensory study was also utilized for investigating the influence of three different juice extraction techniques.

#### MATERIALS AND METHODS

#### **Pomegranate Juice Samples**

Pomegranate fruits of cv. Caner were harvested from the Cukurova University's experimental orchards located in Adana province of Turkey at the full ripeness stage. Ten kilograms of pomegranates were used for each batch. Fruits were washed with tap water, dried with towels, and divided into two parts. Three different juice extraction methods were employed to obtain the juice samples. Pomegranates from the first batch were cut into two pieces and the juice was obtained by a hydraulic stainless-steel bladder press (Speidel, Bayrakli-Izmir, Turkey) at 2 atm pressure and this sample was called as halved pomegranate juice (coded by "HPJ"). The second sample (coded as "AJ") was extracted from the arils by pressing at 2 atm pressure using a hydraulic press without crushing the seeds. For the last sample, arils of the fruit were separated from the skin (membrane) manually then they were crushed without damage to their seeds and kept at 4 °C for 3 h for maceration (coded "MAJ"). After maceration, the juice was extracted with the same procedure used for the AJ sample. Juices



were frozen and stored at  $-20 \circ C$  until analyses.

#### **General Chemical Analysis**

The pH and total soluble solids were determined immediately after obtaining the juice samples with the use of a pH meter (Orion 3 STAR pH Benchtop Meter, Thermo Scientific, Waltham, MA, USA) and a refractometer (Carl Zeiss, Jena, Germany) (expressed as °Brix), respectively. The titratable acidity of the juice samples was determined by titration with NaOH (0,1 N) [16]. The color of juice was quantified using a colorimeter (ColorQuest XE, HunterLab, Reston, VA, USA) and expressed as L\* (lightness), a\* (redness), and b\* (yellowness). The average values of the triplicate measurements were reported.

#### Analysis of Sugars and Organic Acids

Analysis of sugars and organic acids were carried out according to the method of Lee and Coates [17] with slight modifications. Pomegranate juices were centrifuged ( $6000 \times g$ , 15 min), filtered through a 0.45 µm filter, and diluted 1:1 with ultra-pure water. The obtained extract was directly injected to the HPLC system equipped with LC-20AD, SPD20A UV and RID 10 A detectors (Shimadzu, Kyoto, Japan). HRC NH2 column (Biorad,  $150 \times 4.6$  mm, 5 m) was used in both analyses. The mobile phase consisted of 5 mM H2SO4 solution with a flow rate of 0.5 mL min–1. Glucose and fructose standards (Sigma-Aldrich, St. Louis, MO, USA) were used to obtain a calibration curve for the calculation of sugar concentrations. Organic acid concentrations were quantified in same manner with citric, malic, and ascorbic acid standards (Sigma-Aldrich, St. Louis, MO, USA). The limit of detection (LOD) and limit of quantification (LOQ) for the analysis were calculated at a signal-to-noise ratio (S/N) of about 3 and 10, respectively.

#### **Analysis of Individual Anthocyanins**

An Agilent 1100 HPLC system (Agilent Technologies, Palo Alto, CA, USA) operated by ChemStation software was used in the analysis. All juices were centrifuged ( $6000 \times g$ , 15 min) and filtered through 0.45 µm filter (Millipore) before injection. The analysis was performed on a Beckman Ultrasphere ODS (Roissy CDG, France; 4.6 mm × 250 mm) column. The mobile phase consisted of two solvents: Solvent A; water/formic acid (95:5; v/v) and Solvent B; acetonitrile/solvent A (60:40; v/v). Anthocyanins were separated in reference to the method reported in Kelebek and Selli [18]. The compounds were identified using the retention times as spectra were matched to authentic standards. The quantities of different anthocyanins were assessed from the peak areas and calculated as equivalents of representative standard compounds in calibration curves as follows: At 520 nm (anthocyanins), cyanidin 3-glucoside and cyanidin 3-rutinoside, respectively. The results were expressed as mg L–1. The limit of detection (LOD) and limit of quantification (LOQ) were calculated at a signal-to-noise ratio (S/N) of about 3 and 10, respectively.

#### **Antioxidant Capacity Analyses**

The ABTS (2, 20 -azinobis-(3-ethylbenzothiazoline-6-sulfonic acid) and DPPH (1,1-diphenyl-2picrylhydrazyl) assays were used in the determination of the antioxidant capacities of the pomegranate juice samples. In regard the antioxidants, the assays were performed in reference to the method reported in Kelebek et al. [19]. The absorbance of the solution was determined by a UV-VIS Spectrophotometer (UV-1601, Shimadzu, Kyoto, Japan). The absorbance values of the ABTS and DPPH solutions were recorded at 734 and 517 nm, respectively.

#### Analysis of the Volatile Composition

Volatiles of the pomegranate juice samples obtained from three different juice extraction methods (AJ, HPJ, and MAJ) were extracted by the liquid–liquid extraction technique. The procedure was applied with slight modification according to Selli and Kelebek [20]. Dichloromethane was preferred as the solvent in the extraction of the aroma compounds. Briefly, 100 mL centrifuged sample ( $6000 \times g$  for 15 min at 4 °C), 40 mL



dichloromethane, and 5  $\mu$ L of 4-nonanol (43.3 mgL-1) as an internal standard was stirred at 4 °C for 30 min under nitrogen gas. After stirring, the mixture was centrifuged again (9000× g, 15 min at 4 °C) and the organic phase was dehydrated by using anhydrous sodium sulfate. The extract was condensed to 5 mL in a concentrator (Kuderna Danish, Supelco, St. Quentin, France) at 40 °C and thereafter, to 0.2 mL by using a flow of nitrogen [20]. After this stage, each extract was placed in a glass vial of 2 mL with a Teflon-lined cap. Samples were extracted in triplicate.

#### **GC-FID and GC-MS Analysis of Volatile Compounds**

The system of gas chromatography (GC) comprised an Agilent 6890 chromatograph interfaced to an FID (flame ionization detector) and an MSD (mass selective detector) (Agilent 5973, Wilmington, DE, USA). To separate the volatiles of the samples, a DB-Wax column was utilized (0.5  $\mu$ m thickness × 0.25 mm i.d. × 30 m length; J&W Scientific, Folsom, CA, USA). The analysis was applied according to the method used in Keser et al. [21] with a slight modification. Volatiles of the samples were studied based on the retention index and mass spectra on the DB-Wax column using a commercial database of spectra (Wiley 6, NBS 75 k). The volatiles were then quantified utilizing the internal standard with 4-nonanol at 43.3 µg L−1 . Response factors were computed based on the intensity ratio of each volatile to 4 nonanol and ratios of peak areas were corrected by using each constituent's response factor [21,22]. Subsequently, the means and standard deviations were computed for the GC analyses in triplicate. Retention index data for the volatiles were subtracted by utilizing the n-alkane series (C8–C32).

#### Sensory Analysis of Pomegranate Juice Samples

Ten trained panelists (five women and five men aged between 25–47) from Food Engineering Department of Cukurova University (Adana, Turkey) evaluated pomegranate juice samples in terms of overall appearance including color, pulpiness, sourness, sweetness, astringency, fruity, and floral notes. Samples were prepared at room temperature approximately 30 min before the analysis. The juices were served in disposable, odorless plastic cups after shaking. The order of samples was randomized, and the panelists were asked to evaluate the samples on a 10 cm evaluation scale [13].

#### **Statistical Data Analysis**

The data were studied by using analysis of variance (ANOVA) in SPSS (v.24.0, SPSS Inc., Chicago, IL, USA). In addition, principal component analysis (PCA) was employed in XLSTAT software (trial version of 2020, Addinsoft, New York, NY, USA).

#### **RESULTS AND DISCUSSION**

#### **Physicochemical Properties of Pomegranate Juices**

Fruit size, skin, and aril colour are known to directly affect the customer acceptability and these properties depend on variety, climatic, and agricultural conditions. cv. Caner pomegranates used in the study were of medium size compared to the samples reported on Tunisian pomegranate cultures [23]. Fruit skin and arils were observed to have an intensive pink-red colour. It was detected that juice extraction methods did not significantly change pH values. Similar results were reported by other researchers [24,25]. The other characteristics responsible for the determination of juice quality are total soluble solids (°Brix) and titratable acidity. The total soluble solid values were between 14 and 16 °Brix in pomegranate samples. The differences in the total soluble solid values may be related to the tannins formed by the damage of rind cells during the juice extraction process in the HPJ sample. The existence of tannins is reported as an important problem in juices extracted from whole fruits. Accordingly, a bitter taste can develop, and this must be removed by industrial processing in order to meet the consumer demand [26]. The AJ and HPJ samples were revealed to have close values of titratable acidity (TA) while juice obtained from the MAJ had a lower TA value. Vazquez-Araujo et al. [27] reported that no significant differences were found in pH, TA, and TSS values between



Wonderful pomegranate juice samples from arils with albedo homogenate and from arils only.

Significant differences were observed in the HPJ, AJ, and MAJ samples in terms of all L\*, a\*, b\* colour parameters (p < 0.05). Colour is an important food quality parameter, and it influences the consumer's preference and market value of the final product. As displayed in Table 1, the AJ (L\*: 15.7) was found to be darker than HPJ (L\*: 22.0) and MAJ (L\*: 23.9) samples. The colour of pomegranate juices is known to be affected by the treatments applied in processing. The use of juices with low values of L\* parameter (darker red–purple) is recommended in the literature [28,29]. In comparison to the reported L\*, a\*, b\* values in the literature, the colour of the AJ juice sample from the cv. Caner pomegranate was found to be almost twice as dark as the juices of nine Spanish pomegranate varieties [30]. In terms of redness (a\*) and yellowness (b\*), cv. Caner pomegranate juices, independently from the extraction methods, were observed to have higher values than both Spanish and Tunisian cultivars (values are in the range of 3.0–29.7 for a\* and -1.7-23.7 for b\*) [23,30].

#### Sugars and Organic Acids of the Pomegranate Juice Samples

Sugars were detected as glucose and fructose in the samples. Sugar contents varied in the range of 51.0-56.0 g L-1 for glucose and 61.2-69.5 g L-1 for fructose depending on the juice extraction methods. These findings are in agreement with the sugar contents determined in pomegranate juices in other previous studies also reporting the glucose and fructose as the principal component of the juice sugar contents. Individual sugar contents reported in the literature were 57-65 g L-1 for glucose and 60-71 g L-1 for fructose in 40 different Spanish cultivars [31] and 58-76 g L-1 for glucose and 58-71 g L-1 for fructose in the arils from six pomegranate varieties in Turkey [24]. In the present study, citric, malic, and ascorbic acids were found as organic acids in the juice samples (Table 2). Among these, ascorbic acid was present in minor amounts in all samples. These data showed a similar correlation with the previous studies [24,31]. In the present study, the juice sample obtained after maceration (MAJ) had lower amounts of sugars, but the organic acid contents were similar in all three juice samples. When the three samples were compared, it was found that malic and ascorbic acid levels were not significantly changed by the juice extraction methods.

#### Anthocyanin Compositions of Pomegranate Juice Samples

Anthocyanins are crucial quality compounds as they are responsible for the attractive red colour in pomegranate juices [33]. In cv. Caner pomegranate juice samples analyzed in the present study, the total anthocyanin concentration was determined as 408, 555, and 593 mgL-1 in the MAJ, HPJ, and AJ samples, respectively. Thus, the juice extraction process method had a significant influence on anthocyanin composition.

In the present study, a total of six anthocyanins, typical for pomegranates, were detected including cyanidin-3-glucoside (Cya3), cyanidin-3,5-diglucoside (Cya3,5), delph inidin-3-glucoside (Dp3), delphinidin-3,5diglucoside (Dp3,5), pelargonidin-3-glucoside (Pg3), and pelargonidin-3,5-diglucoside (Pg3,5) in all juice samples. Cya3,5 was determined to be the dominant anthocyanin in the studied samples followed by Dp3,5 (Table 3). This compound was reported as the main anthocyanin in Georgian pomegranate juices obtained from cultivars of Rose, Afganski, Nikitski ranni, and Fleshman [25]. In addition, Kelebek and Canbas [34] stated that cv. Hicaz pomegranate juices from Turkey are also rich in cyanidin-3,5-diglucoside. In the current study, among the anthocyanins, Pg3 had the lowest amount in cv. Caner juice samples. Fawole and Opara [35] reported that Cya3,5 was the major anthocyanin followed by Dp3,5 in cv. Bhagwa juices similar to the results found in the present study. They also stated that the amounts of Cya3,5 and Dp3,5 increased with ripening stage. With regard to the effects of juice extraction methods on the anthocyanin contents, maceration of arils resulted in a reduction in their total amounts. This decrement may originate from the effects of light exposure, oxygen, storage time, and temperature [36]. Similarly, Galego et al. [37] reported that anthocyanins are highly sensitive pigments and significant changes in their structure are detected even if these compounds are stored



at -20 °C.

#### Antioxidant Capacity of the Juice Samples

Two methods (ABTS+ and DPPH-) were employed to assess the antioxidant capacities of the pomegranate juices in the present study. These two assays are basically radical scavenging methods based on the decolorization of free radicals [38]. The selection of these two assays was based on the chemical structure of antioxidant substances and the interaction of them with oxidants. ABTS+ can be applied to both hydrophilic and lipophilic antioxidants while DPPH- is applicable to hydrophobic systems as it can be dissolved in organic media [38]. Antioxidant activity values were detected in the range of 6.1-11.3 mmol Trolox L-1 for ABTS+ and 5.8–14.7 mmol Trolox L-1 for DPPH- analyses. These results are consistent with previous studies that reported high antioxidant power in pomegranates of different varieties [12,23,39]. In addition, the AJ sample was found to exhibit higher antioxidant activities than aril juices of cv. Mollar de Elche pomegranate of Spain [13]. In the current study, juice extraction methods were determined to have an influence on the valuable benefit of antioxidant activity as both assays revealed for all three pomegranate juice samples. The HPJ sample had higher antioxidant activities than the AJ and MAJ samples (Table 3). Similarly, Mphahlele et al. [40] reported that cv. Wonderful pomegranate juices obtained from halved fruits showed higher antioxidant activity  $(1337 \mu M \text{ Trolox mL}-1)$  in comparison with the juices from arils and arils plus seeds. With respect to this, the research conducted by Orak et al. [41], which examined different parts of the pomegranate, revealed that the pomegranate peel extract had the highest DPPH scavenging activity compared to the pomegranate juice or seeds. Thus, it can be deduced that the HPJ sample with higher antioxidant activity is related to the phenolics like hydrolysable tannins, punicalagins, anthocyanins, and phenolic acids found in non-edible parts of pomegranates (peel, carpellary membranes, etc.). Additionally, with regards to maceration, it is observed that the process had an at least two-fold diminishing effect on the antioxidant activity of pomegranate juice. Such decrease in the antioxidant activity of the MAJ sample may be due to the degradation of the anthocyanins, which is more probable during maceration.

#### Volatile Composition of the Pomegranate Juice Samples

Pomegranate juices have been reported to have low quantities of volatiles leading to a less intense aroma [30]. To investigate the effects of different juice extraction methods on the aromatic profile of cv. Caner pomegranate juice samples, liquid-liquid extraction was performed to obtain volatile compounds from three juice samples in the present study. A total of 34 volatile compounds including mainly alcohols, esters, and terpenes were identified and quantified. The total aroma composition of the samples was determined in the range of 710.7 and 1872.0  $\mu$ gL-1 with the MAJ having the highest volatile concentration. These results infer that juice extraction processes had a substantial effect on both concentration and composition of the volatile compounds. Alcohols were the dominant chemical group both quantitatively and qualitatively in the AJ and MAJ samples while ketones were detected in higher amounts in HPJ sample. 1-Hexanol was predominant in aril and macerated juices with amounts ranging 185–508 µgL-1 whereas 2,6-di(t-butyl)-4-hydroxy-4-methyl-2,5-cyclohexadien-1-one had the highest concentration with a value of 405  $\mu$ gL-1 in the MAJ sample. 1-Hexanol is a sixcarbon aliphatic alcohol generally found in different parts of pomegranates and their products [35,42–44]. C6 alcohols are generally produced by the lipoxygenase-hydroperoxide lyase metabolic pathways. This compound was reported to contribute to green, grass, and fruity attributes in pomegranate juices [43,45]. The amount of 1-hexanol was found to be higher in the AJ (185  $\mu$ gL-1) than the HPJ (53.5  $\mu$ gL-1) juice sample while the MAJ had the highest value (508  $\mu$ gL-1). Similarly, Mphahlele et al. [40] determined that juices obtained from arils plus seeds had higher 1-hexanol concentration than juices obtained from whole or halved fruits. In another study, maceration of juice with Arbutus unedo L. distillate into pomegranate liquors caused increases in the amount of 1-hexanol level from 1.14 to 5.58 mg 100 mL-1, which was originally absent in the distillate. These literature data are in agreement with our results in the way that the juice exposed to maceration exhibited the highest concentration of this compound. Another important six-carbon alcohol was



determined to be (Z)-3-hexen-1-ol in all three juice samples in the present study. This compound was also found in relatively higher amounts in other pomegranate juices providing a strong fresh green odor due to its higher odor activity value [46]. This compound was also determined by Vazquez-Araujo et al. [47] in fresh and commercial pomegranate juices from the USA. Ketones were the second crucial group of volatiles in all samples in the current study. Among them, 2,6-di(t-butyl)-4-hydroxy-4-methyl-2,5-cyclohexadien-1-one was detected for the first time in pomegranate juice in this current study as volatile compound to the best knowledge of the authors. It was also reported to exist in fermented soy sauce and white strawberry (Fragaria chiloensis) and characterized by caramel odor [48,49]. On the other hand, 2-Nonanone was detected only in the HPJ sample. This was also reported to be an odor active compound responsible for cheesy, fruity, floral, and green attributes in pomegranate samples [43,46]. In addition, this compound was detected in cv. Wonderful pomegranate juices obtained with the peels [43]. Other important aromatic compounds in the juice samples were found to be terpenes and esters (Table 4). Terpenes are secondary metabolites synthesized from isoprene units. Three terpenes were detected including linalool,  $\alpha$ -terpineol, and p-cymen-8-ol while  $\alpha$  terpineol was common in all three samples. This compound was also found in Chinese, American, South African, and other Turkish pomegranate juices displaying floral and fruity attributes [29,43,46,50]. Besides  $\alpha$ -terpineol, linalool and p-cymen-8-ol were also reported to be detected in minor levels in different cvs. pomegranate juices [45– 52]. With regard to esters, in the present study, (Z)-3-hexenyl acetate and isopulegol acetate were detected in minor amounts. In previous studies, (Z)-3-hexenyl acetate was reported to be formed as a result of the conversion of its corresponding six-carbon alcohols [53]. This compound was also previously reported in different Spanish pomegranate juices and attributed to the green, sweet, and banana odor notes [28,52]. Butyrolactone was the only lactone compound found in all juice samples in the present study. This lactone is responsible for sweet caramel odor and was also reported to be found in cv. Wonderful pomegranates [43]. In the current study,  $\beta$ -Ionone is a norisoproneoid typical to berries and was found only in trace levels in aril and halved pomegranate juice samples. This compound was also reported to be found in some Spanish pomegranate juices by Vazquez-Araujo et al. [47].

#### **Sensory Analysis**

Nine sensory attributes were utilized to describe the flavor of pomegranate juice samples: Pulpy, aroma, color, sourness, sweetness, astringency, fruity, floral notes, and general impression. Pulpy was an attribute present in all samples ranging from 3.8 and 5.6. This finding is similar to the data presented by Vazquez-Araujo et al. [47] for pomegranate juice macerated with albedo homogenate. The sweet, sour, floral, and astringency attributes of the samples were found to be very similar and no statistically significant difference was found. HPJ (halved pomegranate juice) samples were characterized by more intense fruity and floral odors. Floral note values were found to be the lowest among all sensory attributes (3.0-3.4) similar to the data reported by Vazquez-Araujo et al. [47]. The largest statistically significant differences (p < 0.05) were for aroma, color, and general impression attributes in the present study. The HPJ sample was less pulpy, but it had high scores in all other attributes as compared to the other two samples, and it was also determined to be the most preferred sample by panelists with the highest general impression score.

#### CONCLUSION

This study was the first detailed approach to elucidate volatiles, anthocyanins, sugars, organic acids, and antioxidant properties of juices of Caner cultivar pomegranate fruits obtained from three different juice extraction methods (halved pomegranate juice, HPJ, aril juice, AJ, and macerated aril juices, MAJ). The experimental results clearly showed that these compounds in all juice samples were significantly altered by the applied juice extraction method. The total quantity of the aroma compounds was detected to be increased as a result of the macerating process (MAJ sample). 1-Hexanol and (Z)-3-hexen-1-ol were the main volatiles in all juice samples. When the three juice samples were compared in terms of volatiles, it was found that MAJ was quite rich in terms of these compounds compared to the HPJ and AJ samples. Among the volatile



compounds, 2,6-di(t-butyl)-4-hydroxy-4- methyl-2,5-cyclohexadien-1-one was determined for the first time in this study as volatile compounds in pomegranate juices to the best knowledge of the authors. The main anthocyanins were found as cyanidin-3,5-diglucoside followed by delphinidin-3,5-diglucoside in all three juice samples. In contrast to the volatiles, it was found that all of the anthocyanin components of the MAJ sample were significantly decreased during maceration due to the degradation of these compounds. Sensory analysis revealed that the general impression of the HPJ sample was higher as compared to the AJ and MAJ samples.

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# Effect of quinoa and germinated wheat flour in physicochemical,

# textural and sensory properties of cupcakes

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#### ABSTRACT

The effect of quinoa flour addition along with germinated wheat flour on the physicochemical, textural, sensory and stability of cupcakes as a convenience product for celiac disorders were investigated. The cupcakes containing quinoa flour exhibited more hardness, elasticity and water activity than the control cupcake. The textural attributes revealed that the cupcakes with quinoa flour or germinated wheat flour were statistically different from those with either quinoa flour or germinated wheat flour as well as the control cupcake. Moreover, cupcakes with quinoa flour had greater acceptance and preference. In addition, L\* demonstrated a high correlation with porosity relative to chemical and textural properties and can therefore be used as a parameter for the prediction of the stiffness and elasticity of formulated products by image processing. As it is more convenient to calculate L\* in food production, it may be advised to use cupcakes prepared with QF and GWF to estimate the textural properties. Cupcakes formulated with composite flours containing 15% of quinoa flour exhibited acceptable technological and sensorial attributes. Consequently, the study also showed that the introduction of QF into germinated wheat flour contributes to sensory and microbiological advantages in cupcakes that extend the cupcakes' shelf life and is recommended as a proper alternative for celiac disorders.

Keywords: Pseudocereals; Quinoa; Germinated wheat flour; Texture: Celiac.



# Effect of Pasteurization on Interaction of Bioactive Compounds in Pomegranate-Sweet Orange Mix Juice

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#### ABSTRACT

The present study deals with the effect of pasteurization on pomegranate, sweet orange and mixed juice of pomegranate and sweet orange (70:30). Thermal effect on TPC, FRAP antioxidant content, DPPH % inhibition, anthocyanins, and ascorbic acid content were analysed. Colour of juices and the microbial load were also analysed using standard methods. For mixed juice, interaction factor (IF) was determined by Gawlik-Dziki method. In all juice samples at high-temperature pasteurization, treatments rise in TPC and antioxidant content was observed. while a decrease in anthocyanin ascorbic acid was reported in all juice samples. The Pearson Correlation Coefficient values of TPC and antioxidant activity for pomegranate juice and mixed juice were found as 0.77 and 0.95, respectively. The IF factor study showed a synergistic effect. No microbial load was found in high pasteurization heat treatments above 70oC. Further, profiling of pomegranate juice performed by advance highresolution Ultra-performance Liquid chromatography revealed the presence of significant health beneficial phenolic compounds in mixed juice (kaempferol, caffeic acid, p-coumaric acid and ferulic acid, narirutin, naringin, hesperidin, neohesperidin, Apigenin 6-C-glucoside). The identification of all phenolic compounds was based on the detection of the precursor ion and one characteristic fragment ion, each with less than 5 ppm of mass error.

Keywords: Bioactive components, Interaction factor, Pasteurization, UHPLC



# Texture and Color Evaluation of Dough and Tortillas Elaborated by Adding Solids from Nixtamalization Waste

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#### ABSTRACT

Nixtamalization or alkaline cooking, is a procedure that provides adequate textural characteristics to maize products like dough and tortilla, but implies a high energy input and releases a considerable volume of waste effluent (nejayote). In this work, the effect of nixtamalization temperature (60 and 90 °C) and the addition of solids from nejayote, on textural properties and on the color of corn dough and tortillas, were studied. It was observed that after nixtamalization at 60 °C the textural properties of corn products did not show a significant difference compared to those processed at 90 °C. Regarding the addition of solids from nejayote, it was observed that the textural properties and the color of the dough and tortillas were not modified, neither at 60 nor at 90 °C, obtaining products with the same attributes as the commercial products. Due to that, considerable energy savings in the process and a decrease in solids of the waste effluents, can be achieved.

Keywords: maize products, nixtamalization, texture, waste effluent



## **Novel Extraction Techniques for Natural Bioactive Compounds**

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#### ABSTRACT

Interest in natural bioactive compounds has greatly increased in the last decade. Natural bioactives are physiologically active components and provide desirable health benefits, reducing the risk of chronic disease and the process of carcinogenesis. Bioactive compounds can be extracted from natural sources such as plants, food by-products, algae, and microalgae and utilized for development of nutraceuticals and functional foods. Bioactive compounds in different commercial sectors such as pharmaceutical, food and chemical industries require most appropriate and standard method to extract these active components. One of the major challenges is to extract these biomolecules from their respective natural sources. Traditional solvent extraction is very time consuming and require relatively large amount of solvents. Moreover, heat applied during solvent evaporation can damage thermolabile bioactive compounds. Additionally, the safety of residual organic solvents in the final product is being questioned. Recently, novel extraction techniques such as ultrasoundassisted extraction, enzyme-assisted extraction, microwave-assisted extraction, supercritical fluid extraction, pulsed electric field extraction and pressurized liquid extraction have been developed for the extraction of bioactive compounds from plants in order to overcome the mentioned shortcomings of the conventional solvent extraction. These methods are better suited for the extraction of thermolabile bioactive compounds, which is not the case with the conventional methods. These novel methods provide shortened extraction time, reduced solvent consumption, improved extraction yields, and enhanced quality of the extracts. At present, some of those techniques are available at both analytical and industrial scale throughout the globe. This present review is aimed to summarize the novel extraction methods along with their principal mechanism for extracting bioactive compounds from the mentioned sources.

Keywords: Bioactive compounds, extraction techniques, novel developments

#### **1. INTRODUCTION**

Bioactives are metabolites synthesized by plants for self defence and other purposes and have the potential to be used by humans for a variety of applications. There is growing interest of consumers towards food bioactives that provide beneficial effects to humans in terms of health promotion and reduction in disease risk (Kumar et al., 2017). Evidence is growing that use of bioactives might help to promote optimal health and reduce the risk of chronic diseases such as cancer, coronary heart disease, stroke and Alzheimer's disease. Bioactives are obtained selectively from plants as specialty chemicals and can be used as nutraceuticals, processed foods to complement a balanced diet or as drug leads (Puri et al., 2012). Phytochemicals can be extracted from natural sources such as plants, food by-products, algae, and microalgae and utilized for development of nutraceuticals and functional foods.

The low content of active molecules in the source material and the complexity of raw material makes it necessary to find alternative methods of effective extraction (Marathe, 2017). One of the main challenges is to extract these biomolecules from their respective natural sources. Different techniques have been reported on these aspects in the literature, each having their own advantages and disadvantages. The choice of technique mainly depends on the type of raw material, environmental concerns, process conditions, and future applications of the bioactives (Marathe, 2017). Conventional solvent-based extraction of bioactives often suffers from low extraction yields, requires long extraction times and the final product often contains traces of organic solvents, which decrease the product quality. Thus, the development of an effective and selective method for bioactive compound extraction is important (Puri et al., 2012). This review describes the utilization



of different novel extraction techniques for extraction of bioactives from plants.

#### 2. METHODS USED FOR BIOACTIVE COMPOUND EXTRACTION

Several methods have been developed to improve extraction of bioactive compounds which include various extraction techniques. The novel extraction techniques are discussed in the following sections (Joana Gil-Chávez et al., 2013).

#### 2.1. Solvent Extraction

Solvent extraction is used to obtain certain compounds from different materials such as fungi, algae and microalgae, and, more commonly, plants. Basically, pretreated raw material is exposed to different solvents, which takes up compounds of interest and also other agents (flavors and colorings). Samples are usually centrifuged and filtered to remove solid residue, and the extract could be used as additive, food supplement or be destined for the preparation of functional foods (Joana Gil-Chávez et al., 2013). Solvent extraction is beneficial compared to other methods due to low processing cost and ease of operation. However, this method uses toxic solvents, requires an evaporation/concentration step for recovery, and usually requires large amounts of solvent and extended time to be carried out. Moreover, the possibility of thermal degradation of natural bioactive components cannot be ignored due to the high temperatures of the solvents during the long times of extraction. Solvent extraction has been improved by other methods such as Soxhlet's, ultrasound, or microwave extraction and supercritical fluid extraction in order to obtain better yields (Kumar et al., 2017).

#### 2.2. Microwave-Assisted Extraction (MAE)

MAE is a relatively new extraction technology which has been widely applied in a variety of natural bioactive compounds with many advantages over conventional extraction techniques including lower environmental pollution, higher extraction efficiency and shorter extraction time (Joana Gil-Chávez et al., 2013).

A microwave is referred to as a nonionizing electromagnetic radiation that has a frequency of 300 MHz–300 GHz (Alternimi et al., 2017). The moisture, when heated up within the plant cell due to microwave effect, evaporates and generates high pressure on the cell wall and results in swelling of the plant cell. This pushes, stretches, and consequently ruptures the cell wall. These facilitate leaching of active constituents from the cells to the solvent, thereby enhancing the yield of bioactive components. This phenomenon could be intensified as the solvent is impregnated into the plant matrix at a high heating efficiency of the microwave (Danlami Jibrin et al., 2014). Generally, elevated temperatures result in improved extraction efficiencies. However, for the extraction of thermolabile compounds, high temperatures may cause the degradation of extracts. In this case, the chosen power during MAE has to be set correctly to avoid excess temperatures, leading to possible solute degradation (Wang & Weller, 2006) Open-vessel microwave extraction at atmospheric pressure condition is better suited to thermolabile species (e.g., organometals) since it uses low temperatures relative to closed-vessel systems (de Castro, 2012). More research is needed to investigate the interaction between microwaves, and plant materials and solvents (Wang & Weller, 2006).

By considering economical and practical aspects, MAE is a strong novel extraction technique for the extraction of nutraceuticals (Wang & Weller, 2006). However, MAE still uses organic solvents, such as hexane, and therefore cannot be considered as a green technology. Furthermore, compared with other modern extraction techniques, an additional stage (filtration or centrifugation) is required for the removal of solid residues (Danlami Jibrin et al., 2014). Moreover, in order to be considered in industrial applications at least two important limitations must be improved including i) the recovery of nonpolar compounds and ii) the modification of the chemical structure of target compounds which may alter their bioactivity and limit their application (Joana Gil-Chávez et al., 2013).

#### 2.3. Ultrasonic-Assisted Extraction (UAE)

Ultrasound-assisted extraction has been used in diverse applications of food-processing technology to extract



bioactive compounds from plant materials. Ultrasound, with levels greater than 20 kHz, is used to disrupt plant cell walls, which helps improve the solvent's ability to penetrate the cells and obtain a higher extraction yield. UAE can use a low operating temperature through processing, maintaining a high extract quality for compounds (Altemimi et al., 2017). Ultrasound-assisted extraction may be a promising alternative to conventional solvent extraction enhancing extraction efficiency and extraction rate, reducing extraction temperature, and increasing the choice ranges of solvents. Therefore, use of ultrasound-assisted extraction is advisable for thermolabile compounds, which may be altered under Soxhlet operating conditions due to the high extraction temperature. However, it should be noted that since ultrasound generates heat, it is important to accurately control the extraction temperature. The sonication time should also be considered carefully as excess of sonication can damage the quality of extracts (Wang & Weller, 2006). Furthermore, pressure frequency, moisture content of sample, milling degree, particle size and solvent are also very important factors in order to obtain efficient and effective ultrasound-assisted extraction. UAE have also been incorporated along with various classical techniques as they are reported to enhance the efficiency of a conventional system (Azmir et al., 2013).

#### **2.4. Pressurized Liquid Extraction (PLE)**

Pressurized liquid extraction is a relative new technology for extraction of phytochemicals under high temperature and pressure (Dai & Mumper, 2010). This method is known by several names; pressurized fluid extraction, accelerated fluid extraction, enhanced solvent extraction, and high-pressure solvent extraction (Azmir et al., 2013).

The temperature and pressure conditions utilized in PLE are within the ranges of 50–200 °C and 3.5–20 MPa, respectively. The elevated pressure causes the solvent temperature to rise above the normal boiling point temperature. The increase in temperature tends to accelerate the extraction rate by increasing solubility and mass transfer rate (Danlami et al., 2014). High temperature and pressure improve analyte solubility and the desorption kinetics from the matrices. Therefore, extraction solvents including water which show low efficiency in extracting phytochemicals at low temperatures may be much more efficient at elevated PLE temperatures (Dai & Mumper, 2010). Also, the increased temperature reduces the viscosity and surface tension of solvents, helping them to spread evenly over the biological matrix and improve the extraction rate (Danlami et al., 2014). The high pressure helps the extraction cells to be filled faster and forces liquid into the solid matrix. These new techniques allow a faster extraction in which less amount of solvents are used and higher yields are obtained in comparison with traditional solvent extractions (Joana Gil-Chávez et al., 2013). Therefore, extraction solvents including water which show low efficiency in extracting phytochemicals at low temperatures may be much more efficient at elevated PLE temperatures (Dai & Mumper, 2010). In some cases, pressurized hot water is employed as a solvent for extraction rather than an organic solvent. This method is called pressurized hot water extraction or subcritical water extraction (Danlami et al., 2014). The use of PLE allows the attainment of food-grade extracts obtained only when water or other GRAS (generally recognized as safe) solvents, such as ethanol are used (Joana Gil-Chávez et al., 2013).

The extraction of natural bioactive compounds by PLE has been demonstrated in numerous studies which have presented several approaches to optimize the extraction conditions or evaluated their efficiency compared with other methods. Despite the advantages over conventional methods, this method is not found to be suitable for thermolabile compounds as high temperature can have deleterious effects on their structure and functional activity (Joana Gil-Chávez et al., 2013). Moreover, this method has other disadvantes such as (a) selectivity is achieved only by varying the solvent type, which tends to be exhaustive and may thus lead to nonselective extractions; (b) postextraction cleanup is necessary; and (c) nowadays, it is used only for analytical purposes because it has not yet been up-scaled (Danlami et al., 2014).

#### 2.5. Supercritical Fluid Extraction (SFE)

Supercritical carbon dioxide (SC-CO<sub>2</sub>) extraction has received significant attention in the last few years as a



promising alternative to conventional solvent extraction for extracting biologically active compounds as it offers a number of advantages, including high extraction efficiency, short extraction time and lack of residue problems (Sahena et al., 2009).

SC-CO<sub>2</sub> extraction has attracted a lot of interest because carbon dioxide (CO<sub>2</sub>) is an inert, low-cost with high purity, nontoxic, and environmentally-friendly solvent that permits extraction at low temperatures and comparatively low pressures. The extraction temperature as low as 30 °C and absence of oxygen and light make this method attractive for the extraction of heat sensible compounds avoiding thermal degradation and decomposition of possible labile compounds. In addition, CO<sub>2</sub> can evaporate instantly when exposed to atmospheric conditions (Danlami et al., 2014). Therefore, SFE can eliminate the concentration process, which usually is time-consuming (Brusotti et al., 2014). In addition, this molecule is environmentally friendly and "generally recognized as safe" (GRAS) by FDA (U.S. Food and Drug Administration) and EFSA (European Food Safety Authority) (Joana Gil-Chávez et al., 2013).

During the process of extraction, raw material is placed in an extraction container equipped with temperature and pressure controllers to maintain the required conditions. Following this, the extraction container is pressurized with the fluid by a pump. Once the fluid and dissolved compounds are transported to separators, the products are collected through a tap located in the lower part of the separators. Finally, the fluid is regenerated and cycled or released to the environment (Kumar et al., 2017). Before SC-CO2 extraction, vegetable substrates are commonly subjected to mechanical pretreatments to improve the rate and yield of the extraction process. The pretreatments may have multiple purposes, including releasing of solutes from cells, facilitating solvent flow through the packed bed, and increasing substrate load onto extraction vessels. One frequently used treatment for high-oil seeds is prepressing to reduce the oil content (Singh, 2012). The extraction efficiency and the characteristics of the products are affected by several parameters, such as particle size and moisture content of the feed material, extraction temperature and pressure, solvent flow rate, extraction time, and the use of a cosolvent (Temelli, 2009). However, because of its low polarity,  $CO_2$  is less effective in extracting highly polar compounds from their matrices. For this reason, the use of other solvent compounds is needed in order to enhance solubility and the selectivity of the process and they must be added only in small quantities. Ethanol is recommended as a co-solvent in SFE because of its lower toxicity and miscibility in  $CO_2$ , although its applications is limited due to its unfavorable properties with respect to safety and environmental considerations (Joana Gil-Chávez et al., 2013).

#### **2.6.** Enzyme-assisted extraction (EAE)

The application of enzymes for complete extraction of bioactives without the use of solvents is an attractive alternative (Puri et al., 2012). Enzyme-assisted extraction offers safe, green, and novel approach for the extraction of bioactives (Marathe, 2017). It is based on the ability of enzymes to catalyze reactions, under mild processing conditions, in aqueous solutions (Kumar et al., 2017).

Plant cell walls contain polysaccharides such as cellulose, hemicellulose, and pectins which act as barriers to the release of intracellular substances. Some enzymes such as cellulase,  $\beta$ -glucosidase, xylanase,  $\beta$ -gluconase, and pectinase help to degrade cell wall structure and depolymerize plant cell wall polysaccharides, facilitating the release of linked compounds. Hence, these enzymes have been proposed as tools to optimize the extraction of compounds from plant (Joana Gil-Chávez et al., 2013). In the EAE process, the operational conditions such as temperature of reaction, pH of system, enzyme concentration, particle size of substrate, and time of extraction are important.

Several researchers have reported this method as a best choice for extraction of bioactives from various sources. Enzyme-assisted extraction has the advantages of (1) Higher extraction yields by breaking down the complex structure of raw material, (2) Removal of unwanted components of raw material selectively, (3) High catalytic efficiency and preservation of the original efficacy of natural products. (4) Reduction in the time of extraction and volume of solvent used (Marathe, 2017). However, enzyme-assisted extraction of bioactive compounds from plants has potential commercial and technical limitations: (i) the cost of enzymes is relatively



expensive for processing large volumes of raw material; (ii) currently available enzyme preparations cannot completely hydrolyze plant cell walls, limiting extraction yields of compounds, including the extraction of stevioside; (iii) enzyme-assisted extraction can be difficult to scale up to industrial scale because enzymes behave differently as environmental conditions such as the percentage of dissolved oxygen, temperature and nutrient availability vary. However, if the above limitations can be overcome, then enzyme-based extraction could provide an opportunity to not only increase extraction yields, but also to enhance product quality by enabling the use of milder processing conditions such as lower extraction temperatures (Puri et al., 2012).

#### 2.7. Pulsed-Electric Field (PEF) Extraction

The application of pulsed electric fields for bioactive compound extraction from plants has received increasing interest during the last 10 years (Shorstkii, 2017). This minimally invasive method allows avoidance of undesirable changes in a biological material (Boussetta, 2012). Pulsed electric field processing is a non-thermal food processing technology based on the application of short pulses of high voltage through a food product, whether in semi-solid or liquid form, which is placed between two electrodes. This process is carried out at either low or moderate temperature and is a promising non-thermal extraction technique (Sotelo et al., 2015). PEF can increase mass transfer during extraction by destroying membrane structure of plant materials for enhancing extraction and decreasing extraction time. PEF has been applied to improve release of intracellular compounds from plant tissue with the help of increasing cell membrane permeability. PEF treatment at a moderate electric field (500 and 1000 V/cm; for  $10^{-4}$ – $10^{-2}$  s) is found to damage cell membrane of plant tissue with little temperature increase. Due to this reason, PEF can minimize the degradation of heat sensitive compounds (Azmir et al., 2013). The application of electroporation through PEF offers a great potential for extraction purposes. The main advantages of PEF pretreatment are those of the non-thermal extraction even at high electric field strength (E > 20-30 kV/cm) due to its short pretreatment time ( $10^{-5}-10^{-2}$  s). This improves extraction rate and extraction yields. Generally, the PEF pretreatment requires lower energy consumption compared to mechanical fractionation and thermal pretreatments. With no or little addition of organic solvents or enzymes, extraction assisted by PEF pretreatment is usually conducted in green solvent such as water or ethanol (Yu et al., 2016).

#### **3. CONCLUSION**

The present review indicates the use of various novel technologies adapted for the extraction of bioactive compounds which can be used as nutraceuticals, dietary supplements and ingredients in functional foods. This review is focused on the principles and mechanisms of green and safe techniques such as enzyme-assisted extraction, ultrasonic-assisted extraction, microwave-assisted extraction, supercritical fluid extraction, pressurized liquid extraction, pulsed electric field extraction as an alternative to traditional solvent extraction in which environmentally troublesome organic solvents are used. These methods are better suited for the extraction of thermolabile bioactive compounds, which is not the case with the conventional methods. At present, some of those techniques are available at both analytical and industrial-scale throughout the globe. The further developments and optimisations in novel technologies may provide innovative approaches to increase the production of specific active compounds for use as nutraceuticals or as ingredients in functional foods. Moreover, detailed economic analysis of these extraction techniques and researches are required for the design and scale-up of these new extraction methods to enhance their lab scale and commercial uses.

TADIC 1. RUCC	Table 1. Recent studies on the use of novel extraction techniques nom unrefent natural products								
Method and conditions	Materials	Bioactive compounds	Applications	Reference					
SFE, SC-CO₂, 1 h, 10-40 MPa, 35- 60℃	Oregano, lemon peel, blackberry marc	Fatty acids and antioxidant compounds	Food and pharmaceutical applications	(García-Pérez et al., 2017)					

Table 1. Recent studies on the use of novel extraction techniques from different natural products



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MAE, Ethanol/water, 2.45 GHz, 60-100°C, 6- 30 min	Juglans regia L. fresh male flowers and unripe walnut seeds		Pharmaceutical, cosmetical and food industry	(Rosa et al., 2017)
MAE, Ethanol/water, 400- 800 W, 15-35 min	Wild apple fruit dust	Flavonoids, polyphenolic antioxidants	Pharmaceutical, cosmetical and food industry	(Pavlić et al., 2017)
UAE, Vegetable oils, 139 W, 20 kHz, 10-60 min, 20-60°C	Pomegranate wastes	Carotenoid Pharmaceutical, cosmetical and food industry		(Goula et al., 2017)
PLE, Water, 0.5-1.5 MPa, 20-30 min, 130-170°C	Grape seed	Resveratrol	Therapeutic and pharmacological activities	(Tian et al., 2017)
PLE, Ethanol/water, 10.34 MPa, 60-120 °C, 10 min	Oak wood waste	Furanic ompounds, terpenes and norisoprenoids	Food Industry	(Alañón et al., 2017)
SFE, SC-CO <sub>2</sub> , 2 h, 400 bar, 80°C	Tomato process wastes	Carotenoids/Proteins	Pharmaceutical, cosmetical and food industry	(Kehili et al., 2016)
PEF, Water, 13.3 kV/cm, 0-564 kJ/kg	Grape pomace	pe pomace Antioxidant Food and compounds pharmaceutical applications		(Barba et al., 2015)
EAE, Water, 40°C, 20-180 min	Saffron tepals	pals Anthocyanins Food additives providing health benefits		(Lotfi et al., 2015)
UAE, Ethanol/water, 25-50 °C, 160-400 W,	Olive leaves	Phenolic	Pharmaceutical, cosmetical and food industry	(Ahmad-Qasem et al., 2013)

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# Development and Characterization of an Exopolysaccharide -Functionalized Acid Whey Cheese Using *Lactobacillus delbrueckii* ssp. *bulgaricus*

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#### ABSTRACT

*Lactobacillus delbrueckii* ssp. *bulgaricus* NCFB 2772 was employed to develop functional acid whey cheese (requesón) added with *in situ* produced exopolysaccharide (EPS) and tested as a carrier of the commercial probiotic *Lactobacillus paracasei* ssp. *paracasei*. The optimum temperature of EPS biosynthesis by the NCFB 2772 strain at pH 4.6 was 37 °C (0.274 mg / mL of whey). Four different requesón formulations were prepared and analyzed in terms of texture properties and color. Addition of EPS significantly increased the obtained yields, and improved some texture parameters such as hardness, elasticity, and adhesiveness. However, colorimetric assays demonstrated that the addition of EPS and a commercial probiotic slightly affected the L\* value, obtaining products with less luminosity. The present study supports utilization of acid whey wastes to obtain functional cheese such as requesón in which the EPS component can improve not only processing yields but also its sensorial characteristics.

Keywords: cheese, exopolysaccharide, Lactobacillus casei, Lactobacillus delbrueckii



# Chapatti (Flat Bread) Characteristics as Affected by Onion Peel Powder

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#### ABSTRACT

The novel approach was made by the addition of onion peel in the form of dried onion peel powder (OPP) in the preparation of chapatti (unleavened flat bread). The OPP was added at (1-3)% level in chapatti. For all doughs with and without OPP, G' (storage modulus) was found to be higher than G" (loss modulus) and exhibited pseudoplastic behavior. The crude fiber content in chapattis significantly increased from 1.75% to 4.09%, respectively. The OPP added chapatti also showed improved antioxidant properties due to rise in the phenolic and flavonoid content. The OPP added chapattis required lower tearing force and had reduced elasticity compared to control ones. Chapatti incorporated with OPP also demonstrated longer shelf life. Thus, this waste could be utilized as a source of bioactive constituents possessing antioxidant and antimicrobial properties due to the presence of phenols. Besides phenols, it is also a rich source of fiber. Thus, for the very first time, onion peel powder was supplemented in the making of flatbread chapattis, being a staple food in many South Asian countries.

Keywords: Antioxidant, chapatti, fiber, Onion peel



# Rheological, Textural and Digestibility Characteristics of Chapatti as Affected by Incorporation of Type 4 Resistant Starch Prepared from Sorghum and Corn Starch

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#### ABSTRACT

 $RS_4$  is a functional ingredient possessing physiological benefits and desirable functional properties. The conventional fibers like cereal brans increase fiber content of food but adversely affects sensory profile. Therefore, the objective of this study was to incorporate corn and sorghum based resistant starch i.e.  $RS_4$  (starch citrates) in chapatti up to 15%.  $RS_4$  substitution produced dough that was stronger and resistant to shear. The corn based  $RS_4$  dough exhibited elastic behaviour throughout the frequency range while sorghum based dough at 15% exhibited viscous behaviour at higher frequency. The % critical strain of corn and sorghum at 10%  $RS_4$  dough was significantly higher than control. The chapattis produced were soft and extensible, having low glycemic index (pGI). The predicted glycemic index (pGI) of chapattis decreased from 57.25 to 44.97, with significant decrease being found in sorghum based chapatti. The RS<sub>4</sub> incorporated chapattis had similar sensory acceptance compared to that of control. With the incorporation of RS in processed foods, the nutritional profile of the products will improve as well as it will make a healthy diet. This study provided an insight of the resistant starch (RS<sub>4</sub>) incorporation in wheat flour-based chapatti that is an important part of south-asian daily diets. Moreover, it will help in the incorporation of RS in other wheat-based products such as breads, cakes, biscuits, tortillas etc. at commercial level.

Keywords: Chapatti, glycemic index, resistant starch, wheat flour



# Oleuropein Extraction from Leaves of Three Olive Varieties (*Olea europaea* L.): Antioxidant and Antimicrobial Properties of Purified Oleuropein and Oleuropein Extracts

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#### ABSTRACT

Olive leaf which is one of the by-products of olive tree cultivation and olive processing industry, is a potential source of phenolic compounds. The most dominant phenolic compound in olive leaf is oleuropein. In this study, it was aimed to extract, purify oleuropein from leaves of different olive varieties and determine antioxidant and antimicrobial activity. For this purpose, olive leaf crude extract, partially purified oleuropein and purified oleuropein were obtained from leaves of Domat, Edremit, Trilye olive tree varieties. Oleuropein amount of olive leaf products ranged between 215.26-958.22 mg/g and the highest oleuropein content was found in Domat. Amount of oleuropein in olive leaf products was increased depend on purification processes (approximately 90% purity). Total phenolic content in terms of gallic acid and oleuropein ranged between 102.36-325.02 mg GAE/g and 308.06-915.33 mg OE/g, respectively. Antioxidant activities of olive leaf products ranged between 104.83-456.50 mg TE/g (ABTS) and 109.27-456.93 mg TE/g (DPPH), respectively. There is an increase in antioxidant activities of products obtained purificiation processes. MIC values for Escherichia coli O157:H7, Listeria monocytogenes, Salmonella Typhimurium and Staphylococcus aureus cultures ranged between 1:1 (50 mg/mL) to 1:64 (0.781 mg/mL). S. aureus is more sensitive to these products compared to other microorganisms, whereas E. coli O157: H7 is more resistant than other microorganisms. In conclusion, crude extracts and oleuropein purified from these crude extracts have potential to extend shelf life of food products due to their determined antioxidant and antimicrobial activities.

Keywords: Oleuropein, Purification, Olive leaf, Antimicrobial, Antioxidant



## **Total Phenolic and Antioxidant Bioaccessibilities of Cookies Enriched with Bee**

#### Pollen

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#### ABSTRACT

The purpose of this study was to investigate the use of bee pollen (5, 10%, 15%) as a natural and functional ingredient in cookies. Evaluation of physicochemical and sensory properties with total phenolic and antioxidant capacities, using ABTS and CUPRAC methods, in extractable, hydrolysable and bioaccesible fractions of enriched cookies was studied. With the addition of pollen, the moisture and carbohydrate content of the cookies decreased, while the ash, total protein and total fat content increased. Parallel to the increased addition of pollen, spread ratio (SR), diameter, the browning index, a \* and b \* values and hardness of the cookies increased, whereas L\* values decreased. Total phenolic contents of the enriched cookies were determined as 352.70-401.13 mg/100 g, of which 92.27-93.16% were hydrolysable phenolic content, only 8.55-9.26% of the total phenolic contents were bioaccesible. The highest TEAC<sub>ABTS</sub> and TEAC<sub>DPPH</sub> results were  $88.01 \pm 0.79$  and  $147.24 \pm 0.62 \mu$ mol Trolox / g (15% pollen addition), respectively, and were higher than the control sample. More than 88% of the total antioxidant capacity values were detected as hydrolyzable fractions and bioaccessibility fraction values were very low as in total phenolic content. In addition, the cookies produced with the addition of bee pollen were accepted in terms of their sensory properties, and the 10% bee pollen added cookies were most liked by the evaluators. In the light of the information obtained, it can be said that bee pollen is an ingredient that can improve the quality criteria while improving the functional properties of the cookies.

Keywords: Bee pollen, cookie, hydrolysable fraction, bioaccesiblity



# Cloud Point Extraction of Lutein and $\beta$ -carotene from Spinach Waste

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#### ABSTRACT

In this study, it was aimed to extract lutein and  $\beta$ -carotene from spinach waste using aqueous solutions of nonionic surfactants by cloud point extraction (CPE) technique. In accordance with this purpose; parameters such as appropriate surfactant type and concentration, phase separation temperature and time, salt concentration have been optimized. In the preliminary trials to see the effect of HLB value of various nonionic surfactants on extraction performance, the best results were obtained with Tween series surfactants having high HLB values. The most suitable surfactant for extraction was determined as Tween 40. Optimum extraction conditions; The surfactant and salt concentration were determined as 10%, the incubation temperature as 70°C, and the incubation time as 40 minutes. Lutein and  $\beta$ -carotene content in the cloud point extract obtained in the pre-determined optimum conditions were quantified by a HPLC system. In parallel, the efficiency of the CPE method in terms of carotenoid extraction from spinach wastes was determined as 66.76% for lutein and 149.89% for  $\beta$ -carotene. The results obtained showed that with the CPE technique, carotenoids from the food matrix can be obtained at a high rate by using aqueous solutions of non-ionic surfactants without requiring an additional recovery or purification step.

Keywords: β-carotene, Cloud point, Green solvents, Lutein, Spinach

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# LC-DAD-ESI-MS/MS Characterization of Phenolic Compounds in Wines from Vitis vinifera 'Shesh i bardhë' and 'Vlosh' Cultivars

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#### ABSTRACT

Phenolic compounds in white wines produced by cv. *Shesh i bardhë*, and red wines produced by cv. *Vlosh*, two native grape cultivars of Albania, were investigated by using Liquid Chromatography-Diode Array Detection-Electrospray Ionization-Tandem Mass Spectrometry (LC-DAD-ESI-MS/MS). A total of 32 phenolic compounds including phenolic acids, flavan-3-ols, flavonols and stilbenes were identified in both wines, with flavanols the main family contributing to total phenolics, from 45.3 % to 89.9 %. Gallic acid, procyanidin dimer and *trans*-caftaric acid were found main compounds in both wines. Significant differences have been found depending on the region and vintage. It was observed that total phenolic content in wines from Durrësi region clearly stand higher compared to wines originating from Tirana region. As a result, significant variations were found among the cultivars in terms of the phenolic profiles and phenolic compounds of the *Shesh i bardhë* wines was much higher compared to the *Vlosh* wines.

**Keywords:** *Shesh i bardhë* cv., white wine, *Vlosh* cv., red wine, phenolic compounds, flavonols, resveratrol, Albania.

#### **INTRODUCTION**

Grapevine (Vitis spp.) is globally known as one of the essential fruit species, evidenced in the production of wine, grape juice, and other food formulations (Torregrosa et al., 2015). Its annual world production reached more than 79 million tons in 2018, being one of the world's largest fruit crops, while wine production over 29 million tones (OIV, 2019). Produced by must fermentation of grape, wine is considered a product of high commercial importance (Markoski et al., 2016). It contains polyphenols such as anthocyanins, flavonols and stilbenes (resveratrol), which originate mainly from red grapes seeds and skins (Flamini et al., 2013). Polyphenols are secondary metabolites, which based on their chemical structure are divided into two major groups: flavonoid and non-flavonoid compounds (Waterhouse, 2002). These natural metabolites arise from either the shikimate pathway or 'polyketide' acetate/malonate pathway, or both, producing monomeric and polymeric phenols (Lattanzio, Kroon, Quideau & Treutter, 2008). The flavonoids' group comprise different flavones, flavonols, flavanones, flavanols, anthocyanins, chalcones, and dihydrochalcones (Waterhouse, 2002; Kennedy, 2008), while non-flavonoids include phenolic acids, and stilbenes (Fernandes et al., 2017). A complex relationship between the factors such as temperature, sunlight, soil, water availability and physiological process of the vine variety influence the wine quality (Lattanzio, Kroon, Quideau & Treutter, 2008; Kelebek, Canbas, Jourdes & Teissedre, 2010). Sensory characteristics such as wines' color, flavor, astringency, and hardness; are directly associated with phenolics, or through their interaction with other wine constituents (El Khaland et al., 2018). Wine phenolics are used as markers of wine quality and authenticity (Merkyte, Longo, Windisch & Boselli, 2020). These compounds have been found to associate with positive effects on health, presenting protective or therapeutic effects on many degenerative and aging-related diseases such as cancer and cardiovascular diseases (Guerrero, García-Parrilla, Puertas, & Cantos-Villar, 2009; Fernandes et al., 2017).



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Shesh i bardhë is a native grape variety, widely planted in Albania, used in the production of one of the highquality dry white wines. Western regions of the country, especially to Durrësi and Tirana present main area of this grape cultivar. The grapevine variety Vlosh, is planted in Vlora region, and produce a light red wine (Susaj, Susaj & Duhanaj, 2018). According to our acknowledge, the influence of vineyard location on the phenolic content of Shesh i bardhë and Vlosh wines and how these factor affects the quality of wines have been never investigated and reported. Therefore, the aims of this research were to detect possible differences in phenolics composition and wine quality manufactured with wines of the Shesh i bardhë and Vlosh wines, according to cultivar, vintage and region. Red wine is the primary source of resveratrol isomers (trans- and cis-) and in its glucoside forms (piceid = resveratrol 3-O- $\beta$ -glucoside). Resveratrol is the parent molecule of a family of polymers named viniferins (Rosario-Bronze, Duarte, & Matias, 2012). The grapevine variety, climate conditions, phytosanitary conditions, and wine storage duration, are some of the influencing factors to the resveratrol content (Radovic, Tesevic, Kodzulovic & Maras, 2015).

Several epidemiological studies support a positive association between red wine consumption and human health. The potent antioxidant activity of phenolic compounds in red wine has been proposed as an explanation for the French paradox (Guerrero, García-Parrilla, Puertas, & Cantos-Villar, 2009). Consumption of wine polyphenols reduces total plasma cholesterol and/or LDL cholesterol, which plays a role in the development of atherosclerosis. Phenolic compounds exhibit health-promoting effects associated with skin protection, antioxidant, antibacterial, anticancer, anti-inflammatory, and anti-diabetic activities, as well as hepatoprotective, cardio-protective, and neuroprotective effects and other health benefits. Disease prevention is related to the potent antioxidant capacity of polyphenols in neutralizing the adverse effects of reactive oxygen species (ROS) (Friedman, 2014; Nassiri-Asl & Hosseinzadeh, 2016). Also, wine consumption is related to reducing the incidence of several cancers, such as renal, gastrointestinal, lung, and prostate cancers (Jose et al., 2014). A positive association between wine consumption and obesity has been claimed, with positive effects on Neurological Diseases by reducing the incidence of dementia and Alzheimer's diseases (Fernandes et al., 2017).

Several studies with a focus on trans-resveratrol have shown its ability to modulates lipid metabolism by protecting lipoproteins from oxidative damage and anticancer, antioxidant, anti-inflammatory, and cardioprotective properties, as well as the ability to inhibit platelet aggregation (Guerrero, García-Parrilla, Puertas, & Cantos-Villar, 2009; El Khawand et al., 2018). Nevertheless, these promising effects of resveratrol support the contention that moderate wine consumption is health-promoting (Fernandes, 2017).

Flavonols of Vitis vinifera red grape cultivars occur as three glycosylated series (3-O-glucosides, 3-O-glactosides, and 3-O-glucuronides) of the six possible flavonoid structures, according to the B-ring substitution pattern (kaempferol, quercetin, isorhamnetin, myricetin, laricitrin, and syringetin). They are yellow pigments classified as flavonoid phenolics located in the grape berry skins, where they are involved in UV screening, with their biosynthesis being light-dependent. They are identified as one of the best phenolics with antioxidant activity in wine, especially in white wines (Hermosín-Gutiérrez, Castillo-Munoz, Gomez-Alonso & Garcia-Romero, 2011).

#### MATERIALS AND METHODS

#### Chemicals

HPLC-grade methanol, acetonitrile, and formic acid (Merck, Darmstadt, Germany) were used after filtration through a 0.45-mm pore size membrane. Protocatechuic acid, procyanidins B1, B2, B3 and B4, resveratrol, all of them were purchased from Extrasynthese (Genay, France). Gallic acid, caftaric acid, coutaric acid, caffeic acid, fertaric acid, *p*-coumaric acid, gallocatechin, quercetin, quercetin-3-*O*-galactoside, quercetin-3-O-glucoside, kaempferol-3-O-glucoside, isorhamnetin-3-O-glucoside, and (+)-catechin and (–)-epicatechin were purchased from Sigma Chemical Co. (St. Louis, MO, USA).



Wine samples analyzed in this study were supplied by wine producers, produced during 2017 and 2018 harvests from vineyards located in three Albanian geographical regions: Vlora (*Vlosh* cv.), Tirana and Durrësi (*Shesh i bardhë* cv.). The *Shesh i bardhë* grape cv is widespread to hilly location across Central Albania. Vineyards is present in hilly locations which reach up to 200 m above sea level. Durresi and Tirana regions constitute the most intensive areas of V. vinifera cultivation, located in the Adriatic Sea coastline. Meanwhile, Vlosh cv. is present in the Vlora region, situated in the Southwestern Albania, shoring the Adriatic and Ionian seas. Altogether, three regions are classified as Csa according to Köppen and Geiger climate classification system (Kottek *et al.*, 2006). With specificities on climatic indicators among them presented in Table 1. Vineyards' locations of the regions: Vlora (40.49° N, 19.46° E), Tirana (41.36° N, 19.68° E), Durrësi (41.30° N, 19.50° E).

e	U X	,	· · ·
	Durrësi	Tirana	Vlora
Annual daily average temperatures (°C)	15.9	15.2	17.0
Daily mean temperatures for January (°C)	8.1	6.7	9.5
Daily mean temperatures for Warmest month, August (°C)	23.9	24.0*	24.5
Annual average precipitation (mm)	1245	1266	995
Lowest monthly precipitation (July) (mm)	31	42	9
Highest monthly precipitations (November) (mm)	186	172	192
Sunny days (days)	284	237	245

Table 1: Climatic indicators for Durrësi-Tirana regions and Vlora region (Kottek et al., 2006; DWD, 2021).

\*July is the warmest month to Tirana region.

Wine samples were taken directly from the wineries and were kept in glass bottles, dark place at 4°C before chemical analysis.

#### Meteorological data comprising vine regions

According to the official EU viticulture classification (EU, 2008), Albania falls into the Climatic Zone C (the warmest) (Figure 1), with three sub-zones C IIIB, C IIIA, and C II based on the viticulture vocation and wine-naming for each zone (WVS, 2018).

- *C IIIB European viticulture zone* covering the coastal and low-hilly western Albania. This zone rises to 400 m elevation and encompasses coastal plains. The yearlong mean temperatures are 15-16°C, the coldest month, January (5.6-7.5°C), the hottest month July, (26.4°C), with the minimum temperature (-5°C), and the days with frost is 5-6 days/year.
- *C IIIA European viticulture zone* Hilly and pre-mountainous regions, away from the sea influence. This zone includes areas between 400-800 m elevations and includes hilly pre-mountainous areas, far from the sea effects. The yearlong mean temperature is 12-13°C, the coldest month, January (2-4°C), the hottest month, July (25°C),
- *C II European viticulture zone* Mountainous areas present in South to North linearity, especially the Eastern regions of the country, with elevations over 800 m. The yearlong mean temperature (10-11°C), the coldest month, January (0.5-2°C), and the hottest month, July, (19-20°C), the minimum temperature (-13°C), days with frost (35-45 days/year).

The solar radiance for these regions is Vlora with 2790 hours, Durrësi with 2600 hours, belonging to the CIIIB European Viticulture Subzone, while Tirana with 2560 hours belongs to the CIIIA European Viticulture Subzone (WVS, 2018).

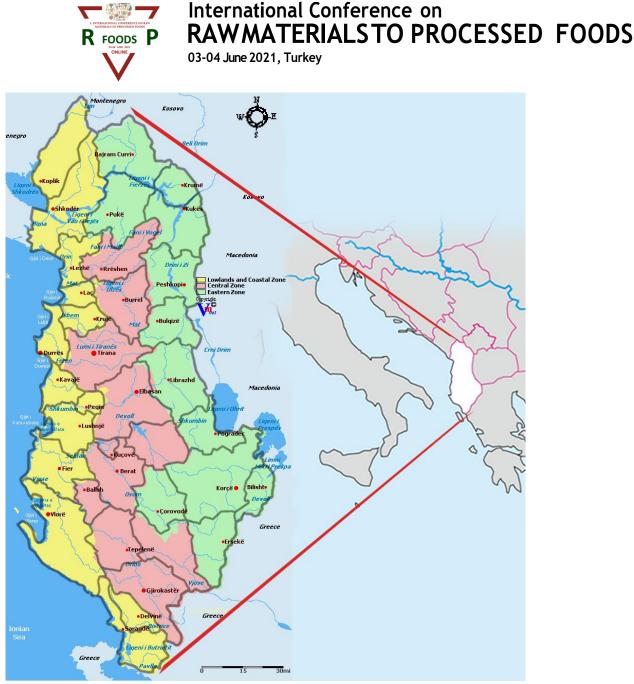


Figure 1: Map of Albania's wine regions (www.wineandvinesearch.com)

#### Standard chemical analysis

Titratable acidity, pH, and total sugar, SO<sub>2</sub>, ethanol, total extract, glucose, and malic acid analyses were performed on wines (OIV, 1990).

#### Liquid Chromatography-Tandem Mass-Spectrometry Analysis

The analysis of phenolic compounds was conducted based on the methods developed by Kelebek and coauthors using LC-DAD-ESI-MS/MS with negative ionization mode (Table 3) (Kelebek Sevidik, Uzlasir & Selli, 2020a; Kelebek, Selli & Sevindik, 2020b). High performance liquid chromatography equipment (Agilent 1260 HPLC; Agilent Tech., Palo Alto, California, USA) was utilized with a diode array detector (G1351D 1260 DAD VL). The system consisted of a binary pump (G1312 B, 1260 Bin pump), a degasser (G1322 A, 1260 Degasser) and an auto sampler (G1367 E, 1260 HIP ALS). The Phenomenex Luna reversed-phase C-18 column with the specification of 4.6 x 250 mm and 5  $\mu$ m (Torrance, California, USA) was employed in the analyses. Two mobile phases were utilized as solvent A, water/formic acid (99:1; v/v) and solvent B, acetonitrile/solvent A (60:40; v/v). Standard curves were obtained by using the commercial standards at concentrations normally exist in extracts (nearly 1–100 mg L<sup>-1</sup>) and obtaining regression values (r<sup>2</sup>) greater



than 0.995. In case of the reference compound absence, the calibration of similar substances was employed by taking the molecular weight correction factor into account. The limits of quantification (LOQ) and limits of detection (LOD) were computed by utilizing the S/N ratio values (signal to noise) of 10 and 3, respectively.

#### **RESULTS AND DISCUSSION**

#### Chemical composition of wines

The chemical compositions of wines from both regions are summarized in Table 2. Titratable acidity, pH, and sugar composition of must was in accordance with previous studies carried out on Vranac wines (Dordevic et al, 2018). However, there were significant differences in the mean values of chemical composition of the wines in relation to regions and years. In the literature, It was reported that in white wines varies between 209.5 and 285.5 mg L<sup>-1</sup>, (Waterhouse, 2002), while for the red wine, the values vary between 1200 – 1800 mg L<sup>-1</sup> (Proestos, Bakogiannis, Komaitis, 2012).

Туре	Shesh i	bardhë	Shesh	i bardhë	Vlosh	
	Durrësi	region	Tirana	a region	Vlora region	
Production year	2017	2018	2017	2018	2017	2018
Volatile acidity (gL-1)	0.33	0.21	0.50	0.42	0.40	0.52
Acidity (gL-1)	6.6	6.0	5.1	5.4	5.1	5.6
Free SO <sub>2</sub> / total SO <sub>2</sub> (mg L- 1)	8/36	6/33	11/40	12/38	12/40	12/45
Alcohol (% Vol)	12.10	13.20	12.90	13.02	12.00	12.20
Total sugars (G/F) (gL-1)	0.14	0.84	0.21	0.67	0.24	0.42
Total extracts (gL-1)	21	24.9	22	23.5	22.9	25.3
Specific gravity (g/ml)	0.9925	0.9926	0.9913	0.9918	0.9910	0.9915

Table 2: Chemical parameters of wines produced by both grape cultivars in different years.

#### Phenolic composition of wines

The identified compounds, depending on different families, with the information by HPLC-DAD-ESI-MS/MS analysis: retention time,  $\lambda_{max}$  in the ultraviolet region, molecular ion, main fragment ions in MS/MS were given in Table 3. A total of 32 phenolic compounds were identified and characterized in wine samples, including phenolic acids, hydroxybenzoic acids, flavanols, flavonols, and stilbenes. The phenolic content of wines was presented in Table 4. When the total amount of these compounds was evaluated, it was found that *Shesh i bardhë* (527.32-633.15 mg L<sup>-1</sup>) wines from the Durrësi region have a higher phenolic content compared to the Tirana region (162.17-175.32 mg L<sup>-1</sup>). It has been also determined that *Shesh i bardhë* wines have approximately 2 times higher phenolic potential than *Vlosh* wines.

#### Phenolic acids

Phenolic acids are a class of non-flavonoid phenolic compounds present in wines. Twelve phenolic acids were detected in both wine cultivars (Table 4). As can be seen, *Shesh i bardhë* wines contained more phenolic acids than *Vlosh* wines. The major phenolic acid in wines is gallic acid. Gallic acid is a naturally abundant plant phenolic compound. It is present in seeds as free gallic acid and as an ester attached to procyanidin polymers. It is also present in grape stems and may be increased by whole cluster fermentations. Gallic acid was the most abundant phenolic acid in both Shesh i bardhë (26.60-380.16 mg L<sup>-1</sup>) and Vlosh wines (30.70-118.15 mg L<sup>-1</sup>) (Table 4). These results are comparable with Greece red wines (10.9–46.3 mg L<sup>-1</sup>) (Kalithraka *et al.*, 2007), red wines from Italy (13.6–90.5 mg L<sup>-1</sup>) (Gambelli & Santaroni, 2004) and Chianti wines from Tuscany (57.0 mg L<sup>-1</sup>) produced by *Sangiovese* and *Trebbiano* cvs. (Burns *et al.*, 2000), Merlot red wines (84.8 mg L<sup>-1</sup>) from



Sicily (La Torre *et al.*, 2006), *Montepulciano* red wine (58.3 mg L<sup>-1</sup>) (Minussi *et al.*, 2003), *Cabernet-Sauvignon* red wines (70.8 mg L<sup>-1</sup>), from Bulgaria (Burn *et al.*, 2000), Spanish red wine (27.2 mg L<sup>-1</sup>) produced by grape cv. *Listan Negro* (Rodriguez-Delgado, Gonzalez, Perez-Trujillo & Garcia-Montelongo, 2002), Turkey red wines (30.5 mg L<sup>-1</sup>) produced by *Merlot* cv. (Kilinc & Kalkan, 2003). Weidner *et al.* (2013) have found that *V. vinifera* seeds contain considerable quantities of gallic acid in addition to other phenolic acids. These results are much higher compared with GA levels found in Italian white wines (3.5 mg L<sup>-1</sup>) produced by *Pinot Grigio* cv according to Minussi *et al.* (2003), Spanish white wines from Canary Islands (0.5 mg L<sup>-1</sup>), or French wines (2.0 mg L<sup>-1</sup>) from *Chardonnay* cv (Rodriguez-Delgado *et al.*, 2001).

The content of tartaric esters of hydroxycinnamic acid esters in all the white wines decreased in the following order: *trans*-caftaric acid > *trans*-coutaric acid > *trans*-caffeic acid > *trans*-fertaric acid. The *trans*-isomeric contents were found higher compared to the *cis*-isomers (Table 4). These trends are in accordance with the literature (Burns *et al.*, 2000; Pajovic-Scepanovic, Wendelin, Raicevic & Eder, 2019). *trans*- Caftaric acid content in the *Shesh i bardhë* white wines varied between 36.44–37.98 mg L<sup>-1</sup> and 37.19–45.79 mg L<sup>-1</sup>, from Durrësi and Tirana regions, respectively. In contrast, the *trans*-coutaric acid content was 7.77 mg L<sup>-1</sup>in 2017 from Durrësi region. *Trans*-caftaric acid levels to white wines from the Durrësi region were higher compared to the varietal origin of the wines (Gómez-Alonso, García-Romero, & Hermosín-Gutiérrez, 2007). The content of *trans*-caffeic acid in *Shesh i bardhë* white wines were found in the interval 4.89±0.08 to 7.55±0.2 mg L<sup>-1</sup>originating from the Durrësi region. Our data are comparable with Italian white wines *Greco di Tufo* (Minussi *et al.*, 2003) or French white wine *Chardonnay* (Teissedre & Landrault, 2000).

Another important phenolic acid is determined in wines as protocatechuic acid. The highest level was found in *the Vlosh* region's wines  $(15.05\pm0.24 \text{ mg L}^{-1})$ . In contrast, the highest concentration to *Shesh i bardhë* white wines belonged to the 2018 vintage from Tirana region  $(6.82\pm0.11 \text{ mg L}^{-1})$ , even though these levels were comparable among Durrësi and Tirana regions. Regarding *Vlosh* red wines, when compared with other publications, protocatechuic acid levels were higher compared to Italian red wines produced by *Montepulciano* cv.  $(7.2 \text{ mg L}^{-1})$  (Minussi *et al.* 2003), or French red wine produced by *Egiadola* cv.  $(5.0 \text{ mg L}^{-1})$  (Teissedre & Landrault, 2000), while referring to other publications, such as Italian red wine produced by *Barbera* cv.  $(2.6 \text{ mg L}^{-1})$  (Minussi *et al.* 2003), or red wines, from the region of Sicily *Merlot* cv.  $(3.2 \text{ mg L}^{-1})$  (La Torre *et al.*, 2006) French red wines produced by *Syrah* cv  $(2.0 \text{ mg L}^{-1})$  (Teissedre & Landrault, 2000). Protocatechuic acid levels from *Shesh i bardhë* white wines compared with published data results on white wines resulted higher than French *Chardonnay* white wines (Teissedre & Landrault, 2000), much higher when compared to Italian white wines produced by *Greco di Tufo* grape cv.  $(1.1 \text{ mg L}^{-1})$ , or *Pinot Grigio*  $(0.1 \text{ mg L}^{-1})$  (Minussi *et al.*, 2003), as well as Spanish white wines  $(0.5 \text{ mg L}^{-1})$  (Rodriguez-Delgado *et al.*, 2001).

The *p*-coumaric acid levels found to highest  $1.29\pm0.02$  mg L<sup>-1</sup>from both 2017 and 2018 vintages to the *Shesh i bardhë* white wines from the Tirana region. The total phenolic acid content in the *Shesh i bardhë* white wines studied was 106.63-109.416 mg L<sup>-1</sup>in wine samples from the Tirana region, to 465.44–576.1077 mg L<sup>-1</sup>in wine samples from Durrësi region. Also, total phenolic acids varied from 106.63±0.17 mg L<sup>-1</sup>originating from the Tirana region of 2017 vintage to 109.41±0.17 mg L<sup>-1</sup>from the Durrësi region of 2018 vintage.

The caffeic acid content to wines varied between 4.89-7.55 mg L<sup>-1</sup>in *Shesh i bardhë* wines and 6.12-10.54 mg L<sup>-1</sup>(Vlosh wines). These findings are in line with Burns *et al.*, (2000) Italian red wines *Sangiovese-Trebbiano* (5.5 mg L<sup>-1</sup>), Gambelli & Santaroni, (2004) *Montepulciano-Troia* red wine from Puglia region of Italy, and Spanish *Listan negro* red wine (Rodriguez-Delgado, Gonzalez, Perez-Trujillo & Garcia-Montelongo 2002). As well as lower compared to French red wine *Pinot Noir* from (19.1 mg L<sup>-1</sup>) (Burns *et al.*, 2000), *Merlot* red wine (19.0 mg L<sup>-1</sup>), and *Cabernet-Sauvignon* red wines (30.0 mg L<sup>-1</sup>) (Teissedre & Landrault (2000).

Table 3: LC-MS/MS data of the phenolic compounds detected in the wines (negative	e mode)
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		Abbreviation	tR	UV-VIS	( <b>M</b> – <b>H</b> ) <sup>–</sup>	
Peak	Compounds		(min)	$\lambda_{max} (nm)$	(m/z)	MS/MS (m/z)



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	Phenolic acids					
1	Gallic acid	GA	13,96	276	169	125
2	3-O-galloyl quinic acid	3-G_QuiA	14,53	274	343	191, 169, 125
3	Protocatechuic acid-O- hexoside	PCAhex	17,25	296	315	153
14	2-S-glutathionyl- caffeoyltartaric acid	2-S-glt_CaTaA	18,89	330	616	484, 440, 272
5	Protocatechuic acid	PCA	20,55	294	153	109
15	cis-Caftaric acid	c cafA	21,94	328	311	179, 149, 135
16	trans-Caftaric acid	t cafA	24,18	328	311	179, 149, 135
17	cis-Coutaric acid	c couA	31,11	310	295	163, 149
18	trans-Coutaric acid	t couA	32,70	314	295	163
19	cis-Fertaric acid	c ferA	34,83	322	325	193, 149
20	trans-Caffeic acid	t CA	35,83	323	179	135
21	trans-Fertaric acid	t ferA	36,16	328	325	193, 149
13	Ethylgallate	etGal	44,39	277	197	169, 125
22	<i>p</i> -Coumaric acid	p-CoumA	45,94	310	163	119
	Flavanols					
5	Gallocatechin	GCat	18,37	274	305	179, 125
6	Epigallocatechin	EpiGCat	25,10	274	305	179, 125
7	Procyanidin B3	B3	26,64	279	577	559, 425, 289
8	Procyanidin B1	B1	29,53	279	577	559, 425, 289
9	Catechin	Cat	30,97	280	289	245, 175
10	Procyanidin B2	B2	33,86	280	577	559, 425, 289
11	Epicatechin	EC	37,56	280	289	245, 175
12	Procyanidin B4	B4	42,93	280	577	559, 425, 289
	Flavonols					
23	Dihydrokaempferol 3-O-ß-d- glucoside	diHKa-3-Glc	38,47	290	449	287
24	Quercetin-3-O-galactoside	Que-3-gal	47,80	360	463	301
25	Quercetin-3-O-glucoside	Que-3-glu	48,14	360	463	397, 301
26	quercetin-3-O-glucuronide	Que-3-gln	48,49	355	477	301, 133
27	Isorhamnetin-3-O-glucoside	Iso-3-Glu	52,32	356	477	315, 301, 300, 299
28	Quercetin	Que	63,38	355	301	151
	Stilbenoids (Resveratrols)					
29	trans-Piceid	t Pic	47,46		389	227
30	cis-Piceid	c Pic	53,29		389	227
31	trans-Resveratrol	t Res	59,4		227	185, 159
32	cis-Resveratrol	c Res	64,34		227	185, 159

#### Flavanols

The flavonoids composition of white wines depends on the grape composition on their extraction into the grape juice and on the subsequent reactions occurring during the vinification, post-fermentation treatments, and wine aging (Rayne, Sheppard, Di Bello & Eggers, 2008). Flavanols constitute a large group of flavonoid compounds, while quantitatively, they are the most abundant secondary metabolites of grape berries. This group is comprised not only by monomers but also oligomers and polymers, extracted during winemaking, like condensed tannins or proanthocyanidins (PAs), being a major qualitative factor in red wines because of their implication in color stability, astringency, and bitterness (Huang, Cheynier & Terrier, 2008).



Nine flavanols; 3-O-galloyl quinic acid, gallocatechin, epigallocatechin, procyanidin B3, procyanidin B1, catechin, procyanidin B2, epicatechin, procyanidin B4. As shown in Table 4, wines obtained from the Durrësi region contained more flavanols than wines obtained from the Tiran region. Moreover, their concentrations were higher for the 2017 vintage in the region than in the 2018 vintage. Among the flavanols, 3-O-galloyl quinic acid was the most abundant in wines. 3-O-galloyl quinic acid in Shesh i bardhë white wines found 54.17±0.85 mg L<sup>-1</sup> and 37.18±0.59 mg L<sup>-1</sup> for 2017 and 2018 vintage, respectively, from the Durrësi region. These levels resulted in much lower in white wine samples from the Tirana region for both the 2017 and 2018 vintage (Table 4). Meanwhile, other gallotannins like gallocatechin, epigallocatechin was found in lower levels. So, gallocatechin levels to the Durrësi region resulted in the interval  $2.77-4.00 \text{ mg L}^{-1}$ , while from the Tirana region, these levels resulted in the interval 1.92-2.64 mg L<sup>-1</sup>. Epigallocatechin levels in wine samples from the Durrësi region belonged to the interval 1.89-3.92 mg L<sup>-1</sup>, while these values resulted in lower wine samples from the Tirana region (0.58-1.79 mg L<sup>-1</sup>). Comparison among gallotannins in Vlosh red wines found that the highest content belonged to 3-O-galloyl quinic acid 6.46±0.10 mg L<sup>-1</sup>to 9.13±0.14 mg L<sup>-1</sup>for 2017 and 2018 vintage, respectively (Table 4). Gallocatechin was found in lower levels,  $5.02\pm0.08$  mg L<sup>-1</sup>in 2017 vintage and  $5.79\pm0.09$  mg L<sup>-1</sup>in 2018 vintage. Epigallocatechin levels resulted in much lower levels. On Vlosh red wine, these results were found lower compared to red wines (2.8 mg  $L^{-1}$ ) from Spain (de Pascual-Teresa *et al.*, 2000). Ethyl gallate levels resulted in wide range values on white wines Shesh i bardhë (3.89±0.06 and  $72.37\pm1.14$  mg L<sup>-1</sup>) including two regions, while in *Vlosh* red wine, these values were found in the interval  $3.70\pm0.06$  to  $28.34\pm0.45$  mg L<sup>-1</sup>.

Catechin and epicatechin, two low molecular weight flavanols, were found in all the studied wine samples. The overall catechin levels were found higher compared to the epicatechin in all white and red wines. Their levels in *Shesh i bardhë* white wines from Durrësi region  $(8.75\pm0.14 \text{ mg L}^{-1})$  from 2017 vintage and  $15.34\pm0.59$  mg L<sup>-1</sup>for 2018 vintage, was lower compared to Tirana region,  $16.79\pm0.26$  mg L<sup>-1</sup>and  $17.48\pm0.28$  mg L<sup>-1</sup>for 2017 and 2018 vintage, respectively (Table 4). Our results show that catechin levels are higher than Italian *Pinot Grigio* (0.6 mg L<sup>-1</sup>) and *Greco di Tufo* cv. (4.9 mg L<sup>-1</sup>) white wines (Minussi *et al.*, 2003), Spanish white wines (1.0 mg L<sup>-1</sup>) (de Pascual-Teresa *et al.*, 2000). Meanwhile, they were found lower than French white wines (28.3 mg L<sup>-1</sup>) (Vitrac *et al.*, 2002). The crucial role of catechin in red wines astringency and bitterness has been widely described (Kelebek, Canbas, Jourdes & Teissedre, 2010).

Epicatechin levels, the second flavanol, in Shesh i bardhë wines originating from Durrësi region, were found in 1.48±0.02 mg L<sup>-1</sup>and 3.06±0.05 mg L<sup>-1</sup>for 2017 and 2018 vintage, respectively, compared to white wines from Tirana region, 3.35±0.05 mg L<sup>-1</sup> and 3.89±0.06 mg L<sup>-1</sup> for 2017 and 2018 vintage, respectively, indicating not a significant difference among two regions. Compared to reported data, the epicatechin concentrations are higher than reported Italian white wines *Pinot Grigio* cv. (0.3 mg L<sup>-1</sup>), and *Greco di Tufo* cv (2.8 mg L<sup>-1</sup>) according to Minussi et al. (2003), or French white wines ranging from 0.7-1.2 mg L<sup>-1</sup>(Arts et al., 2000), but in lower contents in French white wines (33.8 mg L<sup>-1</sup>) according to Vitrac et al. (2002). The catechin levels in *Vlosh* red wines was found at 11.79±0.19 mg L<sup>-1</sup> and 13.79±0.22 mg L<sup>-1</sup> for the 2017 and 2018 vintage, respectively (Table 4). Meanwhile, the epicatechin concentrations were  $3.82\pm0.06$  mg L<sup>-</sup> <sup>1</sup>and 4.63±0.07 mg L<sup>-1</sup> for the 2017 and 2018 vintage, respectively. Our results, regarding the catechin levels, are in line with those on Italian red wines  $(3.5-5.00 \text{ mg L}^{-1})$  published by Goldberg *et al.* (1995), but lower to other red wines from Spain (17.8 mg L<sup>-1</sup>) (de Pascual-Teresa et al., 2000), Italian Montepulciano red wine (14.0 mg L-1) (Minussi et al. (2003), and French Cabernet-Sauvignon red wine Teissedre & Landrault (2000). Compared to published data, the epicatechin levels resulted lower than French red wines  $(32.9 \text{ mg L}^{-1})$  (Vitrac et al., 2002), Spanish red wines 9.2-14.8 mg L<sup>-1</sup> (Rodriguez-Delgado, Gonzalez, Perez-Trujillo & Garcia-Montelongo, 2002), or Italian wines 32.3-69.2 mg L<sup>-1</sup> (La Torre *et al.*, 2006).



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Table 4: Phenolic compounds in *Shesh i bardhë* white wine and *Vlosh* red wine according to regions and the production' year (mg L<sup>-1</sup>)

	and the production' year (mg L <sup>-1</sup> )							
	Shesh i bardhë				Vlosh			
		Durrësi region Tirana region		-	Vlora region			
Peak	Phenolic acids	2017	2018	2017	2018	2017	2018	
1	Gallic acid	380.16±5.99	291.14±4.59	27.50±0.43	26.60±0.42	30.71±0.48	118.15±1.86	
2	3-O-galloyl quinic acid	54.17±0.85	37.18±0.59	8.86±0.14	11.16±0.18	6.46±0.10	9.13±0.14	
3	Protocatechuic acid-O- hexoside	4.27±0.07	3.93±0.06	1.64±0.03	0.99±0.02	7.11±0.11	7.76±0.12	
14	2-S-glutathionyl- caffeoyltartaric acid	3.24±0.05	4.96±0.08	2.96±0.05	3.12±0.05	0.63±0.01	0.77±0.01	
5	Protocatechuic acid	4.15±0.07	8.81±0.14	5.87±0.09	6.82±0.11	6.99±0.11	15.05±0.24	
15	cis-Caftaric acid	0.79±0.01	1.84±0.03	1.40±0.02	1.48±0.02	3.50±0.06	2.65±0.04	
16	trans-Caftaric acid	37.98±0.60	45.79±0.72	36.44±0.57	37.19±0.59	98.97±1.56	100.79±1.59	
17	cis-Coutaric acid	1.72±0.03	2.89±0.05	2.10±0.03	2.08±0.03	4.19±0.07	4.48±0.07	
18	trans-Coutaric acid	7.77±0.12	5.43±0.09	6.16±0.10	6.13±0.10	10.27±0.16	13.36±0.21	
19	cis-Fertaric acid	0.45±0.01	0.24±0.00	0.18±0.00	0.18±0.00	0.19±0.00	0.34±0.01	
20	trans-Caffeic acid	4.89±0.08	7.55±0.12	5.60±0.09	5.61±0.09	6.12±0.10	10.54±0.17	
21	trans-Fertaric acid	3.00±0.05	3.85±0.06	2.89±0.05	2.86±0.05	3.12±0.05	5.37±0.08	
13	Ethylgallate	72.37±1.14	50.61±0.80	3.73±0.06	3.89±0.06	3.70±0.06	28.34±0.45	
22	<i>p</i> -Coumaric acid	1.15±0.02	1.22±0.02	1.29±0.02	1.29±0.02	1.03±0.02	2.05±0.03	
	Total Phenolic acids	576.10±1.58	465.44±1.20	106.63±0.17	109.41±0.17	182.98±0.41	318.76±0.59	
	Flavanols							
4	Gallocatechin	4.00±0.06	2.77±0.04	1.92±0.03	2.64±0.04	5.02±0.08	5.79±0.09	
6	Epigallocatechin	3.92±0.06	1.89±0.03	0.58±0.01	1.79±0.03	0.51±0.01	0.10±0.00	
7	Procyanidin B3	15.75±0.25	17.00±0.27	15.30±0.24	15.75±0.25	30.76±0.48	28.03±0.44	
8	Procyanidin B1	10.98±0.17	13.17±0.21	10.93±0.17	10.98±0.17	20.92±0.33	17.80±0.28	
9	Catechin	8.75±0.14	15.34±0.24	16.79±0.26	17.48±0.28	11.79±0.19	13.79±0.22	
10	Procyanidin B2	4.50±0.07	1.40±0.02	2.12±0.03	4.50±0.07	3.84±0.06	5.17±0.08	
11	Epicatechin	$1.48\pm0.02$	3.06±0.05	3.35±0.05	3.89±0.06	3.82±0.06	4.63±0.07	
12	Procyanidin B4	5.25±0.08	3.48±0.05	0.87±0.01	5.25±0.08	1.80±0.03	$1.34{\pm}0.02$	
	Total flavanols	54.63±0.07	58.12±0.10	51.86±0.11	62.28±0.10	78.46±0.17	76.64±0.15	
	Flavonols							
	Dihydrokaempferol 3-O-							
23	ß-d-glucoside	$1.60\pm0.03$	1.84±0.03	2.35±0.04	2.29±0.04	6.16±0.10	$2.98 \pm 0.05$	
	Quercetin-3-O-							
24	galactoside	$0.02{\pm}0.00$	$0.01{\pm}0.00$	$0.03{\pm}0.00$	$0.01 \pm 0.00$	2.19±0.03	$2.35 \pm 0.04$	
25	Quercetin-3-O-glucoside	0.06±0.00	0.21±0.00	$0.08{\pm}0.00$	$0.08{\pm}0.00$	7.47±0.12	8.97±0.14	
26	Quercetin-3-O- glucuronide	0.56±0.01	1.30±0.02	0.73±0.01	0.72±0.01	7.76±0.12	13.96±0.22	
27	Isorhamnetin-3-O-	0.14:0.00	0.10.000	0.00.000	0.12:0.00	0.00.000	1.01.0.02	
27	glucoside	0.14±0.00	0.10±0.00	$0.08 \pm 0.00$	0.13±0.00	0.98±0.02	1.91±0.03	
28	Quercetin	0.01±0.00	$0.03 \pm 0.00$	$0.04{\pm}0.00$	0.03±0.00	7.59±0.12	5.27±0.08	
	Total Flavonols	2.39±0.04	3.49±0.06	3.32±0.05	3.25±0.05	32.15±0.51	35.43±0.56	
	Stilbenoids							
20	(Resveratrols)	0.01+0.00	0.07+0.00	0.11+0.00	0.02+0.00	0.22+0.00	0.45+0.01	
29	trans-Piceid	$0.01\pm0.00$	$0.07 \pm 0.00$	0.11±0.00	0.03±0.00	0.23±0.00	$0.45\pm0.01$	
30	cis-Piceid	$0.01\pm0.00$	0.13±0.00	0.04±0.00	0.13±0.00	0.13±0.00	$0.40\pm0.01$	
31	trans-Resveratrol	0.00±0.00	0.05±0.00	0.14±0.00	0.14±0.00	0.19±0.00	0.36±0.01	
32	cis-Resveratrol	$0.00 \pm 0.00$	$0.02 \pm 0.00$	$0.07{\pm}0.00$	$0.08 \pm 0.00$	$0.14 \pm 0.00$	$0.05 \pm 0.00$	
	Total Resveratrols	0.03±0.00	0.27±0.00	0.37±0.01	0.38±0.01	0.68±0.01	1.26±0.02	
	TOTAL PHENOLICS	633.15±9.98	527.32±8.31	175.32±2.76	162.17±2.56	294.28±4.64	432.09±6.81	

Four procyanidins B1, B2, B3, and B4, were analyzed in both white and red wine samples. Also, for *Shesh i* bardh $\ddot{e}$  white wines, these contents decreased in the following order Procyanidin B3 > Procyanidin B1 >



Procyanidin B4 > Procyanidin B2; while their order for *Vlosh* red wine Procyanidin B3 > Procyanidin B1 > Procyanidin B2 > Procyanidin B4, changed among Procyanidin B4 and Procyanidin B2. The most abundant from procyanidin dimers, Procyanidin B3, resulted in all *Shesh i bardhë* white wines, despite the vintage and the region origin,  $15.30\pm0.24$  mg L<sup>-1</sup>to  $17.00\pm0.27$  mg L<sup>-1</sup>, with the maximum levels to wines samples belonging to 2018, vintage from Durrësi region.

Procyanidin B3 levels in *Shesh i bardhë* white wines were much higher than French *Chardonnay* white wine  $(0.2 \text{ mg } \text{L}^{-1})$  (Tessedre & Laundrault, 2000). Similarly, the concentration of the procyanidins B1 in *Shesh i bardhë* white wines were found in the interval  $10.93\pm0.17 \text{ mg } \text{L}^{-1}$ to  $13.17\pm0.21 \text{ mg } \text{L}^{-1}$ , with the maximum amount in white wines from Durrësi region. These amounts were found much higher than that on Spanish white wines,  $0.3 \text{ mg } \text{L}^{-1}$ (de Pascual-Teresa *et al.*, 2000). The Procyanidin B2 and B4 were found in much lower concentrations in both Shesh i bardhë white wine and Vlosh red wines.

Procyanidin B2 levels on *Shesh i bardhë* white wines were found in higher levels than other publications on French white wines, 0.3 mg L<sup>-1</sup>(Tessedre & Laundrault, 2000), or Spanish white wines, 0.1 mg L<sup>-1</sup>(de Pascual-Teresa *et al.*, 2000). Procyanidin B4 levels on *Shesh i bardhë* wine were higher than that published by Teissedre & Landrault (2000) and de Pascual-Teresa *et al.* (2000).

Procyanidin B3 in *Vlosh* red wines was found in  $28.03\pm0.44$  mg L<sup>-1</sup>to  $30.76\pm0.48$  mg L<sup>-1</sup>, with the maximum belonging to the 2018 vintage. Procyanidin B3 was found in similar levels to French red wines, *Merlot* (25.0 mg L<sup>-1</sup>) and *Syrah* (25.0 mg L<sup>-1</sup>) (Teissedre & Landrault, 2000), while in lower levels, compared to other red wines such as French *Cabernet-Sauvignon* (44.0 mg L<sup>-1</sup>) and *Grenache*, 80.0 mg L<sup>-1</sup>(Tessedre & Laudrault, 2000). The concentration of the procyanidins B1 was found in 17.80±0.28 mg L<sup>-1</sup>to 20.92±0.33 mg L<sup>-1</sup>from the 2017 and 2018 vintage, respectively (Table 4). Compared with that of French Merlot (50.0 mg L<sup>-1</sup>), and *Cabernet-Sauvignon* (99.0 mg<sup>-1</sup>) red wines, according to Teissedre & Landrault (2000), as well as Italian *Merlot* red wines (La Torre *et al.*, 2006).

Procyanidin B2 levels on *Vlosh* red wine were similar to that published by de Pascual-Teresa *et al.* (2000), while in much lower levels compared with results published by Tessedre & Landrault (2000) such as French red wine *Merlot* (447.0 mg L<sup>-1</sup>), *Cabernet-Sauvignon* (53.0 mg L<sup>-1</sup>), *Syrah* (90.0 mg L<sup>-1</sup>), or Italian *Merlot* (42.4 mg L<sup>-1</sup>) red wines.

Procyanidin B4 levels on *Vlosh* red wine much lower with data published by Teissedre & Landrault, 2000) on French red wine *Merlot* (45.0 mg L<sup>-1</sup>), Cabernet-Sauvignon (53.0 mg L<sup>-1</sup>), or *Syrah* (20.0 mg L<sup>-1</sup>).

The maximum levels on the total HBA and flavonols to *Shesh i bardhë* white wines from the Durrësi region resulted in 569.74  $\pm$ 8.98 mg L<sup>-1</sup>from 2018 vintage; meanwhile, the total HBA and flavonols levels from the Tirana region was 111.73  $\pm$ 1.76 mg L<sup>-1</sup>belonging to 2017 vintage years.

The maximum levels on the total HBA and flavonols to the *Vlosh* red wine resulted in  $255.05 \pm 4.02$  mg L<sup>-1</sup> from the 2018 vintage.

Comparison among gallotannins found that the highest content belonged to 3-O-galloyl quinic acid in *Shesh i* bardhë white wines found  $54.17\pm0.85 \text{ mg L}^{-1}$  and  $37.18\pm0.59 \text{ mg L}^{-1}$  for 2017 and 2018 vintage, respectively, from Durrësi region. These levels resulted in much lower in white wine samples from the Tirana region, for both 2017 and 2018 vintage (Table 4). Meanwhile, other gallotannins like gallocatechin, epigallocatechin were found in lower levels. So, gallocatechin levels to the Durrësi region resulted in the interval 2.77-4.00 mg L<sup>-1</sup>, while from the Tirana region, these levels resulted in the interval 1.92-2.64 mg L<sup>-1</sup>. Epigallocatechin levels in wine samples from the Durrësi region belonged to the interval 1.89-3.92 mg L<sup>-1</sup>, while these values resulted in lower in wine samples from the Tirana region (0.58-1.79 mg L<sup>-1</sup>). Comparison among gallotannins in *Vlosh* red wines found that the highest content belonged to 3-O-galloyl quinic acid 6.46±0.10 mg L<sup>-1</sup>to 9.13±0.14 mg



L<sup>-1</sup>for 2017 and 2018 vintage, respectively (Table 4). Gallocatechin was found in lower levels,  $5.02\pm0.08$  mg L<sup>-1</sup>for 2017 vintage, and  $5.79\pm0.09$  mg L<sup>-1</sup>to 2018 vintage. Epigallocatechin levels resulted in much lower levels. These results, on *Vlosh* red wine, were found lower compared to red wines (2.8 mg L<sup>-1</sup>) from Spain (de Pascual-Teresa *et al.*, 2000). Ethylgallate levels resulted in wide range values on white wines *Shesh i bardhë* (3.89±0.06 mg L<sup>-1</sup>and 72.37±1.14 mg L<sup>-1</sup>) including two regions, while in *Vlosh* red wine, these values were found in the interval  $3.70\pm0.06$  mg L<sup>-1</sup>to  $28.34\pm0.45$  mg L<sup>-1</sup>.

Catechin ( $[M-H]^-$ , m/z 289) and epicatechin ( $[M-H]^-$ , m/z 289), two low molecular weight flavanols were found present in all the studied wine samples. The overall catechin levels were found higher compared to the epicatechin, in all white and red wines. The catechin levels in *Shesh i bardhë* white wines from Durrësi region (8.75±0.14 mg L<sup>-1</sup>) from 2017 vintage and 15.34±0.59 mg L<sup>-1</sup> for 2018 vintage, was lower compared to Tirana region, 16.79±0.26 mg L<sup>-1</sup> and 17.48±0.28 mg L<sup>-1</sup> for 2017 and 2018 vintage, respectively (Table 4). Our results show that catechin levels are higher compared to Italian *Pinot Grigio* (0.6 mg L<sup>-1</sup>) and *Greco di Tufo* cv. (4.9 mg L<sup>-1</sup>) white wines (Minussi *et al.*, 2003), Spanish white wines (1.0 mg L<sup>-1</sup>) (de Pascual-Teresa *et al.*, 2000). Meanwhile they were found lower compared to French white wines (28.3 mg L<sup>-1</sup>) (Vitrac *et al.*, 2002). The crucial role of catechin in red wines astringency and bitterness has been widely described (Kelebek, Canbas, Jourdes & Teissedre, 2010).

Epicatechin levels, the second flavanol, in *Shesh i bardhë* wines originating from Durrësi region, were found in  $1.48\pm0.02 \text{ mg L}^{-1}$  and  $3.06\pm0.05 \text{ mg L}^{-1}$  for 2017 and 2018 vintage, respectively, compared to white wines from Tirana region,  $3.35\pm0.05 \text{ mg L}^{-1}$  and  $3.89\pm0.06 \text{ mg L}^{-1}$  for 2017 and 2018 vintage, respectively, indicating not a significant difference among two regions. Compared to reported data, the epicatechin concentrations are higher than reported Italian white wines *Pinot Grigio* cv. (0.3 mg L<sup>-1</sup>), and *Greco di Tufo* cv (2.8 mg L<sup>-1</sup>) according to Minussi *et al.* (2003), or French white wines ranging from 0.7-1.2 mg L<sup>-1</sup>(Arts *et al.*, 2000), but in lower contents in French white wines (33.8 mg L<sup>-1</sup>) according to Vitrac *et al.* (2002).

The catechin levels in *Vlosh* red wines was found at 11.79±0.19 and 13.79±0.22 mg L<sup>-1</sup> for the 2017 and 2018 vintage, respectively (Table 4). Meanwhile, the epicatechin concentrations were  $3.82\pm0.06$  mg L<sup>-1</sup> and  $4.63\pm0.07$  mg L<sup>-1</sup> for the 2017 and 2018 vintage, respectively. Our results, regarding the catechin levels, are in line with those on Italian red wines (3.5-5.00 mg L<sup>-1</sup>) published from Goldberg *et al.* (1995), but lower to other red wines from Spain (17.8 mg L<sup>-1</sup>) (de Pascual-Teresa *et al.*, 2000), Italian *Montepulciano* red wine (14.0 mg L<sup>-1</sup>) (Minussi *et al.* (2003), and French *Cabernet-Sauvignon* red wine Teissedre & Landrault (2000). Compared to published data, the epicatechin levels resulted in lower than French red wines (32.9 mg L<sup>-1</sup>) (Vitrac *et al.*, 2002), Spanish red wines 9.2-14.8 mg L<sup>-1</sup> (Rodriguez-Delgado, Gonzalez, Perez-Trujillo & Garcia-Montelongo, 2002), or Italian wines 32.3-69.2 mg L<sup>-1</sup> (La Torre *et al.*, 2006).

Four procyanidins B1, B2, B3, and B4, were analyzed in both white and red wine samples. Also, for *Shesh i* bardhë white wines, these contents decreased in the following order Procyanidin B3 > Procyanidin B1 > Procyanidin B4 > Procyanidin B2; while their order for *Vlosh* red wine Procyanidin B3 > Procyanidin B1 > Procyanidin B2 > Procyanidin B4, changed among Procyanidin B4 and Procyanidin B2. The most abundant from procyanidin dimers, Procyanidin B3, resulted in all *Shesh i* bardhë white wines, despite the vintage and the region origin,  $15.30\pm0.24$  mg L<sup>-1</sup>to  $17.00\pm0.27$  mg L<sup>-1</sup>, with the maximum levels to wines samples belonging to 2018, vintage from Durrësi region.

Procyanidin B3 levels in *Shesh i bardhë* white wines were much higher compared to French *Chardonnay* white wine  $(0.2 \text{ mg L}^{-1})$  (Tessedre & Laundrault, 2000).

Similarly, the concentration of the procyanidins B1 in *Shesh i bardhë* white wines were found in the interval  $10.93\pm0.17 \text{ mg L}^{-1}$  to  $13.17\pm0.21 \text{ mg L}^{-1}$ , with the maximum amount in white wines from Durrësi region. These



amounts were found much higher compared with that on Spanish white wines, 0.3 mg  $L^{-1}$  (de Pascual-Teresa *et al.*, 2000).

The procyanidin B2 and B4 were found in much lower concentrations in both Shesh i bardhë white wine and Vlosh red wines.

Procyanidin B2 levels on *Shesh i bardhë* white wines were found in higher levels compared with other publications on French white wines, 0.3 mg L<sup>-1</sup> (Tessedre & Laundrault, 2000), or Spanish white wines, 0.1 mg L<sup>-1</sup> (de Pascual-Teresa *et al.*, 2000). Procyanidin B4 levels on *Shesh i bardhë* wine was higher with that published by Teissedre & Landrault (2000) and de Pascual-Teresa *et al.*, (2000).

Procyanidin B3 in *Vlosh* red wines was found in  $28.03\pm0.44$  mg L<sup>-1</sup>to  $30.76\pm0.48$  mg L<sup>-1</sup>, with the maximum belonging to the 2018 vintage. Procyanidin B3 was found in similar levels to French red wines, *Merlot* (25.0 mg L<sup>-1</sup>) and *Syrah* (25.0 mg L<sup>-1</sup>) (Teissedre & Landrault, 2000), while in lower levels, compared to other red wines such as French *Cabernet-Sauvignon* (44.0 mg L<sup>-1</sup>) and *Grenache*, 80.0 mg L<sup>-1</sup> (Tessedre & Laudrault, 2000). The concentration of the procyanidins B1 was found in 17.80±0.28 mg L<sup>-1</sup> to 20.92±0.33 mg L<sup>-1</sup> from the 2017 and 2018 vintage, respectively (Table 4). Compared with that of French Merlot (50.0 mg L<sup>-1</sup>), and *Cabernet-Sauvignon* (99.0 mg L<sup>-1</sup>) red wines, according to Teissedre & Landrault (2000), as well as Italian *Merlot* red wines (La Torre *et al.*, 2006).

Procyanidin B2 levels on *Vlosh* red wine were similar with that published by de Pascual-Teresa *et al.* (2000), while in much lower levels compared with results published by Tessedre & Landrault (2000) such as French red wine *Merlot* (447.0 mg L<sup>-1</sup>), *Cabernet-Sauvignon* (53.0 mg L<sup>-1</sup>), *Syrah* (90.0 mg L<sup>-1</sup>), or Italian *Merlot* (42.4 mg L<sup>-1</sup>) red wines.

Procyanidin B4 levels on *Vlosh* red wine much lower with data published by Teissedre & Landrault, 2000) on French red wine *Merlot* (45.0 mg L<sup>-1</sup>), Cabernet-Sauvignon (53.0 mg L<sup>-1</sup>) or *Syrah* (20.0 mg L<sup>-1</sup>).

The maximum levels on the total HBA and flavonols to *Shesh i bardhë* white wines from the Durrësi region resulted in 569.74  $\pm$ 8.98 mg L<sup>-1</sup>from 2018 vintage, meanwhile, the total HBA and flavonols levels from the Tirana region, was 111.73  $\pm$ 1.76 mg L<sup>-1</sup>belonging to 2017 vintage years.

The maximum levels on the total HBA and flavonols to the *Vlosh* red wine resulted in  $255.05 \pm 4.02$  mg L<sup>-1</sup>, from the 2018 vintage.

#### Flavonols

Flavonols are yellow pigments that contribute directly to the color of white wines, while their presence in red wines is masked by anthocyanins, the red pigments (Castillo-Munoz, Gómez-Alonso, García-Romero, & Hermosín-Gutiérrez, 2007). Regarding the group of flavonols, quercetin derivatives are the main components present in the white wines (De Beer, Joubert, Gelderblom & Manley, 2005).

Flavonol profiles consist of a mixture of flavonol 3-O-glycosides and free flavonol aglycones released from their 3-O-glycosides hydrolysis compared with those of their grapes (Hermosín-Gutiérrez, Castillo-Munoz, Gomez-Alonso & Garcia-Romero, 2011). Six flavonols were detected in both analyzed wine samples: dihydrokaempferol 3-O-b-D-glucoside, quercetin-3-O-glucuronide, quercetin-3-O-galactoside, quercetin-3-O-glucoside, isorhamnetin-3-O-glucoside, and quercetin. In Shesh i bardhë wines, the highest concentration was dihydrokaempferol 3-O- $\Box$ -D-glucoside ([M–H]<sup>-</sup>, m/z 449) originating in the Tirana region, 2.35±0.04 mg L<sup>-1</sup> and 2.29±0.04 mg L<sup>-1</sup> in 2017 and 2018 vintages, respectively. Quercetin-3-O-glucuronide ([M–H]<sup>-</sup> detected at m/z 477) was the second flavonols found in the highest concentrations with the highest concentration (1.30±0.02 mg L<sup>-1</sup>) belonging to white wine samples from the 2018 vintage originating from the Durrësi region. Quercetin ([M–H]<sup>-</sup> detected at m/z 301) was found in trace amount in the interval 0.01-0.04



mg L<sup>-1</sup>. This finding is consistent with Ragusa et al. (2017) that have presented a study on Italian wines from the Apulia region. The total flavonols content resulted in similar range with wine samples originating from both regions, with the highest level belonged to the Durrësi region,  $3.49\pm0.06$  mg L<sup>-1</sup> to 2018 vintage. Similar levels to *Shesh i bardhë* white wine from the Tirana region,  $3.25\pm0.05$  to  $3.32\pm0.05$  mg L<sup>-1</sup>, were detected in 2017 and 2018 vintage years, respectively.

Total flavonols were found in the interval  $32.15\pm0.51 \text{ mg L}^{-1}$  to  $34.43\pm0.56 \text{ mg L}^{-1}$  from the 2017 and 2018 vintage of *Vlosh wines*. Quercetin levels in *Vlosh* red wine resulted in line with data presenting Bulgarian red wines *Merlot* (7.7 mg L<sup>-1</sup>), *Cabernet Sauvignon* (7.3 mg L<sup>-1</sup>) (Tsanova-Savova & Ribarova, 2002), Spanish *Tempranillo* red wine (Burns *et al.*, 2000), French red wine (5.8 mg L<sup>-1</sup>) and Spanish *Cabernet Sauvignon* (6.3 mg L<sup>-1</sup>) McDonald *et al.* (1998). Meanwhile, these data resulted lower compared to *Montepulciano* red wine from the Molise region, Italy (11.6 mg L<sup>-1</sup>) (Gambelli & Santaroni, 2004), as well as *Sangiovese* and *Trebbiano* cv. (17.5 mg L<sup>-1</sup>) (Burns *et al.*, 2000) *Pinot noir* from France (12.5 mg L<sup>-1</sup>) (Burns *et al.*, 2000).

Among flavonols, both white wines (*Shesh i bardhë*) and red wine (*Vlosh*) contained only the quercetin aglycone. In contrast, kaempferol, isorhamnetin as well as quercetin was detected in their glycosidic units, as isorhamnetin-3-O-hexoside ( $[M-H]^-$  detected at m/z 477) and quercetin-3-O-glucuronide ( $[M-H]^-$  detected at m/z 477). The concentrations of berry flavonols in the resulting wines depend on the grape variety, cultural practices, and environmental factors (Mattivi *et al.*, 2006). Partial hydrolysis reactions decrease concentrations of derived 3-O-glycosides in favor of the corresponding aglycones during the storage of the wine (Castillo-Muñoz *et al.*, 2007). These results are consistent with the literature (Budic-Leto *et al.*, 2017). The total flavonols resulted in 32.15 ±0.51 mg L<sup>-1</sup> and 35.43± 0.56 mg L<sup>-1</sup> in the 2017 and 2018 vintage years.

#### Stilbenoids

The main stilbenes found in grapes are *trans*-resveratrol and *cis*-resveratrol (3,5,4'-trihydroxystilbene) together with two resveratrol glucosides, *cis*-piceid, and *trans*-piceid (Flamini & De Rosso, 2018). Analysis of *Vlosh* red wine samples presented t*rans*-resveratrol glucosides as the dominant forms of stilbenes for all the studied wines in both vintages, as confirmed by literature (Kostandinovic *et al.*, 2012; Ragusa *et al.*, 2017). *Trans*-piceid (m/z 389) content was found 0.23 mg L<sup>-1</sup> and 0.45 mg L<sup>-1</sup> for the 2017 and 2018 vintage, respectively. The second most abundant stilbenes were the *cis*-piceid (m/z 389), 0.13 mg L<sup>-1</sup>, and 0.40 mg L<sup>-1</sup> in 2017 and 2018 vintages, respectively. *Trans*-resveratrol (m/z 227) was found 0.19 mg L<sup>-1</sup> to 0.36±0.01 mg L<sup>-1</sup> in 2017 and 2018 vintage, respectively.

The total resveratrol content was found  $0.68 \pm 0.01 \text{ mg L}^{-1}$  and  $1.26 \pm 0.02 \text{ mg L}^{-1}$  in the 2017 and 2018 vintage years, respectively. These results were in line with those presented in Montenegrin *Vranac* wines, 1.00- 5.70 mg L<sup>-1</sup> (Pajovic-Stepanovic, Wendelin, Raicevic & Eder 2019), Greek red wines  $0.0-9.72 \text{ mg L}^{-1}$  (Kallitraka, Mamalos & Makris, 2007) and Spanish red wines ranging between and  $0.4-5.2 \text{ mg L}^{-1}$ , (Monagas, Suarez, Gomez-Cordoves & Bartolome, 2005). In red wine, the concentrations of the *trans*-isomer, which is the dominant form, generally range between 0.10 and 15 mg L<sup>-1</sup> (Rosario-Bronze, Duarte & Matias, 2012). The levels of trans-resveratrol in *Vlosh* red wine were found in lower levels compared to Italian red wines (3 mg L<sup>-1</sup>) from the Apulia region (Ragusa et al. 2017), or trans-resveratrol in red wines, with *trans*-resveratrol levels, found 1.31 mg L<sup>-1</sup> (Dekic *et al.*, 2008), or North Macedonia red wines with higher concentrations of *trans*-piceid (4.65 ± 0.46 mg L<sup>-1</sup>), *trans*-resveratrol 1.49 ± 0.06 mg L<sup>-1</sup> produced by *Merlot* cv, while the *trans*-piceid 2.24 ± 0.08 mg L<sup>-1</sup> from *Vranac* cv. (Kostandinovic *et al.*, 2012).

Shesh *i* bardh $\ddot{e}$  wine samples revealed the presence of four stilbenes, *cis*- and *trans*-resveratrol, and their glucosides, *cis*- and *trans*-piceid. The *trans*-resveratrol was the most abundant (0.14 mg L<sup>-1</sup>) among stilbenes from the 2017 vintage, belonging to the Tirana region. The *cis*-piceid resulted in 0.13±0.00 mg L<sup>-1</sup>. The total



resveratrol content varied between 0.03 - 0.27 mg L<sup>-1</sup> from the Durrësi region, while  $0.37 - 0.38 \pm 0.01$  mg L<sup>-1</sup> to 2017, from the Tirana region, by concluding that white wines from Tirana region resulted in a higher content of stilbenes. *Trans*-resveratrol content is similar to white wines, 0.005-0.57 mg L<sup>-1</sup>, from Greece (Gerogiannaki-Christopoulou, Athanasopoulos, Kyriakidis & Spanos, 2006), Italian white wines (0.1-0.3 mg L<sup>-1</sup>) from the Apulia region (Ragusa *et al.* 2017), white wines from Serbia with a range from 0.11 to 0.34 mg L<sup>-1</sup>, (Cvejic *et al.*, 2010), and Montenegrin wines with *trans*-resveratrol glucosides varied 0.06-0.21 mg L<sup>-1</sup> (Pajovic-Stepanovic, Wendelin, Raicevic & Eder, 2019).

#### CONCLUSION

This study presents information on the phenolics composition from wines produced by two grape cvs. *Shesh i bardhë* and *Vlosh*. Information about the characteristics of red and white wines from Albania may help national authorities to control wine authenticity, as well as to develop PDO protection certifications intended by the wine sector. The results obtained from the 2017 and 2018 vintage suggest that white wines produced from grape cv. *Shesh i bardhë*, show high content of flavanols, flavonols, phenolic acid and stilbenes in samples from Durrësi terroir, up to 633.15 mg L<sup>-1</sup>, compared to Tirana terroir, up to 175.32 mg L<sup>-1</sup>. Also, dihydrokaempferol-3-O-b-D-glucoside was the most abundant flavonol, procyanidin B3 was the most abundant flavanol, *trans*-caftaric acid was the most dominant phenolic acid, and *trans*-resveratrol among stilbenes, in *Shesh i bardhë* white wines. The total phenolics to *Vlosh* red wines, quercetin-3-O-D-glucoside was the most abundant flavonol; the most abundant flavonol; the most abundant flavonol; the most abundant flavonol; the most abundant flavonol; the most abundant flavanol was found Procyanidin B3; *trans*-caftaric acid was the most abundant phenolic acid, and *trans*-caftaric acid was the most abundant phenolic acid, and *trans*-caftaric acid was the most abundant phenolic acid, and *trans*-caftaric acid was the most abundant phenolic acid, and *trans*-caftaric acid was the most abundant phenolic acid, and *trans*-piceid among stilbenes. Studying wines fermented by local grape cultivars will contribute to the global wines database and increase the perspective of local viticulture in Albania.

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## Effect of Storage on Fatty Acid Composition of Hazelnut (*Corylus avellana* L.) Varieties Cultivated in Turkey

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#### ABSTRACT

This paper aimed to study the effect of storage on the fatty acids profile of Hazelnut (*Corylus avellana* L.). 14 different hazelnut cultivars (cavcava, çakildak, ince kara, kalın kara, kara fındık, kargalak, kuş, mincane, palaz, sivri, tombul, uzun musa, yassi badem, and yuvarlak badem), grown in Turkey and harvested in 2018, 2019, and 2020 were used as samples. The first two samples were stored in room conditions. The oils were extracted in 2020. The major fatty acids were oleic acid and linoleic acid in 42 samples. The results of the analysis showed that the fatty acid profiles of hazelnut cultivars were affected by storage. However, this effect varied according to the cultivars and fatty acids, and the changes did not show a specific characteristic. The biggest deviations were also observed in oleic acid and linoleic acid, while the deviation of the palmitoleic acid was the smallest. It was also revealed that storage had negative effects on the nutritional quality of hazelnut kernels.

Keywords: Corylus avellana L., Effect of Storage, Fatty Acid Composition, Turkey



## The Effect of Maturation Status on Fatty Acid Profile of *Xanthium strumarium* L. Oil

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#### ABSTRACT

Cocklebur is a very precious medicinal plant due to its biochemical contents with antioxidant, antimicrobial, and antioxidants. Besides these properties, it can be evaluated as a source of energy. Cocklebur seeds and seed oil is not edible and contains 67% more biomass than cotton. Therefore, it can be evaluated as a sustainable energy source. Recently, the use of cocklebur as biodiesel makes this plant a point of interest for researchers. However, more studies are needed for evaluating factors that affect the quality of this kind of oil for desire application. The fatty acid profile of matured and immature oil of cocklebur pulp with seeds from Osmaniye province in Turkey was investigated in this study. Although the main fatty acid in the immature sample was linoleic acid with 55.36%, its amount (5.41%) decreased sharply in mature sample oil. Despite Linoleic acid, mature cocklebur oils had higher concentrations of oleic acid (50.17%), palmitic acid (27.38%), and stearic acid (9.35%). These results showed that maturation status significantly affected the concentration and rates of fatty acids in studied Cocklebur. This study will help international researchers on the evaluation of qualified seed oils for application both in industry and biodiesel production.

Keywords: Biodiesel, fatty acids, maturation status, *Xanthium strumarium* L.



## Olive By Products: Low-Cost, Renewable Source of High Added Value Phenolic Compounds and Their Biological and Functional Activities

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#### ABSTRACT

Phenolic compounds, present in plants, are an essential part of the human diet due to their antioxidant properties. These compounds posses an aromatic ring bearing one or more hydroxyl groups and their structures may range from that of a simple phenolic molecule to that of a complex high-molecular weight polymer. Olive oil and olive by-products are the major sources of phenolic compounds in the Mediterranean countries. Indeed, Olea europaea L. organs and by-products such as: leaves, olive mill wastewater, wood, stems and roots represent a major disposal problem for the industry concerned, but they are also promising sources of phenolic compounds which have been associated with numerous in vivo and in vitro biological activities and used for traditional medicinal purposes. In fact, olive oil and some of its by-products has been the subject of investigations and have proven to be effective sources of phenolic antioxidants. Principally, biological activities and characteristic flavour and aroma are due to the presence of unique bioactive compounds both in olives and extra virgin olive oil phenolic compounds such as oleuropein, hydroxytyrosol, verbascoside and derivatives etc., and tocopherols and carotenoids. Several factors, such as agronomical conditions, climate, and level of ripening, olive cultivar and type of production process have the main effects on the profile and activities of bioactive compounds. Accumulating evidence suggests that EVOO may have health benefits; it can be considered as an example of a functional food containing a variety of biologically active phenolic components that may contribute to its overall therapeutic characteristics. This chapter highlights the potential of olive oil and selected by-products as a source of phenolic compounds and their biological and functional properties.



## Assessment of Acrylamide in Potato Chips and French Fries Consumed by the Romanian Population

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#### ABSTRACT

The aim of this study was to estimate the acrylamide (AA) content of potato-based products consumed by the Romanian population and AA intake by consuming such products. 16 samples of potato chips (PC) from the market, 8 French fries ready-to-eat (FF) samples from fast-foods and 34 households FF samples were analyzed for AA content by using the GC-MS/MS and color parameters. The mean AA content of PC, FF from fast-food and prepared in households was 386.67, 149.55, and 530.32  $\mu$ g/kg, respectively. 19% of the PC samples exceeded the benchmark level of 750  $\mu$ g/kg set by EC (2017). 50% of FF samples prepared in households exceeded the established benchmark level of 500  $\mu$ g/kg, while all FF ready-to-eat samples from fast-food had an AA content below the threshold level. A linear correlation between the AA level and the color parameters  $L^*$ ,  $a^*$ , and  $\Delta E^*$  was found for FF samples prepared in households, with the linear correlation coefficients (R) ranging between 0.80 – 0.85. No correlation was found between AA content and color parameters for PC and FF from fast-food samples. The contribution to the total exposure to AA by consumption of PC and FF was calculated to be 45 - 73% of the maximum value estimated by EFSA (0.6  $\mu$ g/kg b.w./day). In order to ensure consumers' safety, food industry producers should control the AA content for the food products delivered to the market. Moreover, consumers should apply appropriate preparation and frying practices in their households, in accordance with the recommendations for reducing the level of AA in FF.

Keywords: Acrylamide, fast-food, household, potato-based products

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### Volatiles of Canned Tuna Fish and The Effects of Different Parameters

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#### ABSTRACT

Tuna is one of the most important commercial fish species due to its economical and high nutritional values. There are many species of fish, which are eaten raw, used in home-cooked dishes and subject to various industrial activities, such as canning. Tuna processing methods are mainly freezing, cooking, smoking, and canned after heat treatment. The species used for canning are mainly yellowfin, skipjack and albacore tuna. The flavor components of tuna vary depending on the processing method. The different temperature conditions applied in tuna canning productions significantly affect the aroma profiles of tuna. The aroma is one of the main indicators on which consumers judge the fish's freshness. Fish flavour quality is changing rapidly according to the freshness of the product, and therefore, sensory analysis of the flavouring is used by consumers, researchers and the fishing industry as a whole to evaluate the quality of fish. Each species of fish has a tender and distinctive aroma, which can be influenced by processing technology, post production and storage methods. Volatile compounds derived from lipid are produced mainly by oxidative-enzymatic reactions and autoxidation of lipids. These are important compounds in fish which give its aroma. Therefore, this review highlights the impact of various parameters on volatiles of canned tuna for use in future studies.

Keywords: Volatiles, canned tuna, freezing, cooking, smoking, aroma

#### **INTRODUCTION**

Tuna is one of the most commercial fish species, due to its excellent economic and nutritional benefits. It can be consumed raw, evaluated under culinary or even industrial processes such as canning. The main processing methods for tuna are freezing, cooking, smoking and canning after being thermally processed (Miao et al., 2017). Generally, the species used for canning are yellowfin, skipjack and albacore tuna (Zhang et al., 2019). Tuna is one of the most widely consumed fish in terms of international seafood production (8%) and is sold commercially all over the world (4.3 million tonnes) (Kumar & Kocour, 2015). Tuna has a lot of

differences compared to other fish in terms of growth rate, size, lifespan, maturity age and spawning period. Species which are restricted to tropical and sub-tropical areas (skipjack and yellowfin tuna) are distinguished by their small to medium size, quick growth, and early ripening. The bluefin tuna, instead, shows the characteristics of a highly variable life with a longer spawning period and a shorter lifetime compared to other tuna species (Fromentin and Fonteneau, 2001). Skipjack tuna has a black and purplish blue upper part and 4 to 6 stripes on the abdomen. Their body shapes are longitudinally round. While these fish prefer waters with a temperature about 25°C during their larval stage, they deserve to live in water at a temperature around 15°C when they reach the growth stage. The depth distribution ranges from about 260 meters from the surface during the day, they live near surface waters. The skipjack spawns all year long in subtropical waters, but it gets shorter and further away from the equator. Female skipjack tuna with a fork length of 41 to 87 cm can lay anywhere between 80,000 and 2 million eggs (FAO, 2020a).

As important commercial species, In 2018, the primary target of purse seine fishing in the Eastern Pacific Ocean was Katsuwonus pelamis with 289,000 tons (IATTC 2019). In China, large quantities of fish byproducts are produced during processing of canned tuna production, including flakes, skin, heads, and viscera, and compounds such as collagens, gelatines, and bioactive peptides are obtained from these bones, heads, and black muscles (Yang et al., 2019; Liu et al., 2015; Chi et al., 2015). Yellowfin tuna is the second tuna species



mostly consumed worldwide and responsible for 27% of the total world catch (ISSF, 2015). Yellowfin tuna reached a maximum length of 208 cm and a maximum weight of 176.4 kg. The black and dark blue color and the silver-colored belly characterize the Yellowfin tuna. Anal and dorsal fins are light yellow in color. These fish usually feed on fish, squid and crustaceans. Although their breeding season is known as summer, they actually multiply all year long (FAO, 2020b).

Seafood is nutritionally rich in protein, omega-3 fatty acids, unsaturated fatty acids, vitamins, micro and macronutrients, and their consumption by humans is increasing worldwide (Pieniak et al., 2010). Fish consumption reduces chronic noncommunicable diseases such as cardiovascular disease, mental disorders, rheumatoid arthritis, as well as several cancers, and promotes normal neuronal development in children (Di Giuseppe et al., 2014; Swanson et al., 2012; Virtanen et al., 2008). Moreover, the consumption of omega-3 fatty acid has proved its role in preventing irregular heart rhythms, controlling rheumatoid arthritis and suppressing breast cancer (Geusens et al., 1994; Belch and Muir, 1998; Nair et al., 1997; Rose and Connolly, 1990 Rose, 1997). Omega-3 fatty acids such as eicosapentaenoic acid (EPA; 20: 5n-3) and docosahexaenoic acid (DHA; 22: 6n-3) have major health benefits: for brain and retina development and the prevention of coronary artery disease (Swanson et al., 2012; FAO / WHO, 2010). Omega3 fatty acids that cannot be synthesized in the body are recommended to be taken from diets and food (Plourde and Cunnane, 2007).

In this review, the effects of different processing parameters (such as production techniques, temperature, drying, cooking, smoking and freezing) on the aroma compounds of canned tuna have been compiled, since no such study has been conducted in the literature before.

#### **VOLATILES OF CANNED TUNA**

The canning process is one of the most important methods of preserving fish for a long time. Canned fish and fish products play a crucial role in human diet. Fish species possess different nutrient compositions and they become stable when the fish is subjected to thermal treatments during canning process. According to a report, lean fish is not recommended in the canning process, since the meat breaks down under high temperatures, thereby losing both its taste and texture (Aberoumand, 2014). The aroma changes during the canning process depending on compounds derived from lipid oxidation and thermal degradation of carbohydrates, or the compounds originating from other reactions such as Strecker degradation and the Maillard reaction. The principal changes occur due to sterilization during the canning process which results in the release of furans, nitrogenous compounds, branched aldehydes and sulphur compounds (De Quirós et al., 2001). The canning process improves the organoleptic properties of fish obtained from canned silver. Canned smokes as well as minced fish from cans possess a marked increase of taste and color. The overall acceptance is gradually declines in all groups of canned fish (Khallaf et al., 1997). In the canning process, both enzymes and bacteria population should be permanently inactivated by heat treatments and when no reinfection or negative interaction takes place with the container, heat-processed fish can be kept for a very long time. On the other hand, a number of adverse effects also exist in the canning process, such as, loss of essential nutrients, release of unwanted compounds, browning, deterioration of lipids and proteins (Lukoshkina and Odoeva, 2003).

Temperature, used in seafood processing, has an enormous influence on the type and quantities of aroma and aroma-active compounds (Moreira et al., 2013). A number of studies have been conducted to aroma and aroma-active compounds from various cooked fish and other types of marine products (Milo et, al., 1993; Tao et al., 2014). Volatile compounds produced by oxidation during heat treatments of fish have been identified in several reports (Medina et al. 1999). De Quirós et al. (2001), identified the volatile compounds present in fresh and preserved sea urchins (Paracentrotus lividus, Lamarck) and reported that the sterilization process gave rise to significant changes in the profile of volatile compounds.

## 2.1. The effect of different production techniques on volatile composition of fish 2.1.1. Effect of drying

Drying is known to be a crucial parameter to prolong the shelf-life of fresh fish and other fishery products. It



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is possible for fishes to be maintained by solar drying, which focuses on reducing the water content to reduce or stop the activity of microorganisms (Farid et al. 2014). By long exposure to sunlight, fish may be oxidized, which can contribute to a reduction in nutrient quality and to increased health risks for consumers (Smida et al. 2014). Apart from health risk and nutritional loss, organoleptic character (such as aroma, color, texture etc.) of fish affects substantially from drying conditions. Aroma normally characterises foods sensory properties, and plays an important role when evaluating nutritional qualities and freshness (Diez-Simon et al., 2019; Dominguez et al., 2019). Previous studies have proven that most volatile compounds in dried meat products originate from fat oxidation (Kawai & Sakaguchi, 1996; Toldrá, 1998; Chung et al., 2007; Czerner et al., 2011). Furthermore, most of these substances are unstable and may undergo further reactions during storage in order to form other stable substances and chemical reactions intermediated by enzymes and micro-organisms continue to produce adverse volatile substances which effect the quality and limit the shelf life of the products (Jia et al., 2019; Shi et al., 2019). So far, most studies have focused on aroma changes during dried fish processing or storage (Ganguly et al., 2017; Leduc et al., 2012; Roseiro et al., 2017). In a recent study, Zhang et al. (2020) focused on the alterations of volatile compounds in dried fish at 4°C and 25°C during storage were investigated by HS-GC-IMS fingerprinting in combination with principal component analysis (PCA). In the 4°C stored samples, the most important volatile compounds were 3-methyl butanal, dimethyl ketone, and hexanal; whereas hexanal, 1-octan-3-ol, and 3- methyl butanal were the dominant compounds of dry cured fish stored at 25°C while butyl methyl ketone was only seen at 25°C. According to results of this study, the PCA differentiated the samples clearly with respect to their storage temperature and time. Moretti et al. (2017) studied the chemical changes and volatile formation during processing and maturation of a traditional salted fresh inland fish product (Alosa fallax lacustris). The researchers observed a sharp increase in volatile compounds of fresh fish to 9, 40, 70 and 100 days maturated and salted samples and the total aroma concentrations were showed a positive correlation with the drying process. In the light of these information, the process of drying is probably the critical stage of the quality of fish and fishery products, assisted by the fact that there are higher levels of volatile substances and malondialdehyde associated with rapid oxidation.

#### 2.1.2. Effect of cooking

There are a number of uses of heating in fish processing, including cooking, baking, grilling, and roasting (Boonsumrej et al., 2007). Heat is an important parameter in the processing of fish for the purpose of improving flavour and taste, and to extend the shelf-life of fish and seafood products. Tuna flavour varies greatly depending upon the processing method used. Although quality features are very important for the economic value of tuna, there is very little information about the volatile and non-volatile compounds in tuna meat over the canning process (Zhang et al., 2019). It is well known that the heating process leads to changes in the complex taste patterns of fish meat. It is estimated that these changes are caused by proteolytic and lipolytic reactions. The changing aroma profile correlates with the taste changes that have been observed after exposure to heat in tuna meat. Various studies have been carried out on the quality characteristics of species of fish affected by heat treatment. An earlier study characterized the volatile composition of boiled and steamed red mullet (Mullus barbatus) (Salum et al., 2017). According to results, hexanal and 2-phenoxyethanol compounds were found in raw fish samples while 3-hydroxybutan-2-one, 2,3-octadienone, (E,E)-2,4-heptadienal, linalool,  $\gamma$ -butyrolactone, 1- methylpyrrolidin-2-one, 2H-furan-5-one and pyrrolidin-2-one were detected in cooked fish samples. Another research focused on the effects of different cooking practices to characterize lipid compositions and volatile profiles of farmed and wild sea bass (Dicentrarchus labrax) (NievaEchevarría et al., 2018). According to results, pyrroles, alkylpyrazines, alkylthiophenes and 2- ethylpyridine compounds were detected only in oven-baked samples. In addition, it was determined that farmed sea bass had richer aromatic compounds than wild samples. Similarly, in the study of Zhang et al. (2019), the effect of the two major tunafish canning operations (steam boiling and canning) on the volatile and non-volatile compounds of tuna was determined. Regarding to the result of the study, 35, 35 and 34 volatile compound have been detected in raw, cooked, and canned tuna patterns respectively. Among these volatiles hydrocarbons and aldehydes have been identified as the most common compounds in all processes. The main contributors to raw tuna flavors were found to be decanal, nonanal, octanal, and (E)-2-nonenal, with their green and fatty properties. Another remarkable finding of this study was the release of 2-pentylfuran (green beans), 2-ethylfuran (rubber, baking) and heptanal (dry fish) during the steam cooking process. Moreover, 2-methyl-3-furanthiol, which contains a fleshy aromatic note also known as an important aromatherapy component, only found in canned tuna (Zhang et al., 2019). Another published study focused on the relationship between the composition of volatile compounds in the bigeye tuna (Thunnus obesus) and the variability through processing temperatures. In this



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study, Sun et al. (2013) examined the volatile compounds of tuna fish by means of HS-SPMEGC-MS" before and after exposure to temperatures of 70°C to 150°C. Researches mentioned that the relative amount of aldehydes, ketones, alcohols, hydrocarbons and heterocyclic compounds increased rapidly with increasing heating temperature. In another study, the oxidation of fish under thermal processing has been investigated using a static head-space gas chromatographic system to determine their volatile production. Various process temperatures and periods have been analysed to simulate the conditions in industrial fish treatment. According to results, acetaldehyde, propanal, heptane, 2-ethylfuran, pentanal and hexanal were the most important volatile compounds. The results showed that 2- ethylfurane can be considered as a marker aroma compound to identify the oxidative stability of fish muscle during heat treatments (Medina et al., 1999).

#### 2.1.3. Effect of smoking

Heat treatments decrease the water activity of fish flesh and this provides an excellent preservation via deactivation of microbial organisms. Thus, minimization of spoilage increases the conservation and so availability of fish for consumers. The smoking is also known as a kind of preservation method providing heat and antimicrobial smoke chemicals such as formaldehydes and phenols, which act as antimicroorganisms providing and opportunity for the fish to produce a unique colour and excellent flavour (Longwe & Kapute, 2016). Changes that arise from the smoking of fish are hard to maintain, especially for heat sensitive nutrient (Adenike, 2014). The smoking does not only give food a particular taste, color and flavor, but also improves its conservation due to the dehydrating, bactericidal and antioxidant properties. The smoking technique is commonly applying to fish as either in cold (28-32°C) or hot (70-80°C) conditions (Alasalvar et al., 2011). Salmon is the most common fish subjected to smoking process in the fish industry. Many aromatic compounds of smoked salmon can be traced back to smoke from wood. A further part of the smoked salmon flavour can either be attributed to the combination of odors of raw fish and an evolution of fish flesh flavoring according to the conditions of the smoking process. In an earlier study the volatile compounds in raw and smoked salmon were studied using two gas chromatography-olfactometry (frequency detection and odorant intensity) and gas chromatography mass spectrometry. For fresh salmon, 49 odorous compounds were identified, and 74 for smoked salmon. In particular, phenolic compounds may be used as indicators of smoke development and process intensity. Once smoked salmon aromatic characteristics and their origin are known, it will become easier to adapt the method of smoking (Varlet, et al., 2006). In another study, Mansur et al. (2002) detected that smoked and baked salmon had twice as many volatile compounds as raw salmon. Among all the processes, smoking is considered the highest aroma components followed by baking, canning, surimi samples (kamaboko and chikuwa), drying and finally salting. In another study, Cardinal et al. (2006) investigated the association between odor characteristics and smoking parameters on smoked herring. The impact of three smoking temperatures (16, 24 and 32 °C) on both traditional as well as liquid smoke atomization has been tested. The findings highlight clear discrimination between products, as some of the odor characteristics are particularly related to smoking. A further study aimed to determine that volatile compounds in cold-smoked salmon products is identified by using gas chromatography in order to determine their suitability for the identification of these compounds quickly as indicators to forecast sensory quality. Smoked salmon odor contributed to guaiacol, cooked potato, and mushroom odors characteristic of degradation of fish fats, and sweet odors associated with the microbial metabolites 3- methylbutanal and 3-hydroxybutanone were the strongest odors. Studies show that smoke-related compounds like furfural, phenol, guaiacol and 4- methyl guaiacol are useful indicators for differentiating between products made by different manufacturers that implement different handling and smoking techniques (Jónsdóttir et al., 2008). In another study, Stołyhwo et al. (2006) showed that the typical smoke taste was largely linked with the phenolic compounds in the smoke. Wood smoking compounds that are most active in traditional smoking are pyrogallol, resorcine, 4- methylguaiacol, and less active are syringol and guaiacol.

#### 2.1.4. Effect of freezing

Freezing is one of the commonly used preservation methods in fresh fish and other seafood. If fish are freezing, however, the result may be physical, chemical and enzymatic changes which ultimately lead to the tissue being in an undesirable state (Magnusse et al., 2008). Texture, flavour and color are some of the effects on quality that are present in frozen foods. Freezing rates, methods of thawing, and different temperatures are some of the factors affecting the magnitude of quality loss (Pourshamsian et al., 2012). The effect that freezing and thawing have on the muscles of frozen fish is a matter for the researchers to consider in order to determine the preservation conditions and textural characteristics of fish products (Díaz-Tenorio et al., 2007). The processing



03-04 June 2021, Turkey

of frozen fish might result in a protein deformity. As soon as protein is denatured, the muscle structure, water holding capacity, color and aroma of frozen fish and fish products are affected, because muscle protein is the main factor for its structural properties (Sriket et al., 2007; Chavan et al., 2008). The freezing of fish results in the loss of important characteristics of quality, with increasing toughness and the formation of large ice crystals. Fish size, shape, and location (extra-cellular or intracellular) are known to have an influence on food quality (Mackie, 1993; Howgate, 1977). Biochemical modifications (mainly in lipids) due to frozen storage are likely to have a significant impact on the sensory properties of fish. In salmon the formation of volatile lipid oxidation products is demonstrated during deep-freeze storage, and free fatty acids derived from lipid hydrolysis are more important for the decay during deep-freeze storage of trout when compared to lipid oxidation (Milo & Grosch, 1996; Refsgaard et al., 1998; Ingemansson et al., 1995). According to study of Iglesias et al. (2009), volatile profile of fresh and frozen-thawed from Italian and Spanish cultured gilthead sea bream fish during 266 days of deep freeze storage were investigated by using SPMEGC-MS methodology. The results showed that aldehydes exhibited the highest peak ranges during deep refrigeration. On the other hand, the volatile composition of Italian fish started to increase after just 6 days of refrigeration. However, after 62 days of frozen storage, the first significant increase in volatility was observed in Spanish samples. In a prior study by Refsgaard et al. (1998) reported that the quantities of free fatty acids and lipid hydroperoxides present in fresh salmon could be used to forecast the sensory quality during the storage process. According to the study of Milo & Grosch (1996), the amount of (E,Z) -2.6- nonaddienal, (Z) -3-hexenal, and (Z,Z) -3, 6nodienal compounds increased as a result of storage of salmon at -13°C for 26 weeks. Similar study by Farmer et al. (1997) showed that there has been no significant change in salmon smell or taste stored at -24°C for 33 weeks. In another research by Refsgaard et al. (1998) reported that not only volatile oxidation products account for significant sensory changes during deep-freeze storage of salmon, but other less volatile compounds could also contribute to the increased intensity of offflavor compounds. In the previous study, Alasalvar et al. (2005) investigated that bream and wild bream were compared for differences in their volatile components a storage period of 23 days in the ice. According to results, a total of 60 volatile compounds have been identified in culture and 78 in wild sea bream. During the whole storage period, the relative concentrations of several compounds (trimethylamine, piperidine, methanethiol, dimethyldisulfide, dimethyl trisulfide, 1-penten-3- ol, 3-methyl-1-butanol and acetic acid) keep increasing and can serve as indicators as to the quality of the bream.

#### CONCLUSION

Fish is not only a popular nutritious water animal but also a food source. For processing that in compliance with the protocol, it represents a reasonable percentage of the food being consumed. Different kinds of processes have an effect on the aroma compounds of fish. When the effects of all processes applied to fish in general on aroma compounds are examined, it is understood that aldehydes and hydrocarbons are the predominant volatile compounds. As a result, consumer preference for fish rich in aroma compounds depends entirely on the best conditions for processing and storing fish.

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## Reduced Salt Spanish Style Green Table Olives (cv. Chalkidiki) Preserved in Flavored Olive Oil

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#### ABSTRACT

The study aimed at examining whether olive oil flavored with essential oils (EOs) can be used as a preservation means for reduced salt Spanish style green table olives (cv. Chalkidiki). Traditional green table olives (cv. Chalkidiki) are an asset among Greek products exported to Europe and elsewhere. Due to their high salt content (fermentation and storage in brine), research efforts focus on reformulation strategies that will balance lower salt content, safety and consumer acceptance. Response surface methodology was applied to organize experimentation and assess data. The EOs used (oregano, lemon balm and bay laurel) are known for their antimicrobial properties and are common seasoning materials for food preparations and salads in the Greek cuisine. Oregano EO is known for the presence of strong antioxidants and antimicrobials such as thymol and carvacrol whereas lemon balm and bay laurel EOs are known for their strong antimicrobial activity due to a variety of non-phenolic terpenoids. The time span set in the experimental design was 1 to 12 month(s). Microbiological parameters (pathogens and fermentation-related microbes), color and firmness attributes, were the responses examined in this experimental design that aimed at extending the effort to introduce a tailormade reduced salt table olives product into the market. Each EO was found to exert a preservative role to maintain microbiological quality of the new product not in expense to its appearance attributes, which were maintained at desirable values. Oregano EO had a profound role against pathogens. Lemon balm and bay laurel EOs were found to be important for yeast population control. The results are promising toward the efforts of the local industry to adopt current global nutritional priorities and consumer preferences for natural preservatives.

Keywords: Essential oils, flavored olive oil, olives cv. Chalkidiki, Spanish style reduced salt green table olives

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## Application of Response Surface Methodology (RSM) to Optimize the Concentrations of Essential Oils in Olive Oil Used as a Preservation Means for Reduced Salt Green Table Olives

International Conference on

03-04 June 2021, Turkey

**RAWMATERIALSTO PROCESSED FOODS** 

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#### ABSTRACT

RSM is applied to study the relationships between independent (factors) and dependent variables (responses) with the main objective to obtain an optimal response. This statistical approach is widely used to optimize food manufacturing processes (e.g. extraction). The central composite design (CCD) is typically used to build a second-order model for the response variable in a cost-effective way, ensured by less experimental runs. In the present study, a CCD was adopted to examine the optimum combination of concentrations of specific essential oils (EOs) used as preservative agents in a tailor-made reduced salt table olives product. Olive oil served as the EO carrier. As factors, the concentrations of the EOs used (oregano, lemon balm and bay laurel) and time of storage under vacuum were set. Selection of EOs levels (oregano: 0-1%, w/w; lemon balm and bay laurel: 0-0.5%, w/w each) to prepare the flavored olive oils was based on minimum inhibitory concentration (MIC) values for pathogens and fermentation-related microbes as well as olfactory thresholds of EOs major volatiles. The annual production of table olives determined the time span of the experiment (time: 1-12 months). Secondorder models were fitted to the experimental data of Staphylococcus, lactic acid bacteria, yeasts, chroma (C\*), hue (h\*) and firmness responses. The simplified form of models showed that: (a) oregano EO was determinant for the safety of the product, (b) lemon balm and bay laurel EOs managed to control the yeast population while (c) appearance attributes remained almost unaffected. The predicted values for the responses after applying the optimization approach, seem encouraging toward the direction of the production of tailor-made reduced salt table olives preserved under mild conditions.

Keywords: Essential oils, food safety, reduced-salt table olive preservation, response surface methodology

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## Reduction or Partial Substitution of NaCl: What Effects Sensory and Biochemical Properties of Semi-Hard Cheeses

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#### ABSTRACT

Salt plays a fundamental role in human physiology, but an excessive intake can generate various diseases (e.g. hypertension). In the recent decades, the NaCl reduction has become a priority for the public health and a challenge for the food industries and particularly in dairy industry because cheese has a high sodium content. In this study, the impact NaCl reduction or its partial substitution with KCl on sensory and biochemical properties was investigated on semi-hard cheeses (Cantal-type cheese a traditional French cheese). Five formulations of cheeses were manufactured using 2% (Wt/Wt) salt content in the following proportions: C100 (100% NaCl- control cheese), R50 (50% NaCl), R25 (25%NaCl), S50 (50:50 NaCl:KCl), S25 (75 NaCl:25KCl) and ripened for 30 days. Biochemical parameters (e.g. dry matter, chloride content, protein, fat) were measured by standard methods and sensory features (i.e. appearance, aroma, taste and texture) were evaluated by a trained panel on a 10 cm-unstructured scale intensity.

After using different statistical methods (e.g. PCA, ANOVA) the results demonstrated that salt content of cheeses affected sensory properties, in particular, texture and visual features. In comparison to the control, R25 cheese showed the lowest firmness and the highest melting, sticky and dry texture. Regarding the salty perception, substituted cheese had a similar salty taste compared to the control cheese (C100). On the contrary, R25 and S50 cheeses presented lower saltiness. Moreover, several biochemical parameters (proteolysis, mineral content-Ca, P or K-) were affected by the reduction or the substitution of Na content.

Overall, despite of some observed modifications on the biochemical characteristics, the NaCl reduction in cheese with partial substitution at a level of 25% and 50% by KCl may be a suitable alternative to preserve cheeses standard sensory qualities.

Keywords: Semi-hard cheeses; salty perception, sensory properties, biochemical parameters



## Delineation of Molecular Structure Modification of Camel Milk and Cow Milk Mixtures during Coagulation by 2D-cross Correlation Spectroscopy Coupled with Molecular Fluorescence Spectroscopy

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#### ABSTRACT

Synchronous fluorescence spectroscopy (SFS) coupled with two-dimensional correlation spectroscopy (2DCOS) was employed to evaluate the impact of five mixture ratios of camel's milk (CaM) and cow's milk (CM) (100% CaM, 75% CaM:25% CM, 50% CaM:50% CM, 25% CaM:75% CM and 100% CM) in order to appraise the development of a rapid, accurate and feasible analysis method to monitor milk and to distinguish between molecular structure difference in the milk formulations during coagulation. This study demonstrated that 2DCOS-SFS is a successful strategy to discriminate milk mixtures and to monitor molecular structure modifications during coagulation process. The dissimilarities among the different formulations are clearly observed on the synchronous 2DCOS-SFS. In addition, according to the cross-peak symbols in synchronous and asynchronous spectra, the response speed of modification in riboflavin, protein and vitamin A corroborated with common coagulation phenomena usually reported during chymosin coagulation (360 nm - riboflavin/lumichrome->450 nm -riboflavin->403 nm -aldehydes and amino acids ->298 nm - tryptophan-> 322 nm-vitamin A-).

Keywords: milk, mixture, spectroscopy, two-dimensional correlation spectroscopy



## Biofilms Obtained from an Enriched Arabinoxylan Fraction. Estimation of Thermodynamic Parameters by Thermal Analysis

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#### ABSTRACT

Biopolymers have gained importance as a raw material to develop sustainable materials, due to their natural origin. Biopolymers from vegetable origin, like polysaccharides, can be extracted from by-products of the food industry. Brewers' spent grain (BSG) is a by-product of the brewing industry, which is rich in hemicellulose, including arabinoxylans (AXs). AXs have the capacity to form films, with properties that favor their use in pharmaceutical, cosmetic, and food industry, as coating or support materials, or matrices for release of compounds. However, the thermal stability of these films is needed to take advantage of their properties, in order to be used in a certain temperature range, and without the risk of decomposition. Therefore, the aim of this work was to evaluate the thermal stability of thermoplastic films prepared from a fraction rich in AX (BSG-AX) obtained from by brewers' spent grain (BSG). For this, several films were formulated, in which, plasticizers (polyethylene glycol and glycerol) were added to increase their decomposition temperature. The thermograms were obtained, subsequently, the pyrolysis evaluation was carried out near decomposition temperature (258.17-309.83°C) and the kinetic was analyzed using the Coats-Redfern model, showing that the experimental curves were adjusted to first-order and second-order. Additionally, activation energy (Ea), Gibbs free energy, enthalpy and entropy were calculated from this model. According to the results, the thermoplastic films showed greater thermal stability than the films prepared only with BSG-AX; in fact, the plasticizer addition improves the stability of the films, due to increase their activation energy by up to 43%.

Keywords: Arabinoxylans, Biofilms, Thermal stability, Thermogravimetric analysis.



## 03-04 June 2021, Turkey Delineation of Molecular Structure Modification During Coagulation of Mixed

**RAWMATERIALSTO PROCESSED FOODS** 

#### Delineation of Molecular Structure Modification During Coagulation of Mixed Camel and Cow Milk by Mid-Infrared Spectroscopy and Parallel Factor Analysis

International Conference on

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#### ABSTRACT

Molecular structure modifications of camel milk (CaM) and cow milk (CM) mixtures during coagulation were investigated combining mid-infrared (MIR) spectroscopic monitoring with Parellel Factor Analysis (PARAFAC) and particle size measurements. To evaluate the structure evolution at a molecular level, five different milk formulations were prepared using the following volume fractions of CaM in the mixtures: 100%, 75%, 50%, 25%, and 0%. Regarding MIR spectroscopy, wavelength ranges located between 3000–2800 cm–1 corresponding to fatty acids, 1700 and 1500 cm–1 related to amide I and II bands and 1500–900 cm–1 region called fingerprint region were considered for the characterization of milk coagulation kinetics. MIR spectroscopy with PARAFAC allowed identifying the modifications at a molecular level depending on the coagulation time and milk composition. This was also confirmed by canonical correlation analysis (CCA) that showed a high correlation level between the casein particle size distributions and MIR spectra measured during coagulation.

Keywords: Coagulation, Milk, MIR Spectroscopy, PARAFAC analysis, Particle size.



## **Functional Food Formulated with Food Industry By-Product**

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#### ABSTRACT

The valorization of fruit or vegetable by-products particularly in cereal products has been growingly increasing and attracting the interest of researchers and food producers. Olive pomace has been well known its bioactive and both soluble and non-soluble fiber content. Therefore, this study aimed at formulating the breadstick with olive pomace which was the by-product of olive oil processing and analyzed the effects of olive pomace addition on the physicochemical, bioactive and sensorial properties of product. While the control breadstick was formulated with wheat flour, other formulations was obtained with the replacement of wheat flour with olive pomace at the ratio of 5, 7.5 and 10 %. Olive pomace preparation involved its obtainment from olive oil plant, drying under sun and grinding. Final olive pomace had  $10.03 \pm 0.01\%$  of moisture content,  $4.60 \pm 0.04\%$ of ash content and  $15.78 \pm 0.02\%$  of fat content and  $57.96 \pm 1.26\%$  of total dietary fiber (on wet basis). Total phenolic content was determined using Folin-Ciocalteu method and antioxidant activity was detected by 1,1diphenyl-2-picrylhydrazyl (DPPH) test. The addition of olive pomace reduced  $L^*$  and  $b^*$  of products and increased significantly total phenolic content and antioxidant activity. The highest total phenolic content and antioxidant activity was found as  $1.04 \pm 0.09$  mg gallic acid equivalent, GAE/g dry matter, DM and  $8.14 \pm$ 0.23 µmol trolox equivalent, TE/g DM in product containing 10% olive pomace. In sensorial analysis, products with 10% olive pomace resulted in lower scores in terms of taste, color, appearance, and as well overall acceptability compared to products containing 5 and 7.5% olive pomace. As a result, the use of olive pomace in foods has a great potential to produce functional foods.

Keywords: Bioactive, by-product, functional food



## An Assessment of Using Pea and Brown Rice Proteins to Formulate Flexitarian and Vegan Burger Patties

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#### ABSTRACT

The present research focused on the development of vegan and flexitarian burger patty formulations utilizing pea protein (PP) and brown rice protein (BRP). Five different formulations were prepared as follows: A control formulation that contained 100% beef (C), two flexitarian formulations that contained 50% beef + 50% PP (F-PP) and 50% beef + 35% PP + 15% BRP (F-PPR), and two vegan formulations that contained 100% PP (V-PP) and 70% PP + 30% BRP (V-PPR). Higher moisture content was recorded in V-PP samples compared with C samples, and the addition of BRP was effective to decrease moisture (p < 0.05). F-PPR and V-PPR samples had the highest protein content among treatments (p < 0.05). The lowest fat content belonged to V-PPR patties (p < 0.05). The lowest L\* value belonged to C patties while V-PPR patties gave the highest L\* values (p < 0.05). 0.05). a\* values of the samples generally did not differ except F-PPR samples, while the highest b\* values were recorded in vegan groups (p < 0.05). Vegan patties had lower hardness, springiness, cohesiveness, and chewiness compared with that of control samples (p < 0.05). Addition of BRP to vegan formulations resulted in a harder texture when compared with using PP alone (p < 0.05). V-PP and V-PPR patties showed higher moisture and fat retention and lower diameter reduction, while F-PP patties had higher cook yield compared to that of C patties (p < 0.05). Cook yield of V-PPR samples was also higher than that of C samples (p < 0.05). Although texture scores of C patties were the highest among samples and flavor of C samples was also higher than in vegan samples (p < 0.05), no differences were obtained in appearance, juiciness, and overall impression scores. Consequently, the findings of the study were encouraging for the development of flexitarian and vegan burger patties by utilization of PP enriched with BRP that could provide products having nutritious and technological advantages as well as acceptable sensory quality.

Keywords: meat analog, healthier meat products, sustainable diet, vegetable protein, non-meat protein

#### INTRODUCTION

The human population across the world is increasing exponentially with each passing year, and it is estimated that the global population will rise to ten billion by 2050 (Hellwig et al., 2020). Within this context, the necessity of providing sustainable, nutritious, and high value-added foods that will protect both the environment and the life of other living creatures comes to the fore. The world's first scientific targets on healthy and sustainable food systems were determined by the EAT-Lancet Commission in 2019, a diet under the name of "planetary health diet" was designed within the scope of these goals, and the daily consumption ranges of each food group were defined in this diet. The main purpose of this diet is to increase the amount of high-quality vegetable protein consumed and to reduce animal origin foods (Bánáti, 2020).

Despite the fact that meat is a major source of high biological valued proteins, valuable nutrients, and various health-promoting compounds such as bioactive hydrolysates, connective tissue components, conjugated linoleic acids, and antioxidants (Öztürk and Serdaroğlu, 2017), it could not be regarded as a sustainable food source. Animal-based protein is mentioned as problematic since increased numbers of livestock could cause water depletion, climate changes, disruption of phosphorus cycle, nitrogen cycle, and biodiversity, as well as could seriously influence human health and animal welfare (Michel et al., 2021). Therefore, the multifaceted pressure on animal meat production has encouraged many consumers to reduce meat consumption in their



diets. Besides, today further-processed meat products are less preferred by consumers due to high amounts of animal fat, cholesterol, saturated fatty acids, and additives that could trigger certain health risks (Öztürk and Serdaroğlu, 2017). In this context, consumers have started to change their eating habits by adopting new diet styles. Considering all the factors mentioned above, it is necessary to substitute meat and meat-origin ingredients by utilizing healthy and nutritious non-meat protein sources.

Individuals who desire to completely stop or limit their meat consumption adopt different diets. Vegan consumers are "strict vegetarians". Veganism is defined as a lifestyle and a nutrition form in which any animal products including secondary ones (egg, milk, and their products) cannot be used and consumed (Tunçay, 2018). Apart from this, a sizeable group of people who limits their meat intake yet still include meat in their diets. Those flexibly vegetarian individuals are called "flexitarians" (Rosenfeld et al., 2020).

Pea protein (PP) is an emerging alternative to soy protein due to its high adaptability, hypo-allergenicity, and high functionality, and is often used in combination with other sources to improve the nutritional and textural properties of meat analogs (Boukid, 2020). Legumin and visillin, which constitute 65-80% of the total proteins in peas show high emulsifying properties (Liang and Tang, 2013). Brown rice protein (BRP) is another important alternative as a high-quality protein source, having a total amount of amino acids around 78%, and 36% of the proteins in its structure consist of essential amino acids and 18% of branched-chain amino acids (Kalman, 2014).

In the light of the above-mentioned information, no study has yet been conducted regarding the utilization of PP and BRP to substitute meat in burger patty formulations. Therefore, the present work aimed to investigate the chemical, physical, technological, textural, and sensory quality of vegan and flexitarian burger patties formulated with PP enriched with BRP.

#### MATERIAL AND METHODS

Post-rigor aitchbone was purchased from a local butcher located in Izmir (Turkey) and transported to the laboratory maintaining the cold chain. Granular PP (Veggy<sup>®</sup>, 66.5% protein, 16.9% carbohydrate, 7.6% lipid, 4.5% fiber) was provided from Vegan Dükkan (Istanbul, Turkey) while BRP concentrate (80% protein, 3.8% fiber, 3.8% carbohydrate, 3.5% lipid) was purchased from Proteinocean Food Co. (Ankara, Turkey). Smoke flavor, yeast extract, and garlic powder were supplied from Smart Chemicals Co. (Izmir, Turkey), soy protein was supplied from Kimbiotek Chemicals Co. (Istanbul, Turkey), and red beetroot powder was purchased from Sultanveli Organic Food Market (Izmir, Turkey). The rest of the ingredients and spices were bought from local market. All the chemicals used in the analysis were analytical grade (Merck KGaA, Germany).

#### Experimental design and production of burger patties

In the trial, five different burger patty formulations were prepared. Accordingly, a control treatment (C) containing 100% beef was formed, and flexitarian and vegan treatments were prepared by replacing beef with 50% or 100% vegetable proteins. In those treatments, PP was used either alone (50%) or in combination with BRP (35% PP plus 15% BRP). In the production of the C group, minced beef (3 mm) was mixed with olive oil, salt, spices, and other ingredients in a food processor (Vorwerk, Termomix, Germany) at 200 rpm for 2.5 min. Except for the C group, hot water (1:3) was added to PP to obtain a protein paste to be further used in flexitarian and vegan treatments. For the production of flexitarian groups, minced meat and PP paste were firstly mixed at 100 rpm for 1 min. Afterward, breadcrumbs (10%), soy protein isolate (8%), onion powder (1.5%), olive oil (1.5%), salt (1.5%), red beetroot powder (0.2%), garlic powder (0.15%), yeast extract (0.15%), smoke flavor (0.1%), sugar (0.1%), and other spices (7.5%) were added, and mixing was continued at 200 rpm for 2 min. Meanwhile, vegan groups were prepared by mixing the PP paste with other ingredients at 200 rpm for 2 min. In the formulations enriched with BRP, this protein was mixed with the PP paste at 200 rpm for 2 min prior to the addition of other ingredients. After that, the rest of the ingredients were added, and mixing was carried out as mentioned above. After all the processes, water was added to each formulation in the amount determined as a result of preliminary trials, and mixing was done at 200 rpm for an additional 2.5 min.

The dough obtained was portioned using stainless steel molds (9 cm diameter and 1.5 cm height). The patties were then cooked in a fan oven heated at 165°C on non-flammable and non-stick baking papers until the core temperature reached 73°C. The samples were then immediately cooled to room temperature and afterward were kept at 4°C before the analysis.



#### Methods

Total moisture (oven drying), protein (Kjeldahl), and ash (dry combustion) contents were determined according to AOAC (2012). Total fat content was analyzed by chloroform-methanol extraction (Flynn and Bramblett, 1975). Total carbohydrate content was proportionally calculated over the chemical composition.

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pH value was measured using a portable pH-meter (WTW pH 330i/SET, Germany). CIELAB color parameters (luminosity-L\*, redness-a\*, and yellowness-b\*) were measured using a portable colorimeter (Konica Minolta, CR-200, Japan). Hardness (N), springiness (mm), cohesiveness, and chewiness (N × mm) of the samples  $(3\times3\times1.5 \text{ cm})$  were determined with a TA-XT2 texture analyzer (Stable Micro Systems, UK) using a post-test speed of 2 mm/s, a crosshead speed of 1 mm/s, and a test speed of 1 mm/s with 30 kg of a load cell.

Moisture retention, fat retention, cooking yield, and reduction in diameter were calculated according to Serdaroğlu et al. (2018) for assessment of technological quality. Sensory evaluation was performed with 8 trained panelists who were asked to score the samples in terms of appearance, texture, flavor, juiciness, and overall impression on a five-grade scoring scale. Patties were served warmed to panelists after they with randomly coded 3-digit numbers.

Data were statistically analyzed using SPSS statistical package program (IBM, version 21.0, USA). The intergroup comparison of the means was achieved with one-way analysis of variance (ANOVA), and Duncan's Post-hoc test was used significance was determined. Probability values (p < 0.05) were considered statistically significant.

#### **RESULTS and DISCUSSION**

#### **Chemical composition**

The proximate composition and pH of the patties are presented in Table 1. V-PP samples had higher moisture content than C samples, whilst the lowest moisture was recorded in F-PPR (p < 0.05). In both flexitarian and vegan treatments, the addition of BRP was effective to decrease moisture (p < 0.05), which could be attributed to the increase in dry matter content. Accordingly, F-PPR and V-PPR patties had the highest protein content among treatments (p < 0.05). This result was a good indicator of protein enhancement when BRP was introduced to both flexitarian and vegan formulations. Similarly, Razavizadeh et al. (2019) reported increased protein content in vegan burger patties formulated with fermented soy press cake (okara). The lowest fat content belonged to vegan patties with PP and BRP, and carbohydrate content was also higher in that samples compared with C (p < 0.05), while the rest of the samples had similar fat and carbohydrate contents when compared with control samples. Ash content of the samples did not differ from each other. Flexitarian and vegan samples solely including PP had higher pH values compared with others (p < 0.05), but the addition of BRP led to a decrement in pH values. pH values of PP treatments were in agreement with Moreno et al. (2020) who stated that PP isolate suspensions had pH values close to neutral.

Treatments*	Moisture (%)	Protein (%)	Fat (%)	Ash (%)	Carbohydrate (%)	pН
С	55.44 <sup>b</sup> ±0.70	19.33 <sup>b</sup> ±0.07	13.20 <sup>ab</sup> ±1.24	2.43±0.19	9.60 <sup>b</sup> ±0.76	6.33°±0.02
F-PP	56.47 <sup>ab</sup> ±1.22	17.47°±0.30	13.11 <sup>ab</sup> ±1.26	2.42±0.21	10.53 <sup>ab</sup> ±0.59	6.69ª±0.05
F-PPR	46.07 <sup>d</sup> ±1.19	26.48ª±0.63	14.61ª±1.00	2.18±0.26	10.65 <sup>ab</sup> ±1.48	6.37°±0.09
V-PP	57.61ª±0.83	17.23°±0.18	11.61 <sup>b</sup> ±0.19	2.41±0.07	11.13 <sup>ab</sup> ±1.01	6.76 <sup>a</sup> ±0.02
V-PPR	50.72°±1.14	25.69ª±1.71	9.65°±0.62	2.20±0.18	11.74 <sup>a</sup> ±0.16	6.55 <sup>b</sup> ±0.05

Table 1: Ch	emical Comp	position and	1 pH of the	Burger Patties

\*C: Control treatment consisted of 100% beef, F-PP: Flexitarian treatment consisted of 50% beef + 50% PP, F-PPR: Flexitarian treatment consisted of 50% beef + 35% PP + 15% BRP, V-PP: Vegan treatment consisted of 100% PP, V-PPR: Vegan treatment consisted of 70% PP + 30% BRP.

Data was presented as mean ±standard deviation. a, b, c, d: Different letters within the same column represent a significant difference (p<0.05).

#### **Instrumental quality**

Color and textural parameters of the burgers are presented in Table 2. Significant differences were recorded in both color and texture of flexitarian and vegan samples when compared with control samples containing only



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meat. Luminosity was the lowest in C patties while vegan patties with PP and BRP (V-PPR) gave the highest L\* values (p < 0.05). This data showed that the treatments containing meat (control and flexitarian) were darker than non-meat burger patties (vegan treatments) which could be due to the formation of cooked meat color due to myoglobin denaturation upon cooking. On the other side, vegetable proteins resulted in lighter tints, vegan burgers were even lighter when they were formulated with added BRP. a\* values of the samples generally did not differ from each other except F-PPR samples. The addition of red beetroot powder to all formulations as a natural colorant could be the reason that provided similar redness. The highest b\* values among samples were recorded in vegan patties (p < 0.05), which could be associated with natural pigments such as chlorophyll and carotenoids which are co-extracted during the solubilization and extraction of pea protein isolate (Swanson, 1990).

		Color		Texture			
Treatments	L*	a*	b*	Hardness	Springines	Cohesivenes	Chewiness
*	$L^*$	a	D**	(N)	s (mm)	S	$(N \times mm)$
С	39.86 <sup>d</sup> ±1.2	7.77 <sup>a</sup> ±0.3	13.36°±0.9	106.58 <sup>b</sup> ±3.1	0.704+0.02	0.32ª±0.04	26.13ª±2.1
	5	5	5	7	$0.78^{a}\pm0.02$		1
F-PP	45.31°±1.3	7.82ª±0.1	17.67 <sup>b</sup> ±2.0	(7.050)1.20	0.70h 0.00	0.29ª±0.01	13.78 <sup>b</sup> ±1.7
	2	8	1	67.95°±1.29	0.70 <sup>b</sup> ±0.06		4
F-PPR	44.45°±2.2	7.08 <sup>b</sup> ±0.3	17.11 <sup>b</sup> ±0.9	152.56 <sup>a</sup> ±4.8	0.59010.01	0.201 0.01	24.85ª±0.5
	1	9	9	1	0.58°±0.01	0.28ª±0.01	6
V-PP	51.91 <sup>b</sup> ±0.5	8.07 <sup>a</sup> ±0.1	22.65 <sup>a</sup> ±0.3	29.261.2.50	0.1.00.01	0.14b+0.00	0.000+0.10
	0	3	1	38.36 <sup>e</sup> ±3.50	$0.16^{d} \pm 0.01$	0.14 <sup>b</sup> ±0.00	$0.90^{\circ} \pm 0.18$
V-PPR	55.22ª±0.5	7.73ª±0.4	22.96ª±0.4	40.07d+0.01	0.1 <i>c</i> d+0.00	0.12h+0.01	
	3	0	7	48.27 <sup>d</sup> ±2.01	$0.16^{d}\pm0.00$	0.12 <sup>b</sup> ±0.01	0.90°±0.06

\*C: Control treatment consisted of 100% beef, F-PP: Flexitarian treatment consisted of 50% beef + 50% PP, F-PPR: Flexitarian treatment consisted of 50% beef + 35% PP + 15% BRP, V-PP: Vegan treatment consisted of 100% PP, V-PPR: Vegan treatment consisted of 70% PP + 30% BRP. Data was presented as mean  $\pm$ standard deviation. a, b, c, d: Different letters within the same column represent a significant difference (p<0.05).

The highest hardness among treatments was recorded in F-PPR samples (p < 0.05), indicating that the use of meat, PP, and BRP together led to hardening that could be a result of protein-protein interactions. In a similar manner, the addition of BRP to vegan formulations resulted in a harder texture when compared with using PP alone (p < 0.05). Springiness was the highest in C samples, and springiness of both control and flexitarian patties was higher than the vegan patties (p < 0.05). This data might be due to the higher elastic recovery of meat proteins. Cohesiveness of control and flexitarian samples were similar to each other, while vegan samples had lower cohesiveness than those groups (p < 0.05), which was related to the lower internal stickiness of vegan samples. The addition of BRP did not affect cohesiveness in either flexitarian or vegan treatments. In line with hardness, chewiness was significantly higher in control and flexitarian groups when compared with vegan burgers (p < 0.05). The inclusion of BRP led to an increase in chewiness of flexitarian burgers but it did not lead to any significant difference in vegan samples. Similar to the findings of the present study, Samard et al. (2021) reported lower cohesiveness, chewiness, and hardness in burger patties produced with textured vegetable proteins (isolated soy protein, wheat gluten, and starch) compared with commercial meat patties.

#### **Technological features**

Figure 1 depicts the technological quality parameters of the burgers. Moisture retention, fat retention, cook yield, and diameter reduction of the samples ranged between 79.5-84.1%, 67.7-89.4%, 88.9-93.4%, and 2.2-6.9%, respectively. Vegan patties had higher moisture retention than traditional patties (p < 0.05), while flexitarian patties showed similar moisture retention to control samples. Control patties showed the lowest fat



retention (p < 0.05), whilst all the other samples gave similar values. Thereby, it could be concluded that both water and fat retention abilities were better in vegan samples containing PP (alone or in combination with BRP), underlining higher fat- and water-holding capacity related to the functional characteristics of PP. Cook yield of flexitarian burgers with PP (F-PP) and vegan burgers with PP and BRP (V-PPR) had higher cook yield compared with C burgers (p < 0.05), and cook yield of other samples was similar to that of C samples. That result pointed out the advantageous cooking properties of both flexitarian and vegan samples. Conformably, a lower diameter reduction was recorded in vegan samples when compared with C and F-PP (p < 0.05), meaning that shrinkage and deformation would be less in vegan burgers after cooking. In a similar study, it was found that cooking loss and shrinkage in diameter and thickness of meatless burger patties containing soy protein, wheat gluten, and starch were lower than that of commercial meat patties (Samard et al., 2021).

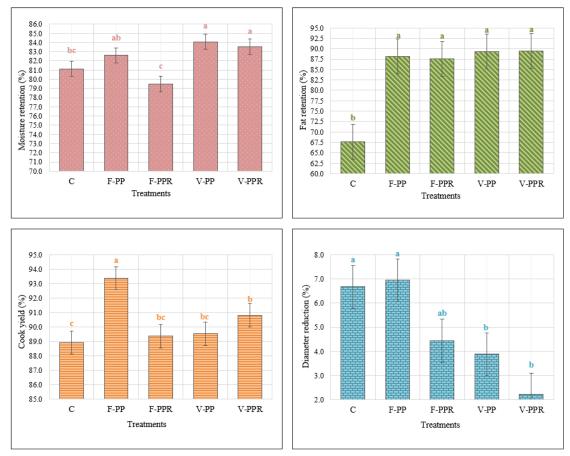


Figure 1: Technological Features of the Burger Patties

**C:** Control treatment consisted of 100% beef, **F-PP:** Flexitarian treatment consisted of 50% beef + 50% PP, **F-PPR:** Flexitarian treatment consisted of 50% beef + 35% PP + 15% BRP, **V-PP:** Vegan treatment consisted of 100% PP, **V-PPR:** Vegan treatment consisted of 70% PP + 30% BRP. Data was presented with the error bars. a, b, c, d: Different letters within the same-colored column represent a significant difference (p < 0.05).

#### Sensory quality

Sensory scores of the patties are shown in Table 3. Texture scores of traditional meat patties were the highest among samples, but the addition of BRP to vegan formulations was effective to increase texture scores (p < 0.05). Flavor of control samples was also higher than in vegan samples (p < 0.05), meanwhile, the flavor of F-PP samples was similar to that of control. Therefore, meat flavor was still perceivable in flexitarian burgers. Here it should be borne in mind that since the trained panelists were not vegan consumers, the expected standard taste in burgers would possibly not be appealing to all members. Nevertheless, both texture and flavor scores in flexitarian and vegan burgers were in acceptable ranges. Appearance, juiciness, and overall impression scores were very promising because no significant differences were obtained between control and either flexitarian or vegan groups. Therefore, the general sensory acceptability of half-meat and non-meat



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patties was advantageous in terms of visual, mouthfeel, and overall scores. Hellwig et al. (2020) compared the sensory quality of fungi-based burgers with commercial vegan and traditional meat burgers. They reported that most of the participants liked the characteristics of fungi burgers, but the chewiness and bitterness should be improved to be commercially accepted.

Treatments*	Appearance	Texture	Juiciness	Flavor	Overall impression
C	4.33±0.82	4.17ª±0.75	3.67±1.03	4.50ª±0.55	4.33±0.82
F-PP	3.33±1.03	2.50 <sup>cd</sup> ±0.55	3.33±0.82	3.67 <sup>ab</sup> ±1.21	3.67±0.82
F-PPR	4.00±0.63	3.00 <sup>bc</sup> ±0.63	2.67±0.52	3.50 <sup>bc</sup> ±0.55	3.50±0.55
V-PP	3.50±1.05	2.17 <sup>d</sup> ±0.41	3.67±1.21	2.33 <sup>d</sup> ±0.52	3.50±1.05
V-PPR	3.67±1.21	3.33 <sup>b</sup> ±0.82	3.17±0.41	2.67 <sup>cd</sup> ±0.52	3.83±0.75

 Table 3: Sensory Scores of the Burger Patties

\*C: Control treatment consisted of 100% beef, F-PP: Flexitarian treatment consisted of 50% beef + 50% PP, F-PPR: Flexitarian treatment consisted of 50% beef + 35% PP + 15% BRP, V-PP: Vegan treatment consisted of 100% PP, V-PPR: Vegan treatment consisted of 70% PP + 30% BRP. Data was presented as mean  $\pm$ standard deviation. a, b, c, d: Different letters within the same column represent a significant difference (p < 0.05).

#### CONCLUSION

The findings of the present work revealed that utilization of PP and BRP to formulate flexitarian and vegan burger patties gave encouraging results by especially leading to improvement in technological quality features. Enhancement of PP with BRP in vegan formulations led to considerable enhancement of nutritional profile in terms of increasing total protein. Although the results of instrumental features (color and texture) reflected challenging quality alterations, sensory scores of the samples gave promising signs of acceptability. Keeping the mentioned points in view, long-term studies would be beneficial for the development of flexitarian and vegan meat products using different non-meat protein sources.

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# Characterization of *Citrus Latifolia* by-products (peel and pomace) and their incorporation effect on the quality of cookies

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#### ABSTRACT

The current study was performed on the waste valorisation of *Citrus latifolia*. The work evaluated the chemical composition, phytochemicals and antinutritional properties of *Citrus latifolia* (CL) peel and pomace powder. The peel and pomace powders were added in formulation of functional cookies to investigate the physical and sensory parameters. Four different treatments of CL peel and pomace were designed by replacing dough as 0%, 5%, 15% and 25% respectively for preparation of cookies. The cookies were baked at lower temperature 50 °C for long time period (75 min) to prevent the nutritional loss. The chemical composition results revealed that CL peel powder have more mineral content, phytochemical and antinutritional composition (P<0.05). This research work is novel in food industry as nutritional perspectives by using *citrus latifolia* by-products as natural carrier and reduced the load of citrus waste.

Keywords: Citrus Latifolia, Functional properties, Cookies, Hadonic Scale, Phytochemicals



## Liposomal encapsulation of omega-3 and lipoic acid conjugate for cow milk fortification

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#### ABSTRACT

 $\alpha$ -Linolenic acid (ALA) and  $\alpha$ -lipoic acid (LA) are well known bioactives. However, they are susceptible to oxidation. The present study was conducted to encapsulate chia oil as the source of ALA and LA, together in a liposomal formulation, to protect these biomolecules. LA inclusion complex with hydroxyl propyl  $\beta$ cyclodextrin exhibited encapsulation efficiency of 86.09±2.17%. The inclusion complex was characterized using FTIR, NMR, and DSC. Nanoliposome formulated with LA inclusion complex and chia oil showed encapulation efficiency of  $80.24\pm0.49\%$  and  $76.38\pm0.58\%$ , respectively, with an average particle size of 52.19±7.0 nm, and moderate repulsion among the particles. Cow milk fortified with LA and chia oil nanoliposome showed supplementation of 236.16 mg LA and 719.52 mg ALA in one serving (240 mL) of milk. This fortification would contribute to the functionality of cow milk, by improving its nutritional attributes even further than naturally present.

Keywords: α-lipoic acid, α-linolenic acid, inclusion complex, nanoliposomes, milk fortification



## The Study of Elimination Potential of Sulfur Blankit (Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub>) and Recovery of Sugar Juice Specification in Sugar Factories Using Membranous Nano-Filtration Method

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#### ABSTRACT

In the sugar industry using membrane filtration process designed to remove Blankit chemical substances, impurities and non-sugar compounds, keeps the nutritional value of sugar, reduces energy consumption and increases crystallization efficiency (Lancrenon and Kientz, 1993; Mohammad et al., 2007). Since most Nanofiltration membranes have superficial load, in Nano-filtration membrane processes, in addition to the mechanism of action (depending on the size of the molecules of the soluble components and the size of the membrane pores), the phenomenon of (electrostatic) caused by the surface charge of the membrane, in isolation and the percentage of excretion Organic compounds are effective (Darwish et al, 2007). In this study, in order to remove the Blankit sulfur substances (Na2S2O4)- because of its Irrecoverable effects on people's health - and improve the properties of the waste water (color, ash, turbidity, non- sucrose rejection percentage, sucrose remove percentage, purity rate) - considering the effect of parameters of pressure difference at three levels (5, 10, 15) times, the temperature at three levels (20, 40, 60) degrees Celsius and time in three levels (20, 55, 90) minutes on Nano-filtration process efficiency, statistical pattern of response level to the mentioned traits was used (Schneider, 1978). Results showed that at the high levels of pressure and temperature, the removal percentage of Blankit, color, ash, turbidity and non- sucrose rejection percentage reduced. But as more time passes due to the compactness polarization layer growth, removal rate (Blankit, color, turbidity, ash and non- sucrose substances) was increasing. The rejection percentage of sucrose in high-pressure surfaces reduced. The maximum percentage was in 20 degrees Celsius and 5bar pressure and the minimum percentage was in 60 degrees of Celsius and 15bar pressure. The higher purity was the result of applying lower pressure and lower temperature in longer times. Results showed that the quantity of Blankit, color, turbidity and ash in syrup obtained from the use of Nano-filtration membrane process compared with the traditional method significantly (98%) decreased. With increasing pressure from 5 to 15 bar the amount of Blankit, color, turbidity and ash in syrup obtained from Nanofiltration membrane respectively (48.86%, 57.54%, 78.72%, 66.67%) and traditional methods (44.28%, 36.67%, 75.29%, 50%) was reduced. The results reflect the positive impact of treatments on the syrup produced by the membrane Nano-filtration process.

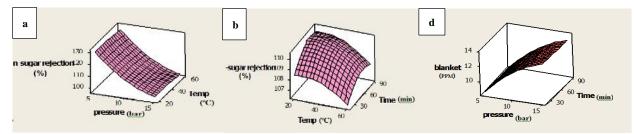


Figure 1. Interaction effect of Temperature and pressure on non-sugar rejection (%); b. Interaction effect of Temperature and time on non-sugar rejection (%); d. Interaction effect of pressure and time on Blankit (ppm)

Keywords: Blankit; purity; non-sucrose material rejection; Nano-filtration



## Investigation of the Bio-Dynamic Commands Use Effect on Mucilage Content and Germination Behavior in 3 Ecotype of Basil (*Ocimum Sp.*)

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#### ABSTRACT

One of the most popular and useable of Aromatic plants are kinds of Basilica (Ocimum sp., Lamiaceae). This genius has different characteristics in behavior germination. Biodynamic agriculture is a new science in the research and especially, the position of stars and planet relative to each other, position of moon around the earth and seasonal change and Solar and lunar eclipses (Steiner, 1924). We must to demonstrative of this theory. In this experiment, we want to record the effect of based on tow calendars (biodynamic and Astronomic) on 3 ecotype of Basilica seed germination. The based on this experiment randomized block design with 3 replications. We use 50 seeds in Falcon tube (value=15ml) and record the weight and value of seed in tube. Then added 5ml of water in tube and shacked after moisturized and control and record of Mucilage value and another behavior of seed germination in lab condition. Results showed that different position of some planets such as moon and Mars and Jupiter had the highest effect, positive and significant effect on mucilage percent (p>95%), germination speed and length of root. Therefore, we could be express the lunar position had the highest effect on root length of local type and the lowest effect on purple basil. Mucilage percentage of seed coat in green basil had the highest content relative to another ecotype and this content was significant (p>95%). Then, the best suggest for produce of mucilage from the basil seeds, the best time is the first quadrature of moon.

Keywords: Basil; Bio-dynamic; Germination; Mucilage; ecotype



## Phytochemical Analysis of Phenolic Compounds, Antioxidant and Anti-Acetylcholinesterase Activities of Extracts from Eight Populations of *Jatropha curcas* L.

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#### ABSTRACT

This study was conducted to evaluate, the phytochemical composition of leaves and roots of eight populations of *Jatropha curcas*. Levels of total phenolic contents, total flavonoids, and condensed tannins of methanolic extracts were determined by UV-spectrophotometer. Some phenolic compounds were identified and quantified by RP-HPLC (reverse phase HPLC). Epicatechin, naringin, rutin, vitexin, and p-coumaric acid were detected. GC–MS analysis revealed that the jatropha oils contained mainly the palmitoleic, palmitic, stearic, oleic, and linoleic acids. The antioxidant properties of leaves, roots, and oils of jatropha showed that the best results were obtained for the leaves extracts of Suriname (P8) (IC<sub>50</sub> = 8.4 µg/ml by DDPH test and IC<sub>50</sub> = 0.55mg/ml by ABTS test). Besides, the anti-acetylcholinesterase activity of leaves and roots extracts of *J. curcas* was investigated revealing that these extract were effective against the acetylcholinesterase enzyme (AChE). Compared to the galantamine (IC<sub>50</sub> = 0.55±0.02 µg/ml), the most promising extracts were the leaves extracts of Mato Grosso Brazil (P5) (IC<sub>50</sub> = 0.54 µg/ml), Vale do Jequitinhonha Brazil (P7) (IC<sub>50</sub> = 0.47 µg/ml), and Suriname (P8) (IC<sub>50</sub> = 0.57 µg/ml). These results indicate that these extracts could be used as natural antioxidant source and in the treatment of the Alzheimer disease too, since they may contribute to increase acetylcholine in cholinergic neurons.

**Keywords**: *Jatropha curcas*; anti-acetylcholenesterase; Alzheimer disease; antioxidant activity; RP-HPLC; GC-MS; phenolic compounds.



## Optimization Of Iron-Oligofructose Formulation On Wheat Flour Of A High Extraction Rate On Dough Rheological Properties

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#### ABSTRACT

The aim of this study was to optimize the iron (FE) and oligofructose (OF) content in order to obtain wheat flour dough of a high extraction rate with the best rheological properties by using response surface methodology. The wheat flour used in this study was of a strong one for bread making with the fallowing chemical composition: 14.3% moisture content, 1.25% ash content, 14.3% protein content and 35% wet gluten content. The levels used for oligofructose variable were between 2.5 and 10% and for iron ions from lactate salt were between 3 and 5 mg/100g wheat flour. The dough rheological properties were analyzed by using the Farinograph, Alveograph Amylograph, Falling Number and Rheofermentograph properties devices. The response surface methodology (RSM) using central composite rotatable design (CCRD) was used for optimization. Dough development time, dough tenacity, extensibility, gelatinization temperature decreased with the increase level of oligofructose and iron addition. Iron salt from the lactate form presented a significant negative effect on dough development time and extensibility. Oligofructose presented a negative effect on water absorption and degree of softening and a positive one on dough stability and peak viscosity.

#### Keywords:wheat flour, rheological properties, oligofructose, iron salt, optimization



## Phytochemical Evaluation and Antimicrobial Activity of Selected Pigmented Plants: Garcinia mangostana, Clitoria ternatea, Ardisia colorata var elliptica and Syzygium cumini

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#### ABSTRACT

Phytochemical compounds in plants are the backbone of antimicrobial activity and natural pigment, therefore are gaining interest as natural preservatives and colourants in food industry to replace the synthetic ones. In recent years, because of the great consumer awareness and concern regarding synthetic chemical additives, foods preserved with natural additives have become very popular. There are concerns about using synthetic chemical additives because of the reported negative effects on human health. Hence, the main objectives of this study were to evaluate the phytochemicals and antimicrobial activity of selected pigmented plants namely, Garcinia mangostana peel, Clitoria ternatea flower, Ardisia colorata var elliptica fruit and Syzygium cumini fruit. The phytochemicals presences in the selected pigmented plants were determined based on the phytochemical screening and reverse phase high performance liquid chromatography (RP-HPLC) analysis, while antimicrobial activity analysis was carried out using the disc diffusion technique. Phytochemical screening revealed that Garcinia mangostana peel exhibited strongest indications for the presence of flavonoid, leucoanthocyanidin, quinone, tannin and anthocyanidin as compared to the other three pigmented plants studied. Findings from RP-HPLC analysis revealed that Garcinia mangostana peel possessed significantly high (p<0.05) amount of phenolic compositions which consists of protocatechuic acid, vanillic acid, chlorogenic acid, p-coumaric acid, ferulic acid, quercetin, rutin, epicatechin, catechin and cyanidin 3-sophoroside. The antimicrobial activity analysis also showed that among the four pigmented plants studied, Garcinia mangostana peel exhibited the strongest inhibition on the fungi Aspergillus niger and gram positive bacteria i.e. Bacillus cereus, Bacillus subtilis and Staphylococcus aureus with minimum inhibitory zone of 6.50 mm, 8.50 mm, 6.70 mm, and 7.20 mm, respectively. Therefore, our results suggested that the antimicrobial activity of Garcinia mangostana peel was associated with their specific phenolic compounds. From a practical point of view, Garcinia mangostana peel may be a good candidate for functional foods and pharmaceutical applications.

Keywords: antimicrobial activity, phytochemical, pigmented plant, RP-HPLC.



## The Effects of Hydrocolloids on The Physical Properties of Sponge Cakes

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#### ABSTRACT

The suitability of hydrocolloids as an improver in bakery products is still doubtful. The rheological characteristics (flow behaviour and viscoelasticity) of batter were studied to relate with the physical properties (volume, texture and microstructure) of sponge cake. The effect of xanthan gum (XG) and hydroxypropylmethylcellulose (HPMC) on cake volume, hardness and sensory evaluation scores of baked sponge cakes were investigated. Batter density and specific gravity were significantly (p<0.05) correlated with volume of cake (r <sub>batter density</sub> = -0.64 and r <sub>specific gravity</sub> = -0.70). Low complex viscosity was observed in hydrocolloids cake during heating at 25-95°C. Hydrocolloids cakes (both XG and HPMC) had significant (p<0.05) low volume compared to control cake. Consistency index and viscosity were significantly (p<0.05) correlated with hardness (r <sub>consistency index</sub> =0.71; r <sub>viscosity</sub> =0.63), which able to determine the crumb texture of sponge cake. For sensory evaluation, sponge cake containing xanthan gum was less preferred by the panellists compared to other sponge cake formulations since it presented the lowest mean score in overall acceptability (6.25).

Keywords: Hydrocolloids, Physical properties, Xanthan gum, Sponge cake



## The Stability of Mayonnaise Model System Incorporated with Black Cumin (Nigella sativa) Seed Oil

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#### ABSTRACT

Incorporation of black cumin (*Nigella sativa*) seed oil (BCSO) (0.5%, 1.0%, 1.5% and 2.0%) in palm oil in dispersion phase of mayonnaise model system is believed can preserve health by delivering beneficial bioactive compound within the BCSO known as thymoquinone. The physical analyses of mayonnaise model system involved the viscosity measurement using viscometer while the stability analyses included parameters such as: mean droplet size (D<sub>32</sub>), droplet size distribution (DSD), creaming index (CI) and dispersion stability by using lumifuge analyzer. The oxidative rancidity test involved rancimat analysis and peroxide value (PV). All mayonnaise model systems showed no visible separation of oil until the end of storage day (day 28) in creaming index and these results were well correlated with creaming stability analysis. Significant changes (p<0.05) were observed in oil mean droplets size (D<sub>32</sub>) for all mayonnaise model systems ranged from 4.14  $\mu$ m to 6.43 $\mu$ m until the end of storage period. The droplet size distribution of mayonnaise model systems showed unimodal distribution up to day 21. Viscosity analyses indicated that there was a significance decrease (p<0.05) in viscosity of mayonnaise model system over time. The addition of BCSO in all mayonnaise model system solve value and induction time measured at 110°C and 120°C in rancimat analysis. Thus, show no development of oxidative rancidity in mayonnaise model system over time.

Keywords: black cumin (Nigella sativa) seed oil, mayonnaise, oxidative rancidity, stability



### Development of slow melting dietary fiber-enriched ice cream formulation using bacterial cellulose and inulin

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#### ABSTRACT

Effect of addition of bacterial cellulose (BC) and inulin on properties such as melting rate, over run, and sensory properties of ice cream was investigated. BC-supplemented ice creams were characterized by a significant reduction in the melting rate, while inulin contributed to enhance the over run and sensory properties. Sensory perceptions varied for BC at 17% and 30% (wet weight) when compared to inulin addition at 0.7% and 1.4% (dry weight). Eight formulations of ice cream were prepared, varying inulin concentration (0.7 and 1.4 g/100 g), BC concentration (17 and 30 g/100 g), and additional water (13.6 ml/100 ml) and two control samples were prepared. An interaction effect among BC, inulin, and additional water content was found for tested parameters. According to the model obtained, the ice cream formulation with 17 g/100 g BC and 1.4 g/100 g inulin observed significant differences for reduction in melting rate and increased fiber content.



## Screening of the antioxidant, nutritional, physical and functional properties of bran obtained from six Indian wheat cultivars

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#### ABSTRACT

In this research paper, the proximate, antioxidant, functional, color, and scanning electron microscopy properties bran were screened in six different cultivars of wheat viz. HUW-234, PBW-373, WH-1105, PBW-502, HD-2967, and PBW-343. The results showed that carbohydrates ranged from 66.74% to 83.94%, proteins 3.45%-17.73%, fats 0.16%-3.16% among the wheat bran of different cultivars, and dietary fiber content ranged from 39.38% to 44.70%. The DPPH content of wheat bran ranged from 5.74% to 9.14% and the decreasing order for a radical scavenging activity was WH-1105 > PBW-502 > HUW-234 > HD-2967 > PBW-373 > PBW-343 between cultivars. The imperative of water absorption capacity was high in PBW-373 and minimum in HUW-234. Swelling capacity was found to increase with increasing temperature (50–90°C). The whiteness and yellowness Index values were found higher in cultivar HD-2967. The bran is being used for the production of functional food products which able to prevent various degenerative diseases.



### Determination of Catechol in Water Extract of Tea using CPE Modified with Banana tissue

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#### ABSTRACT

Tea, one of the most popular consumed worldwide beverages, is known to be rich in polyphenols, more particularly in catechin and catechol. Carbon paste electrode modified with banana crude tissue for determination of catechol in green tea was developed. Polyphenol oxidase enzyme that is present in banana fruit tissue was immobilized into carbon paste electrode CPE-B and the analytical performance of the biocomposit is investigated. The electrochemical properties were assessed by using square wave voltammetry in phosphate buffer 0.1 M, pH 7 using standard solutions of catechol. This biosensor exhibits a high sensitivity 2.32  $\mu$ A/mg and a low detection limit 0.1 mg/L. The linear zone between peak high and catechol concentration was found from 1.4 to 15.7 mg/L with correlation coefficient 0.9990. Reproducibility of the signal obtained in 1.4 mg/L catechol resulted with relative standard deviation 3.6 % (n=4). The storage stability was also studied; the biosensor retained successively 85% and 75% of its initial response after 24 and 40 days. The biosensor was applied for the determination of catechol in green tea samples using two extraction methods (by water solution (141 ± 8.3 mg/L) and phosphate buffer solution (478.5±6.8 mg/L). The calculated recovery was between 89% and 92%, proving that the proposed CPE-B biosensor can be an alternative analytical tool for determination of catechol in tea.

Keywords: Green tea, carbon paste biosensor, crude tissue, PPO enzyme, catechol, square wave voltammetry.



### Nutraceuticals and Functional Foods from Agri-Horticulture Waste of Indo-Argentina

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#### ABSTRACT

Fruits, vegetables, cereals, legumes and other food processing wastes are produced in large quantities. Waste utilization from food processing industries is highly indispensable and challenging task all over the world. Studies have proved that the agri-horticultural wastes are rich source of valuable phytochemicals and nutrients that can serve as a potential source of nutraceuticals and functional foods, and can be beneficial as adjuvant in the management of several health issues like ageing, hypertension, cancer, cardiovascular and other degenerative diseases. The full utilization of agri-horticultural produce is in demand all over the world including Argentina and India to implement low-waste technology in their agribusiness. Several scientific studies have shown that these wastes could be considered as valuable by-products in the form of phytochemicals of nutraceutical importance if they are treated scientifically and technically. Research proved generation of important phytochemicals, antioxidants, dietary-fiber, food ingredients like pectin, natural colour, vitamins, antibiotics and proteases apart from ethanol or biogas from agri-horticultural waste.

Agri-horticultural waste are cost effective natural sources of various phytochemicals like phenols (tannins, lignins, anthocyanins, isoflavones, flavonones, flavanoids), isoprenoid derivatives (terpenoids, carotenoids, saponins, tocotrienols, tocopherols, terpenes), ascorbic acid, oligosaccharides, non-starch polysaccharides, fatty acid and structural lipids (n-3 PUFA, CLA, MUFA, sphingolipids, lecithins), amino acid derivatives, microbes, probiotics, prebiotics, and minerals. Phytochemicals play key role in human health as antioxidants, antibacterial, antifungal, anti-inflammatory, anti-allergic, antispasmodic, chemo-preventive, hepato-protective, hypolipidemic, neuro-protective, hypotensive agents, preventing aging, diabetes, osteoporosis, cancer and heart diseases, induce apoptosis, diuretic, CNS stimulant, analgesic, protects from UVB-induced carcinogenesis, immuno-modulator and carminative.

**Keywords:** Agri-horticultural waste, Health benefits, Phytochemicals, Antioxidants, Nutraceutical, Functional foods



### Effect of CMC and Guar Gum on Oil Absorption and Sensory Quality of Banana (*Musa acuminate*) Fritters During Repeated Frying

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#### ABSTRACT

This study aims to determine the effects of carboxymethyl cellulose (CMC) and guar gum on the oil uptake and acceptability of banana fritters during repeated deep fat frying. Banana of variety "Awak" was deep fried intermittently in cooking oil for 6 hour daily without oil replenishment over 3 consecutive day at  $170\pm5^{\circ}$ C. The moisture, oil content and color were evaluated at first and every  $10^{\text{th}}$  frying cycles whereas the texture and sensory acceptability of banana fritters were evaluated only at the first frying cycle. Results indicated that the moisture and oil content of banana fritters were dependent on the frying cycles. The oil content increased while the moisture decreased with increased in frying cycles. There was significant reduction (p<0.05) in oil content of banana fritters dipped in batter containing guar gum compared to CMC and control. The highest reduction in oil content was banana fritters also had significantly (p<0.05) the highest moisture content (54.27%). The lightness value decreased with increased in frying cycle and 1% guar gum treated fritters had the lowest value. The treated banana fritters especially guar gum significantly increase the crust hardness (9.42N) compared to control (2.32N). No significant different (p<0.05) in the appearance, color, oiliness and taste between control and treated fritters. Despite the crispiness and overall acceptability of control and treated fritters were different, there is a potential of adding CMC and guar gum into the batter to reduce oil absorption in banana fritters.

Keywords: Banana fritters, CMC, Guar gum, Oil absorption



## Effect of Adding Muskmelon (*Cucumis melo L.*) Fruit on Physico-Chemical Properties and Sensory Attributes of Yoghurt

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#### ABSTRACT

Muskmelon (*Cucumis melo L*.) or cantaloupes is a rich source of vitamins, carbohydrates, sugars and protein, it was used as dessert and also eaten alone. This study is done with the objective of evaluating the effect of adding muskmelon either before or after processing of the yoghurt. It was also meant to assess some of the chemical constituents (total solids, fat, protein and ash contents), acidity and sensory attributes (flavor, color, texture, taste and overall acceptability) of muskmelon yoghurt. Fresh raw whole cow milk (6 liters) was obtained from Khartoum University farm. The cow's milk was first examined for chemical composition before the processing. Then the milk was heated at 95°C for 1 minute and cooled to 45°C. The milk was divided into 3 portions; in the first the slices of muskmelon (3%) were added before the incubation (45°C) of yoghurt and in the second the slices of muskmelon were added after the incubation, while the third portion was kept as a control (plain yoghurt). The starter culture was added at a rate of 3% and mixed well with the milk and other ingredients and then the different yoghurt samples were prepared and distributed into plastic bags (150 ml). The yoghurt samples were stored in a refrigerator (5-8 °C for 10 days) and the physico-chemical analysis and sensory evaluations were carried out on day 2, 5 and 10. The data showed non-significant (P>0.05) variations in the physico-chemical content of plain (control) yoghurt and those to which the muskmelon was added before or after incubation. Also the result indicated non-significant variations for total solids, fat and ash content during the storage period of yoghurt, while significantly ( $P \le 0.01$ ) higher values were found for protein (2.86%) and acidity (0.72%) at day 2 of storage period. Similarly significant differences were found for flavor and texture (P $\leq$ 0.05) and taste and overall acceptability (P $\leq$ 0.01) of yoghurt. This study concluded that when adding slices of muskmelon to yoghurt, it is recommended to be done before incubation in order to give better flavor and overall acceptability and to avoid the high level of acid taste.

Keywords: Muskmelon slices, fruit yoghurt, incubation, chemical content, sensory properties, storage



### **Storage of Herbal Raw Materials**

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#### ABSTRACT

Herbal medicinal raw materials, if harvested in due time and dried correctly, but stored in inappropriate conditions, i.e. exposed to direct sunlight, high temperature and high humidity, it can become damp, moldy. As a result, the medicinal raw material loses its normal appearance and basic active substances in connection with their decomposition, therefore, both pharmacological properties and therapeutic effect. Therefore, medicinal raw materials should be stored in appropriate (clean, well-ventilated, dry and protected from direct sunlight) windows on shelves in a packaged form. We must try to prevent rodents and barn pests from attacking medicinal raw materials. Poisonous, highly odorous essential oil and carbohydrate-rich medicinal raw materials, which are often attacked by rodents and insects, should be stored separately.

**Keywords:** storage, drying, herbal medicinal raw materials



### Monosaccharide Removal and Effects of *Komagataeibacter Xylinus* Fermentation on Antioxidant Capacity and Flavor Profile of Chinese Wolfberry Juice

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#### ABSTRACT

Chinese wolfberry is a widely used traditional medicine-food homology plant with diverse functions. However, as Chinese wolfberry contains abundant monosaccharides, it cannot be consumed by diabetics and obese people, which reduces its health and commercial value. To remove monosaccharide, raw Chinese wolfberry juice was fermented by *Komagataeibacter xylinus*. Glucose was exhausted after 10 days, and maximum 3.145g/L bacterial cellulose was obtained. The total organic acid and amino acid concentration increased from 2974.32 mg/100 mL and 409.19 mg/100 mL to 4217.7 mg/100 mL and 655.1 mg/100 mL, respectively. Fermentation promoted the generation of esters, volatile acids, aldehydes and ketones, but no observed change in polysaccharide concentration was detected. Fermentation enhanced the contents of flavonoid and polyphenol, and antioxidant ability was also increased.

**Keywords:** Chinese wolfberry; monosaccharides removal; flavor components; active ingredients; *Komagataeibacter xylinus* 



### **Drying Herbal Raw Materials**

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#### ABSTRACT

Drying is one of the important stages in the procurement of vegetable medicinal raw materials. The purpose of this work is to ensure subsequent long-term storage of raw materials without changing the composition and content of biologically active substances. Timely and proper drying of the collected raw materials will ensure the production of high-quality medicinal raw materials. The time interval from the moment of collection to the start of drying should be minimal. This is because the biochemical processes in the freshly picked plant and its individual parts not only do not stop, but rather, their intensity increases markedly, which leads to a significant decrease in the content of active substances. In conditions of lack of water, enzyme systems cannot exhibit high activity. At the same time, the possibility of the activity of microorganisms and fungi is limited, which prevents decay and moldiness of medicinal plant materials.

Keywords: raw materials, drying, herbal



### Development of Cakes with Almond Baru Flour: Characteristics and Their Correlations with the Texture Profile Analysis

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#### ABSTRACT

Baru (*Dipteryx alata* Vog.) is a native fruit from Brazilian Cerrado. It has an important contribution to the nutritional profile of the products manufactured with them, but the influence of the addition of Baru in bakery products on technological properties still a rare explore. The aim of this study was to evaluate of cakes developed with almond Baru flour. Cakes were manufactured with 20, 40, 60 and 80% of replacement of almond Baru flour by wheat flour. The Baru flour obtained showed high content of protein and lipids (2.8 and 47-fold greater than wheat flour, respectively). The formulation with 80% of baru flour and 20% wheat flour showed 30.3% of protein and 72.1% of lipids. The correlation analysis showed that the Baru flour is a great option to increase protein and lipids, but occasioned harder, more chewy texture and less cohesive and springiness texture into the product.

Keywords: Baru; cakes; texture; nutritional value.



### The Comparison Effect of Hydro Alcoholic and Hydro Distilation Extract of Melissa officinalis o Acne and Pimple

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#### ABSTRACT

One of the famous plants in Lamiacae family is Melissa officinalis and this is the most important herbs for health and Medicine. Possitive effects of melissa recorded to pharmacopet global and get sertificate registered (Ben Erick and vinck, 1956). This plant has positive treatment effect and introduce in the german drag commission. Melisa has 2 grades in both of professional Europ commission of medicenal plantes and WHO. Melisa has rosmarinic acid and flavenoid and fenolic acid (Jayman and Rezaiee, 2006). These components have thitning and rejonation effect on skin. Also, it has monoterpen and dyterpen that there are anti bactrial and skin cratolisasion (Hadji Akhondi and Baligh, 2014). Essential oil of Melisa has good complex with sebom, so it can decrease inflimantory and pain of acne affect. The first produced of hydrodistilation extract of melisa. There for we use 30 gr. Of dry leaves and stems of melisa in 500 m.lit. of distiled water. For hydro alcoholic extract we use 150 gr of dry matter (leaves and stems) in 500 m.lit. of etilic alcohol 25 degrees during 10 days. Then alcoholic extract mixed in 5 values of water. Client were 20 person and 3 month were under experiment. Treatments were 3-5 times spreyed on skin. The results showed that all clients get the best result of this treatment. After apply of water extract of melisa, we observed 75% of clients improved the skin pore, 65% of clients get the improved neurogical problems and infectional Acne and 92% Recovery of Hyper secretion of Sebaceous glands. We observed same results about treatments effect of Hydro Alcoholic extracts. 75% recovery of clients improved the skin pore, 95% of clients get the improved neurogical problems and infectional Acne and 90% recovery of Hyper secretion of Sebaceous glands. These results showed that the best effects of using Melisa extract and value of applying on some skin problems.

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### Advances in Extracting and Understanding the Bioactivities of Marine Organism Peptides: A Review

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#### ABSTRACT

Marine organisms are important foods for humans. Marine organisms contain large amounts of high-quality proteins. Biologically functional peptides have great potential for use as functional foods and drugs, in which can be extracted from marine organisms. Biologically functional peptides with anticancer, anticoagulant, anti-diabetic, antihypertensive, antimicrobial, anti-oxidant, and cholesterol-lowering activities have been described. It is very difficult to screen, extract, and purify biologically active substances from marine organisms, particularly using traditional chemical methods. Here, recent advances in extracting peptides from marine organisms are reviewed and the biological activities of various marine organism peptides are summarized.

Keywords: Peptides, Marine organisms, Biological activities, Extraction, Purification



### Investigating the Effect of the Maltodextrin Gel Usage on Oil Cake Formulation

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#### ABSTRACT

The aim of this research was investigating the effect of the maltodextrin gel usage on oil cake formulation. The specific volume of dough as well as physicochemical properties of the cake including height, moisture, tissue and sensory properties of the product were evaluated. The obtained results cleared that increasing the amount of maltodextrin gel resulted in reduction of the specific volume of the dough. The results of the test conducted on the cake revealed that increasing the amount of maltodextrin gel increased that increasing the amount of maltodextrin gel increased that increasing the amount of maltodextrin gel increased the height of the cake samples. Also, making use of maltodextrin gel increased the moisture content of the cake samples. The results related to texture test also showed a decrease in texture stiffness as a result of increasing maltodextrin gel. So, maltodextrin gel increased texture softness compared to the blank sample. The results related to sensory evaluation showed that the sample containing 3% maltodextrin gels achieved the highest score by the sensory evaluators in terms of taste, color and texture. The findings of overall desirability also exhibited the desirable conditions of the sample containing 3% maltodextrin gel in all the testable properties.

Keywords: Cake, Maltodextrin Gel, Physicochemical Properties. Hygiene



### Identification of Fungal Species from Turkish Mold-ripened Cheeses and the Morphological and Genetic Diversity of *Penicillium roqueforti* Isolates\*

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#### ABSTRACT

In this study, we aimed to identify the filamentous fungi of Turkish mold-ripened cheeses and spesify the morphological and genetic diversity of *Penicillium roqueforti* isolates. We identified 148 filamentous fungi isolated from traditional cheese samples (n=62) comprising Konya Kuflu Tulum, Erzurum Kuflu Civil, Divle Cave Tulum, Karaman Tulum, Kars Kuflu Chechil, Sivas-Zara Tulum and Rize-Ardesen Golot using internal transcribed spacer (ITS) region or beta-tubulin (benA) gene. These isolates mainly consisted of Penicillium roqueforti (81%) in addition to other Penicillium species, P. biforme, P. crustosum, P. paneum, P. nordicum, P. brevicompactum, P. roseomaculatum, and P. solitum, and other species isolated such as Alternaria alternata, Albifimbria verrucaria, Cladosporium cladosporioides, Cladosporium macrocarpum, and Talaromyces kabodanensis. Fingerprinting analyses were conducted using rep-PCR (GTG5 primer) and RAPD-PCR (M13 primer). The primers were discriminative at the species level. While one distinctive band in 2750 bp was obtained in RAPD-PCR, rep-PCR resulted in an identical pattern for all P. roqueforti isolates except a single isolate. PCR-screening for the Wallaby and CheesyTer loci (horizontal gene transfer regions) demonstrated the presence of both loci in all 120 P. roqueforti isolates besides P. biforme, and P. solitum. The mating type distribution of *P. roqueforti* isolates was skewed in favor of *MAT1-2* (95%). Morphological investigation of P. roqueforti isolates were also conducted on four different media, potato dextrose agar (PDA), yeast extract sucrose agar (YES), malt extract agar (MEA) and oatmeal agar (OA). Future studies will include the use of microsatellites for further information about the genetic diversity of *P. roqueforti* isolates.

Keywords: Mold-ripened cheeses, filamentous fungi, Penicillium roqueforti, diversity

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## Impact of Low phHon The Textural and Structural Characteristic of Canned

### Meatballs (Rista)

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#### ABSTRACT

Traditional canned meatballs (*rista*) pretreated with varying concentrations of lactic acid (LA) were evaluated for physicochemical, microbial load, textural, structural and sensory parameters. LA was used at the level of 0% (control), 0.5% (LA<sub>1</sub>), 1% (LA<sub>2</sub>) and 1.5% (LA<sub>3</sub>). Among treated *rista*, LA<sub>3</sub> maintained a significantly lower value (P $\leq$ 0.05) of FFA (1.17%), TBARS (0.75 mg MDA/kg), peroxide (7.22 meq/kg), tyrosine (2.74 mg/100 g) and carbonyl content (6.06 nmol/mg protein) at the 12 months of the storage period. During the 12month storage period, the pH of LA3 was the lowest (4.7) compared to the control *rista* (6.3). The total plate count (TPC) of the control *rista* was found as log<sub>10</sub> cfu/g (4.39), while in the case of LA<sub>1</sub>, LA<sub>2</sub> and LA<sub>3</sub> the TPC was log<sub>10</sub> cfu/g 2.11, 2.03 and 1.51 respectively. Treated *rista* exhibited significantly higher (P $\leq$ 0.05) sensory and textural scores throughout the storage period.

Keywords: Meat, Meatballs, Meat preservation, Rista, Lactic acid



## Novel Methods for The Extraction of Bioactive Components and Essential Oils from Foods

**RAWMATERIALSTO PROCESSED FOODS** 

International Conference on

03-04 June 2021. Turkev

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#### ABSTRACT

The extraction of bioactive compounds from natural food sources via novel non-conventional methods is gaining popularity due to their numerous advantages over conventional methods. In this review article conventional (e.g. Soxhlet extraction, hydro distillation (water distillation, steam distillation), combined water and steam distillation, maceration, cohobation, enfluerage and heating reflux extraction and non-conventional ultrasound-assisted extraction (UAE), microwave-assisted extraction (MAE), high-pressure (HP), pressurized liquid extraction (PLE), negative pressure cavitations assisted extraction (NPCE), subcritical water extraction (SWE), supercritical fluid extraction (SFE), enzyme-assisted extraction (EAE), pulsed electric field assisted extraction (PEF) and accelerated solvent extraction (ASE) method of extraction has been discussed with more emphasis on nonconventional/green extraction. The bioactive compounds extracted by novel techniques have great potential in functional food market as they are extracted under mild processing conditions. Further research is required to evaluate these green extraction techniques in terms of their safety, scalability, consumer acceptability, challenges, legal aspects and potential feasibility.

Keywords: Green extraction; negative pressure cavitations; subcritical; enzyme-assisted; accelerated solvent



### Moderation of Polyphenol Composition in The Cranberry Extract Powders by Spray Drying Parameters and Carrier Addition

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#### ABSTRACT

*Vaccinium macrocarpon* L. is an exceptional source of bioactives including polyphenols responsible for its relatively high antioxidant properties. The composition of these constituents influences the taste of cranberry fruit, being unattractive to consumers especially in a fresh form. For this reason, the conversion of cranberry into novel food ingredients or nutraceuticals produced from cranberry juice extracts in the form of powder has become of high interest. Such transformation requires processing that significantly influences the composition of polyphenolics. One of the promising approaches to obtain cranberry extract powders is spray drying. Taking the above into consideration, the study aims at the evaluation of different inlet air temperatures and application of carrier, i.e., inulin, on the quality of the spray-dried powder in order to provide recommendations for its production.

Application of the inlet air temperature ranged from 150 °C to 190 °C during spray drying of cranberry juice extract enabled for the obtainment of powders without and with the addition of inulin (freeze drying technique was used as a control). In the powders, four major groups of polyphenolic compounds were identified: flavonols, anthocyanins, phenolic acids, and flavan-3-ols. The inlet air temperature of spray drying significantly affected the polyphenolics content in cranberry extract powders and the changes were different when the inulin was added, pointing a strong role of carrier on the retention of selected groups of polyphenols during drying. The content of polyphenols in spray-dried cranberry extract powders were similar or even higher when compared to freeze drying (control), indicating that this drying technique can be successfully applied for the production of cranberry powders.

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Keywords: cranberry, extracts, polyphenols, spray drying



## Effect of Jam and Marmalade Processing and Storage on Phytochemical Properties of Currant Cultivars (*Ribes Spp.*)

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#### ABSTRACT

Currant fruits are recognized as being an important dietary source of health-related compounds, such as anthocyanins and ascorbic acid. In this study, changes in phytochemical properties of different currant (red, black, Ojebyn) cultivars while processing them into jam and marmalade and throughout the storage were investigated. Total phenolics content (TPC), total antioxidant capacity (TEAC and FRAP), total anthocyanins (TA), polymeric color (PC) and hydroxymethylfurfural (HMF) analyses were performed in fresh currant fruits at the beginning of the storage. Also, the same analyses were carried out on jams and marmalades at the end of 6-month storage period. Decreases were observed in TPC, TEAC, FRAP and TA values of jam and marmalade samples of all types of currant varieties at the end of the storage. At the end of 6-month storage period, average TPC values varied between 405.74 -657.51 µg GAE/g; TEAC values between 9.47 - 19.07 µmol TE/g; FRAP values between 7.97- 17.44 µmol TE/g and TA values varied between 26.08 - 341.09 µg cy-3 glu/g. Ojebyn blackcurrant (OBC) jam and marmalade samples had the greatest values at the end of 6-month storage.

Keywords: Antioxidant capacity, FRAP, polymeric color, TEAC



### Effect of Endogenous Lipids and Proteins on the Antioxidant and Pasting Properties of *Sorghum bicolor* Flour

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**RAWMATERIALSTO PROCESSED FOODS** 

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#### ABSTRACT

The functionality of flour, including its bioactivities and industrial applications, reflects its chemical constituents, which may exist either in free or bound state. Hence, this study evaluated the effect of endogenous lipids and proteins on the antioxidant and pasting properties of *Sorghum bicolor* flour (SBF). Different portions of SBF were defatted, deproteinized, or defatted and deproteinized, to remove the endogenous lipids and proteins. The raw (control) and treated flours were analyzed for phytochemicals (total phenolics, tannins, flavonoids, saponins and anthocyanins) levels, antioxidant activities (ABTS<sup>\*+</sup> and DPPH<sup>\*</sup> scavenging activities, reducing power and Fe<sup>2+</sup> chelating capacity), starch and amylose contents, and pasting properties. The raw flour contained high levels of the phytochemicals tested and exhibited potent antioxidant activities. However, defatting (DF), deproteinization (DP), and defatting and deproteinization (DF-DP) significantly (*p* < 0.05) reduced the levels of the phytochemicals and antioxidant activities of the flour, with DF-DP resulting in the highest reduction. The starch and amylose contents of the flour increased significantly as a result of DF, DP and DF-DP. This was accompanied with an increase of the trough viscosity and peak time of the flour, but a decrease of the peak and breakdown viscosities. Therefore, endogenous lipids and proteins may enhance the antioxidant and pasting properties of SBF; removing them from the flour is not recommended.

Keywords: Antioxidant activities, Endogenous lipids and proteins, Pasting properties, Sorghum bicolor flour



### First Principles Study of Structural and Electronic, Properties of Sc<sub>x</sub>ga<sub>1-x</sub>n Alloys

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#### ABSTRACT

Using the first principles total energy calculations within the full-potential linearize muffin-tin orbitals méthod, we have investigated the structural and electronic properties of  $Sc_xGa_{1-x}N$  alloys, with Sc concentration varying from 0% up to 100%, in zinc blend structure (the ground state configuration of GaN) and in rocksalt structure (the ground state configuration of ScN). It is found that for Sc concentrations equal or less than 0.25%, the favored structure is zinc blend, while for Sc concentrations greater than 0.25%, the favored structure is rocksalt. It is also found that for zinc blend crystals, the fundamental gap is large and direct. For the rocksalt crystals the fundamental gap is small and indirect.

Keywords: Sc<sub>x</sub>Ga<sub>1-x</sub>N, LMTO, LDA, zinc blend structure, rocksalt structure



### The Effect of Different Fermentation Temperatures on Şalgam Quality

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#### ABSTRACT

In this study, the effect of different fermentation temperatures on physical, chemical, microbiological and sensorial characteristics of salgam was evaluated. Salgam produced by traditional method and five different temperatures (15 °C, 20 °C, 25 °C, 30 °C and 35 °C) were applied during carrot (main) fermentation. Fermentation time was shortened with increased temperature. Fermentation was successfully completed in 15 days for experiment performed at 35 °C, while it was terminated in 22 days for the trial at 15 °C. pH and total acidity levels (as lactic acid) of salgam ranged from 3.59 to 3.85 and from 5.45 g/L to 8.23 g/L at the final product, respectively. Total anthocyanin and total phenolic contents of salgam diminished with rising temperature. The highest total anthocyanin (as cyanidine-3-glycoside) and total phenolic content (as gallic acid equivalent) were determined as 201. 45 mg/L and 845.43 mg/L in experiment conducted at 15 °C, respectively. Colour scores of produced salgams were assessed according to CIELAB colour system. Darkness of the salgam decreased with increasing temperature, while redness value increased with rising temperature. The lowest L\* (the highest darkness) and the highest a\* (highest redness) values were determined in salgam produced at 15 °C and 35 °C, respectively. From a microbiological point of view, it has been determined that lowering the fermentation temperature does not have any negative effect on the growth of microorganisms and a notable number of lactic acid bacteria and yeast was detected in all experiments. According to the sensorial analysis (scoring and preference test) salgam produced at 15 °C was clearly the most favoured one while the salgam produced at highest temperature (35 °C) was the least preferred. As a conclusion, lessening fermentation temperature of salgam to 15 °C positively influenced the most of the quality parameters of salgam.

Keywords: Black carrot, Fermentation, Lactic acid, Şalgam, Temperature



### Characterization of Aroma-Aroma Active Compounds of Qvevri White Wines Produced from Ketengömlek Grapes with Gas Chromatography–Mass Spectrometry–Olfactometry (GC–MS–O)

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#### ABSTRACT

The Qvevri Wine, produced in amphorae in which alcoholic fermentation and the aging of wine occur is getting popular among many winemakers in Georgia, Europe and Turkey as well. In this study, the white wine was produced by spontaneous fermentation without using special yeast and any chemicals. The Ketengömlek grape, a local variety from ancient times in the Cappadocia Region, was used for making wines. The quality characteristics of this wine were determined. The aroma and aroma active compounds of this qvevri white wine have been qualitatively and quantitatively studied for the first time by using gas chromatography-mass spectrometry-olfactometry (GC-MS-O) and the findings obtained have been compared with the wine produced by the commercial method.

A total of 62 aroma compounds (283 mg/L) including esters (16), alcohols (16), acids (9), lactones (5), the C-6 compounds (4), phenols (8), ketones (2), terpene (1) and N-compound (1) were identified and quantified in the qvevri wine. The wine produced by commercial method consists of alcohols (13), esters (13), acids (10), lactones (4), the 6-C compounds (3), phenols (2), ketone (1), aldehyde (1) and N-compound (1) had 48 aroma compounds at the concentrations of 203.4 mg/L. In both wine samples, alcohols and esters were the predominant compounds in terms of number and amount. The qvevri wine came to the forefront with the notes of earthy, mineral, mushroom, honey, linden and sherry among determined 19 aroma active compounds. In the commercial wine 14 aroma active compounds giving floral, fruity, rose and linden scents were identified.

Keywords: amphorae, aroma compounds, olfactometry, qvevri wine



### Comparison of Volatile Compounds in Sesame Oil and Sesame Cake Extract

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#### ABSTRACT

Sesame (*Sesamum indicum*) is one of the most important oilseed crops (due to its high level of lipid) in the world. In recent years, consumers' demand for this oil has increased due to the positive effects on human health. Cold press oils are seed oils obtained without changing the quality of the oil and processed only mechanically (such as pressing). These oils can only be purified by water, filtration or centrifugation. Therefore, they preserve their beneficial ingredients. Sesame seeds have high amounts of fat, protein, mineral, phenolic and volatile components. In addition to these components of sesame seeds, the obtained sesame oil, sesame cake and shell also contain a high proportion of these components. The sesame cake is a by-product of the edible oil industry which could be recovered and used as a value added product. In this study, it was aimed to compare volatile compounds in cold press sesame oil and its cake extract.

The volatile compounds were extracted using purge and trap extraction and identified by gas chromatographymass spectrometry (GC–MS). The main volatile compounds were composed of aldehydes, alcohols, terpenes, pyrazines, ketones, furans and lactones in the samples. In oil samples, these compounds were identified as the dominant aroma group and a significant reduction was found in the amount of these compounds in the sesame cake samples. Among these, hexanal was found in high level in sesame oil while it was not found in cake extract.

Keywords: sesame, sesame oil, aroma compounds, volatile compounds

#### **INTRODUCTION**

Sesame (Sesamum indicum) is an oil-bearing plant that contains %50-60 oil and %25 protein in its seeds (Dong et al., 2012; Li et al., 2016). This plant, whose homeland is thought to be Asia or East Africa, has been cultivated since ancient times. Today, although the sesame production is widespread, the leading sesame producers are Sudan, Venezuela, India, Nigeria, China, Mexico and Burma. In Turkey, sesame is grown mainly in Southeast Anatolia, Mediterranean, Aegean and Marmara regions (Arslan et al., 2018). World sesame production is approximately 3.5 million tons per year. Sudan is the country that produces the most sesame seeds (981000 tons) in the world. In Turkey sesame oil production is 33600 tons in 2018 (FAOSTAT, 2018). Sesame seeds and oils are rich in health beneficial components. Sesame seeds contain high amounts of oil, protein, mineral substances, phenolic compounds and volatile compounds. In general, oil can be obtained from sesame in two different ways as from roasted or unroasted seeds. To preserve the beneficial and delicate compounds like volatiles in seeds, cold press is generally preferred in obtaining the oil. Aroma is one of the most important criteria that determines the quality and consumer acceptability of a food product. The overall aroma of sesame oil significantly depends on the roasting conditions, oil extraction conditions, and the changes in the relative concentrations of the volatiles. The aroma profile of sesame oil is closely related to its manufacturing processes as mentioned previously (Tamura et al., 2011; Dong et al., 2012). Applied processes like roasting in obtaining sesame oil were reported to have a favorable effect on the volatile compounds such as pyrazines, furans and other thermally driven compounds (Shimoda et al., 1997). Dong et al. (2012), investigated the roasting effect



on the volatiles of sesame oil and reported that the process had an increasing effect on pyrazines, furans and sesamol compounds.

There is limited information in the literature on comparison of volatile compounds between cold press sesame oil and sesame cake extract. Therefore in this study, it was aimed to compare volatile compounds in cold press sesame oil and its cake extract by the application of purge and trap extraction and GC-MS characterization.

#### MATERIALS AND METHODS

#### Materials

Cold pressed sesame oil and sesame cake were purchased from a local producer in Adana, Turkey. The standard aroma compounds were purchased from Sigma-Aldrich (Steinheim, Germany). Dichloromethane, was supplied by Merck (Darmstadt, Germany). All chemicals and solvents used in this study were of analytical and chromatography grade.

#### Methods

#### **Extraction and Analysis of Volatile Compounds**

The aroma compounds of sesame oil and its cake were determined by purge and trap (PT) method. The PT extraction process consisted of a source of nitrogen controlled by a flow-meter (LZT 4-M, Union-Tek Instrument, China). The needle of the source of N2 and the cartridge were installed through the septum to purge and trap the aroma compounds. The temperature of the vial sample was controlled by a thermostat, 3 ml of sample transferred into a 20 ml vial, then the sample was pre-incubated at the extraction temperature (60°C) for 10 min. (Sonmezdag, Kelebek, and Selli, 2018).

#### GC-MS analysis of volatile compounds

The GC system consisted of an Agilent 6890 equipped with a flame ionization detector (FID), and an Agilent 5973N – mass selective detector (MSD). Aroma compounds were separated on a DB-Wax (30 m x 0.25 mm, 0.5  $\mu$ m thickness; J&W Scientific, Folsom, CA). A total of 3  $\mu$ L of extract was injected each time in pulsed splitless (40 psi; 0.5 min) mode. The injector and FID were set at 270 and 280 °C, respectively. The flow rate of carrier gas (helium) was 1.5 mL/min. The oven temperatures was first increased from 50 to 200 °C at a rate of 5 °C/min and then to 260 °C at 8 °C/min with a final hold at 260 °C for 5 min. The mass detector was operated in scan mode, with electronic impact ionization energy of 70 eV. The GC-MS interface and ionization source temperatures were set at 250 and 180 °C, respectively. Identification and quantification were performed in full scan mode scanning a mass range of m/z 30-300 at 2.0 scan/s. The compounds were identified by comparing their mass spectra output with those in Wiley 9 and NIST 11 mass spectral data libraries and an inhouse library created from previous laboratory studies using similarity searching of MS fragmentation (Selli and Kelebek, 2011). Compounds were taken into account if they had a similarity match of at least 80% with the MS fragmentation in the databases. The chromatogram obtained was analysed, and each peak was checked by determining the per cent area on the chromatogram, the retention time, the spectrum and the base peak and then referring to the characteristic mass spectra of compounds listed on the National Institute of Standards and Technologies (NIST) using the software of Agilent ChemStation. Each sample was analysed in triplicate. Linear Retention Indices (LRI) was calculated Linear retention index calculated on DB-WAX capillary column.

#### **RESULTS AND DISCUSSION**

#### Volatile composition of samples

It is very important to choose the most suitable method for the determination of aroma compounds. Purge and trap extraction method gave the best results among the another tried-and-tested methods (Liquid-liquid and solid phase). A total of 23 volatile compounds were identified in two samples. The main volatile compounds



were composed of aldehydes, alcohols, terpenes, ketones, furans and lactones in the samples. In the Table 1. results  $(\mu g/l)$  of the GC-MS analyses of triplicate extractions were given. In oil, these compounds were identified as the dominant aroma group and a significant reduction was found in the amount of these compounds in the sesame cake sample. Aldehydes, the main significant components in flavor of oils (Xu-Yan et al, 2012; Zhao et al., 2013). The major aldehyde compounds in sesame oil include hexanal, 2-methylpropanal, nonanal and 2-methyl-butanal. Hexanal was found in high level (255,1 µg/l) in sesame oil while it was not found in cake extract. In some studies, it was reported that hexanal is the important compound among the aldehydes in sesame samples (Zhao et al., 2013; Hou et al., 2019). Other aldehydes can not be detected in cake also, except nonanal. But content of nonanal decreased in cake. Results show that the concentration of total alcohols increased in sesame oil. 1-pentanol, 1- hexanol, 1-heptanol and 1-octanol were identified in sesame oil while can not be detected in sesame cake. 2-Ethyl-1-hexanol, 3-Penten-2-ol and 3-hexanol were detected in both samples. In addition 3-hexanol has the highest concentration among alcohols. Pyrazines were not detected in oil, but were detected in cake. Pyrazines are likely formed during the Maillard reaction between sugar, proteins, lipids and their lysates (Maga,2002). In literature, pyrazines, pyrroles, thiazoles, thiophenes, and furans have been identified as the major volatile compounds responsible for the aroma of sesame oil (Takei, 1988; Schieberle, 1996; Xu-Yan et al., 2012; Hou et al., 2019). Technological processes such as roasting temperature and duration and the method of extraction of the sesame oil have significant effects on the nature of the aroma. In this study sample was raw and cold pres oil. Therefore, pyrazines were not detected in the oil. But 2,6-dimethyl-pyrazine and 2,5-dimethyl-pyrazine were detected at low concentrations in cake. A total of three different organic acids were found in the oil and cake samples examined and it was observed that acetic acid had the abundance. Acetic acid a common volatile compound in oils (Zhao et al., 2013). In sesame oil, acetic acid has the highest concentration  $(61.4\mu g/l)$  among acids, followed by hexanoic acid and octanoic acid. Other compounds detected in the oil but not in the cake are D-Limonene, 2-Pentylfuran, Dimethyl sulfoxide and 5-ethyl-2(5H)-furanone. In addition, p-Cymene was identified in both samples. As a result, when the cold pressed sesame oil and cake were compared in terms of aroma compounds, it was determined that the aroma compounds were higher in the oil and most of the compounds did not pass into the cake.

No	LRI	Compounds	SO	SC
1	983	Pentenal	193,8	Ν
2	1053	Hexanal	255,1	Ν
3	1204	D-Limonene	15,6	Ν
4	1230	1-Pentanol	18,5	Ν
5	1248	<i>p</i> -Cymene	94,9	35,6
6	1245	2-Pentylfuran	23,9	Ν
7	1254	2-methyl butanal	11	Ν
8	1328	3-penten-2-ol	169,85	113,1
9	1350	1-Hexanol	114,1	Ν
10	1378	3-Hexanol	251,1	225,6
11	1382	Nonanal	67,4	27,7
12	1388	2-Hexanol	27,7	25,2
13	1413	Acetic acid	61,4	Ν
14	1498	1-Heptanol	4,4	Ν
15	1532	2-Ethyl-1-hexanol	97,4	84,2
16	1554	1-Octanol	11,9	Ν
17	1675	Dimethyl sulfoxide	23,9	Ν
18	1802	2-Methyl-propanal	4,8	Ν
19	1810	Hexanoic acid	46,7	14,5N
20	2072	5-Ethyl-2(5H)-furanone	9,1	Ν
21	2073	2,6-Dimethyl- pyrazine	Ν	23,7
22	2075	2,5-dimethyl-pyrazine	Ν	17,5
23	2083	Octanoic acid	58,4	15,2

Table 1. Aroma composition of sesame oil and sesame cake



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\*LRI: Linear retention index calculated on DB-WAX capillary column; N: Not found; The results are the means of % peak areas obtained from 3 different injections.

\*\*SO: Sesame oil; SC: Sesame cake

#### CONCLUSIONS

It was aimed to compare volatile compounds in cold press sesame oil and its cake extract. Total of 23 aroma compounds with the majority of aldehydes were identified in samples. Hexanal was found dominantly in all samples and as a result of pressing, it was observed that this compound did not pass into cake. In the light of the findings, it was determined that the amount of volatile compounds in sesame oil was higher than the cake. The resulting data might be useful for the pressing process, improvement of product flavor and increase of consumer acceptability.

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### Larvicidal Activity on *Culex pipiens* L. Fourth Instar and Phytochemical Characteristics of Aqueous Extracts from Leaves and Roots of Two Species from the Genus *Plantago*: *Plantago major* L. and *Plantago lagopus* L.

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#### ABSTRACT

Nowadays, a large number of aromatic and medicinal plants have very important biological properties that have many applications in various fields, as industry, medicine, food additive... Among these plants we can mention the *Plantaginaceae* family and precisely the genus *Plantago* which has several biological activities, this is due to its richness in various secondary metabolites (polyphenols, etc.), among these activities we can cite antioxidant activity, antiviral activity, and reduction of the immunosuppressive effects of anticancer drugs. However, there has been no previous research on the insecticidal activities of extracts of the genus *Plantago* in the world. Indeed, the larvicidal activity of aqueous extracts from leaves and roots from *Plantago major* L. and Plantago lagopus L. were evaluated on fourth instars of Culex pipiens L. Plant material was collected in two regions of Tunisia, Beja and Bizerte. Differences of toxicity between P. major leaves extracts according to geographical origin resulted insignificant during the observation period. While leaves extracts from P. lagopus collected in Bizerte resulted more toxic compared to those of Beja up to 12 h, nonetheless differences became not significant at 24 h. Distinct figures ensued for root extracts, as geographical origin influenced toxicity and P. lagopus extract from roots collected in Bizerte yielded a significantly greater mortality compared to those from Beja, even at the last observation. With respect to leaf extract concentration, larvae mortality levels at the last survey were similar for both species. ranging from 80 to 100% with 50 and 100 ppm, respectively. As a general role, with 10 and 25 ppm the extracts of *P. major* leaves resulted more effective. Root extracts had a lower toxicity, and mortality after 24 h ranged between 20 and 60% with the highest values attained with 100 ppm of P. lagopus extract. The median lethal concentration (LC<sub>50</sub>) was about 1.50 %. Quantitation of total polyphenols, flavonoids and condensed tannins, evidenced clear differences between species, organs, and geographical origin. Indeed, root and leave extracts from P. major collected in Beja owned higher concentrations compared to those from Bizerte. While, for P. lagopus leaves extracts the ones from Beja had the highest concentrations. These preliminary results pave the way to develop a novel natural bioside to control mosquito. In addition, we have found that there is a good correlation between the larvicidal activity and the content of phenolic compounds, which allows us to conclude that they are partly responsible for this larvicidal activity.

**Keywords**: *Plantago major* L.; *Plantago lagopus* L.; Mosquito; Total polyphenols, Flavonoids; Condensed tannins; Larvicidal activity



### Influence of the Carrier Type and Drying Methods on the Physico-Chemical Properties of Sustainable Powders Gained from Chokeberry Pomace Extracts

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#### ABSTRACT

Chokeberry is one of the richest plant-based source of bioactives, mainly polyphenols. Its processing generates a considerable amount of by-products that could be used for recovering of those constituents with proven health-promoting properties. One of the ways to preserve polyphenols from chokeberry by-products its obtainment of extracts and their conversion into easy-to-handle form of sustainable powders. Such conversion is a complicated process and requires numerous processing steps (extraction, application of carrier, selection of drying techniques and its parameters) that will modify the initial composition of raw material. The study aims at evaluation of influence of addition of maltodextrin, inulin, and trehalose (10%; w/w) and drying techniques (freeze- and vacuum drying at 60 °C and 90 °C) on the polyphenols content and antioxidant capacity of chokeberry pomace extract powders.

Powders gained after vacuum drying had lower moisture content and water activity when compared to products gained after freeze drying. The bulk density was lower in case of powders obtained after freeze-drying, except products gained with trehalose. In this case, the increase in temperature of vacuum drying resulted in higher values of bulk density. In powders, 3 major groups of polyphenolic compounds were identified: anthocyanins, flavonols, and phenolic acids. When the drying techniques were concerned, freeze-drying caused the highest retention of polyphenols, regardless the carrier applied for their preparation. In case of application of selected carrier, the highest retention of sum of identified polyphenols was noted for powders gained after freeze-drying with the addition of maltodextrin and trehalose, whereas inulin resulted in the highest content of polyphenols when vacuum drying at 60 °C was used. Thus, the type of carrier and drying techniques had a strong influence on the physical properties and significantly influence the retention of polyphenolics in powders.

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Keywords: chokeberry, extracts, polyphenols, drying



### **Experimental Studies of the Drying Process of Plant Materials**

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#### ABSTRACT

The developed solar water heating dryer is designed for food and pharmaceutical companies to produce high-quality products - dried and concentrated extracts of vegetables, fruits and herbs, as well as syrups, mashed potatoes and dry concentrates with the preservation of biologically active substances and healthy ingredients based on local raw materials. Experimental studies were performed on a laboratory solar-water convective heating unit and curves were obtained for the duration and temperature of the drying process of medicinal plants. Based on the results of experimental studies of the dehydration process in a laboratory unit, an engineering methodology has been developed for calculating the design and technological parameters of an industrial solar-water convective installation. A comparative analysis of the behavior of the constituent extractives of medicinal plants, as well as the ash content and final moisture content of medicinal plants under the implementation of various drying methods, was carried out.

Keywords: solar water heating dryer, medicinal plants.



### Antiglycation Potential of Freeze-Dried Powders Obtained from Different Fruit Fractions

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#### ABSTRACT

Fruit powders, due to their versatile applications, are increasingly consumed as food additives in everyday people diets. However, there is a significant lack in the literature on the biological properties of such products. One of them is the ability to inhibit the formation of advanced glycation end products (AGEs) formed during the glycation reaction in the human body, particularly damaging in conditions of diabetes and other civilization diseases.

For this reason, this study aimed to evaluate the antiglycation activities (Bovine Serum Albumin (BSA)glucose, BSA- methylglyoxal (MGO), and MGO-arginine model systems) *in vitro* of powders obtained from four different fractions (fruit, pomace, juice and sugar-free extract) of blackcurrant, Japanese quince, haskap berry and rosehip fruits. The total polyphenolic content (TPC) as well as antioxidant capacity (ABTS and FRAP assay) was also examined.

The antiglycation activities of fruit powders are affected by the specific chemical composition of each type of fruit as well as the fraction type. Interestingly, in the case of BSA-glucose model the formation of fluorescent AGEs was more influenced by the fruit composition, while for the other models it was the fraction type. Among the analysed fruits, the highest antiglycation activity was observed in haskap berry and Japanese quince powders. Comparing the different fractions, the most effective inhibitor of AGEs formation proved to be the powdered sugar-free extract rich in polyphenolics, while the powders obtained from pomace showed the lowest antiglycation activity, except for rosehip in the BSA-glucose model. Here, it was the powdered juice that showed the lowest inhibition values. Furthermore, strong correlations were found between the antiglycation activity determined by BSA-MGO and MGO-arginine and the antioxidant capacity as well as TPC. Therefore, the type of fruit and its fractions significantly influence the antiglycation potential of freeze-dried powders.

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Keywords: fruit powders, Advanced Glycation End products, polyphenols, antioxidant capacity



### A Comparison of the Acid Gelation Properties of Nonfat Cow, Sheep and Goat Milks with Standardized Protein Contents

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#### ABSTRACT

Acid gelation of the milk is essential for the production of many dairy products that involve fermentation such as yogurt. Previous studies showed major differences between cow, sheep and goat milk yogurts. However, there is not much study that compares acid gelation and gel properties of standardized cow, sheep and goat milks. In this study we standardized the total protein, total solids and fat contents of the cow, sheep and goat milks: and after heat treatment we used Glucono Delta Lactone (GDL) for slow acidification to mimic the fermentation process. Acid coagulation was monitored by measuring the turbidity and viscosity in every 10 min. and change in the pH was measured every min. throughout the incubation. Chemical composition (total solids, fat, protein, casein, bound and soluble Ca, P, Fe, Mg, K, Na), pH, acidity, SDS-PAGE analysis of the milk samples and viscosity, serum separation and SEM images of the acidified milk gels revealed several differences between standardized cow, sheep and goat milk samples and their acidified milk gels. During the incubation highest decrease in pH observed with cow milk while sheep milk pH was higher and was above pH 4.6 at the end of incubation. Cow milk gels had the highest viscosity, lowest serum separation and more homogenous microstructure. Sheep milk gels had a lower viscosity than the cow milk and goat milks formed the weakest acid gels of all. Results showed that, although the total protein and dry matter content of milk samples were standardized, acid coagulation properties of milk samples showed dramatic differences. These differences were likely as a result of the differences between proteins, particularly the different ratio and amounts of caseins ( $\alpha s_1, \alpha s_2, \beta$  and  $\kappa$ ) and serum proteins found in the different milk types and differences in heat induced interactions between caseins and serum proteins.

Keywords: Cow milk, Sheep milk, Goat milk, Acid coagulation



### Chemical and Sensory Characterization of Kalecik Karası Wines Produced from Two Different Regions in Turkey Using Chemometrics

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#### ABSTRACT

Kalecik Karası (KK) is one of the important native grape varieties used in red wine production of Turkey. Kalecik/Ankara is the origin province for KK wines. However, this variety is also grown in other wine regions, such as Güney/Denizli. As an extension of a previous project that profiled the sensory characteristics of KK wines from different geographic regions of Turkey, this study aimed to investigate the chemical composition of KK wines belong to different vintages from two different regions (Kalecik province in Ankara and Güney province in Denizli) of Turkey with using chemometric methods. Identification/quantification of aroma, aroma-active compounds and sensory evaluations were carried out by GC/MS/FID, GC-O and Descriptive Analysis, respectively. PLSR was used for determining the correlation between chemical and sensory data. 26 aroma-active compounds have identified for both regions. Depend on PLSR, it was determined that common aroma descriptors of KK wines include red fruit (strawberry, raspberry, and apple), dried fruit (raisin, fig), cotton candy, flower and spice odors (sweet spices, nutmeg). Red fruit attribute in wines was positively correlated with isoamyl acetate and ethyl hexadecanoate, Ethyl-4-hydroxybutanoate, 2-phenylethyl acetate. Dried fruit attribute has shown a significant positive correlation with ethyl-2-methyl-propanoate, ethyl-2methyl butanoate and ethyl-3-methyl butanoate compounds. Cotton candy attribute on nose was positively correlated with 2-Phenylethanol, 2-phenylethyl acetate, ethyl-4-hydroxybutanoate, gamma-butyrolactone. On the other hand, attribute of cotton candy on palate has showed positive correlation with ethyl-2-hydroxy-4methyl pentonate, isoamyl lactate and ethyl butanoate.

Keywords: Aroma, Aroma-active, Kalecik Karası, PLSR, Sensory



### The Use of Torulaspora delbrueckii Yeast for the Production of Beer

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#### ABSTRACT

In this study, the effect of utilization of five different *Torulaspora delbrueckii* (Y1018, Y1026, Y1013, Y1031, C134V4) strains in combination with the lager type Saccharomyces cerevisiae on physical, chemical, microbiological, aroma composition and sensory profile of beer were examined. Ethyl alcohol fermentations were completed in 12 days and specific gravity of beers were ranged from 1.005 g/cm<sup>3</sup> to 1.007 g/cm<sup>3</sup> at the end of the fermentation. Ethyl alcohol content of produced beers was changed between 5.46 % (v/v) and 5.93 % (v/v), while the highest alcohol amount was obtained by using single culture of S. cerevisiae (control). The pH and total acidity (as lactic acid) level of beers was ranged from 4.27 to 4.41 and 0.30 g/L to 0.34 g/L, respectively. At the end of the fermentation, the highest Saccharomyces and non-Saccharomyces yeast numbers were determined in experiment that used the mixture of strain C134V4 with S. cerevisiae as 6.84 log cfu/mL and 6.79 log cfu/mL, respectively. The lowest total yeast number were detected in control group as 6.11 log cfu/ml, while minimum number of non-Saccharomyces yeast was found as 5 log cfu/ml in beer produced with the mixture of strain Y1018 and S. cerevisiae. The prominent compounds among the aroma compounds of beer were determined as acetaldehyde, n-propanol, 3-methyl-1-butanol, 2-methyl-1-butanol, ethyl acetate, isoamyl acetate, free 2,3-butanedione and free 2,3-pentanedione. It was ascertained that the total amount of higher alcohols and esters were higher in all mixed culture samples than control group. In terms of sensory evaluation, beer produced with mixed culture of S. cerevisiae and T. delbrueckii Y1031 was the most preferred one characterized to be high in hop aroma and full-bodied. As a result, it was demonstrated that the use of Torulaspora delbrueckii as a non-conventional yeast in mixed fermentations with S. cerevisiae was effective on beer quality.

Keywords: Aroma, Beer, Ethyl acetate, Fermentation, Non-Saccharomyces, Sensory



# Sensory Lexicon and Major Volatiles of Rakı Using Descriptive Analysis and GC-FID-MS

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#### ABSTRACT

Raki is a traditional and Protected Designation of Origin (PDO) alcoholic beverage that is distilled from grape distillate, suma with aniseed (Pimpinella anisum) in copper pot stills in Turkey. This study focused on the development of a sensory lexicon, the creation of a sensory wheel and determination of major volatiles by GC-FID-MS for Rak1. 37 Rak1 samples representing different producers within the various style and quality categories were used for volatile and sensory evaluation. The experts identified 78 attributes and references for the lexicon. The main attributes were spicy, anise, sweet, resinous, fruity, dry fruit, floral, head&tail aroma and white colour. The Raki sensory wheel was created to provide a graphical display of its sensory attributes, with three sensory modalities. For validation of the lexicon, 18 Rakı samples were evaluated using descriptive analysis. The results were subjected to principal component analysis (PCA) to examine the relationship of the Raki samples with the defined sensory attributes. The PCA results show that there is a significant relationship between the Rakı categories and sensory terms and flavour intensities. The GC-MS analyses depicted the following major volatile compounds n-propanol, 2-methyl-1-propanol, 2 and 3-methyl-1-butanol, ethyl acetate, acetal, acetaldehyde, trans-anethol and estragol. Grape distillate based total volatiles ranged between 104.7-220.9 g/hl PA and the anise based total volatiles ranged 1029.5-2118.6 mg/L in Rakı. This is the first study in the literature to determine the sensory lexicon and sensory wheel of the distilled beverages of Rakı. The characterization and presentation of the product using its most distinctive sensory descriptors are important tool and can be used for the industry, marketing, consumer education and for scientists researching Raki products. These findings can help to define and improve new styles of products and maintain the quality of existing ones.

Keywords: Rakı, Volatiles, Lexion, Sensory Wheel, Descriptive Analysis



### Modeling of Hibiscus Anthocyanins Transport to Apple Tissue During Ultrasound Assisted Vacuum Impregnation

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#### ABSTRACT

This study reports the mathematical modeling of hibiscus anthocyanins transport to apple tissues under various vacuum impregnation (VI) conditions (Control, VI 100, 250, and 500 mbar; ultrasound-assisted VI 100, 250 and 500 mbar). *Azuara, Peleg, Diffusive, Z & L* and *Weibull* mathematical models were used to describe the water gain and anthocyanin gain's mass transfer kinetics. *Azuara* model successfully described the kinetic parameters of mass transfer during impregnation treatment. The equilibrium anthocyanin ( $AG^{\infty}$ ) and water gain ( $WG^{\infty}$ ) values predicted by the Azuara model varied between 38.4001-58.2536 and 24.0863-33.0245 mg/kg, respectively. Anthocyanin and water diffusion coefficients of the samples ranged between 0.406x10<sup>-9</sup>, -1.68x10<sup>-9</sup>, 0.117x10<sup>-9</sup> and 0.715x10<sup>-9</sup> m<sup>2</sup>/s, respectively. Ultrasound treatment increased moisture and anthocyanin gain during vacuum impregnation. At the end of the 30 min process, the highest anthocyanin content was determined as 38.85 mg/kg in samples treated ultrasound-assisted vacuum at 100 mbar.

Keywords: Apple, anthocyanin, modeling, ultrasound, vacuum impregnation



### Phytochemicals, Antioxidant Attributes and Larvicidal Activity of *Mercurialis* annua L. (Euphorbiaceae) Leaf Extracts against *Tribolium confusum* (Du Val) Larvae (Coleoptera; Tenebrionidae)

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#### ABSTRACT

This study reports the link between larvicidal activity and the phytochemical composition of male and female leaf extracts of Mercurialis annua L. (M. annua) from four Tunisian regions: Bizerte, Jandouba, Nabeul and Beja. Their antioxidant activity was evaluated using DPPH (2, 2-diphenyl-1-picrylhydrazyl) assays. Phenolic compounds were identified and quantified using liquid chromatography coupled with a UV detector and mass spectrometry (LC-UV-ESI/MS). Higher antioxidant activity (AOA) was found in the leaves of male plant extracts than of female ones. The leaves of male and female plant extracts from Bizerte exhibited the highest AOA: 22.04 and 22.78 mg Trolox equivalent/g dry matter (mg TE/g DM), respectively. For both sexes, plant extracts from Beja had the lowest AOA with 19.71 and 19.67 mg TE/g DM for male and female plants, respectively. Some phenolic compounds such as narcissin, gallocatechin, rutin, epigallocatechin and epicatechin were identified and quantified using LC-MS, which highlighted the abundance of narcissin and rutin in the male leaves of *M. annua*. We noted that the interaction between the sex of plants and the provenance had a significant effect on TFC (F = 6.63; p = 0.004) and AOA (F = 6.53; p = 0.004) assays, but there was no interaction between sex and origins for TPC (F = 1.76; p = 0.19). The larvicidal activity of aqueous leaf extracts of M. annua against Tribolium confusum (Du Val) (T. confusum), an insect pest of flour and cereal seeds, showed that the mortality could reach 100% after 48 h of exposure in the Bizerte region. The  $LC_{50}$  values for the leaf extract were low in Bizerte, with 0.003 and 0.009 g/mL for male and female plants, respectively, succeeded by Jandouba, which displayed 0.006 and 0.0 24 g/mL for male and female plants, respectively. Nabeul showed 0.025 g/mL for male plants and 0.046 g/mL for female plants and Beja showed 0.037 and 0.072 g/mL for male and female plants, respectively. This is the first time that a study has revealed a negative correlation between the antioxidant activity and the larvicidal activity of the leaf extracts of *M. annua* with the following correlation coefficients of Pearson: r = -0.975 and r = -0.760 for male and female plants, respectively.

Keywords: Mercurialis annua L.; antioxidant activity; LC-MS; larvicidal activity



# Targeted Quality Evaluation of Hard Wheat: Application of Solvent Retention Capacity Test with Supplementary Solvents

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#### ABSTRACT

Solvent retention capacity (SRC) is a solvation test for wheat flour in which functional contributions of different polymeric components are predicted based on their swelling behavior with different diagnostic solvents. SRC can be used for all wheat classes but has been used largely for soft wheat flours. Hard wheat and strong gluten flours require a better determination of glutenin and gliadin contributions since products made from these flours are dependent on gluten functionality. The SRC test was conducted according to AACCI method 56-11.01 utilizing the supplementary solvents: 55% aqueous ethanol, 0.75% sodium dodecyl sulfate (SDS), 0.006% sodium metabisulfite (MBS), and 0.75% SDS + 0.006% MBS. The ethanol solvent is related to gliadin content. MBS promotes the thiol-disulfide exchange for glutenins, and the SDS with or without MBS is relevant to the alignment of glutenin macropolymers. Commercial flours and hard red spring (HRS) wheat flours from different varieties were evaluated for quality and SRC. Traditional and supplemental solvents were able to discriminate between commercial flour samples of different types. The supplementary solvents were able to provide additional information about the quality of HRS wheat flours. Samples with higher protein content had higher ethanol SRC and higher MBS SRC. The SDS SRC showed a decreasing trend for samples with higher protein content. The flour samples had a wide range of quality attributes that could be evaluated and selected based on the use of the supplementary SRC solvents. Often, HRS flour will have similar protein contents, but the quality of the protein will be critical to the end-use quality of the flour. A more detailed evaluation of protein quality can determine end-use applications for HRS wheat flours. Understanding the interaction of these solvents with the polymeric components of the flour will allow for a targeted evaluation of hard wheat flour quality.

Keywords: Hard wheat, Quality tests, Solvent retention capacity, Supplementary solvents

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### A Sensory Observation at a House for Different Fish Species Stored at Room Temperature

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#### ABSTRACT

Total mesophilic bacteria count, pH, TVBN values, texture, and sensory (taste, odor, color, and texture) analysis are the main significant factors to observe quality changes of seafood. However, the consumers do not have a chance to make microbiological and chemical analyses. Therefore, the consumers pay should attention to sensory properties or characteristics of fish meat when they buy it. After harvesting or cutting, the changes in odor, color, and texture of stored fish meat should be known and revealed to the consumers. In this study sensory changes of four fish products (red mullet: RM, horse mackerel: HM, bluefish: BF, gilthead seabream: GS) were monitored every hour from the initial hour at room temperature (23±3°C). The overall acceptance results of the study showed that RM, HM, BF, and GS samples were unacceptable at 12<sup>th</sup>, 11<sup>th</sup>, 15<sup>th</sup>, and 17<sup>th</sup> hours, which can be seen also from visual analysis by photographs. The obtained results will give knowledge to the consumers regarding the maximum possible storage period of these samples at room temperature.

Keywords: Fish Quality, Sensory Analysis, Odor, Color, Texture

#### **INTRODUCTION**

Food is a unique source for humankind to sustain a healthy life. Therefore, after harvesting, some food preservation methods are applied to prevent rapid spoilage. In this respect, seafood can be evaluated as a highly perishable food; especially their preservation is the most critical issue for the consumers. In this regard, some preservation methods can be costly while some of them need less cost (Ceylan, 2019). For example, food irradiation is one of the widely used food preservation methods, which limits rapid microbial spoilage (Ceylan and Ozogul, 2020). Food additives mostly chemical origins are preferred to provide food safety. Natural-based ones such as nisin cause high costs in a food preservation system (Ceylan and Mol, 2015). Different food packaging applications such as modified atmospheric packaging (MAP), vacuum packing, and some basic sealing applications can be utilized to provide food safety and delay chemical, sensory, and especially microbiological spoilage.

Besides these mentioned conventional food preservation methods, novel techniques have been tried and used. In this sense, nanotechnology applications (nanoparticles, nanofibers, nanoencapsulation, nanoemulsions, nanoliposomes), time-temperature indicators (TTIs), cold plasma applications, and some microencapsulation applications have been treated with some food products (Yazgan et al., 2017; Gülgün F. Ünal Şengör et al., 2018; Ceylan, 2019; Cetinkaya et al., 2021). Different biopolymers, bioactive materials, essential oils, or bacteriocins could be successfully used. In addition to novel methods, some cooking methods like sous vide could be tested for better preservation in ready-eat foods (Ceylan et al., 2018; Uçar, 2020; Park et al., 2020).

As could be seen from the mentioned above, large scope of food preservation methods could be used to delay the rapid spoilage (sensory, chemical, etc.). Especially, for meat and meat products like fish, total mesophilic bacteria, pH, TMA, TVBN, TBA, texture, and sensory (taste, odor, color, and texture) analysis are very significant to reveal the quality changes (Ceylan et al., 2018; G.F. Ünal Şengör et al., 2019; Durmus, 2020). On the other hand, the consumers do not have any chance to analyze them. Therefore, the consumers pay attention to their sensory properties or characteristics while buying something from the markets or wholesale markets. After harvesting or cutting, the changes in odor, color, and texture of fish meat samples stored at room temperature conditions should be known and revealed for the consumers.

The aim of the study is to reveal the sensory changes in different (small and bigger) fish species stored at room



temperature for the consumers. The obtained knowledge can be shared with the consumers who want to consume fish samples under these conditions.

### MATERIAL AND METHODS

All food samples were obtained as fresh from an international supermarket in Istanbul. The present study consists of four fish species (gilthead seabream: GS, red mullet: RM, horse mackerel: HM, bluefish: BF). Four of the fish species (GS, RM, HM, and BF) were placed at room temperature ( $23\pm3^{\circ}$ C). The samples stored at room temperature were analyzed per hour for 18 hours. To reveal the consumer awareness at home conditions, sensory characteristics were summarized as odor, texture, color, and overall acceptance. Sensory scores for all parameters were applied between 0-10 points. But, 5 points were accepted as the limit value for the rejection (Ceylan et al., 2018). Also, all sensory character (ANOVA) in order to determine the sensory quality changes. GraphPad Prism software (California Corporation, USA) was applied to reveal significant differences between groups by ANOVA. Once a significant (p<0.05) main effect was obtained, mean values of the samples were further analyzed using the Tukey's multiple range comparison test.

### **RESULTS AND DISCUSSION**

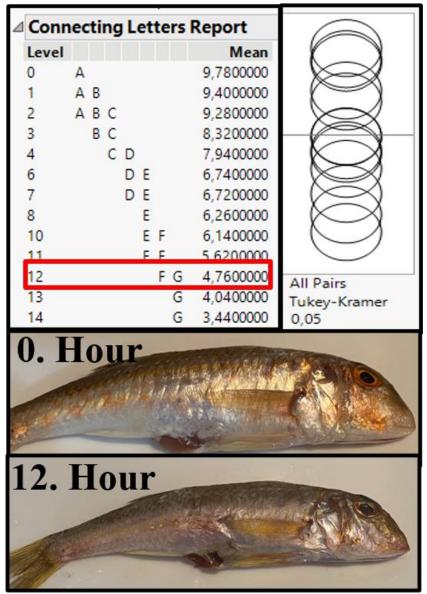
### **Red Mullet**

Overall sensory evaluation of red mullet samples placed at room temperature are presented in Figure 1. As known; the storage of RM samples at room temperature is an undesired event. Unfortunately, it is widely known that these storage or sales conditions are applied for fish samples in different cities of our country. Although RM is a small species of fish, it has a well economic value. So, the importance of household applications for food safety has been reported with the present study. On the initial day of the experimental period for RM samples, odor, color, texture scores were found to be 9.80, 9.82, and 9.82, respectively. Depending on the increase of storage period, deterioration for the mentioned parameters was clearly observed (p<0.05). After 12<sup>th</sup>, 12<sup>th</sup>, and 13<sup>th</sup> hours for odor, color, and texture, respectively, RM samples were defined as unacceptable. Moreover, the initial overall acceptability of RM samples was 9.78. The RM samples were observed as unacceptable with 4.76 scores at the 12<sup>th</sup> hour of the experimental period (p<0.05).

Besides chemical, physical and sensory changes in fish meat, microorganism activity beginning after rigor mortis in fish meat is the main factor, which limits the shelf life of fish. In this regard, especially, psychrophilic and mesophilic bacteria play a key role in the fish spoilage (Ceylan et al., 2020). As could be seen from the figure of RM samples, the consumers can use this study as a guiding document for the evaluation of RM samples.



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# OVERALL SCORES-RM

Figure 1: Statistical overall score results of RM samples with initial and 14th hour photographs

### **Horse Mackerel**

In Figure 2, overall sensory evaluation of HM samples at room temperature can be seen from statistical results. Although HM is also small species of fish, it has been widely preferred by consumers in Turkey. So, the importance of household applications for horse mackerel is also presented in this study. On the initial day of the experimental period for horse mackerel samples, odor, color, texture scores were found to be 9.64, 9.72, and 9.64, respectively. Furthermore, the overall acceptability of HM samples on initial day was 9.64. With the increase of the storage period, deterioration was observed for the mentioned parameters (p<0.05).

The HM samples were observed as unacceptable with 4.794 scores at the  $11^{\text{th}}$  hour of the experimental period (p<0.05). On the other hand, odor, color, and texture were defined as unacceptable after the  $12^{\text{th}}$ ,  $12^{\text{th}}$ , and  $10^{\text{th}}$  hour respectively.



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# **OVERALL SCORES-HM**

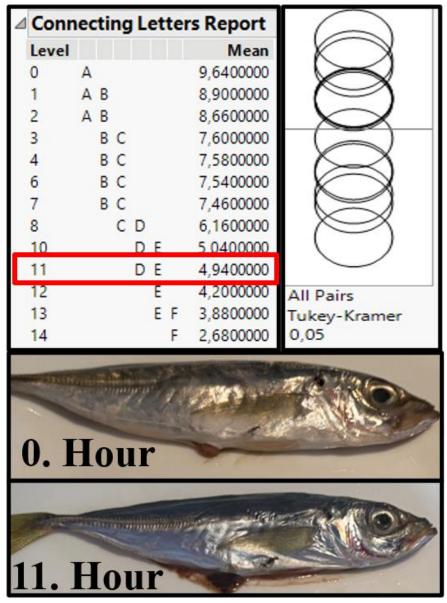


Figure 2: Statistical overall score results of HM samples with initial and 11<sup>th</sup> hour photographs

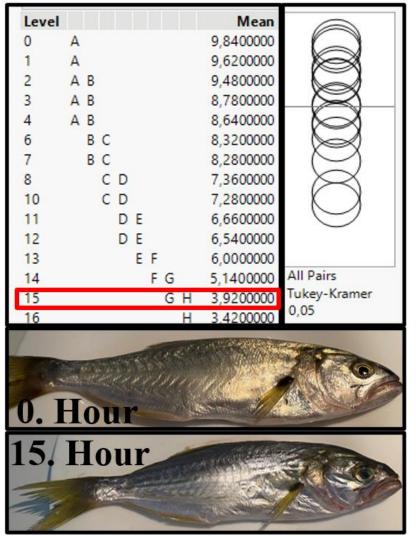
There are different studies related to HM in the previous literatures. In this respect, according to the result of sensory evaluation of frozen HM, wild and cultured hot-smoked HM samples had 7 months of shelf-life (Koral et al., 2015). This is an actually expected result for the frozen and smoked samples. In the case of placing the samples at room temperature, extreme sensory quality changes could be observed.

### Bluefish

Overall score results of BF samples stored at room temperature are presented in Figure 3 with statistical figures and photos. On the initial day of the experimental period for BF samples, odor, color, texture scores were found to be 9.86, 9.76, and 9.72, respectively. From 0. hour to  $16^{th}$  hour on the increase of storage period, deterioration for the mentioned parameters was clearly observed (p<0.05). After  $15^{th}$ ,  $13^{th}$ , and  $13^{th}$  hours for odor, color, and texture, respectively, BF samples were defined as unacceptable. The initial overall acceptability of BF samples was 9.84. However, they evaluated as unacceptable with 3.92 scores at the 15th hour of the experimental period (p<0.05).



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# OVERALL SCORES-BF

Figure 3: Statistical overall score results of BF samples with initial and 15th hour photographs

BF can be processed with different methods. In this respect, different sensory properties can be gained. For example, fish burgers prepared from BF and hake but the burgers prepared from BF gained much more acceptability in terms of sensory (Di Monaco et al., 2009). So, processing conditions could play a key role, in some cases, these processed food samples could be prepared at room temperature, which is not expected. With the present study, the consumers and the producers have been informed.

### **Gilthead seabream**

Overall sensory evaluation of gilthead seabream samples at room temperature are presented in Figure 4. Seabream is one famous fish species that preferred by consumers and has a huge economic value. The importance of household applications for GS has been also reported in the present study. On the initial day of the experimental period for GS samples, odor, color, texture scores were found to be 9.84, 9.8, and 9.82, respectively. Furthermore, the overall acceptability of GS samples on initial day was 9.86. With the increase of the storage period, deterioration was observed for the mentioned parameters (p<0.05). The overall acceptability of GS samples was observed as 4.14 scores at the 17th hour of the experimental period (p<0.05). On the other hand, odor, color, and texture were defined as unacceptable at the 17th hour.



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# **OVERALL SCORES-GS**

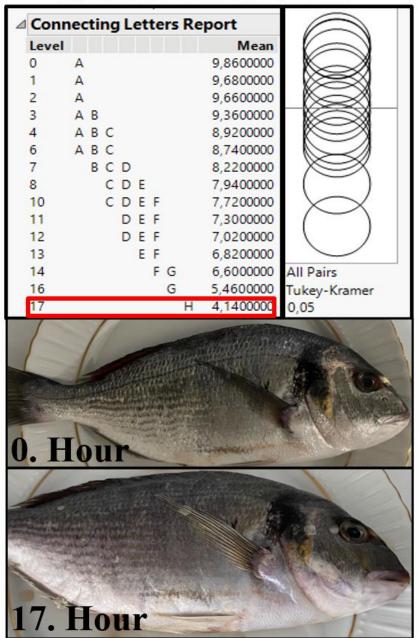


Figure 4: Statistical overall score results of GS samples with initial and 17th hour photographs

GS is bigger fish as compared to the other fish samples. Therefore, it has different properties connective tissues, skin, and eyes, than others. Furthermore, with image analysis, some quality changes in GS could be observed (G.F. Ünal Şengör et al., 2019). As stated by (Ceylan et al., 2018), After 5 days of cold storage, putrid and fishy odor in control samples stored at  $4\pm1^{\circ}$ C was determined by sensory assessment. In the present study, at 17<sup>th</sup> hour, the GS samples were not fit for human consumption in terms of sensory evaluation.

### CONCLUSION

Shelf life and quality of seafood are important for consumers. Total mesophilic bacteria count, pH, TVBN values, TMA values are the main parameters to observe quality changes of seafood. However, these analyses are time-consuming and could be expensive as many materials are needed for experiments. To overcome this



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issue, sensory analysis can be seen as an important quality parameter. Sensory evaluation provides information about the shelf life of fish products in a short time and does not cost any money. So, the consumers can determine sensory characteristics such as odor, color, and texture by the naked eye in a short period of time. For this reason, in the present study sensory evaluation of 4 fish products is examined by odor, color, texture scores at room temperature. From initial day, samples were scored and photographed until they reach the unacceptable score of 5. Results showed that GS had higher scores compared to other samples at the 11th hour (overall acceptability:7.3) and was accepted as consumable even after 16 hours. However, RM and HM samples were evaluated as inconsumable on the 12<sup>th</sup> and 11<sup>th</sup> hour, respectively. Color change, oil sealing, drying on the surface can be seen clearly by visual photography at the end of storing time. Furthermore, a bad odor (4.5 on 12th hour) has been observed for HM. These lower scores for RM and HM are attributed to the weight and size of the samples as they were smaller than GS and BF. The overall acceptance results of the study showed that RM, HM, BF, and GS samples were unacceptable at 12th, 11th, 15th, and 17th hours, which can be seen also from visual analysis by photographs. Longer shelf life for GS could be attributed to the thick skin layer which preserves it from deterioration compared to other samples. Drying on the surface and textural deterioration can be clearly seen in the photograph for BF. On the 15th hour, odor, color, texture, and overall scores (3.98, 4.5, 4.54, and 3.92 respectively) showed that BF was also had longer shelf life than RM and HM. When all results evaluated together it is highly recommended consuming small fish species such as RM and HM less than 12 hours of storage time. On the other hand, fish species with bigger size and weight such as GS could be consumed up to the 17th hour. The obtained results in this study will give important knowledge to consumers regarding the average shelf life of small and big fish species at room temperature.

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# Effect of Cultivar and Maturity on Functional Properties, Low Molecular Weight Carbohydrate and Antioxidant Activity of Jackfruit Seed Flour

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# **Nutritional Values in Fresh Beans**

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### ABSTRACT

Beans are used extensively in human nutrition as fresh, canned and dried. Since the bean plant is a plant that growing mild climate, it is affected by hot and cold climates. While it is generally grown as the first crop on the Mediterranean coasts in the spring season or in the summer season in places where altitude is high, 11 bean varieties were investigated as a second crop in 2018 in Kahramanmaraş conditions and the nutritional values of fresh beans were examined. In this study Protein, Ca (Calcium), Mg (Magnesium), K (Potassium), P (Phosphorus), ADF (Acid Detergent Insoluble Fiber), ADP (Acid Detergent Insoluble Protein), NDF (Neutral Detergent Insoluble Fiber) ratios of fresh bean varieties were researched. It has been recorded as Protein (20.00-22.92%), Ca (0.30-0.90%), Mg (0.21-0.31%), P (0.39-0.43%), K (3.00-3.61%) ADF (20.07-26.31%), NDF (27.17-33.68%) ratios of bean varieties. As a result of the research carried out, it was concluded that the bean plant planted in July or August can be harvested as green beans, and there will be problems in obtaining dry grains due to the cooling of the weather.

Keywords: Bean Varieties, Nutritional Content, Second Crops

### INTRODUCTION

People need proteins of vegetable and animal origin for quality, adequate and balance nutrition in the world. A large part of the world population cannot get enough protein due to the high price of proteins of animal origin and cannot be provided for various reasons and limiting of cereals proteins in terms of some amino acids. The utilizable of legumes which are not restricted by amino acids the according to the price of animal proteins, plays an active role in eliminating the protein deficit in the nutrition of developing countries as an alternative source. Legumes have an important place in terms of nutrition, the absolute essential lysine amino acid, calcium, phosphorus, iron, B1, B2 vitamins and minerals, with a protein content of 18-31.6%. It should be emphasized the requirement of including to an important product group, due to reduces the risk of cancer with its antioxidant content, also and cardiovascular diseases and diabetes diseases for reasons such as low



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cholesterol content, high fiber content and low-fat content. Vegetable proteins and carbohydrates of human and animal nutrition are provided from legumes 22% and 7%, 38% and 5% in the world respectively. The legumes should be considered to in the more efficient and economical in solving the nutritional problem and eliminating the protein deficit in nutrition. In other words, legumes constitute the protein source of 2 billion people in the world (Adak ve ark., 2010). The bean plant, which is in the legume group, is an important field plant that is consumed in large quantities in Turkey and in the world, because of its high nutritional value, being traded as fresh, canned and dry, an indispensable food of kitchens. Beans, whose homeland is South America, were brought to Europe at the beginning of the 16th century, its agriculture increased over time and began to be grown in almost every part of the world. Bean cultivation in Turkey, on the other hand, dates back to about 250 years ago (Nemli 2013).

Today, when the popularity of environmentalism and sustainable agriculture is increasing, legume plants increase their nutrient intake from the soil, while forming in root nodosites, by connecting the free nitrogen of the air to their roots thanks to the Rhizobium bacteria, they meet their own needs and leave some nitrogen to the next plant (Rurangwa et al., 2018). Nitrogen binding capacity is generally around 5-20 kg / ha per year and may vary depending on the variety and environmental conditions (Schirali, 1998). For this reason, legumes are seen as a good crop rotation plants. Rotation of the bean plant with other field crops plays an important role in the reduction of fertilizer and energy use in arable land and thus in the reduction of greenhouse gas emissions (Stagnari ve ark. 2017). Legumes reduce the use of commercial nitrogen fertilizers, enrich the soil with organic matter, aerate the soil and increase the water holding capacity of the soil, as well as providing an economic advantage by obtaining two products a year for plants including crop rotation. Legumes also play an important role in animal nutrition, as the stalk and straw are low in cellulose. The high level of digestion of crude proteins (78%) in the stems and cereals of beans compared to grains encourages the increase in production (Azkan, 1999). This increase also creates additional feed resources, with its protective effect on agricultural lands. The use of plant residues as feed constitutes another positive contribution for animal production. In addition to nitrogen and protein sources, bean seeds contain calcium, potassium, phosphorus, magnesium, copper, zinc, iron, sulfur and manganese (Suarez-Martinez ve ark., 2016).

Bean cultivation is carried out by farmers in every region where climatic conditions allow. The bean is stakerooted, it has a temperate climate loving, very sensitive to cold. The bean plant requires a soil temperature of at least 8 ° C for germination, an optimum germination temperature of 18 ° C and a growth temperature of 20-25 ° C. The yield decreases are take place due to the slowing down of the development at a temperature below 15 ° C during the development period and the prevention of fertilization above 32 ° C (Porch and Jadn, 2001). The bean plant is generally grown in the spring (as the first product) in places with a Mediterranean climate. The quality criteria of the fresh broad beans of 11 bean varieties were examined, different from the knowledge of the ability of the beans to be adapted and to contribute to the plant to be planted in winter by making autumn (as a second crop) cultivation in Kahramanmaraş region.

### MATERIALS AND METHODS

In the study; Önceler, 98, Göynük 98, Yunus 90, Topcu, Alberto, Aras 98, Bermaz, Noyanbey 98, Akman 98, Göksun and Karacasehir 98 were used as materials. Bean varieties were obtained from Agricultural Research Institutes and private companies. The research was conducted in 2018 in Kahramanmaras province, which is 568 m above sea level, which has the effect of the Mediterranean climate. The minimum (23.2, 21.0, 14.6, 9.2 <sup>o</sup>C), maximum (36.8, 34.7, 26.4, 17.6 <sup>o</sup>C), average temperature (29.1, 27.2, 19.8, 2.7 <sup>o</sup>C) average humidity (48.3%, 38.4, 51.5, 66.7%) and precipitation (4, 8, 45, 70 mm) values for August, September, October and November covering the months of the study were recorded respectively (Anonim, 2018). Soil properties, saturation, pH, salt, lime, organic matter, potassium, phosphorus values were determined, respectively 85.80%, 7.28, 0.30%, 1.00%, 2.08%, 266.8 mg/kg, 10.46 mg/kg for 0-30 cm depth, 86.35, 7.31, 0.26%, 1.10%, 1.79%, 291.7 mg/kg, 4.92 mg/kg for 30-60 cm depth, 83.60, 7.30 0.23%, 2.90%, 1.23%, 293.9 mg/kg, 3.65mg/kg for a depth of 60-90 cm (Anonim, 2019). The soil structure of the trial area has a slightly alkaline and clayey structure, medium level of organic matter, slightly salty and sufficient lime, phosphorus and potassium. Wheat plant was grown as a preveous plant in the experimental area. After the wheat harvest, the field was plowed. After being plowed with second-class agricultural tools before planting, the roller was pulled and made ready for planting. The research was planted with hand on 7 August 2018, in 4 rows, in three replications, on 5meter-long plots with 50 cm row spacing and on 10 cm intra spacing. Before planting, 6 kg / da phosphorus



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and 2.3 kg / da nitrogen fertilizer were applied. With the completion of the planting, the drip irrigation system was established and irrigation was carried out. When the plants reached a height of 15 cm, 3.7 kg / da nitrogen fertilizer was applied. Hoeing was done 3 times manually, for weed control. The drip irrigation system has been irrigated 12 times about 7 hours throughout, since there is almost no rainfall in August and September that during the period when the bean plant continues its development. The middle two rows of each parcel were harvested for observations, measurements and analysis on November 6-10, due to insufficient climatic conditions for the dry harvest of bean varieties. The pods of the harvested bean varieties were separated and kept in the oven at 105 ° C for 72 hours and dried pods into flour. Nutritional analysis of 48 samples taken as flour were made using the WINISI package program on the FOSS 6500 NIR system device in laboratory. In this study Protein, Ca (Calcium), Mg (Magnesium), K (Potassium), P (Phosphorus), ADF (Acid Detergent Insoluble Fiber), ADP (Acid Detergent Insoluble Protein), NDF (Neutral Detergent Insoluble Fiber) ratios of fresh bean varieties were researched. In the statistical analysis of two soltained in the study, the means of the variety were compared using the SAS package program, analysis of variance according to the Anova procedure, and the means according to the Duncan (P <0.05) multiple test.

### **RESULTS AND DISCUSSION**

The averages of protein, Ca (Calcium), Mg (Magnesium), K (Potassium) of 11 fresh bean varieties were given in Table 1, the other averages of P (Phosphorus), ADF (Acid Detergent Insoluble Fiber), ADP (Acid Detergent Insoluble Protein), NDF (Neutral Detergent Insoluble Fiber) ratios of 11 fresh bean varieties were given in Table 2.

Table 1. Averages and groups of nutritional values of fresh bean varieties				
varieties	Protein *	Calcium **	Magnesium *	Potassium **
Karacaşehir 98	22.92 a	0.90 a	0.31 a	3.00 c
Bermaz	20.00 c	0.44 bc	0.24 b	3.55 a
Önceler 98	21.62 ab	0.50 bc	0.22 b	3.38 abc
Akman 98	22.19 ab	0.53 b	0.26 ab	3.09 bc
Topçu	21.43 abc	0.45 bc	0.22 b	3.46 ab
Noyanbey 98	21.28 bc	0.46 bc	0.21 b	3.57 a
Göksun	20.72 bc	0.30 c	0.23 b	3.61 a
Göynük 98	21.17 bc	0.53 b	0.24 b	3.20 abc
Aras 98	20.69 bc	0.59 b	0.26 ab	3.28 abc
Yunus 90	22.15 ab	0.50 bc	0.22 b	3.56 a
Alberto	21.69 ab	0.39 bc	0.23 b	3.59 a

Table 1. Averages and groups of nutritional values of fresh bean varieties

\*\* : p < 0.01; \*: p < 0.05

### Protein (P)

It was determined that the protein values of the bean varieties examined in Table 1 vary between 20.00% and 22.92%. It was observed that there was a statistically significant difference in protein values among bean varieties. In terms of protein value, it was recorded that the highest was obtained from Karacaşehir 98 variety with 22.92% and the lowest from Bermaz variety with 20.00% and the two varieties made a statistically significant difference from each other. The values protein of Akman 98, Yunus 90, Alberto, Öneler 98, varieties were 22.19%, 22.15%, 21.69%, 21.62%. While these cultivars were placed in the same transition group and statistically were not made a significant difference from the Karacaşehir 98 variety, but it were seen to make a difference from Bermaz cultivar. It was determined that there were statistically significant difference between the varietie of Karacaşehir 98 with Aras 98, Göksun, Göynük 98, Noyanbey 98 and Topçu varieties in terms of protein. These cultivars with other cultivar among was no significant difference except for Karacaşehir cultivar.

Results from previous researchers' studies were reported that Gülümser et al. (2005) found that the protein ratio in the grain was between 20.27-23.15% as a result of the boron application on foliar and soil in the Black Sea Region, Ülker (2008) found that the protein ratio in the grain was between 18.53-27% as a result of harvesting in two different locations in the Central Anatolia ecological conditions, In finding of Kahraman



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(2008) in Konya region, the protein ratio in broad beans in 3 different planting times for two years was between 20.11-28.59%, in study of Demir (2011) in Ordu region changed between 18.18-23.6% after the harvest of green bean varieties, Kuyucuoğlu (2016) found that protein rate varied between 20.17-23.15% in broad beans in Konya ecological conditions. It was stated that the protein value varied between 17.75-23.60% of 6 bean varieties with three years by of Kazydup et al. (2017) Previous research were shown that the protein ratios could differ according to the climatic factors, temperature, planting time and the amount of fertilizer applied, although the protein values are characteristic of the variety(Peterson et al. 2005; Johanson et al. 2003; Kazydup et al. 2017)).

### Calcium (Ca)

It was observed that there was a statistically significant difference in calcium values among bean varieties. It was determined that there were 3 different groups in terms of Ca values in bean varieties and their values varied between 0.30-0.90%. The highest Ca value was obtained from Karacaşehir 98 variety with 0.90% and it was significant difference than other ten bean varieties. Aras 98 with 0.59%, Akman 98 and Göynük 98 with 0.53% were in the second group and these varieties had statistically significant differences between Karacaşehir 98 and Göksun varieties that was third group, but not were statistically significant difference among other varieties. The lowest value in terms of Ca value was observed in Göksun variety with 0.30%. Önceler 98, Yunus 90, Noyanbey 98, Topçu, Bermaz and Alberto varieties did not create statistically significant differences among themselves, but formed the transition group between the second and third groups (Table 1).

Demir (2011) found that the calcium content of the fresh bean varieties was between 0.36-0.69% after the harvest in the Ordu region, and Kahraman (2008) found that the calcium content in the fresh bean was between 0.01.0.19% in three different planting times for two years in the Konya region. Ca concentration was affect by environmental and genetic factors in dry bean seed (Moraghan and Grafton, 1997). As can be understood from the previous findings, it is understood that the calcium ratio is affected by environmental factors as well as the variety feature (Moraghan and Grafton, 1997). Calcium is a requirement element for humans and animals. The Ca need of human body, fattening animals, cattle for day are 0.8-0.9, 20-22,20-30 g respectively (Demirci 2016; Işık 1996; Sevgican 1996).

### Magnesium (Mg)

In the bean varieties used in the study, it was determined that 2 different groups were formed in terms of Mg values that varied between 0.21-0.31%. The highest Mg value was observed in Karacaşehir 98 variety with 0.31%. The lowest Mg value was obtained from Noyanbey 98 variety with 0.21%. Noyanbey 98 variety with Önceler 98, Yunus 90, Topçu, Alberto, Göksun, Göynük 98, Bermaz varieties were place in the same group. While Noyanbey 98 variety created statistically significant differences with Karacaşehir 98 varieties, it was observed that there was no significant statistical difference with other varieties in terms of Mg. It was noted that Akman 98 and Aras 98 varieties were in a transition group with 0.26% and statistically wasn't differ significantly from the Karacaşehir 98 variety. Mg value is different according to the varieties. The actual performance of cultivar depends on the environmental conditions (Saleh et all., 2018). Kahraman (2008) determined that the Mg ratio in pods varied between 0.01% and 0.13% for 3 different planting times and two years in Konya region. Demir (2011) determined, the Mg ratio varied between 0.17-0.29% with the grinding of the broad bean after the harvest of the green bean varieties in Ordu region. Calcium, Mg and K were been the main cations of common bean. Mg or K concentrations had lower variable than Ca concentration (Moraghan and Grafton, 1997).

### Potassium (K)

In this study, the K values examined in different bean varieties varied between 3.00-3.61%. The highest K value was found in Göksun variety with 3.61%. In terms of K value, the lowest value is seen in Karaçaşehir 98 variety with 3.00%. While Karaçaşehir 98 variety did not create statistically significant difference with Önceler 98, Aras 98, Göynük 98 and Akman 98 varieties, it was determined that there was statistically



significant difference between other cultivars. It was noted that Göksun variety with Alberto, Noyanbey 98, Yunus 90, Bermaz varieties were place in the same group, and these cultivars waren't statistical differences with Karacaşehir and Akmam 98.

Kahraman (2008) noted that the K ratio in pods varied between 0.11-2.03% in two years and 3 different planting times in Konya ecological conditions. Demir (2011) reported that the K content of green bean varieties varied between 1.19-2.44% in his study in Ordu region. Potassium content of dry bean varied between 14.2 to 18.4 g kg-1 in two regions with fifty genotypes made study by Mario Paredes et al (2009).

	Phosphorus	Acid	Acid Detergent	Neutral
varieties	**	Detergent	Insoluble	Detergent
varieties		Insoluble Fiber	Protein	Insoluble Fiber
		**		**
Karacaşehir 98	0.43 a	26.31 a	0.68	33.68 a
Bermaz	0.39 b	24.61 ab	0.59	31.60 ab
Önceler 98	0.39 b	24.06 abc	0.53	33.16 a
Akman 98	0.40 b	23.61 abc	0.57	32.74 ab
Topçu	0.40 b	22.92 bcd	0.48	30.02 abc
Noyanbey 98	0.41 b	22.89 bcd	0.52	30.33 abc
Göksun	0.40 b	22.62 bcd	0.50	30.60 abc
Göynük 98	0.39 b	22.00 bcd	0.50	30.37 abc
Aras 98	0.40 b	21.66 bcd	0.52	30.66 abc
Yunus 90	0.40 b	20.39 cd	0.50	28.56 bc
Alberto	0.40 b	20.07 d	0.49	27.17 с

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Table 2. Averages	and groups	of nutritional	values of	fresh bean	varieties

\*\*: p < 0.01; \*: p < 0.05

### Phosphorus (P)

In terms of the P value in the pods of the bean varieties used in the experiment, it was determined that 2 groups were formed and the values varied between 0.39-0.43%. The highest P value was obtained from Karacaşehir 98 variety with 0.43% and it was recorded that there was a statistically significant difference from other varieties. In terms of P value, the lowest value was obtained from Bermaz, Önceler 98 and Göynük 98 varieties with 0.39%. The lowest value was followed by Alberto, Akman 98, Yunus 90, Topçu, Aras 98, Göksun with 0.40% Noyanbey 98 with 0.41%, they were had the same group and made a significant difference compared to the Karacaşehir 98 variety.

Kahraman (2008) determined that the P ratio in pods varied between 0.10-0.53% for three different planting times and two years in Konya ecological conditions. In study of Demir (2011) determined that the P values of the green bean varieties changed between 0.38-0.85% with the grinding of the broad bean after the harvest in Ordu region. P value is different according to the varieties. The actual performance of any cultivar depends on how its genetic parameters interact with the environmental conditions (Saleh et all., 2018).

### Acid Detergent Insoluble Fiber (ADF)

The ADF values in pods of the bean varieties studied varied between 20.07-26.31%. The highest ADF value of 26.31% was obtained from Karacaşehir 98 variety. In terms of ADF value, it was determined that Topçu, Noyanbey 98, Göksun, Göynük 98, Aras 98 varieties were in the same transition group and differed statistically from Karacaşehir 98 variety, and did not create statistically significant difference between other varieties. The lowest ADF value was recorded in Alberto cultivar with 20.07%. It was determined that Alberto cultivar made



statistically significant differences with Karacaşehir 98, Bermaz, Öneler 98 and Akman 98 varieties, but not statistically significant difference between other cultivars.

According to roughage quality standards, it is known that the best quality class was below 31% of ADF, the first class was between 31-35% and the lowest roughage value was (5th class) 45% upper (Güney 2016). Kobal Bekar et al. (2019) found that the highest amount of ADF among the pole bean genotypes varied between 29.75-28.38% and the lowest was 19.28% in Black Sea conditions. Since ADF values are used as an important criterion in determining the quality of the feed, the ADF values in previous studies on other plants; the dried hay yield of soybeans was 26.56-34.61% ADF in Kayseri conditions as Akıncı (2019) finding. Yalçınkaya (2019) determined that the dried plants from fresh grass samples taken during flowering period from meadow pasture taxons was 15.39-31.56% ADF in the Southeastern Anatolia Region. In finding of Kavak (2019) was determined the meadow pasture vegetations varied between 12.53-34.5% ADF in the Southeastern Anatolia Region. Aydoğan et al. (2014) noted the ecological conditions of Konya varied between 28.63% and 33.12% ADP in different shape times in artificial pastures. As understood from previous studies, it is understood that the ADF value creates differences according to the plant type, the development level and the time of taking the material taken, the climate and soil structure and the structure of the taxonomy. Since the high ADF value will decrease the best quality class, the values we achieved shows that it is of very good quality.

### Acid Detergent Insoluble Protein (ADP)

The ADP ratios in pods of different bean varieties as second crop grown were no statistically significant differences in terms of varieties. It was noted that the highest value was obtained from Karacaşehir 98 with 0.68% and the lowest value was obtained from Topçu variety with 0.48%, where the average values varied between 0.48 and 0.68%. Kavak (2019) determined that the ADP values of some astragalus taxonomy varied between 0.64-1.40% in Southeastern Anatolia Region. The finding of Başbağ (2018) was cited that ADP values of forage crop in the flowering period varied between 0.08-0.63% in the ecological conditions of Diyarbakır. It was showed that the values in the literature as previous research were differ according to the acquisition time of the material and the structure of the taxonomy.

#### **Neutral Detergent Insoluble Fiber (NDF)**

The NDF value in pods of bean varieties tested as the second crop varied between 27.17-33.68%. It was recorded that the highest NDF value was in Karacaşehir 98 variety with 33.68%, followed by Önceler 98 variety with 33.16% and was in the same group. Akman 98, Bermaz, Aras 98, Göksun, Göynük 98, Noyanbey 98 and Topçu varieties in terms of NDF values were in the transition group and as statistically no were significant differences with the varieties of Karacaşehir 98 and Previous 98. The lowest value in terms of NDF value was noted in Alberto cultivar with 27.17%. It was determined that Alberto cultivar made statistically significant differences with Karacaşehir 98, Previous 98, Akman 98 and Bermaz varieties, but not statistically significant difference between other cultivars. According to roughage quality standards, it was known that the best quality class was 40% below, the first class had between 40-46% and the lowest roughage value had 65% upper in terms of NDF (Güney 2016). NDF values are used as an important criterion in determining the quality of the feed. Kobal Bekar et al. (2019) found the highest value of NDF between 36.53-36.01% and the lowest NDF as 29.10% on the pole bean genotypes at the Black Sea Agricultural Research Institute. Dried hay yield of soybeans was 38.43-44.85% NDF in Kayseri conditions as Akıncı (2019) finding. Yalçınkaya (2019) determined that the dried plants from fresh grass samples taken during flowering period from meadow pasture taxonomy was 28.28-49.04% NDF in the Southeastern Anatolia Region. Beycioğlu (2016) found that the NDF content of cowpea plant as dry grass varied between 24.51-42.55% NDF in Kahramanmaraş conditions. Kaplan et al. (2014) determined that the NDF values on pea grains varied between 18.65-36.48% NDF. Since the increase in the NDF ratio will decrease the digestion rate, the findings obtained in our study are more digestible. Based on the previous studies, it is seen that it creates differences according to the plant type, the development



level of the material taken and the time of taking it, the climate and soil structure and the structure of the taxonomy.

### CONCLUSION

In the study investigating the cultivation possibilities of 11 bean varieties as a second crop in the agricultural areas left empty after the wheat harvest in the Kahramanmaraş region with the Mediterranean climate, the nutritional values of the green bean pods used as vegetables were examined. The fact that the protein values of bean varieties grown as a second crop are between 20-23% will make a great contribution to human nutrition. Good ADF, ADP and NDF values obtained in terms of feed values were increased the protein value together with the cereal group plants, so also the feed resources would be increased in the regions with Mediterranean climate. Considering that it contributes to the soil in terms of organic agriculture and that it leaves free nitrogen for the plant to be planted after it, it is suggested that fresh bean cultivation can be done as the pre-plant of winter plants in the summer season. The second crop bean cultivation will contribute to the scarcity of literature in terms of the characteristics examined and will guide the future studies.

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### The Impact of Various Sowing Applications on Nutritional Value of Quinoa Dry Herb

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### ABSTRACT

The present study aimed to determine the availability of the quinoa plant residues, which is mostly grown as grain crops as fodder and the impact of various cultivation applications on nutritional value. For this purpose, the Ca, DM, K, Mg, P, protein content, tetany and milk fever incidences of the post-harvest residues of the quinoa plant, cultivated in different sowing times and with various row spacing applications, were investigated. Thus, it was determined that the Ca, DM, K, Mg, P, protein content, tetany and milk fever incidences content in quinoa straw were 0.933-3.330%; 91.080-87.937%; 0.730-2.237%; 0.200- 1.240%; 0.223-0.303%; 4.403-6.980%; 0.166-2.669 meq, and 3.352-12.268 meq. The study findings demonstrated that the quinoa straw mineral content and balance were suitable for use as an alternative fodder source. However, due to the high milk fever incidence, it was concluded that mono and intensive use of quinoa straw may be risky.

Keywords: Milk fever, plant density, protein, sowing time, tetany



03-04 June 2021, Turkey

# **Dioxins as Environmental Pollutants**

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#### ABSTRACT

Dioxins and dioxin-like compounds are highly toxic, widespread, and stable environmental pollutants, which could be found almost everywhere in the environment throughout the world. The dioxin term is often used to refer to a group of substances structurally and chemically related to the polychlorinated dibenzo-p-dioxins (PCDD) and polychlorinated dibenzofurans (PCDF). Polychlorinated biphenyls (PCB), which share similar structures and toxic properties, are called dioxin-like compounds. There are about 420 congeners of dioxin and dioxin-like compounds. Dioxins are often originated in human activities. They generally occur as an undesirable by-product during industrial processes. Only a tiny part of dioxins is of natural origin. High levels of dioxins were reported in soils, sediments, and some foods. Because of their hydrophobic nature and slow degradation properties in the environment, they tend to accumulate in the food chain, especially in fatty tissues such as dairy products, eggs, meat, and fish. Dioxins are among the most dangerous chemicals that exhibit potential risks to human health and the environment. Human exposure to high levels of dioxins may negatively affect the development of the nervous system and reproductive function, disrupt the immune and endocrine systems, and cause cancer. 2,3,7,8-tetrachlorodibenzo-para-dioxin (TCDD) has been considered the most toxic congener. It has been classified as a carcinogen based on limited evidence in humans and sufficient evidence in experimental animals (Group 1) by the International Agency for Research on Cancer (IARC). In the present study, the structure and properties of dioxin and dioxin-like compounds, their sources, their distribution, and potential toxicity have been reviewed.

**Keywords:** Dioxin, dioxin-like compounds, polychlorinated dibenzo-p-dioxins, polychlorinated dibenzo furans, polychlorinated biphenyls, health, environment



03-04 June 2021, Turkey

# **Black Garlic in the Food Industry**

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#### ABSTRACT

Black garlic is a type of garlic product produced by heating fresh garlic under controlled temperature (60–90°C) and humidity (80–90%) for a while (> 1 month). Many chemical and physicochemical changes occur during the production process, which results in a new derivative with a darker color, softer texture, and sweeter taste and does not release the strong off-flavor of fresh garlic. In addition, many sulfur-containing compounds are formed during production, which contribute to health benefits. Literature data indicate that black garlic and its extracts exhibited antioxidant, antimicrobial, antiobesity, hepatoprotective, anti-inflammatory, antiallergic, and anticancer activities. However, the production conditions (temperature, humidity, and period) strongly affect the black garlic quality and bioactivity. Minimizing the unpleasant taste and odor of black garlic compared to fresh garlic and its enhanced bioactivity, it becomes a popular food item in high-end cuisine and many food products. Black garlic and/or its extracts were added to various types of food products such as sponge cake, bread, jam, jelly, yogurt, candies, ice creams, sausage, and meatballs. Herein, some properties and health benefits of black garlic and its application in the food industry, especially meat products, have been reviewed.

Keywords: black garlic, bioactivity, food industry



# Antifungal Activities of Essential Oils Against Mycotoxigenic Fungal Agent *Fusarium incarnatum*, Causal Disease Agent of Pepper Fruit Rot

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### ABSTRACT

Chilli pepper (*Capsicum annuum* L.), is one of the most commercially important, widely grown and consumed vegetables in Turkey. During a survey conducted from June to September of 2017, *C. annuum* plants showing typical fruit rot symptoms were observed in several inspected fields and local retailers/bazaars in the Hatay province of Turkey. Among the 40 isolates obtained, six isolates caused typical disease symptoms on artificially inoculated pepper fruits. Based on morphological characteristics of fungal structures and molecular analysis, all isolates were identified as *Fusarium incarnatum* (Desm.) Sacc. 1886. To our knowledge, this is the first report of fruit rot disease on chilli pepper caused by potentially mycotoxigenic fungal agent *Fusarium incarnatum* in Turkey. Antifungal activities of six different essential oils (EOs) were evaluated against mycelial growth inhibition fungal agent. The complete mycelial growth inhibition was recorded at the relatively low concentrations used for EOs of *Origanum syriacum* (4.0 µl petri<sup>-1</sup>) followed by *Thymbra spicata* (6.0 µl petri<sup>-1</sup>) and *Thymus vulgaris* (6.0 µl petri<sup>-1</sup>). Essential oils of *Foeniculum vulgare, Laurus nobilis* and *Eucalyptus camaldulensis* have showed antifungal activities against fungal isolate at the relatively higher concentrations used (20.0-25.0 µl petri<sup>-1</sup>). Higher antifungal efficacies of essential oils of Thyme-like plants against fungal agent suggesting their use in different food technologies as biopresevative.

Keywords: Pepper, Fruit rot, Essential oils, Antifungal, Fusarium incarnatum



# Antioxidant Potential of Milk Obtained from the Most Important Breeds of Dairy Cattle in Poland

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#### ABSTRACT

Consumption of food products which are natural sources of antioxidants improves the body's antioxidant status and reduces the risk of a number of civilization diseases. One such product is milk, which contains a number of bioactive substances with antioxidant activity, including whey proteins and vitamins. Whey proteins, mainly  $\beta$ -lactoglobulin ( $\beta$ -LG), have the highest antioxidant status of all proteins in the human diet. Among vitamins, the most important antioxidant in milk is vitamin E, which activity prevents lipid peroxidation by scavenging singlet oxygen or oxidizing cholesterol. The aim of the study was to determine variation in the antioxidant potential of milk depending on the breed of cow and the feeding season. Individual milk samples were collected from cows of the Simmental (SM) and Holstein-Friesian (HF) breeds during two feeding seasons – winter (W) and summer (S). In the summer season, the cows grazed in a pasture and additionally received haylage, hay, and concentrate feed. In the winter season maize silage was introduced to the diet. A total of 275 milk samples were collected, i.e. 120 from SM cows (60 in each season – W and S) and 155 from HF cows (75 in season W and 80 in season S). The following determinations were made in all samples: content of crude protein, fat, lactose, and dry matter (Infrared Milk Analyzer; Bentley Instruments, USA), casein (AOAC, 2000), and somatic cell count (Somacount 150; Bentley Instruments, USA). In addition, in samples in which SCC did not exceed 400,000 cells/ml, the content of selected whey proteins, i.e.  $\alpha$ -lactalbumin ( $\alpha$ -LA),  $\beta$ -lactoglobulin ( $\beta$ -LG), lactoferrin (Lf), and serum albumin (BSA), as well as fat-soluble vitamins (A, D<sub>3</sub> and E) was determined by RP-HPLC using a ProStar 210 system (Varian, USA). Cholesterol content was determined by spectrophotometry (Varian Cary BIO, USA; wavelength 570 nm), and total antioxidant status (TAS) using tests (Randox Laboratories Ltd, UK). In addition, the degree of antioxidant protection (DAP) was calculated as the molar ratio between antioxidants and oxidants according to Pizzoferrato et al. (2007). Milk from SM cows, irrespective of the production season, had significantly (p≤0.01) higher content of crude protein and casein than the milk of HF cows. It also had significantly (p≤0.05) higher levels of bioactive compounds, i.e. whey proteins (except for BSA) and vitamins. It should be emphasised that milk obtained in the pasture season was a significantly (p≤0.01) richer source of bioactive substances, irrespective of the breed of cow. Significant  $(p \le 0.01)$  differences in TAS and DAP were also shown between seasons, in favour of the pasture season. In addition, significant positive correlations were obtained between TAS and the content of vitamins A and E and of β-LG, which indicates that the content of these compounds largely determines the antioxidant potential of milk. DAP was positively ( $p \le 0.01$ ) correlated with TAS and negatively ( $p \le 0.01$ ) with cholesterol content. Importantly, the higher TAS and DAP values during the pasture season ensure greater stability and quality of milk and dairy products.

Keywords: degree of antioxidant protection, milk, total antioxidant status



# Change in the Antioxidant Activity and Total Phenolics with Thermal Treatment

and Incorporation Way of *Pistacia Terebinthus* in Ice cream

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### ABSTRACT

Pistacia terebinthus belongs to the same family with Pistacia vera and contains many bioactive compounds having antioxidant, antimicrobial, anti-inflammatory and cytotoxic activities. Although P. vera is a common flavor used in ice-cream in Turkey, P. terebinthus consumption is limited to being a snack food or coffee-like drink. In this study P. terebinthus is added to the ice-cream after heat treatment at different temperatures (A: 0, B: 100, C: 125 and D: 140°C for 20 min.). Unheated and heated P. terebinthus seeds were milled, hard shells were removed and added to the ice-cream mix before pasteurization of the mix. Change in the antioxidant activity and phenolic content of *P. terebinthus* due to any possible interactions with milk proteins and sugar when heating the ice-cream mix is also examined by comparing the control P. terebinthus solution with milk solutions (9% milk powder, 10% sugar) where P. terebinthus is added before and after heating the milk solution to 80°C for 1 min. Stage of adding P. terebinthus didn't appear to influence total phenolics and antioxidant activity according to ABTS assay. DPPH test showed a slight decrease in antioxidant activity when it is added to milk solution and heated together, however that was statistically insignificant. Antioxidant activity and total phenolics were determined by DPPH, ABTS assays and Folin-Ciocalteau total phenolics assay. The physical, chemical and sensory properties of ice creams (A, B, C and D) were compared with control (K) that doesn't contain P. terebinthus. Adding P. terebinthus to ice-cream reduced its pH, lengthened its melting time, however didn't affect its viscosity as compared to K. Heat treatment of P. terebinthus increased its total phenolics and antioxidant activity. Highest antioxidant activity and total phenolics were observed at ice-cream C. Sensory evaluation was also found the C better in terms of general acceptability.

Keywords: Ice cream, Pistachia terebinthus, Antioxidant activity, Heat treatment



# Differently Structured Systems as a Carrier for the Bioactive Sea Buckthorn Pomace Extract

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### ABSTRACT

The objective of this study was to produce differently structured aerogels as a carrier for the bioactive sea buckthorn pomace extract intended for food applications. Kognac glucomannan cryogels were produced with different concentrations (0.2, 0.3 and 0.4 M) of sodium carbonate by applying a subsequent lyophilization process. Whey protein alcogels (20 %) were prepared by removing pore fluid using supercritical CO<sub>2</sub> drying. The aerogels were analyzed by their morphology, encapsulation efficiency and antioxidant capacity of extract. The properties of konjac glucomannan cryogels depended on the concentration of sodium carbonate used for deacetylation. The pores in the konjac glucomannan aerogels were irregular in shape and the decrease in total pore volume (0.026 to 0.019 cc/g) as well as surface area (12.39 to 11.40 m<sup>2</sup>/g) after increasing the carbonate concentration was observed. Meanwhile, pores in the whey protein alcogels were spherical with a total pore volume of 0.135 cc/g. Cognac glucomannan cryogels were found to have better encapsulation efficiency properties of hydrophilic sea buckthorn pomace extract (17 to 20%) in comparison with whey protein alcogels (0.05 to 0.36 %). It was observed that the antioxidant binding capacity of the extract encapsulated in both type aerogels increased during storage. However, whey protein aerogels presented higher antioxidant capacity (48 to 150 µc) than kognac glucomannan (70 to 181 µc).

The obtained aerogels (especially kognac glucomannan-based ones) showed potential to be applied in food industry as a carrier of bioactive sea buckthorn pomace extract.

Keywords: food-design, encapsulation, aerogels, structure, sea buckthorn



# Chemical Composition and Functional Properties of *Cynara cornigera* Lindley Shoot System Extract

International Conference on

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**RAWMATERIALSTO PROCESSED FOODS** 

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#### ABSTRACT

The aim of this study is to explore the chemical composition and potentiality (such as antimicrobial, antioxidant and antidiabetic substances) of *Cynara cornigera* L. shoot system extract harvested from the Turkish Republic of Northern Cyprus. Methanolic extract of *C. cornigera* shoot system displayed no antibacterial effect on all tested microorganisms. Although the methanolic extract possessed a significant radical scavenging activity for TPC, TFC, DPPH<sup>-</sup>, ferric reducing capacity, metal chelating and phosphomolybdenum assay, it did not show any antidiabetic activity. In addition, no essential oil was determined in the chemical composition of the extract. These results indicated that the presence of antioxidant substances of shoot system extract of *C. cornigera* can be effective against harmful effects of free radicals. Due to this property, its use in human nutrition in Northern Cyprus becomes even more important.

Keywords: Antidiabetic, Antimicrobial, Antioxidant activity, Cynara cornigera, methanol, Northern Cyprus

#### 1. INTRODUCTION

*Cynara cornigera* is a wild edible plant, belonging to family *Asteraceae*, originating from Mediterranean countries. This plant is mostly grown in some regions of Mediterranean countries such as Egypt, Libya and Cyprus (Sonnante et al., 2007; Ahmida, 2011; Elsayed et al., 2012; Hegazy et al., 2015). It is known as 'Hostes', 'Gafurez' and 'Diken Otu' in Northern Cyprus among the local people. It is a dwarf, perennial, prickly plant having about 30 cm height with numerous base leaves. Its flower head is up to 5 cm with off-white color. It is collected mostly in winter and early spring season. Its stem and receptacle organs are used for human diet. Thorns of the plant must be peeled before being consumed by humans. Then it should be cut into small pieces. It is washed with water before cooking. Its stew is made either plain or with potatoes/dried bean. The boiled meal of the cowpea - Hostes mixture is made and it can be also fried with eggs and cooked with meat (Kaya Yıldırım, 2010; Yilmaz et al., 2012).

Various plants having therapeutic activity, less toxic and not side effects are consumed by people all over the world (Elsayed et al., 2012). Family of *Asteraceae* is accepted as a crucial family of plants with strong hypoglycemic effects (antidiabetic activity) and hepatoprotective activities. It involves abundantly the natural antioxidants, mostly polyunsaturated fatty acids, vitamins such as C, K,  $\alpha$ -tocopherol and  $\beta$ -carotene. It is also rich in polyphenols, especially caffeoylquinic acids and luteolin. (Ahmida, 2011; El Sohafy et al., 2016). Since the ancient times, the members of this family which are rich in phenolic compounds, have been used in herbal medicine for their beneficial and therapeutic effects. Phenolic compounds play a crucial role against cancer, cardiovascular diseases, osteoporosis, diabetes mellitus and neurodegenerative diseases for human nourishment (El Sohaimy, 2014).

Cynara cornigera is used for treatment of several diseases including hepatitis, hyperlipidemia, hepatobiliary



dysfunction, digestive complaints, irritable bowel syndrome, hyperlipoproteinemia, obesity, dyspeptic disorder, diabetes mellitus, liver complaints and for improving liver regeneration after partial hepatectomy in folk medicine in different Mediterranean countries (Ahmida, 2011; Elsayed et al., 2012; Hegazy et al., 2015).

Environmental pollution, polluted waters, radiation, heavy metals, pesticides and oxygen metabolism in living cells are known to cause the formation of free radicals in the human body. Free radicals are source of many diseases, especially cancer. Antioxidants protect the human body against all harms caused by free radicals. Therefore, the significance of food products including antioxidants should be known and they should be consumed in order to obviate the spread of many diseases.

Other point is the emerging pathogenic microorganisms resistant to antibiotics and reduced effectiveness of these antibiotics. Currently, many studies are proceed to find influential solutions against drug-resistant microorganisms all over the world. The discovery of new antimicrobial substances originating from plants leads to new approaches to minimize antibiotic resistance and provides to benefit in preventing side effects caused by antibiotic resistance for human health (Sumengen Özdenefe et al., 2018; Sumengen Ozdenefe et al., 2020).

Diabetes mellitus is a metabolic disorder that affects many people around the world. In recent years, the incidence of diabetes has increased in all countries and many studies have been carried out in this area (Ahmida, 2011).

The absence of studies on the *Cynara cornigera* in Northern Cyprus or Turkey makes this study unique and worthwhile. This research therefore aims to investigate chemical and functional properties of methanolic shoot system extract of *C. cornigera*.

### 2. MATERIALS AND METHODS

### 2.1. Collection and Preparation of Plant Material

Stem and leaves of *C. cornigera* were collected from the Kyrenia region of Northern Cyprus in February 2020. Total wet weight of samples was 5300 grams. The collected shoot system was cleaned by paper towel. The shoot system was cut into small parts with a knife and dried in an oven at 50°C for 6 hours. Total dry weight of samples was 720 grams. The dried samples were powdered and stored at +4°C refrigerator for further analysis.

### 2.2. Extraction

The shoot system of *C. cornigera* was extracted by the methanol (1:10 [w/v]) under shaking conditions for 72 hours at room temperature. 10 g of *Cynara cornigera* L. shoot system was used for extraction. Then, extract was filtered by Wattman No. 4 paper. The extract was kept refrigerated at +4°C for chemical composition and biological analyses.

### 2.3. Antimicrobial Activity

Antimicrobial activity of extract was carried out on Mueller Hinton Agar (MHA) by the standard method (Owusu et al., 2021) following Kirby-Bauer disk diffusion method according to the Clinical Laboratory Standard Institute (CLSI) guidelines (CLSI, 2012). The turbidity of the overnight bacterial cultures were adjusted to 0.5 McFarland standard reference range. 10  $\mu$ L of the each microbial suspension was taken with a pipette and transferred to Mueller Hinton agar and then spreaded homogeneously on the surface with a wooden cotton applicator stick. The sterile antimicrobial blank discs impregnated with 20  $\mu$ L of the extract were placed away from each other. Following the inoculation, the plates were incubated at 37°C for 12-24 h. Then, the inhibition zones around the discs were evaluated.

6 different commercial antibiotics including Erythromycin (E; 15 μg/disc) for *Salmonella typhimurium* ATCC 14028 and *Escherichia coli* ATCC 25922; Penicillin (P; 10 units/disc) for *Bacillus cereus* ATCC 11778; Methicillin (M; 5 μg/disc) for *Staphylococcus aureus* ATCC 25923 and Polymxin B (PB, 300 unit/disc) for *Pseudomonas aeruginosa* ATCC 27853 were tested as the positive controls. Methanol was used as negative control.



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### 2.4. Total phenolic (TPC) and flavonoid content (TFC)

The total phenolic content was performed by Folin-Ciocalteu's colorimetric method as gallic acid equivalents (mg GAE)/g (Stankovic, 2011). The total flavonoid content was determined by Sharm and Vig (2013) method as mg of routine equivalent (mg RE)/g.

### 2.5. Gas chromatography headspace analyses

For determining of essential oil composition in extract, 3 g dried and powdered shoot system sample was analyzed using gas-chromatography coupled with headspace system After optimization processes, headspace conditions were detected as follows: Sample volume ( $\mu$ L): 1; incubation time (min): 30; incubation temperature (°C): 80; syringe temperature (°C): 80. HP-5 MS capillary column with mass selective detector 7890B GC-5977MSD (Agilent, Santa Clara, USA) were used in the analysis. The GC conditions were set as follows; initial column temperature at 50°C for 2 min, then ramped to 150 °C at 10 °C/min and held at same temperature for 5 min. Thereafter, temperature was raised gradually to 240°C at same condition and kept at for 5 min. Samples were injected automatically with split ratio 50:1 (Sevindik, 2020) Components were identified by electronic NIST14 library (2014 version). Percent amounts (%) of each component in sample were calculated from total peaks area by apparatus software.

### 2.6. Antioxidant Activity Analyses

### 2.6.1. DPPH' radical scavenging capacity

Free radical scavenger method declared by Blois (1958), Ucan Turkmen and Mercimek Takci, (2018) is based on turning colorless of the 1,1-diphenyl-2-picryl-hydrazil (DPPH<sup>-</sup>) reagent solution depending the electron or proton-transfer ability of samples. For this analysis, 100  $\mu$ L of the extract was added to 3.9 mL of DPPH<sup>-</sup> reagent (0.025 g/L in methanol) solution prepared in methanol (0.1 mM). For allowing the chemical reaction, this mixture was incubated at room temperature in the dark for 30 min. Following incubation, the absorbance of the mixture was spectrophotometrically measured at 517 nm (Biochrom, Libra S60, B, England) against methanol blank DPPH<sup>-</sup> scavenging activity is expressed as Trolox equivalent (mg TE/g).

### 2.6.2. Ferric Reducing Capacity Assay (FRAP)

In this assay, the reducing  $Fe^{3+}$  to  $Fe^{2+}$  capability of antioxidant substances in extract was tested (Oyaizu, 1986). The absorbance of Prussian blue color formed by adding FeCl<sub>3</sub> in reaction mixture was measured. In brief, the extract (1 mL) was mixed with 2.5 mL of 0.2 M sodium phosphate buffer (pH 6.6) and 2.5 mL of 1% potassium K<sub>3</sub>Fe(CN)<sub>6</sub>. This reaction solution was incubated at 50°C for 20 min. To terminate the activity, 10% TCA was added and the mixture was centrifuged at 2500 rpm for 10 min. The equal volume of distilled water and 0.5 mL FeCl<sub>3</sub> (0.1%) were added to 2.5 mL of supernatant. The absorbance of reaction mixture was measured at 700 nm (Biochrom, Libra S60, B, England). Reducing capacity of extract is expressed as Trolox equivalents ( $\mu$ g TE/g).

### 2.6.3. Metal chelating activity

Fe<sup>2+</sup> chelating activity of extract was performed according to method described by Dinis et al. (1994). This method is based upon the competition of metal-binding compounds in the extract with ferrozine, a strong iron chelating agent. The compounds having high metal ions binding capacity avoid the red Fe<sup>2+</sup>/ferrozine complex formation. Briefly, 3.7 mL distilled water and 100  $\mu$ L of 2 mM FeCl<sub>2</sub> were added to 1 mL of extract. After incubated at room temperature for 30 min, 200  $\mu$ L of 5 mM ferrozine solution was added to the reaction and mixed through 10 min. And the absorbance of reaction mixture was measured at 562 nm (Biochrom, Libra S60, B, England). Chelating activity was expressed as % inhibition using following equation.

% chelating activity =  $(1-(A_{sample}/A_{control})) \times 100$ 

### 2.6.4. Phosphomolybdenum method

The total antioxidant capacity was determined as spectrophotometrically by using the phosphomolybdenum method (Zengin et al., 2014). 3 mL of reactive solution (0.6 M sulfuric acid, 28 mM sodium phosphate, and 4 mM ammonium molybdate) was mixed with 300  $\mu$ L extract rapidly. After incubation at 95°C for 90 minutes, the absorbance was measured at 695 nm (Biochrom, Libra S60, B, England). Total antioxidant capacity was expressed as equivalents of trolox ( $\mu$ g/TE g). Each spectrophotometric analysis was repeated at least three



times.

### 2.7. Antidiabetic activity

For  $\alpha$ -amylase assay, the test tube including 1 mL extract, 1 mL of starch solution (1% w/v) and 1 mL of 20 mM sodium phosphate buffer (pH:6.9) was incubated at 37°C for 5 min. Thereafter, 1 mL of  $\alpha$ -amylase solution was started the reaction by adding into this tube. The reaction was stopped with 1 mL of color reagent prepared with 5.31 M sodium potassium tartrate solution, 2 M NaOH and 96 mM 3,5-dinitrosalicylic acid solution after 30 min. This mixture was boiled for 5 min and the absorbance was measured at 540 nm (Biochrom, Libra S60, B, England) (Başyiğit, et al. 2020).

For  $\alpha$ -glucosidase assay, a volumetric flask including 10  $\mu$ L of extract and 40  $\mu$ L of  $\alpha$ -glucosidase enzyme solution was incubated at 37°C for 5 min. Then, 950 µL of 0.7 mM 4-nitrophenyl-α-D-glucopyranoside solution containing 100 mM NaCl and 50 mM phosphate buffer was added into this flask. After incubation at 37°C for 15 min, the reaction was stopped by adding 1000 µL of 0.5 M Tris. The absorbance was measured at 400 nm (Biochrom, Libra S60, B, England) (Güngör Bilen, 2004). All antidiabetic activitiy analyses were performed three repetitions.

### 3. RESULTS

In this study, extract of Cynara cornigera shoot system was not demonstrated the antibacterial activity against all tested microorganisms. Negative control was not shown any inhibitory effect on all tested strains (Table 1). Commercial Penicillin, Erythromycin, and Polymxin B showed antibacterial activity towards Bacillus cereus ATCC 11778, Salmonella typhimurium ATCC 14028, Escherichia coli ATCC 25922 and Pseudomonas aeruginosa ATCC 27853, respectively. On the contrary, Methicillin did not demonstrate antibacterial activity against Staphylococcus aureus ATCC 25923. The total phenolic, total flavonoid content, antioxidant and antidiabetic activity results of Cynara cornigera methanol shoot extract was shown in Table 2. TPC and TFC values were calculated as 0.176mg GAE/g and 0.1038mg RE/g, respectively. DPPH' radical scavenging activity of extract was evaluated by comparing it with the standard antioxidant activity of Trolox equivalent (mg TE/g). DPPH of extract was determined as 86.2% (0.55 mg TE/g). Results of reducing capacity of Fe<sup>3+</sup>, metal chelating activity and phosphomolybdenum assay were calculated as  $14.1 \mu g TE/g$ , 91.4% and  $53.54 \mu g$ TE/g, respectively. These results indicated that the existence of antioxidant substances of extract could be efficient towards the damaging effects of free radicals.  $\alpha$ -amylase and  $\alpha$ -glucosidase activities of extract were not detected. Table 3 shows the identified compounds their retention times and area percentages by gas chromatography headspace analysis. In GC/MS, 18 compounds were identified using the NIST14 mass spectra library and the main active constituents were ethylene oxide (51.30%), acetaldehyde (31.58%), pentanal (4.87%), hexanal (3.1%), formic acid, methyl ester (CAS) (2.55%) and isobutyraldehyde (1.85%). These results indicated that chemical composition of extract not include any essential oil.

	<i>Cynara cornigera</i> extract	Positive Control	Negative Control
Bacillus cereus ATCC 11778	-	40 (Penicillin)	-
Staphylococcus aureus ATCC 25923	-	- (Methicillin)	-
<i>Salmonella typhimurium</i> ATCC 14028	-	16 (Erythromycin)	-
<i>Pseudomonas aeruginosa</i> ATCC 27853	-	20 (Polymxin B)	-
<i>Escherichia coli</i> ATCC 25922	-	16 (Erythromycin)	-

**Table 1** Diameter of the inhibition zone (mm) of *Cynara cornigera* extract

(-) represents a no inhibition zone against microorganism.

**Table 2** The total phenolic, total flavonoid content, antioxidant and antidiabetic activities of Cynara cornigera methanol shoot extract



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	Cynara cornigera methanol shoot extract
Total phenolic content (mg GAE/g)	$0.176{\pm}0.005$
Total flavonoid content (mg RE/g)	$0.1038 \pm 0.001$
DPPH (% / mg TE/g)	86.2±0.001 / 0.55±0.001
Ferric reducing capacity (µg TE/g)	14.1±0.957
Metal ( $Fe^{2+}$ ) chelating activity (%)	91.4±0.069
Phosphomolybdenum (Total 53.54±0.099	
antioxidant capacity) (µg TE/g)	
α-amylase activity	ND
α-glucosidase activity	ND

Values are mean  $\pm$  Standard deviation (SD) of three replicate analyses. ND: Not detected.

Table 3         The retention time (n	n) and percentage (%) of chemical composition of <i>Cynara cornigera</i>
	methanol shoot extract

Commonia	Cynara cornigera methanol shoot extract		
Compound	Retention time (min)	%	
Ethylene oxide	2.366	51.30	
Carbon dioxide	2.366/2.476	51.30/31.58	
Acetaldehyde	2.476	31.58	
Isobutyraldehyde	3.973	1.85	
1-Propanol	4.884	2.55	
Methyl formate	4.884	2.55	
Formic acid, methyl ester (CAS)	4.884	2.55	
Butanal, 3-methyl-	5.134	4.87	
Pentanal	5.134/6.003	4.87/0.80	
Hexanal	7.487	3.11	
1-Penten-3-ol	8.618	0.34	
Furan, 2-pentyl	9.732	0.49	
1-Pentanol	9.966	0.39	
Acetic acid	12.728	1.22	
2-Furan-carboxaldehyde	13.087	0.94	
Furfural	13.087	0.94	
Butanoic acid, 3-methyl	15.510	0.54	
1,2-Hydrazinedicarboxaldehyde	15.510	0.54	

### 4. DISCUSSION

Two Gram-positive, three Gram-negative bacterial strains were tested for antimicrobial activity of the *Cynara* cornigera shoot system methanol extract. Extract did not show antibacterial activity against all tested microorganisms.

El Sohaimy (2014), reported methanol extracts of globe (*Cynara cardunculus* L) and baby artichoke (*Cynara scolymus*) which showed antimicrobial activity against *Proteus vulgaris* ATCC 6830 ( $2.63\pm0.15$  and  $3.42\pm0.32$  cm), *Escherichia coli* 0-143 ( $2.76\pm0.21$  and  $3.54\pm0.25$  cm), *Staphylococcus aureus* 0006 ( $1.75\pm0.13$  and  $3.45\pm0.19$  cm), *Klebsiella pneumonia* 8961 ( $1.60\pm0.14$  and  $2.12\pm0.16$  cm) and *Bacillus subtilis* ATCC 6633 ( $1.86\pm0.23$  and  $2.67\pm0.26$  cm), respectively. The MIC of methanol extract of globe and baby anzio artichoke were determined as 100 and 75 mg/ml, respectively. Methanol extract of *Cynara scolymus* was more effective than *Cynara cardunculus* L. extract against all tested bacterial strains (El Sohaimy, 2014). Falleh et al. (2008) reported that the methanolic leaf extract of *Cynara cardunculus* L. showed antibacterial activity against *S.aureus* ATCC 25923 ( $25.7\pm0.6$  mm) and *E.coli* ATCC 35218 (22.3 mm). In addition, leaf extract



showed inhibitory effect on tested other microorganisms such as *S. epidermidis* CIP 106510 (20.3 mm), *Micrococcus luteus* NCIMB 8166 (21.7±0.6 mm), *E. feacalis* ATCC 29212 (16.3±0.6 mm), *L. monocytogenes* ATCC 19115 (9.3±0.6 mm), *P. aeruginosa* ATCC 27853 (13.7±0.6 mm). The leaf extract did not have any inhibitory effect against *Salmonella typhimurium* LT2 (Falleh et al., 2008).

In the study of Zhu et al. (2004) the *n*-butanol fraction of *Cynara scolymus* L. leaf extract showed more antimicrobial activities than that of chloroform and ethyl acetate fractions against *B. subtilis*, *S. aureus*, *A. tumefaciens*, *M. luteus*, *E. coli*, *S. typhimurium* and *P. aeruginosa*. 2.5 mg/mL of ethyl acetate fraction did not show any inhibitory effect to all tested microorganisms. Similary, any inhibitory effect did not observe for chloroform fraction in same concentration except *Bacillus subtilis*. *M. luteus*, *S. typhimurium* and *P. aeruginosa* were resistance to 2.5 mg/mL concentration of n-butanol fraction. In addition, *M. luteus* and *P. aeruginosa* were resistant to 5 mg/mL chloroform fraction. *S. aureus*, *A. tumefaciens* and *P. aeruginosa* were also resistant to 5 mg/mL concentration of ethyl acetate fraction. Briefly, only *P. aeruginosa* was resistant to the 2.5, 5 and 10 mg/mL concentration of ethyl acetate fraction of leaf extract.

The EtOAc extract of *C. cardunculus* displayed the most effective antibacterial activity, followed by the EtOH, CHCl<sub>3</sub>, water and n-BuOH extracts. *S. typhimurium* was found to be the most resistant. However, *E. coli* was the most sensitive against all tested extracts (Kukic et al., 2008). In the study of El Sohaimy (2014), Falleh et al. (2008), Kukic et al., (2008) extracts exhibited antibacterial activity against *E. coli*, *S. aureus* and *P.aeruginosa*. However, no inhibition zone against *E. coli*, *S. aureus* and *P. aeruginosa* was observed in the methanolic extract in this our study. Antibacterial activity result of *Cynara cornigera* extract against *S. typhimurium* and *P. aeruginosa* was similar to the results of Kukic et al. (2008), Zhu et al. (2004) and Falleh et al. (2008). Since there was no previous research on the antibacterial activity of *Cynara cornigera* methanol extract, the authors could not compare the results against same species. For this reason, a comparison has been made with studies conducted with different species of the same genus.

Nowadays it is accepted that phenolics make the greatest contribution to the antioxidant activity of plant foods. Therefore, it is important to determine the phenolic content of the extract. TPC and TFC results of Cynara cornigera methanol shoot extract was determined as 0.176±0.005 mg GAE/g and 0.1038±0.001 mg RE/g, respectively. The TPC and TFC results obtained from the several studies with different species of the Cynara genus are given following. According to Stumpf et al. (2020) TPC (TPC is referred to as not total phenolic content but total phenolic concentration in their study, mg GAE g<sup>-1</sup>DM) and total flavonoids (% DM) of leaves of globe artichoke (Cynara cardunculus var. scolymus L.) were determined by Ph. Eur (extraction according to European Pharmacopoeia), UAE (ultrasound assisted extraction) and HW (hot water extraction) extracts. TPC and total flavonoids values of Ph. Eur., UAE and HW extracts were as  $32.7\pm1.8$ ,  $31.9\pm1.7$ ,  $29.5\pm1.8$ ,  $0.68\pm0.04$ ,  $0.60\pm0.03$  and  $0.53\pm0.03$ , respectively. Salem et al. (2017) reported that maximum TPC value of Cynara scolymus L. leaves extract was obtained in EtOH extract with 54.54±1.26 mg GAE/g DW. TPC values of ethyl acetate, aqueous, butanol and hexane extracts were determined as 53.07±0.47, 49.49±0.39, 41.66±2.23 and 39.91±9.36 mg GAE/g DW, respectively. TFC values of extract were  $12\pm0.83$  and  $8.19\pm0.6$  mg CE/g DW for EtOH and hexane extracts. The total phenolic content (TPC) of globe artichoke (Cynara cardunculus L) and baby anzio (Cynara scolymus) species of methanol extract were reported as 30.70±1.87 and 38.31±0.96 mg GAE/g DW (El Sohaimy, 2014). Lutz et al. (2011) revealed that total phenolics (TP) concentration of mature and baby artichokes (Cynara scolymus L.) in AE (Aqueous extract) and HE (hydroalcoholic extract) extracts. TP of mature and baby artichokes were detected as 5.40±0.13 and 4.55±0.14 mg TAE/100 g d.w., whereas the values of HE was 5.76±0.09 and 5.93±0.15 mg TAE/100 g d.w., respectively. In other study, leaf and seed methanolic extracts of Cynara cardunculus L. were declared the total polyphenol content as 14.79 and 14.33 mg GAE/g DW). However, flower extract was 6.96 mg GAE/g DW. Flavonoid content of leaf, seed and flower extracts were 9.08, 9.78 and 5.58 mg CE/g DW, respectively (Falleh et al., 2008).

Total phenolic contents of EtOAc, n-BuOH, EtOH,  $H_20$  and CHCl<sub>3</sub> extracts of *Cynara cardunculus* whole involucral bracts were determined as  $0.203\pm0.018$ ,  $0.062\pm0.019$ ,  $0.050\pm0.010$ ,  $0.046\pm0.007$  and  $0.026\pm0.002$  mg of gallic acid equivalent/mg dry weight, respectively (Kukic et al., 2008). The total phenol content of different parts of five artichoke varieties (*Cynara cardunculus* L. var. *scolymus* (L.) Fiori) were notified by Fratianni et al. (2007). TPC values of receptacle, inner, intermediate, outer bracts and leaves were reported with ranging from 3.09-1.32, 8.03-2.33, 1.74-0.50, 1.79-0.51 to 2.30-052 mM/g. Similar to our results,



literature data indicate that different species of the genus of Cynara have different amount of phenolic content.

DPPH<sup>·</sup> of *Cynara cornigera* methanol shoot extract was determined as 86.2%. Furthermore, results of ferric reducing capacity, metal chelating activity and phosphomolybdenum assay were detected 14.1µg TE/g, 91.4% and 53.54µg TE/g, respectively. Determination of antioxidant activity was done with applying similar and different methods in previous studies. Antioxidant capacity of *Cynara cardunculus* var. *scolymus* L. was determined by using the oxygen radical absorbance capacity (ORAC) assay. ORAC values of Ph.Eur., UAE and HW extracts were  $524\pm30$ ,  $518\pm30$  and  $442\pm27$  µmol TE/g DM, respectively (Stumpf et al., 2020). The antioxidant activity of Artichoke Leaves Extracts (ALE) was performed by using four different methods. Results of the EtOH extract of *C. scolymus* of DPPH, ABTS, FRAP and β-carotene test were found 94.23%, 499.43 mmol Trolox/g DW, 527.79 µmol Fe<sup>2+</sup> DW and 70.74%, respectively (Salem et al., 2017).

Free radical scavenging activity, reduction capability, superoxide anion scavenging activity, metal chelating activity, total antioxidant capacity (ABTS) of *Cynara cornigera* methanol extract were determined by Hegazy et al. (2016). Compound 1 displayed both a great metal chelating activity and a high total antioxidant capacity (ABTS) compared with 2-7 as well as the standard butylated hydroxytoluene. Free radical scavenging activity (DPPH) of compounds 2, 4, and 6 exhibited approximately the same inhibition level as 96% with an even greater activity than BHT. The ferric reduction capability of compound 4 and 6 were exhibited a significant reducing power at 5  $\mu$ g/mL. In superoxide anion scavenging activity assay, compound 6 achieved the most influential scavenging activity as 100% at 20 and 40  $\mu$ g/mL concentrations rather than BHT which has lowest effect at all concentrations.

El Sohaimy (2014) reported that the antioxidant activity of globe and baby anzio artichoke methanol extracts were determined by DPPH-Free radical scavenging activity.  $IC_{50}$  of globe artichoke extract was  $55.12\pm0.31\%$ in concentration of 75  $\mu$ g/ml, while IC<sub>50</sub> of baby anzio artichoke extract was 49.52 $\pm$ 0.16%. The antioxidant activity of leaf extracts of the wild artichoke (Cynara cornigera) was analysed by using DPPH radical scavenging activity. 80% methanol, ethyl acetate fractions, compound 1 (luteolin 7-O-glucoside) and compound 3 (luteolin -7- O-rutinoside) displayed the maximal antioxidant activities as 88.46%, 89.77%, 90.2% and 90.5%, respectively (Elsayed et al., 2012). The antioxidant activites of the aqueous and hydroalcoholic extracts in mature raw and baby raw of Cynara scolymus L. were found 30.50±0.14, 5.02±0.17, 28.46±0.33 and 22.97±0.39% (Lutz et al., 2011). These values were lower than of methanol extract of this study. Falleh et al. (2008) were evaluated the antioxidant activity of Cynara cardunculus L. organs extracts by free radical-scavenging and superoxide anion-scavenging activities methods. The authors reported that seeds extract possessed the highest DPPH as compared to leaves and flowers.  $IC_{50}$  values of seeds, leaves and flowers were 23, 53 and 118, respectively. Antioxidant activity of *Cynara cardunculus* extracts were examined using the ferric reducing antioxidant power assay (FRAP) and DPPH radical assay. FRAP values of EtOAc, n-BuOH, EtOH, H<sub>2</sub>O and CHCl<sub>3</sub> extracts were  $0.38\pm0.01$ ,  $0.36\pm0.01$ ,  $0.35\pm0.01$ ,  $0.34\pm0.01$  and  $0.12\pm0.02$ µmol Fe<sup>2+</sup>/mg DW, respectively. On the other hand, SC50 (the concentration of extracts that caused 50% of neutralization) values of EtOAc, n-BuOH, EtOH and H<sub>2</sub>O extracts for DPPH were revealed  $21.50\pm1.87$ , 127.10±0.88, 157.00±0.16 and 173.15±0.65 µg/ml, respectively. The SC50 value of CHCl<sub>3</sub> extract did not achieve 50% of DPPH neutralisation at the highest concentration tested. Authors determined that EtOAc extract had the strongest activity among all extracts (Kukic et al., 2008). When this study was compared with previous studies according to antioxidant results, it was determined that the results of Salem et al. (2017), Hegazy et al. (2016) and Elsayed et al. (2012) were consistent with the results of this study.

*Cynara cornigera* shoot system methanol extract was not exhibited any antidiabetic activity. Unlike the abovementioned studies, there is an *in vivo* study. The different findings regarding the antidiabetic effect of *C. cornigera* extract have been reported by Ahmida. The author informed that antidiabetic activity of aqueous extract of the roots of *Cynara cornigera* in alloxan-induced experimental diabetes mellitus. Application of aqueous extract (1.5 g/kg) and glibenclamide (10 mg/kg) orally in diabetic rats were resulted in a apparent reduction on blood glucose level from 330.80±10.11 mg/dLto 229.70±7.94 and 195.50±6.53 mg/dL for extract and glibenclamide, respectively. Additionally, serum insulin levels (from  $18.00\pm1.23 \ \mu\text{L/mL}$  to  $27.00\pm1.19$  and  $32.00\pm1.73 \ \mu\text{L/mL}$  for extract and glibenclamide, respectively) and liver glycogen content (from  $14.80\pm1.10 \ \text{mg/gto} 33.65\pm1.96$  and  $39.10\pm2.47 \ \text{mg/g}$  for extract and glibenclamide, respectively) were reclaimed through *Cynara cornigera* aqueous extract (Ahmida, 2011).



To our knowledge, this is the first study regarding the chemical composition of *C. cornigera* shoot system. For this reason, the authors discussed chemical substances of its to the other *Cynara* species. Hadaruga et al. (2009) expressed that  $\beta$ -cubebene was the main compound in flowers, stem and root extracts (38%, 39% and 25%, respectively) of *C. scolymus* L.; (*E*)-2-hexenal was the major biocompound (20%) in leaf extract. A previous study reported that 25 compounds were identified of which hexadecanoic acid (27.6%), methyl hexadecanoate (9.3%) and methyl 9,12-octadecadienoate (12.6%) in flowers extract of *C. cardunculus* L. (Mucaji et al., 2001). Dabbou et al. (2017) identified sesquiterpenes hydrocarbons as the main chemical class of *C. scolymus* in particular  $\beta$ -selinene, followed by  $\beta$ -caryophyllene. In the other study, Saucier et al. (2014) found a total of 130 compounds including oxygenated monoter-penes, sesquiterpenes, oxygenated sesquiterpenes, norisoprenoids, lactones, alcohols, ketonesand aldehydes in *C. scolymus* L. leaves. Similar to previous study, Nassar et al. (2013) observed 37 compounds in the volatile oil, the majority including mono and sesquiterpenes for *C. scolymus* L. methanol extract.

In conclusion, these findings demonstrated that phenolics and flavonoids are a key factor for the antioxidant activity of *Cynara cornigera* extract. The present paper is the first study which describes the identification of chemical constituents of the *C. cornigera* methanolic shoot extract. Our results related to antioxidant activity of the extract are emphasized the importance of its consumption as a beneficial food product and the traditional medicinal usage of *Cynara cornigera*.

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### Quality Evaluation of Pumpkin (*Cucurbita Pepo*) Powder Produced Using Three Different Drying Methods

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### ABSTRACT

Matured pumpkins (*Cucurbita pepo*) are sweet with orange flesh rich in  $\beta$ -Carotene. They are valuable sources of functional components such as carotenoids, lutein and selenium but underutilised. Pumpkin has potential as food and industrial crop. Poor postharvest handling contributed to wastage during bumper. Value-added products such as pumpkin powder will create shelf stable product with appreciable keeping quality. Pumpkins were cleaned, cut and peeled. The seeds were scrapped off and flesh cut into uniform sizes. Samples were blanched in hot water at 95°C for 1 minute, drained and cooled to room temperature. They were then dried using three different methods (cabinet, tent and open sun). The dried samples were grounded into powder using laboratory mill and sieved using 125-micron mesh. The samples were evaluated for proximate composition, functional properties, microbial and sensory qualities. Proximate analysis results showed relatively low moisture content across all methods. The moisture content of the powders ranged from 4.690±0.410 to 85.42±1.66 with cabinet dryer having the least moisture of 4.690±0.410. Samples were within limits for safe storage. Cabinet dried sample had the highest protein, crude fibre, ash and  $\beta$  carotene compared to others. The results indicated that increase in drying temperature are accompanied by decreases in the water solubility, water and oil absorption capacities of the resulting powders. Tent and sun-dried samples had better functional properties. Results of microbial analyses revealed low bacterial and fungal counts with two samples (fresh and cabinet dried) recording no fungal growth. Results of sensory evaluation of *Mivar taushe* prepared from the samples with fresh sample serving as control revealed that cabinet dried pumpkin powder had good sensory quality in terms of colour, taste, flavour and acceptability. Good quality pumpkin powder rich in nutrients with good sensory qualities that could be utilised for culinary purposes could be produced using cabinet dryer.

Keywords: Drying, Pulp, Pumpkin, Powder



# **Grain Quality Characteristics of Local Popcorn Populations**

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### ABSTRACT

Popcorn is a healthy consumption material because it has low calories. Popcorn has an important place in human nutrition due to its popping property and the nutrients it contains. In this study, seventeen local popcorn populations and three registered popcorn varieties were grown as second crops in 2019 under Kahramanmaras conditions and their some quality characteristics were investigated. In this study, ash, oil, moisture, protein and starch ratios were determined in the grain samples that were ground into flour, thousand grain weights and popping volume of each population were determined. According to the results of this research, thousand grain weights of local popcorn populations were found between 210.44 - 97.43 g and the highest thousand grain weight value was obtained from Ordu Kabartur population. The ash rate in the grain was found between 1.73-1.48% and the highest value was recorded in Nermin Cin popcorn variety. Fat ratio was found between 6.69-4.35% and the highest value was obtained from Çanakkale Red population. The moisture content in the grain was between 10.99-10.33% and the highest value was found in Ordu Catalpinar population. Protein ratio is between 10.66-8.88% and statistically no difference was observed between the populations. The starch rate in the grain was found between 67.11-61.65% and the highest starch rate was obtained from Samsun White. In the study, the popping volumes of popcorn varied between 28 and 7.33 cm<sup>3</sup> g<sup>-1</sup>. In the research, Samsun bafra Kasu line had the highest popping volume with 28 cm<sup>3</sup> g<sup>-1</sup>, while the Ordu Persembe Kovanlı line had the lowest popping volume with 7.33 cm<sup>3</sup> g<sup>-1</sup>.

Keywords: Popcorn, Quality Charecteristics, Ash, Protein, Starch

### **INTRODUCTION**

People, plants and animals living on the soil constantly explore their environment and provide a living space for themselves. Humans, the most intelligent of these creatures, use the animals and plants around them to shape them and use them for their own welfare and nourishment. The increasing human population increases the need for plant and animal foods. Grain group plants are at the top of the herbal products used by people most in the world. At the same time, the most fed product for animals are cereals. For this reason, studies on cereal crops from past to present maintain their continuity.

Cereal plants known as Gramineae family are divided into two groups as cool and warm climate cereals according to the climates in which they are grown. While cool climate cereals show the largest distribution area in the world, the cultivation of hot climate cereals with higher temperature demand is more limited than cool climate cereals. Corn, which is included in the Maydeae tribe of the Gramineae family, is a C4 plant that can make the best use of solar energy and gives the highest yield in a short time. The corn plant has seven varietal groups due to the fact that it forms different grain shapes in its evolution. Horsetooth corn, hard corn, popcorn and sugar corn varieties of corn plant are cultivated economically (Kırtok, 1998). The economic usage areas of maize differ according to the varieties. Pop corn has popping ability. It has been determined that the temperature permeability and mechanical strength of the popcorn grain is 2.2-2.9 times higher than the other corn varieties, increasing the burst quality (Silva et al. 1993). Since popcorn poping when it is heated, it has become a snack that is loved by people. Popcorn (Zea mays L. everta Sturt.) is the maize which has the hardest and smallest grains. It has a short vegetation period, is generally considered as a snack and finds buyers at high prices.(Özkaynak and Samanci 2003). A significant part of the world's popcorn production is in the USA. Consumption of popcorn, which is mostly consumed in the USA, is also increasing in our country. Popcorn is a preferred food item in terms of nutrition because of the vitamins and minarets it contains. It is a good dietary



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product with its satiating and stomach acid absorbing feature (Ülger, 1998).

Due to the growing has a short periot and high yield of the corn plant, commercial companies had to constantly develop new corn varieties. Local maize populations and gene resources are destroyed by manipulate the genes of maize (İdikut et al., 2012) ). The spread of hybrid varieties, the use of too many inputs and the increasing commercialization rate in agriculture have narrowed the production area of local plant species. Although highly productive hybrid varieties are predominate, local varieties are still cultivated in some areas.Local corn varieties have generally succeeded in yielding products in adverse conditions by completing their natural selection in their environment. Even within themselves, the characteristics of having different genetic variations can complement each other's deficiencies and be superior to commercial varieties in terms of some characteristics (Allard and Bradshaw, 1964). For this reason, studies that will protect the gene resources of our local varieties should be supported for the transfer of next generations.

Agricultural practices differ according to regional conditions. These differences are due to climate, soil and water sources. For this reason, agricultural practices are planned according to regional conditions. In some regions, climatic conditions allow corn to be grown as a monoculture, while in some regions it allows it to be grown as a second crop after the main crop. When the areas with irrigation facilities are evaluated for second crop corn, it contributes to animal production due to the use of stubble residues left in the field for grazing (Sarikurt and Bengisu 2020).

Although the yield of commercial varieties is higher than local varieties (Özkaynak and Samancı, 2003), local genotypes are more resistant to adverse conditions. Due to the necessity of protecting local populations and maintaining agriculture, 20 popcorn varieties were grown as a second crop in Kahramanmaraş region, which has a Mediterranean climate, and grain quality characteristics were examined.

### MATERIAL AND METHOD

As research material, 2 commercial varieties (Ant cin, Nermin cin) and local popcorn population obtained from different regions of Turkey were used. The grain material used in the research was named after the region from which it was obtained. In the research, Ant (1), Çanakkale Red (2), Samsun Yellow (3), Ordu Çatalpınar (4), Edirne White (5), Tokat Erbağ (6), Çanakkale White (7), Çanakkale Yellow (8). ), Sakarya Han Village (9), Samsun Bafra Kasu Village (10), Nermin (11), Balıkesir (12), Kadirli (13), Ordu Perşembe Kovanlı Village (14), Balıkesir White (15), Ordu Kargan Tepealan (16), Ordu Çamaş Giden District (17), Samsun White (18), Ordu Kabartur Çukurca Mahallesi (19), Composite 13 (20) genotypes were used. A total of 20 maize genotypes were grown in Kahramanmaraş conditions in the second crop growing season (between June and September) in 2019.

The minimum temperatures for the months of June and October 2019, in which the experiment was conducted, were 19.9 °C, 21.80 °C, 29.3°C, 17.7 °C, maximum temperatures 35.9 °C, 36.6 °C, 38 °C, 35.2 °C, average temperatures 27.2 °C, 28.4 °C., 29.3 °C, 26.0 °C, 21.30 °C humidity were 50.1%, 49.8%, 50.5%, 43.3%, and the average precipitation was recorded as 5.2, 0.2, 0, 1, 174.60, respectively. It has been determined that the experimental area has a clayey texture, rich in useful potassium, calcareous, neutral and medium organic matter soil. The experiment was planted on 19.06.2019 according to the randomized blocks experimental design with three replications, 70 cm row spacing and 20 cm row spacing in 4 rows. 18-46 DAP fertilizer was applied with sowing, so that 6 kg net phosphorus fertilizer per decare. When the plant was 50 cm tall, urea fertilizer was applied so that a total of 25 kg nitrogen per decare was dropped. Tractor hoe was applied to the plants in the experimental area twice and the plants were watered 9 times during the growing period. The trial was sprayed twice for the for the corn worm. The data obtained at the end of the research were analyzed using the SAS statistical program according to the ANOVA method. The means of the features that differed statistically significantly were grouped according to the Duncan multiple comparison test.

### FINDINGS AND DISCUSSION

In the research, thousand grain weight, ash, oil, moisture, protein, starch ratios and popping volume of popcorn genotypes were examined. Except for the protein content in the grain, other properties showed significant differences.

**Table 1**. The averages and groups of thousand grain weight (g), ash (%), oil (%), moisture (%), protein (%), starch (%) and popping volume (cm<sup>3</sup> g<sup>-1</sup>) of local popcorn genotypes.



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Genotypes	Thousand Grain Weight (g)	Ash (%)	Oil (%),	Moisture (%)	Protein (%)	Starch (%)	Popping volume (cm3 g <sup>-1</sup> ) **
1.Ant cin	190.53 a	1.610 a- d	5.93 а-с	10.73 a-d	9.35 a	64.09 ab	21.00 b-d
2.Çanakkale K.	97.43 e	1.686 а- с	6.696 a	10.33 d	9.64 a	63.01 ab	18.53 ef
3.Samsun Sarı	148.30 b	1.593 a- d	5.95 a-c	10.65 a-d	9.34 a	64.76 ab	19.00 de
4.Ordu Çatalpınar	184.68 a	1.566 a- d	6.353 ab	10.99 a	9.49 a	65.05 ab	9.26 ıj
5.Edirne Beyaz	185.05 a	1.603 a- d	4.41 bc	10.94 ab	9.59 a	64.47 ab	13.60 h
6.Tokat Erbağ	117.14 с- е	1.596 a- d	5.13 а-с	10.70 a-d	10.44 a	64.75 ab	15.06 gh
7.Çanakkale Beyaz	128.69 b- d	1.720 ab	4.79 a-c	10.70 a-d	10.66 a	63.51 ab	22.20 b
8.Çanakkale sarı	125.53 b- e	1.483 d	4.35 c	10.45 b-d	10.00 a	66.07 ab	26.53 a
9.Sakarya han köyü	141.88 b- c	1.706 ab	6.16 a-c	10.45 a-d	10.47 a	61.65 b	16.66 fg
10.Samsun B. Kasu	100.95 de	1.566 a- d	6.496 a	10.79 a-d	9.33 a	64.15 ab	28.00 a
11.Nermin Cin	136.35 bc	1.730 a	6.06 a-c	10.80 a-d	10.59 a	63.21 ab	20.20 b-e
12.Balıkesir Cin	136.99 bc	1.616 a- d	6.22 a-c	10.67 a-d	9.82 a	62.91 ab	19.33 с-е
13.Kadirli Cin	144.29 bc	1.626 a- d	5.45 a-c	10.85 a-c	10.09 a	64.22 ab	21.53 cb
14.Ordu P. Kovanlı	195.56 a	1.506 cd	4.99 a-c	10.38 cd	9.10 a	66.47 a	7.33 j
15.Balıkesir Beyaz	139.92 bc	1.553 b- d	5.12 a-c	10.56 a-d	9.40 a	67.10 a	20.33 b-e
16.Ordu K. Tepealn	205.36 a	1.623 a- d	5.95 a-c	10.33 d	9.49 a	63.18 ab	9.73 1
17.Ordu Ç. Giden	188.99 a	1.643 a- d	6.433 a	10.69 a-d	10.01 a	63.71 ab	9.73 1
18.Samsun Beyaz	107.43 de	1.503 cd	4.97 a-c	10.83 a-d	8.88 a	67.11 a	19.13 de
19Ordu K.	210.44 a	1.576 a-	5.56 a-c	10.70 a-d	10.09 a	64.23 ab	8.53 ıj
Çukurca		d					
20. Kompozit 13	143.05 bc	1.553 a- d	5.89 a-c	10.65 a-d	9.53 a	65.10 ab	20.00 b-e

### **3.1.** Thousand Grain Weight (g)

Differences between genotypes were found to be statistically significant in terms of thousand grain weight (p<0.01). It was determined that the thousand grain weights of local popcorn genotypes varied between 210.44 g - 97.43 g, the highest value was 210.44 g in genotype 19, and the lowest value was in genotype 2 with 97.43 g. Genotype number 19 with the highest thousand grain weight was statistically included in the same group with genotypes 1, 4, 5, 14, 16, and 17, and it was determined that these genotypes differ significantly from other genotypes. Genotype 3 formed a second group with a weight of 148.30 g, and there was a statistically significant difference between other genotypes except for genotypes 7, 8, 9, 11, 12, 13, 15 and 20. While the



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genotype 2, which has the lowest thousand grain weight, did not make a significant difference between the genotypes 6, 8, 10 and 18, and there was a statistical difference between the other genotypes. The thousand-grain weight of Nermin Cin, Ant Cin 98 and Composite 13 local commercial varieties were 136.99, 190.53 g and 143.05 g (Table 1). The thousand-grain weights of some of the local popcorn population showed higher values than the registered local varieties. From the researches made with a thousand grain weight of popcorn as the first crop; Özkaynak and Samancı (2003) found the highest 114.8 g in 15 popcorn lines and 115.2 g in crosses, İdikut et al. (2021) determined the thousand grain weights of popcorn genotypes between 181.06-127.12 g, Marques et al. (2015) reported that it was between 131.43-137.99 g, and Kahramanoğlu (2019) found that between 241.81 g and 149.06 g. In the study conducted as the second crop more variation was recorded than the results obtained by previous researchers. The excess of variation is a genetic gain. This may be due to the large number of varieties used and the fact that they were grown as a second crop.

### **3.2.** Ash (%)

In this study carried out with local popcorn genotypes, ash values were found between 1.730 and 1.483. The highest value was observed in genotype 11, while the lowest value was observed in genotype 8. The ash ratios of Nermin Cin, Ant Cin 98 and Composite 13 local commercial varieties were found to be 1.730, 1.610 and 1.553. The genotype 11 with the highest ash content created a significant difference between the genotype 8 which has the lowest ash value and the genotypes 14, 15, 18.

### **3.3.** Oil (%)

In the study, the oil values in the popcorn grain were found in the range of 6.69 - 4.35%. While genotype 2 has the highest oil value with 6.69%, it is followed by genotype 10 with 6.49%. Genotype 17 took the third place with a rate of 6.43%. It has been reported that the genotypes in the first three ranks are in the same group. It was determined that genotype 8 had the lowest fat ratio with 4.35% and formed a different group. The other 16 genotypes were included in the transitional groups between these two groups (Table 1). Ratkovic and Dumanovic (1993) determined the lowest oil rate in popcorn and the highest rate in standard type in 6 hybrid maize. Idikut et al. (2021) reported that the oil ratio of the local gin corn genotypes used in their research was between 3.003-6.650%. Kahraman (2016) stated that the fat ratio in the second product varied between 3.0-4.6%. Although it is expected that the oil ratios are low in the popcorn genotypes, the variation is also important due to the local population.

### **3.4.** Moisture (%)

The moisture values of the study were found in the range of 10.99 - 10.33. It was observed that the highest moisture value was in genotype 4 (10.99) and the lowest moisture value was in genotype 2 (10.33%). It was noted that the varieties formed two main groups in terms of moisture content and the other varieties were in the transition groups between these two main groups. Storage humidity is expected to be 13-14% in cereals under normal conditions. The results obtained are the expected result, as the low moisture content of popcorn contributes positively to the popping feature.

### **3.5. Protein** (%)

There was no statistically significant difference between genotypes in terms of protein content in grain. In this study carried out with local popcorn varieties, the protein ratio in the grain was found to be in the range of 10.59 -8.88. The genotype with the highest protein content was found as 11, and the genotype with the lowest protein ratio was found as 18. In Nermin Cin, Ant Cin 98 and Composite 13 varieties, the protein ratios were determined as 10.59%, 9.35% and 9.53%. Some of the local popcorn populations were found to have a much higher protein content than registered local varieties (Table 1). Banarjee et al. (2004), in their study in India, the protein content was found to be 9.19% on average, Idikut et al. (2021) found the protein ratio between 8.435-16.650% in their study with local popcorn genotypes and Kahramanoğlu (2019) found the protein ratio between 11.40% and 9.30%. Taş (2020) reported that they found the protein ratios of the second crop of maize to be between 7.67–14.5% in their study which is conducted in Şanlıurfa conditions. It was observed that the findings obtained in previous studies supported the findings of this study. The protein ratio of the corn varieties used for feed or oil is important, the popcorn grain is mostly used as a snack.

### **3.6.** Starch (%)



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When the popcorn genotypes were examined in terms of starch ratio, it was determined that the differences between the genotypes were statistically significant (p<0.01). The starch ratio of the local popcorn genotypes used in the study varied between 67.11 and 61.65%. It was recorded that genotypes 18, 15, 14 had the highest starch ratio with 67.11, 67.10, 66.47 % values, respectively, and they were in the same group. The lowest starch ratio are group between these two groups. Starch ratios were determined as 63.21%, 64.09 and 65.10% for Nermin Cin, Ant Cin 98 and Composite 13. Some of the local popcorn populations have been found to have higher starch values than registered local varieties. (Table 1). Sweley et al. (2012) reported that the starch ratio in grain for the first crop varied between 76.76 - 80.17%. The starch value obtained in the study was below the values obtained by other researchers due to its cultivation as a second product. Variety, precipitation, temperature, soil type and growth conditions sometimes can be more effective than genetic conditions in affecting starch properties in grains (Beckles and Thitisaksakul, 2014; Lu et al., 2015).

### **3.7.** Popping Volume (cm<sup>3</sup> g<sup>-1</sup>)

When the popcorn lines were examined according to the burst volume criterion, the difference between the lines was found to be statistically significant at the level of 1%. In the study, the popping volumes of popcorn varied between 28 and 7.33 cm<sup>3</sup> g<sup>-1</sup>. In the research, Samsun bafra Kasu line had the highest explosion volume with 28 cm3 g-1, while the Ordu Perşembe Kovanlı line had the lowest explosion volume with 7.33 cm<sup>3</sup> g<sup>-1</sup>. Öztürk et al. (2016) reported that they found the popcorn volume to be between 8.31 cm<sup>3</sup> g<sup>-1</sup> at the lowest and 29.30 cm<sup>3</sup> g<sup>-1</sup> at the highest.

### CONCLUSION

As a result, as can be seen from the examined characteristics, it was noted that the local popcorn populations had higher values for some characteristics than the local commercial varieties Nermin Cin, Ant Cin and Composite 13. Obtaining variations in terms of examined traits is important in terms of genetic diversity. Growing as a second crop in the center of Kahramanmaraş, where the Mediterranean climate is dominant, will also contribute to the increase in the cultivation area. Due to the genetic diversity of local genotypes, their high adaptation to the environment in which they are grown, and their being valuable breeding materials, it is necessary to ensure their breeding and continuity. In addition, local varieties have an indispensable genetic material diversity in breeding studies. It is thought that the study with local genotypes is important and will be important in terms of leading the genotype development and breeding studies.

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### **Nutritional Values of Chickpea Hulls**

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### ABSTRACT

Chickpea is an important field crop grown in semi-arid regions and used in human nutrition due to its high protein content. After the chickpea grain is taken, the remaining plant parts are used as animal feed or left in the field. However, there are some macro and micro nutrients in other plant parts that are not used as human food. For this reason, the nutritional values of the remaining chickpea hulls were investigated after the chickpea bean was removed. In the pods of 17 chickpea varieties grown in Kahramanmaraş conditions in 2020; Dry Matter (DM), Protein, Ca (Calcium), Mg (Magnesium), K (Potassium), P (Phosphorus), ADF (Acid Detergent Insoluble Fiber), ADP (Acid Detergent Insoluble Protein), NDF (Neutral Detergent Insoluble Fiber) rates have been examined. The chickpea pod of DM, Protein, Ca, Mg, K, P, ADF, ADP, and NDF were observed 87,71-86,91 %, 8,93-10,83 %, %1.60 - 33 %, 0.17 - 12 %, 0.66 - 0.09, 22-0,17 %, 47,52-42,15 %, 1,05-0,93 %, and 64,28-54,90 %, respectively. According to the nutritional values it was understood that chickpea pod could be used in animal nutrition.

Keywords: Chickpea, nutrition, pod

### **INTRODUCTION**

Legume plants have an important place in human and animal nutrition. Legumes are an important source of vegetable protein and are grown as an alternative protein source in countries with low meat consumption. Chickpea is the most cultivated edible legume in Turkey and ranks second in the world after beans (FAO, 2020; TUIK, 2020). India, which realizes approximately 65% of the world chickpea production, is in the first place in the world in terms of production amount. Australia, Turkey and Pakistan follow India respectively. The annual total production of chickpeas in our country is around 500 thousand tons. Our country, which has an important share in world legume production, is also an exporter country (Sayar and Karatas, 2017). Chickpeas have an important place in human nutrition thanks to 20-25% protein, 40-60% carbohydrate, 4.5-5.5% fat, phosphorus and calcium in their grains (Karaagac et al., 2019). While dry grains with rich protein content are used in human nutrition, post-harvest products such as under-sieve chickpeas, chickpea straw and chickpea hulls have an important place in animal nutrition (Uçar et al., 2020).

The sources where animals meet their roughage needs are meadows and pastures, forage crops, threshing residues, green and succulent feeds. Roughages produced in our country cannot adequately meet the needs of animals Among the reasons for this situation are the insufficiency of natural meadows and pasture areas, low amount of grass per unit area and low forage crop cultivation areas (Güngör et al., 2008).

Chickpeas can be used as a high energy and high protein feed source in animal diets to support milk, meat and egg production (Bampidis and Christodoulou, 2011). There are many studies on the chemical composition of chickpea grain in the literature. Approximately 80% of the grain composition consists of carbohydrates and proteins. It has been noted that chickpeas contain an average of 24% protein, 55% carbohydrate, 4% mineral substance, 6% fat and 10% dietary fiber on dry matter basis (Williams and Sing., 1987). It has been reported that the amount of crude fiber in chickpeas consists of cellulose and hemicellulose, and that the grain varies directly proportional to the hull amount (Javan et al., 1987). It is stated that the hull amount of chickpea grain is in the range of 5-19% (Sing et al., 1980). Considering that chickpeas produced in the world and in Turkey are about 15% of broad bean pods, it will make a significant contribution to the source of animal feed when considered as animal feed. When the hull parts of many legume varieties consumed in the world are examined in terms of both chemical composition and nutritional properties, it is known that the grain has different properties compared to the other parts. In the literature, there are many studies on the functional properties of the chemical composition of the hull parts of various legumes and some important features in terms of nutritional health. However, there is no study examining the chemical composition and nutritional properties



of chickpea hulls.

In this study, it was aimed to investigate the nutritional value of the chickpea shell remaining after the grains of the chickpea plant with high nutritional value and evaluate its usability as animal feed.

### MATERIAL AND METHOD

The experiment was carried out between November 2019 and June 2020 in Kahramanmaraş Sütçü İmam University, Faculty of Agriculture, Field Crops Research area, by establishing three replications and applying the randomized blocks trial design plan. Trial materials in the study were obtained from Research Institutes and commercial companies. The varieties of Aslanbey, Aksu, Çağatay, Seçkin, Hasanbey, İnci, Azkan, Arda, Küsmez, Damla, Cevdetbey, Cantez, Ubet, Gulumser, Borabey, Zuhal, Sezenbey were used in the experiment. Average temperature values in 2019 November, December, 2020 January, February, March, April, May, June, respectively, 13.5, 8.4, 6.3, 6.1, 12.5, 15.9, 15.9, 25.4 0C, average relative humidity 56.2, 81.9, 69.3, 68.3, 67.3, 58.2, 47.2, 46.9% and monthly total precipitation were reported as 39.1, 198.5, 88.0, 72.7, 173.4, 61.8, 18.5, 0.3. According to the analysis results of soil samples taken from a depth of 30 cm, water saturation was 69.96%, pH 7.71, organic matter rate 1.58, lime rate 6.09, salinity 0.05, phosphorus 2.84 kg/da and potassium 55.51 kg/da. The soil structure of the trial area is clay loam, salt-free, medium calcareous, low in organic matter, potassium content above the adequacy level and very low in phosphorus. In the experiment, pure 2 kg/da N and 6 kg/da P (urea and triple Super phosphate) fertilizers were applied to the soil before planting. The sowing was carried out on 26 November 2019 in a randomized block design with 10 rows, 5 m long, three replications, with 50 cm row spacing. Manual weeding was applied twice. When the plants were 15-20 cm, net 3 kg da-1 nitrogen fertilizer was applied. The chickpeas that reached the harvest maturity were handharvested and the chickpea grains were separated from the chickpea hull. The separated chickpea shells are grinded in a grinding device and turned into flour and the Dry Matter (DM), Protein, Ca (Calcium), Mg (Magnesium), K (Potassium), P (Phosphorus), ADF (Insoluble Fiber in Acid Detergent), ADP (Acid Detergent Insoluble Protein), NDF (Neutral Detergent Insoluble Fiber) ratios were investigated. The data obtained at the end of the research were analyzed using the ANOVA method using the SAS statistics program. The averages of statistically significant differences were grouped according to the Duncan multiple comparison test.

### FINDINGS AND DISCUSSION

The average values of the investigated properties and Duncan groups between these averages are given in Table 1 and Table 2. As a result of the statistical analysis, Dry Matter (DM), Protein, Ca (Calcium), Mg (Magnesium), K (Potassium), P (Phosphorus), ADF (Acid Detergent Insoluble Fiber), ADP (Acid Detergent Insoluble Protein), NDF (Insoluble Fiber in Neutral Detergent) ratios were found to be significant.

### 3.1 Dry Matter

The highest dry matter ratio in the bean hull of 17 chickpea varieties grown in Kahramanmaraş conditions was 87.71% and this value was found to belong to the Arda variety. It has been noted that there are no statistically significant differences between Arda variety and Canıtez, Sezenbey, Aslanbey, Borabey, Zuhal varieties, but there is a significant difference between the other varieties in terms of dry matter. Hasanbey variety has the lowest value with 86.91% dry matter. It was noted that there was no statistically significant difference between Hasanbey cultivar and Çağatay, Seçkin, İnci, Ubet cultivars, and there was a significant difference between other cultivars. It has been determined that Aksu, Azkan, Küsmen, Damla, Cevdetbey and Smileer varieties are between 87.00-87.36% values and form the transition groups.

Since studies on the dry matter values of chickpea bean hull were not encountered in previous studies, literature studies with chickpea grains and other legumes were compared with the results obtained. Kaya et al., stated that the dry matter ratio in the grain of local chickpea genotypes ranged from 60.49 to 32.27. Şehu et al. (1999) determined the dry matter values of wheat fodder, rice fodder, barley fodder, oat fodder, chickpea fodder, vetch fodder, lentil fodder, oat fodder and medicago sativa fodder 90.20, 90.23, 93.15, 92.53, 88.65, 90.15, 90.75, 88.37 and 91.67 respectively. It is an expected situation that the dry matter ratio of the bean hull is less than the plant stems.



Table 1: Averages and Groups of Chickpea Cultivars for Protein, Calcium, Magnesium, Potassium, Phosphorus Values

Varieties	Protein**	Ca**	Mg**	K**	P**
Aslanbey	10.356 b-d	1.390 fg	0.153 a-c	0.613 ab	0.200 cd
Aksu	9.363 gh	1.460 с-е	0.140 с-е	0.150 fg	0.170 1
Çağatay	8.933 h	1.473 bc	0.153 a-c	0.183 d-f	0.170 1
Seçkin	9.686 fg	1.346 gh	0.120 f	0.150 fg	0.203 cd
Hasanbey	9.916 d-f	1.500 bc	0.153 a-c	0.160 fg	0.186 e-g
İnci	9.403 g	1.473 bc	0.143 dc	0.093 g	0.193 d-f
Azkan	9.756 e-g	1.393 fg	0.123 ef	0.483 a-c	0.193 d-f
Arda	10.830 a	1.380 f-h	0.170 a	0.353 dc	0.223 a
Küsmen	10.606 ab	1.466 cd	0.163 ab	0.353 dc	0.193 d-f
Damla	10.156 с-е	1.413 d-f	0.133 d-f	0.373 dc	0.196 d-e
Cevdetbey	10.283 b-d	1.523 b	0.156 a-c	0.336 с-е	0.193 d-f
Canıtez	10.573 а-с	1.333 h	0.143 dc	0.480 a-c	0.216 ab
Ubet	10.140 с-е	1.603 a	0.166 a	0.433 bc	0.173 hı
Gülümser	9.630 fg	1.493 bc	0.140 с-е	0.146 g	0.180 g-1
Borabey	9.643 fg	1.416 d-f	0.140 с-е	0.536 abc	0.183 f-h
Zuhal	10.626 ab	1.366 f-h	0.146 b-d	0.663 a	0.210 bc
Sezenbey	9.370 gh	1.496 bc	0.166 a	0.446 bc	0.173 hı

Table 2: Averages And Groups Of Chickpea Varieties For Dry Matter, Fiber Insoluble In Acid Detergent,Protein Insoluble In Acid Detergent, Fiber Insoluble In Neutral Detergent

Varieties	Kuru Madde**	ADF**	ADP**	NDF**
Aslanbey	87.53 а-с	42.15 h	0.933 gh	54.90 g
Aksu	87.36 b-e	47.41 a	1.056 a	64.28 a
Çağatay	87.04 g-1	47.52 a	1.040 a-c	62.49 a-c
Seçkin	87.00 h-1	46.91 a-c	1.053 ab	64.06 ab
Hasanbey	86.91 1	45.38 с-е	1.010 b-e	60.33 dc
İnci	87.06 f-1	47.15 ab	1.026 a-d	64.08 ab
Azkan	87.23 d-h	46.17 a-d	1.026 a-d	61.26 cd
Arda	87.71 a	43.19 f-h	0.946 gh	57.15 e-g
Küsmen	87.31 c-f	44.41 e-g	0.970 e-h	58.54 d-f
Damla	87.29 c-g	45.08 de	1.003 c-f	59.44 de
Cevdetbey	87.28 c-g	44.79 d-f	0.993 d-f	59.38 de
Canıtez	87.60 ab	42.91 gh	1.000 c-f	57.11 e-g
Ubet	87.15 e-1	42.94 gh	0.976 e-g	56.38 fg
Gülümser	87.31 c-f	45.55 b-e	1.033 a-d	61.15 cd
Borabey	87.49 a-c	44.43 d-g	0.936 gh	59.46 de
Zuhal	87.48 a-d	42.16 h	0.930 h	55.28 g
Sezenbey	87.59 ab	45.31 с-е	0.963 f-h	60.39 cd



### 3.2 Protein

The Arda variety had the highest protein value in the bean hull of the 17 chickpea varieties examined in the study with 10.830%. It has been noted that there is no statistical difference between Arda variety and Zuhal, Küsmen, Cantez Damla varieties, but there is a significant difference between the other varieties. Çağatay variety had the lowest protein content with 8.933%. It was determined that Cağatay variety has a significant difference in protein from other varieties except Aksu and Sezenbey varieties. In terms of protein, the Inci variety made a statistically significant difference between the other cultivars except Aksu, Seçkin, Azkan, Düşüner, Borabey, Sezenbey varieties. Aslanbey, Hasanbey, Damla and Ubet varieties did not create statistical difference between them with 9.916-10.356% protein values. There are no literature studies on the protein values of chickpea bean hull. For this reason, studies on protein values in cereals and legumes were examined. In studies on the protein ratio in chickpea hull, the protein ratio was determined 22.37 (Hayıt and Gül, 2019), 25.82-26.83% Ceran (2015), 22.53-23.69 (Carillo et al., 2000), 20.60-26.70 (Kaur and Singh, 2004), 20.50 -23.20 (Tayyar et al., 2008), 21.00-24.00 (Kopaç Kork, 2009), 17.90-22.06 (Bayrak, 2010), 21.99-27.15 (Doğan, 2011), 18.83-20.43% (Erdin & Kulaz, 2014). Türksoy (2018) reported protein values in wheat, chickpeas, green lentils, red lentils, peas and beans as 12%, 19.50%, 20.70%, 21.60%, 24.90%, and 22.50%, respectively. Idikut et al. recorded the protein content of the second crop corn varieties as 7.6-9.6%. In the conducted study, the protein ratio in the bean hull of chickpea was 10,830 - 8,933, when compared to previous studies, it is understood that the bean shell has an important value in terms of feed. In other studies, the amount of calcium in chickpea grain (ppm); It has been reported in the range of 400.00-1600.00 (Wang & Daun, 2004), 1096.00 (Patane, 2006), 878.23-1635.85 (Bayrak, 2010), 887.33ppm-916.88 ppm (Ceran, 2015). Başaran et al. Stated that the Ca ratio in 2017 silage maize varied between 0.32-0.69%.

### 3.3. Calcium

According to the results of the analysis, when the averages of calcium in the chickpea hull were listed, the highest value was 1.603% in the Ubet variety and it was recorded that there was a statistically significant difference from the other varieties. Cevdetbey variety followed the Ubet variety with a Ca value of 1.503%. It was determined that there was no statistically significant difference in Ca between Cevdetbey cultivars and Çağatay, Hasanbey, İnci, gülür, Sezenbey cultivars, but there was a significant difference with other cultivars. With the lowest calcium value in the chickpea hull of 1.333%, the Cantez variety has created a statistically significant difference with the other varieties except Seçkin, Arda, Zuhal. Köse and Mut (2019) found the calcium (Ca) content in the local chickpea grain between 963.1-1908.4 mg kg-1.

### 3.4. Magnesium

According to the data obtained as a result of the study, the variety with the highest amount of magnesium in the chickpea hull was Arda with 0.170, followed by Sezenbey and Ubet varieties with 0.166 values and they were in the same group, except for Aslanbey, Çağatay, Hasanbey, Küsmen, Cevdetbey cultivars, they created statistical differences from other cultivars. While Seçkin variety with the lowest 0.120 magnesium value in chickpea hull did not create statistical difference between Azkan and Damla varieties, it did create significant differences between other varieties. Köse and Mut (2019) found the Mg value in grains of local varieties between 844.2-1267.2 mg/kg. In other studies, the amount of magnesium in chickpea grain was reported as 1942.00 ppm (Ahmad et al., 2002), 1890.00 ppm (Patanae, 2006), 1086.34-1269.02 ppm (Ceran 2015). Başaran et al. reported that the Mg content in corn silage in 2017 varied between 0.25 and 0.33%.



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### 3.5. Potassium

When the potassium values in the chickpea shell were examined, the highest potassium value (0.663) in the hull was obtained from the Zuhal variety. It was followed by the Aslanbey variety with a value of 0.613. The variety with the lowest potassium value in chickpea hull was found to be Inci variety with 0.093. In other studies, conducted on chickpeas, the average potassium values are between 7314.3-9980 ppm (Köse and Mut, 2019) 7168.66-8215.93 ppm range (Ceran,2015), 12360 ppm (Haq et al., 2007), 4698.16-7423.69 ppm range (Bayrak, 2010) was recorded.

### 3.6. Phosphorus

The highest phosphorus ratio in the chickpea shell examined in the study was obtained from Arda variety with 0.223. In terms of phosphorus, it has been noted that the Arda variety does not make a significant difference with the Canitez variety, and is significantly different from other varieties. It was noted that Aksu and Çağatay cultivars had the lowest phosphorous (0.170) in the hull, significantly different from other cultivars except for Ubet, Gülümser and Sezenbey. In studies with chickpea grain, phosphorus amounts are respectively 3246.33-4148.07 ppm (Ceran, 2015), 2460 ppm (Haq et al., 2007), 2257.01-3590.37 ppm (Bayrak, 2010), 1969.9-3705.3 ppm (Köse and Mut., 2019). ) have been reported in the range. Başaran et al., 2017, reported that the phosphorus content in silage corn varied between 0.25-0.29%.

### 3.7. ADF

According to the analysis results, it was noted that the highest ADF rate in the shell was 47.52% and 47.41% in Çağatay and Aksu varieties and it was found to be in the same group, and it was statistically significant difference from other varieties except Seçkin, İnci, Azkan varieties. The lowest ADF value belonged to Aslanbey variety with an average of 42.15%. Except for Arda, Cantez, Ubet, Zuhal cultivars, Aslanbey cultivar showed statistically significant differences from other cultivars in terms of ADF. Other cultivars were included in the transition groups with ADF values of 44.43-45.38. Şehu et al., (1999) were reported that wheat straw, rice straw, barley straw, oat straw, chickpea straw, vetch straw, lentil straw, oat hay and medicago hay ADF values of 51.20, 36.76, 45.23, 39.12, 48.76, 43.35, 46.49, 42.01 and 40.09% respectively. According to roughage quality standards, it is known that the best quality class is below 31% of ADF, the first class is between 31-35% and the lowest roughage value (5th class) is over 45% (Güney 2016). ADF is a feed value that is mostly used to determine the digestibility of a forage by the animal. It is understood that the ADF value of chickpea hull is in the middle in the direction of feed standards.

### 3.8. ADP

It was observed that the highest 1.056% ADP in chickpea pods was found in Aksu cultivar, with the exception of Çağatay, Seçkin, İnci, Azkan, Gülümser cultivars, which made a statistically significant difference from other cultivars. It was determined that Zuhal cultivar with the lowest 0.930% ADP differed significantly from the cultivars except Aslanbey, Arda, Küsmen, Borabey, Sezenbey cultivars. Other varieties were in the transition group with 0.976-1.01% ADP value. Acid detergent insoluble protein (ADP) is a value that expresses the rate of protein that loses its digestibility by binding to cellulose and lignin due to adverse environmental and storage conditions. Basbag et al. (2018) reported that they found the ADP value in the range of 0.08-0.63% in the digestibility analysis of some forage grasses such as bread and durum wheat and rye. Kavak (2019) determined that the ADP values of some astragalus taxonomies in the Southeastern Anatolia Region varied between 0.64-1.40%.

### 3.9. NDF

In the study, the highest NDF value (64.28) in the bean shell of chickpea was obtained from Aksu variety. While Aksu cultivar did not make a significant difference between Çağatay, Seçkin, İnci cultivars, it was determined that there was a significant difference between other cultivars in terms of NDF. The lowest NDF value in the bean hull of chickpea was obtained from Aslanbey (54.90%) and Zuhal (55.28%) cultivars, and these cultivars formed statistically significant differences from other cultivars except Arda, Cantez and Ubet cultivars. Other varieties were in the transition group with ADF values of 61.26-58.53%. Şehu et al. (1999) found NDF values of wheat straw, rice straw, barley straw, oat straw, chickpea straw, vetch straw, lentil straw, oat hay and alfalfa hay to 84.04, 72.08, 85.89, 69.73, 70.24, 65.44, 62.98, 73.60 and 54.36 respectively. According to roughage quality standards, NDF was known to be 40% below the best grade, 40-46% for the first grade, and 65% higher for the lowest roughage (Güney 2016). NDF values are used as an important



criterion in determining the quality of feed. NDF is a feed value used to determine the availability of roughage by animals. As it can be understood from previous studies, it is seen that the NDF value of chickpea peel is at a better level than the stalks used as roughage.

### CONCLUSION

Chickpea plant has an important cultivation area in the world and in Turkey. While the grain is used as human food, the plant remains are used as roughage. For this reason, the nutritional value of the bean shells of 17 chickpea cultivars grown in the city center of Kahramanmaraş, which has a Mediterranean climate, was investigated. Considering the need for animal feed, the protein value in chickpea shell being 8.93-10.83 and being close to ADF, ADP and NDF feed standards will contribute to feed sources. It is thought that the lack of studies on the nutritional value of the bean hull of chickpea will contribute greatly to the literature and future studies.

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# **Development of Special Designed Meatballs Technology**

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### ABSTRACT

Pregnant and breastfeeding women require special nutrition needs. To come to their aid, this study was conducted in order to develop a novel food product. Four variants of pork and turkey meatballs, with lemon balm (*Melissa officinalis* L.) and wild thyme (*Thymus serpyllum* 1.) aqueous extracts were obtained. These extracts were used due to their antioxidant, antibacterial and galactogenic potential, as well as for flavouring purpose. The traditional frying of meatballs, which leads to unhealthy compounds, was replaced with baking (180 °C for 36 minutes) or steam cooking (100°C for 15 minutes).

The obtained meatballs were further analysed for a complete characterisation. The functionality of the plants extracts was proved by flavonoids and total polyphenols content, as well as by the antioxidant activity, determined by DPPH assay. When trans-anethole and estragole standard were used for the FT-IR determination, similar peaks were obtained. The acceptance of the consumers was investigated by sensorial analysis, sustained by instrumental determination of colour and texture. The best antioxidant activity was shown by the meatballs containing lemon balm extract. In these samples the highest amounts of flavonoids were determined, both for pork and turkey meatballs. Contrary, polyphenols registered the greatest concentration in the samples containing wild thyme extract. From the colour point of view, the baked pork meatballs registered the best intensity of red and yellow colour, making them the most appreciated by the consumers. In the same time, steam treatment determined a smoother texture, with an increased juiciness, both for pork and turkey samples.

In conclusion, this study revealed that pork and turkey meat, with added lemon balm and wild thyme aqueous extracts, could be successfully used to obtain meatballs for special destination. All samples presented very good acceptability, but the complexity of this issue is given by the subjective considerations of each consumer.

Keywords: antioxidant activity, consumers acceptability, galactogogue potential, meatballs



# The Development of Melon Sorbets with Acacia or Lavender Syrup

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### ABSTRACT

Sorbet is known since ancient times being considered the ancestor of ice-cream, recognized for its specific structure and texture. These issues could be obtained by using a fine-dispersed comminution of fruits and freezing.

The aim of this paper is to develop a healthy melon sorbet with acacia or lavender syrup addition. For this goal were used three melon varieties (Medena Rosa, Honeydew Green Flesh and Ducral) and two types of syrup obtained from acacia and lavender flowers with brown sugar. The samples were prepared by mixing the melon with the syrup, then the mix was put into an ice-cream machine for 10 minutes until the sorbet was prepared. The samples were stored at -18°C in a freezer for 24 hours. After that, the samples were analyzed by the phytochemicals, overrun, melting down, color, textural, confocal and sensorial analysis. It seems that the lavender syrup has accelerated the melting time (90 min.) reported to the samples with acacia syrup (110 min.). The total carotenoids, lycopene, polyphenols and flavonoids were influenced by the melon variety, the highest values being gained by Honeydew Green Flesh. Similarly, the color measurements (L\*-55.57 and b\*-19.99) and the sensorial attributes analysis determined that this variety was the dominant one. A double penetration test was applied in order to investigate the sorbet texture parameters (firmness, adhesiveness, cohesiveness and springiness). The texture analysis shows that the addition of acacia or lavender syrup did not significantly affect the texture parameters, while the raw material did. Medena Rosa melon variety induced the firmest texture and the lowest cohesiveness. Therefore, the quality of the sorbet samples depends on the melon variety more than on syrup addition.

Keywords: acacia, lavender, melon, sorbet, syrup



# Effects of Different Drying Methods on the Physicochemical and Antioxidant Content of 'Cempedak' (*Artocarpus integer* L.) Powder

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### ABSTRACT

"Cempedak" is an aromatic fruit, which can be converted into powder with a longer shelf life. The effects of different drying methods (spray-drying, freeze-drying, convection oven-drying, and vacuum-drying) on "cempedak" powders produced were evaluated in their physicochemical properties (moisture content, water activity, wettability, flowability, water solubility index, hygroscopicity, and degree of caking). In addition, the color and antioxidant content (carotenoid and DPPH scavenging activity) of "cempedak" powder were determined as well. The process yield of convection oven-dried "cempedak" powders is higher (62.98%) as compared to spray-dried powder (25.08%). On the contrary, the total color change for freeze-dried powder is lesser (12.31) compared to other drying methods. The scavenging activity of freeze-dried powder had the greatest antioxidant effect, which is 52.12% at 50 µg/ml, while the carotenoid content of freeze-dried powder is 15.83 mg/g. Freeze-drying can produce higher quality "cempedak" powder as compared to spray-drying, convection oven-drying.



# Prediction and Qualitative Analysis of Sensory Perceptions over Temporal Vectors Using Combination of Artificial Neural Networks and Fuzzy Logic: Validation on Indian Cheese (Paneer)

**RAWMATERIALSTO PROCESSED FOODS** 

International Conference on

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### ABSTRACT

The present study investigated the feedforward-backpropagation artificial neural network (ANN) architectures to predict the sensory scores at different moisture levels (40%–50%) of paneer. Paneer was produced aseptically and packed in low density polyethylene (LDPE) bags, in a semi-organized commercial dairy plant. Samples were evaluated at regular intervals (8 days) for biochemical content and microbial counts, while subjected every day for sensory evaluation. Three layered (input-hidden-output) ANN was able to produce similar sensory responses using biochemical and microbiological data, for both single (best combination 10-7-1; R2  $\approx$  0.99) and conjugated (10-9-4; R2  $\approx$  0.97) parameters for an extent of 25 sensory output nodes (10-35-25; R2  $\approx$  0.90). The comparison of ANN and predicted sensory scores using linear regression model (with R2 = 0.99) suggests that paneer at 42%–44% and 47%–49% moisture was best for consumption as fresh and for storage. The developed method for predicting and analyzing sensory data of produced paneer opens new possibilities for improving food product's likeness.



# Feasibility of a chromameter and chemometric techniques to discriminate pure and mixed organic and conventional red pepper powders: A pilot study

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### ABSTRACT

Food adulteration is a major problem causing significant economic and health risks for consumers. Nondestructive, quick and inexpensive methods are needed for food authentication. Organic foods have gained importance in last decades with better taste, nutrition and health benefits but higher costs. Research studies on mixing similar type conventional product into organic ones have been very limited. No study has been found on the adulteration of organic pepper powders. This study examined the feasibility of color data (CIE L\*a\*b\*) from a chromameter to discriminate pure and adulterated (mixed; 0-100%) organic sweet red pepper powders obtained from intermittent microwave drying with three power rates (150, 300 and 450 W) for the first time. PCA (principal component analysis), SIMCA (soft independent modelling of class analogy), and PLSR (partial least squares regression) were used for the data analysis. The PCA exhibited a very good distinction based on product type (organic and conventional) and drying powers (150, 300 and 450 W) while the SIMCA effectively classified the samples as organic or conventional with an overall correct classification ratio of 94%. Also, the adulteration rates (%) of the organic pepper powders were predicted using PLSR with promising results (R<sup>2</sup>=0.90; RMSECV=10.9%). To summarize, a chromameter has a good potential to classify the red pepper powders as organic or conventional.

Keywords: Organic pepper powder, spice, adulteration, microwave drying, chromameter



# Effect Of Chia (Salvia hispanica L.) Seed Mucilage on Lipid Oxidation of Reduced-Fat Beef Patties

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### ABSTRACT

In this study the effects of using chia mucilage (CM) and different cooking methods (grilling and frying) on lipid oxidation levels of beef patties were evaluated. For this purpose, four different patty formulations were prepared where beef fat was substituted with CM as C (0% CM), C25 (25% CM), C50 (50% CM), and C75 (75% CM). TBARS and peroxide values of the samples were determined during the storage at 4°C for 7days. Utilization of CM decreased TBARS values in fried samples while it was increased in grilled samples. The effects of different cooking methods on the oxidation level were found to be significant (p<0.05). The control group which was fried (KC) had the highest (0.76±0.04) TBARS values on 7<sup>th</sup> day and beef patty formulated with 25% chia mucilage (KC25) had the lowest  $(0.27\pm0.02)$  on  $3^{rd}$  day of storage. The peroxide values of the fried samples decreased as the amount of chia mucilage increased. Peroxide values generally decreased with the addition of CM in both cooking methods (p<0.05). L\* values of samples increased and a\* values decreased with the addition of CM during storage (p < 0.05). The Hue value of the treatments showed a negative correlation with the a\* value and a positive correlation with the b\* value. Yellowness (b \* values) of the samples increased with the addition of CM due to the color of the chia mucilage. These results indicated that utilization of CM presents the opportunity to prevent oxidation as a natural antioxidant for fried patties. The authors are thankful for the financial support of Ege University Scientific Research Projects Coordination under project number FHD-2021-22370.

		IC	IC25	IC50	IC75	КС	KC25	KC50	KC75
TBA alonaldhyde /kg)	0	0.40±0.06 <sup>bx</sup>	0.55±0.04 <sup>ax</sup>	0.51±0.04 <sup>ax</sup>	0.54±0.09ª	0.29±0.00 <sup>ct</sup>	0.35±0.02 <sup>bcx</sup>	0.32±0.04 <sup>ct</sup>	0.29±0.03 <sup>cy</sup>
	3	0.33±0.02 <sup>cy</sup>	0.31±0.03 <sup>cdy</sup>	0.39±0.02 <sup>by</sup>	0.43±0.06 <sup>ab</sup>	0.40±0.01 <sup>bz</sup>	0.27±0.02 <sup>dy</sup>	0.47±0.02 <sup>ay</sup>	0.29±0.03 <sup>cdy</sup>
	5	0.44±0.00 <sup>cx</sup>	0.35±0.02 <sup>dy</sup>	0.46±0.02 <sup>bcxy</sup>	0.51±0.01ª	0.52±0.02 <sup>ay</sup>	0.38±0.04 <sup>dx</sup>	0.49±0.02 <sup>aby</sup>	0.30±0.03 <sup>ey</sup>
(mgm	7	0.28±0.03 <sup>dy</sup>	0.34±0.02 <sup>cdy</sup>	0.39±0.06 <sup>cy</sup>	0.51±0.05 <sup>b</sup>	0.76±0.04 <sup>ax</sup>	0.40±0.03 <sup>cx</sup>	0.54±0.01 <sup>bx</sup>	0.49±0.06 <sup>bx</sup>
e	0	6.45±0.49 <sup>ax</sup>	4.64±0.57 <sup>cx</sup>	4.43±0.47c <sup>dy</sup>	4.41±0.48 <sup>cdy</sup>	5.56±0.24 <sup>by</sup>	2.99±0.90ez	3.58±0.14 <sup>dez</sup>	4.02±0.37 <sup>cdy</sup>
e Vali 2/kg)	3	4.24±0.48 <sup>by</sup>	2.93±0.98 <sup>cy</sup>	1.96±0.98 <sup>dz</sup>	3.92±0.11 <sup>by</sup>	5.92±0.42 <sup>ay</sup>	3.82±0.03bcz	5.74±0.05 <sup>ay</sup>	5.86±0.08 <sup>ax</sup>
Peroxide Value (meqo2/kg)	5	4.45±0.47 <sup>ey</sup>	4.32±0.56exy	8.77±0.64 <sup>cx</sup>	10.23±0.52bx	15.65±0.33ax	8.82±0.98 <sup>cx</sup>	6.53±0.57 <sup>dx</sup>	5.73±0.13 <sup>dx</sup>
P.	7	6.43±0.52 <sup>cx</sup>	4.23±0.65 <sup>dxy</sup>	3.96±0.99 <sup>dy</sup>	4.65±0.59 <sup>dy</sup>	15.33±0.17 <sup>ax</sup>	7.56±0.06 <sup>by</sup>	5.71±0.06 <sup>cy</sup>	5.69±0.13 <sup>cx</sup>
	0	38.26±0.75 <sup>cz</sup>	41.26±1.34 <sup>by</sup>	42.46±0.30 <sup>aby</sup>	43.32±0.95 <sup>ay</sup>	37.05±1.04 <sup>cz</sup>	34.43±1.69 <sup>dy</sup>	34.10±0.64 <sup>dy</sup>	34.97±0.31 <sup>dy</sup>
L*	3	37.57±1.38 <sup>bz</sup>	41.81±1.38 <sup>ay</sup>	43.53±1.18 <sup>ay</sup>	44.49±0.70 <sup>ay</sup>	38.80±1.09 <sup>by</sup>	42.69±3.42ax	43.94±1.41 <sup>ax</sup>	43.98±0.99 <sup>ax</sup>
	5	43.00±0.62bx	45.68±0.98 <sup>ax</sup>	46.12±0.84 <sup>ax</sup>	46.33±1.26 <sup>ax</sup>	38.20±0.43 <sup>dyz</sup>	40.90±1.64 <sup>cx</sup>	42.34±0.79bcx	42.81±0.93bx
	7	41.62±0.15 <sup>dy</sup>	44.39±0.71bx	45.91±0.68 <sup>ax</sup>	46.14±0.84 <sup>ax</sup>	42.46±0.84 <sup>cdx</sup>	43.45±0.75 <sup>bcx</sup>	43.77±1.35 <sup>bx</sup>	43.88±0.61bx
	0	5.18±0.21 <sup>bcx</sup>	5.06±0.32bcx	4.60±0.13 <sup>dx</sup>	4.82±0.37 <sup>cdx</sup>	4.54±0.24 <sup>d</sup>	5.42±0.34 <sup>abx</sup>	5.65±0.15 <sup>ax</sup>	5.63±0.37ax
a*	3	5.48±0.72 <sup>ax</sup>	4.55±0.33bcxy	4.25±0.53 <sup>bcxy</sup>	3.88±0.42 <sup>cy</sup>	4.67±0.34 <sup>b</sup>	4.21±0.36 <sup>bcy</sup>	4.17±0.22 <sup>bcy</sup>	4.67±0.15 <sup>by</sup>
	5	4.38±0.24 <sup>bcy</sup>	4.35±0.23 <sup>bcy</sup>	3.93±0.19 <sup>cdyz</sup>	3.52±0.03 <sup>dy</sup>	4.63±0.47 <sup>b</sup>	4.60±0.46 <sup>by</sup>	4.36±0.27 <sup>bcy</sup>	5.34±0.19 <sup>ax</sup>
	7	4.82±0.21 <sup>axy</sup>	4.56±0.36 <sup>abxy</sup>	3.48±0.32ez	3.87±0.17 <sup>dy</sup>	4.22±0.13 <sup>cd</sup>	4.30±0.17 <sup>bcy</sup>	4.07±0.07 <sup>cdy</sup>	4.28±0.15 <sup>bcz</sup>
b*	0	6.23±0.18 <sup>cy</sup>	9.32±0.05 <sup>ay</sup>	9.46±0.86 <sup>ax</sup>	9.10±0.53 <sup>az</sup>	5.16±0.32 <sup>dz</sup>	5.85±0.40 <sup>cy</sup>	6.20±0.47 <sup>cz</sup>	7.60±0.32 <sup>by</sup>
	3	9.19±0.43 <sup>bcx</sup>	9.98±0.78 <sup>abx</sup>	9.77±0.34 <sup>abx</sup>	10.30±0.80 <sup>axy</sup>	7.95±0.48 <sup>dy</sup>	8.48±0.50 <sup>cdx</sup>	9.09±0.33bcx	9.13±0.50bcx
	5	8.55±0.69 <sup>cx</sup>	8.56±0.10 <sup>cz</sup>	9.98±0.28 <sup>bx</sup>	11.29±0.93 <sup>ax</sup>	9.84±0.51bx	8.91±0.29 <sup>cx</sup>	9.43±0.60bcx	9.40±0.61 <sup>bcx</sup>
	7	8.57±1.01 <sup>bcx</sup>	7.99±0.23 <sup>cdz</sup>	8.04±0.37 <sup>cdy</sup>	9.59±0.56 <sup>ayz</sup>	7.62±0.54 <sup>dy</sup>	8.35±0.21 <sup>bcdx</sup>	8.24±0.54 <sup>bcdy</sup>	8.98±0.56 <sup>abx</sup>
Hue	,	50.30±1.82 <sup>bc</sup>	61.55±1.44 <sup>a</sup>	63.99±1.50ª	62.02±2.75ª	48.66±2.44°	47.20±2.53°	47.62±2.32°	52.35±1.47 <sup>b</sup>

Keywords: beef patty, chia mucilage, cooking methods, lipid oxidation

xyz:Different superscripts in the same column indicate statistically significant differences (p <0.05).

abcde: Different superscripts in the same row indicate statistically significant differences (p < 0.05)



# FT-NIRS and Chromameter-Based Estimation of Applied Microwave Power of Black Carrot Powders Obtained from Intermittent Microwave Drying

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**RAWMATERIALSTO PROCESSED FOODS** 

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### ABSTRACT

Drying is one of the preservation methods for fruits and vegetables; however, the drying method and drying parameters have significant impacts on the quality of the final product. Intermittent microwave drying (IMWD) is used with significant advantages including better drying rate, shorter drying time, reduced cost, and better final product quality. Applied microwave power (AMWP) is a critical parameter that influences the product quality in IMWD. It is very difficult to assess the AMWP of the powdered product by naked eye. The goal of this work was to appraise the AMWP of black carrot powders by utilizing two electro-optical instruments: FT-NIRS and chromameter. The drying tests were conducted with nine AMWP levels (100-500 W with 50 W increments) with four replications (n=36). NIR reflectance and colour data were analysed using PLSR (partial least square regression). Research findings revealed that the AMWP was a key factor on the colour and NIR reflectance. The NIRS system can predict the AMWP with considerably better performance ( $R^2$ =0.99; SEP=15.3 W) than a chromameter ( $R^2$ =0.79; SEP=57.6 W). In sum, a NIRS system can be utilized to predict the AMWP of black carrot powders with acceptable accuracy better than a chromameter.

Keywords: Black carrot, microwave drying, power rate, colour, NIRS



Aroma Enhancement of Ocimum basilicum L. via Drought Stress Induced by

03-04 June 2021, Turkey

**Polyethylene Glycol (PEG)** 

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### ABSTRACT

Drought is a well-known stress factor that inhibits growth period and productivity and effects the release of secondary metabolites from the plants resulting the aroma enhancement. The present study was aimed to examine the effect of applied drought stress using different rates of PEG 6000 (polyethylene glycol) solution, on morphology, stomatal openness, chemical structure and volatile profile of basil (*Ocimum basilicum* L.). Expectedly the highest shoot and root lengths observed at Control (16.2 cm) and 15% PEG treatments (7.5 cm) respectively. Stomatal openness apparently closed with increasing rate of drought stress and aroma profile of dominant volatiles. Terpenes were found to be the most abundant chemical group and their concentrations altered significantly with PEG treatments. Methyleugenol was detected as the major terpene in control, 5% PEG, 10% PEG samples while linalool was the prominent compound in 15% PEG sample. FTIR spectrum supported the GC results being differentiated significantly particularly in the fingerprint region (below 1400 cm<sup>-1</sup>).

Keywords: Basil, Volatile, Aroma enhancement, Polyethylene glycol, FTIR, SEM



# **Effect of Ultrasound -Assisted Extraction Application on Total Phenolic** Substance, Catechin and Caffein Amounts of Green Tea (Camellie sinensis) Extract

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### ABSTRACT

In this study, it was aimed to investigate the changes in the amount of water soluble dry matter (<sup>o</sup>Briks), total phenolic substance, caffeine and some important catechins of green tea extracts obtained by using ultrasound technique. For this purpose; different tea: water ratios (5: 100; 10: 100) and brewing times (5, 10 and 20 minutes) at 70 ° C were applied and the obtained green tea extracts were treated with tannase enzyme. The water-soluble dry matter content of green tea extracts was determined between 1.30-3.65.6 °Brix. Due to the increase in the brewing time and the tea: water ratio, there is usually an increase in the water soluble dry matter values, the highest 'Briks value (3.65 'Briks) was measured in the extract obtained in 20 minutes brewing at 70 ° C with 10: 100 tea water ratio and applied tannase enzyme. The total phenolic content of the extracts ranged from 2.68-3.87 g GAE / 100 g dry green tea. The highest total phenolic content (3.87 g GAE / 100 g dry green tea) was determined at 10: 100 tea: water ratio, 20 min at 70 ° C and applied tannase extract. The caffeine, EGC, EGCG, EC, ECG amounts of the extracts according to tea: water ratio, brewing time and tannase enzyme usage varied respectively; 609.44-1212.11 mg/L; 42.68-130.13 mg/L; 39.57-145.44 mg/L; 2.42-9.86 mg/L; 5.52-117.66 mg/L. When compared with the control samples, EGCG and ECG amounts of the extracts decreased and the amount of EGC and EC increased depending on the enzyme application.

Keywords: Catechin, Green tea, phenolic substance, tannase



# Effect of Modified Atmosphere Packaging on Quality Parameters of Fresh-cut 'Deveci' Pears

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### ABSTRACT

'Deveci' is a popular local cultivar of pears in Turkey. There is potential for developing fresh-cut pear product from this cultivar but no information on respiration rate and modified atmosphere packaging of fresh-cut 'Deveci'pears exist in literature. Respiration rate of fresh-cut pears (Pyrus communis L. cv 'Deveci') were determined as a function of O<sub>2</sub> and CO<sub>2</sub> concentration at 4°C and the data were fit to Michaelis-Menten type enzyme kinetic models. A modified atmosphere package was designed based on optimum O<sub>2</sub> and CO<sub>2</sub> leves at which aerobic respiration rate was minimum. Fresh-cut pears were dipped in a solution containing 1% citric acid + 1% calcium chloride for 5 minutes to prevent softening and enzymatic browning. They were packaged under 2.15% O<sub>2</sub>+8.70% CO<sub>2</sub> equilibrium atmosphere and stored at 4°C for 8 days. In the equilibrium gas concentration,  $O_2$  consumption rate (RO<sub>2</sub>) and CO<sub>2</sub> production rate (RCO<sub>2</sub>) were calculated as 0.98 ml O<sub>2</sub> kg<sup>-1</sup>  $h^{-1}$  and 1.98 ml CO<sub>2</sub> kg<sup>-1</sup>  $h^{-1}$ , respectively. It was found that the most suitable model for the respiration rates was the no-inhibition model. The K<sub>m</sub> values in the model was calculated as 30.23 and 26.25 for RO<sub>2</sub> and RCO<sub>2</sub>, respectively. The high K<sub>m</sub> values indicated that the respiration rate of fresh-cut 'Deveci' pear slices was low. Packaging and storage time did not have a significant effect on total soluble solid, pH and titratable acidity of the fresh-cut pears throughout storage. The firmness of pear slices was measured as 6.9 N in the control samples, while it was 13.6 N in MA packaged samples at the end of the storage period. Modified atmosphere packaging resulted in a lower total color change, a\*-value, chroma value, and a higher hue angle and L\*-value compared to air-controls during 8 days of cold storage. In conclusion, the modified atmosphere packaging preserved the quality parameters very well during a short term storage period.

Keywords: Fresh-Cut Pear, Modified Atmosphere Packaging, Respiration Rate, Quality



# Migration of Lead and Cadmium from Ceramic Kitchenware and Estimation of Sampling Uncertainty

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RAWMATERIALSTO PROCESSED FOODS

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#### ABSTRACT

The aims of this study were (i) to evaluate the existing analysis capacity for lead and cadmium leached from ceramic articles, (ii) to determine the migration of lead and cadmium from glazed ceramic kitchenware sold on Turkish market, (iii) to estimate the sampling uncertainty. Traditional ceramic cooking ware in brown color (from thirteen sales point) and Chinese breakfast hollowware (in blue, white and pink colors) (from one sale point) were purchased randomly from different sales points located in Bursa in 2018. From each site, six identical samples were taken (totally n=96). Ceramic articles were subjected to the migration conditions specified in the European Union Directive 84/500/EEC, and the amount of lead and cadmium was determined with the inductively coupled plasma and mass spectrometry. It was found that the current analytical method can quantify lower levels of the metals with acceptable method performance parameters. The amount of lead and cadmium that migrated from all ceramic articles were far below the current permissible limits set in the legislation. Relative sampling uncertainty was estimated as 66.2% and this result was thought to be caused by heterogeneity between the articles sampled from the same lot. This study can be regarded as a case surveillance study by providing updated data that can be used by authorities to make risk assessments on consumer health.

Keywords: Ceramic articles, cadmium, heavy metals, lead, migration, sampling uncertainty

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# Characterization of Volatile Composition of Mint and Oregano Obtained from Different Drying Methods

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### ABSTRACT

Folia mentha piperitae and Herba origani tytthanthi are endemic mentha species from the Republic of Uzbekistan. In the present study, volatile compounds of mint and origano samples dried by three methods (water heating infrared convective drying (WHICD), convective drying (CD), infrared drying (IRD)) were investigated using the HS-SPME-GC-MS technique. The volatile components of the samples consist of terpenes, esters, ketones, alcohols, furans, aldehydes, and aromatic hydrocarbons. Terpenes were the predominant aroma group in all the studied samples. Among the drying methods, WHICD provided better results compared to other methods for preserving volatiles. *Herba origani tytthanti* had higher amounts of *p*-cymene, thymol and carvacrol, while menthone, *L*-menthol, limonene, and eucalyptol were found abundantly in *Folia mentha piperitae* samples.

Keywords: Folia mentha piperitae, Herba origani tytthanthi, combined drying, volatile compounds, HS-SPME-GC-MS

### INTRODUCTION

Aromatic plants have been widely used in food, perfumes, pharmaceutical, and natural cosmetic products since ancient times (Baatour et al., 2010; Sonmezdag et al., 2017). The cultivation and export of aromatic plants are constantly increasing because of the bioactive compounds they contain. The Lamiaceae plant family is also referred to as the Labiatae family and is one of the most various and widespread plants in the world in terms of ethnomedicine and its medicinal value based on the volatile oils content (Venkateshappa & Sreenath, 2013).

Mint (*Mentha ssp.*) is one of the most significant members of the Lamiaceae family. This genus has been used for its characteristic aroma throughout human history (Venkateshappa & Sreenath, 2013; Masi et al., 2017). It is widespread mainly in Europe, Asia, South Africa, Australia, and the United States (Almeida et al., 2012; Cirlini et al., 2016). Mentha is widely used in food, cosmetics, and medicines (Silva & Camara, 2013; Bokic et al., 2020). Among the *Mentha* species, pepermint (*Mentha piperita L.*), cornmint (*Mentha arvensis L.*) and spearmint (*Mentha spicata L.*) are commonly used as spices (Díaz-Maroto et al., 2003; Masi et al., 2017).

*Herba origani tytthianthi* is a medicinal plant native to the Republic of Uzbekistan (Ait-Kaddour et al., 2019; Abderrahmane et al., 2019). Origanum is from the Lamiaceae or Labiatae family. Origanum, another important genus of the Lamiaceae family, is used in flavoring foods and traditional medicine due to its pharmacological properties (Ali et al., 2020). Origani tytthanthum is especially used in Central Asian folk medicine in the treatment of gastritis, colitis, bronchitis, and pneumonia (Dzumayev et al., 1999) Origanum species are



commonly found in North Africa, the Eastern Mediterranean, and Siberia (Ali et al., 2020). Because of its composition and properties, both fresh and dried origanum has been used for flavor in fish, meat, vegetables, and wine since the 7th century BC (Altintas et al., 2013; Matlok et al., 2020).

Drying is one of the most common techniques used to increase food stability, to extend shelf life, and to prevent biochemical reactions that could alter the organoleptic characterics. The volatile compounds lost during drying is not only mainly dependent on the drying air temperature and time, but also on the biological properties of the plants (Altaf et al., 2020; Rajkumar et al., 2020; Yildiz et al., 2021). Volatile compounds are the most sensitive ingredients in the food drying process (Sellami et al., 2011).

Volatile compounds are important because they effect food quality and consumer preferences (Yildiz et al., 2021). The volatile extract of aromatic herbs consists of monoterpene hydrocarbons, sesquiterpene hydrocarbons, aliphatic hydrocarbons, aldehydes, ketones, acids, and esters (Mondello et al., 2005; Altaf et al., 2020). The most important of volatile compounds for mentha is carvone, limonene, 1,8-cineole, menthone, menthol and eucalyptol; according to previous study data, the main volatile compounds of origani include thymol and carvacrol (Díaz-Maroto et al., 2003; Altintas et al., 2013; Cirlini et al., 2016; Sonmezdag et al., 2016).

The extant literature contains many studies examining the effects of different drying techniques such as oven, freezing, sun, shade, vacuum, and microwave drying on the characteristic properties of mentha and origano such as color, aroma, antioxidant activity, and total phenolic compounds (Ozer et al., 2018). It is important to determine the correct drying method without losing the medicinal and aromatic properties of the plants. Therefore, this study was conducted to determine the volatile compounds of the fresh and dried mint and oregano samples utilizing headspace solid-phase microextraction gas chromatography-mass spectrometry (HS-SPME/GC-MS). Several drying procedures were applied to commercial and wild herbs including convective drying, infrared drying, and the combined version of these two named water heating infrared convective drying to evaluate the effects of drying on volatiles.

### MATERIALS AND METHODS

### Samples

Mint is cultivated in Uzbekistan and Asian mint grows in almost all regions of the country on the banks of the irrigation ditches and other humid places from the foothills to the middle belt of the mountains. All aerial organs of peppermint contain essential oil: leaves up to 2.75%, inflorescences up to 6%, and stems up to 0.3%. The main components of the oil are menthol 40-70%, menthone 10-25%, pulegon, mentofuran, and esters of menthol and acetic and valeric acids (Mukhamedjanov et al., 2017). The oil is colorless with a yellowish or greenish tint as well as a refreshing, pleasant taste and smell. For medicinal purposes, the aerial part – the grass (leaves) – is collected during the budding or flowering of the plant and then dried.

Oregano grows on rocky slopes and pebbles in the lower and middle belt of the mountains in the Tashkent, Fergana, Samarkand, and Surkhandarya regions of Uzbekistan. The nutritional value of oregano is as follows: calorie content – 24.8 kcal, proteins – 1.5 g, carbohydrates – 5 g, dietary fiber – 0.5 g, organic acids – 0.1 g, water – 90 g, mono- and disaccharides – 4 g, starch - 0.5 g, ash – 1 g. Oregano also contains the following vitamins: A – 0.1 mg, PP – 0.5 mg, B1 (thiamine) – 0.03 mg, B2 (riboflavin) – 0.03 mg, C – 10 mg, and PP (neocene equivalent) – 0.749 mg. The content of macro- and microelements includes: Ca – 40 mg, Mg – 30 mg, Na – 70 mg, K – 260 mg, P – 50 mg, Fe – 0.5 mg, and I - 9 µg (Mukhamedjanov et al., 2017). As a medicinal purpose, the aerial part is collected in the grass during the flowering of the plant and then dried.

To conduct a study on drying mint and oregano, we collected the aerial part of the leaves during the month of



June germinated on the territory of the specialized forestry in the Namangan region of Uzbekistan. Multiple experiments were carried out to investigate the optimal drying method and technology. For each repetition of the experiment, 10 kg of mint and oregano were loaded.

### **Drying methods**

This study used the water heating infrared convective drying, convective drying, and infrared drying methods. The drying methods are explained in detail below.

### Water Heating Infrared Convective Drying (WHICD)

Freshly cut *Folia menthae piperitae* and *Herba origani tytthanthi* were sorted from weeds and other impurities were cleaned. They were cut with a size of 5 cm and folia menthae piperitae was pre-dried in the shade in the open air for one to two hours to reduce initial humidity. It is kept away from direct sunlight to preserve the biologically active material of the samples. Before drying, sliced samples pieces are evenly placed in stainless steel mesh pallets, each with a useful area of 0.8 m<sup>2</sup> and a depth of 5 cm. The drying process of folia menthae piperitae and finely colored *Herba origani tytthanthi* were carried out by supplying a coolant (bottom to top) at a temperature of 45-55 °C for 120-180 and 110-120 min., respectively.

A convective water heater intensifies the technological process of drying plant materials from medicinal herbs, tubers, fruits, and flowers. It has several advantages like saving up to 80-90% of biologically active substances and receiving good quality products with improved presentation and chemical composition. Additionally, this drying technique contributes to energy and resource conservation by not using any flue gases or solid fuel.

#### **Convective Drying (CD)**

Sampling and preprocessing was done in the same way as WHICH. Before drying, sliced samples pieces were evenly placed in stainless steel mesh pallets, each with a useful area of  $0.8 \text{ m}^2$  and a depth of 5 cm. Drying of *Folia menthae piperitae* was carried out by supplying hot air at 90 °C for 280-300 min., while drying of finely colored *Herba origani tytthanthi* was done by supplying a coolant at 90 °C for 260-280 min.

The advantage of this method is its simplicity, the ability to vary the temperature of the dried material. The temperature gradient is directed in the opposite direction to the moisture content gradient, which inhibits the removal of moisture from the material. This feature can be attributed as the disadvantage of the method.

#### Infrared Drying (IRD)

Sampling and preprocessing was done in the same way as WHICH. Before drying, sliced samples pieces were evenly placed in stainless steel mesh pallets, each with a useful area of 0.8 m<sup>2</sup> and a depth of 5 cm. The drying process of *Folia menthae piperitae* and finely colored *Herba origani tytthanthi* were carried out by supplying infrared rays with a temperature of 75 °C for 220-250 and 180-190 min., respectively.

Advantages of the method are the ability to regulate and maintain the temperature inside the product. The disadvantages include the contact of the product with oxygen contained in the air; overheating of the surface due to the temperature gradient; possible formation of a hard crust on the surface; increase in cost due to the use of electricity.

### Volatile Compounds Analysis HS-SPME-Procedure

The volatile fraction of mentha and origani samples were characterized following the protocol of Silva & Camara (2013) with minor modifications. One-gram samples were placed into a 20 mL SPME glass vial. A 2 cm long 50/30  $\mu$ m PDMS/DVB-CAR fiber was utilized for analysis. First, the glass vial was kept in the headspace heater block for 10 min. at 40 °C before incubation. Then, it was extraction for 60 min. at 40 °C.



After extraction, the fibre was inserted into the injector port of the GC–MS system equipped with a quadrupole mass analyser where the metabolites were thermally desorbed in the splitless mode at 250 °C for six min. and transferred directly to the analytical capillary column. Each analysis was done in triplicate.

### **GC–MS analysis conditions**

The identifications and amounts of the volatiles were carried out by using "Agilent 6890N" gas chromatography (Wilmington, Delaware, USA) and the associated mass spectrometer "Agilent 5975B VL MSD" (Wilmington, Delaware, USA). The separation of volatiles was performed using a DB-WAX capillary column ( $60 \text{ m} \times 0.25 \text{ mm} \times 0.25 \text{ µm}$ ). Column temperature was programmed to be set as the following: waiting for three min. to  $60^{\circ}$ C, then increasing by  $2^{\circ}$ C/ min. to  $220^{\circ}$ C, and subsequently increasing by  $3^{\circ}$ C/ min. to  $245^{\circ}$ C and remained constant at this temperature for 20 min.. Helium was utilized as a carrier gas (pure:99.99%, flow rate: 1.5 mL/ min.) with an injector and detector temperature of  $250^{\circ}$ C. Volatile compounds were identified based on the retention index, reference aroma compounds, and mass spectra using a commercial database of spectra (Wiley 6, Flavor 2L, NBS 75 k) (Sonmezdag et al., 2018). These compounds were quantified utilizing the internal standard (2-octanol; 41.5 µg/kg and 2-methylbutylacetate 41.5 µg/kg). Response factors were calculated based on the intensity ratio of each compound to internal standards and ratios of peak areas were corrected by using each volatile's response factor (Sevindik et al., 2020, Kelebek et al., 2015). The means and standard deviations were calculated for the GC analyses in triplicate.

### Statistical data analysis

Data were evaluated by analysis of variance (ANOVA) using SPSS 18.0 (SPSS Inc., Chicago, Illinois, USA). Additionally, XLSTAT software (Addinsoft, New York City, NewYork, USA) was utilized for the principal component analysis (PCA).

### **Result and Discussion**

#### Volatile compounds of the samples

A total of 36 and 48 aroma compounds were determined in the *Herba origani tytthianthi* and *Folia mentha piperitae* samples, respectively. The aroma components of the samples consisted of terpenes, esters, ketones, alcohols, furans, aldehydes, and aromatic hydrocarbons. In all examples, terpenes make up most of the aroma compounds.

L-menthol (40.8-51.19%), menthone (27.89-36.66%), eucalyptol (4.0-6.04%), limonene (1.15-4.15%), piperitone (3.31-4.12%) and carvone (0.83-1.27%) are the predominant compounds in *Folia mentha piperitae*. These molecules give the product characteristic odors such as mint, phenolic, and floral aromas. The results of this study supported previously published data on the content of menthol and menthone in *Mentha* species (Salehi et al., 2018; Bokic et al., 2020). Mokhtarian et al. (2020) found that menthone is the main compound in *Mentha piperita L*. via new solar drying methods. In addition, Hassanpouraghdam and Hassani (2014) in their study investigating the impact of drying methods on volatile compounds of *Mentha pulegium* L. found that menthone has a higher amount in sun drying. In our study, the amount of mentone was found to be higher in the WHICD method compared to other drying methods.

Mint has a different flavor due to the existence of menthol, an alcoholic terpene. This compound is known as the primary aromatic compound and is used in medicine for gastrointestinal ailment (Cirlini et al., 2016). In our example, *L*-menthol was 40.18% in the main product, while the highest value (51.19%) was achieved in the IRD. Also, while the main compound in our sample is *L*-menthol, Mokhtarian et al. (2020) reported that the principal compound in fresh Iranian peppermint sample is isomenthol. However, *L*-menthol has not been detected in other mint studies (Silva & Camara, 2013; Cirlini et al., 2016; Ghani & Rowshan, 2017).



Limonene and eucalyptol are other important aroma compounds in *Folia mentha piperitae*. The amounts of these compounds are higher with the WHICD method compared to other drying methods and are approximately the same as fresh produce. In addition, as determined in previous studies, monoterpenes were detected in small amounts in our samples as follows: Sabinene,  $\beta$ -myrcene,  $\alpha$ -terpinene,  $\gamma$ -terpinene, p-cymene, terpinolene,  $\beta$ -caryophyllene,  $\alpha$ -terpineol, isoborneol, viridiflorol, citronellal, linalool, isopulegone, (*E*)-calamenene, (+)-spathulenol, thymol and carvacrol (Díaz-Maroto et al., 2003; Shah & Mello, 2004; Park et al., 2016).

Ghani & Rowshan, (2017) found the compound (*Z*)-jasmone between 2.8 and 0.8% in their study on the endemic mint (*Mentha mozaffarianii* Jamzad) of Iran. In our study, while this compound was not detected in the source product, it was found in the samples between 0.07 and 0.10% in the WHICD, CD, and IRD drying methods.

Another aroma group determined in mentha samples are alcohols. These compounds are (Z)-3-hexenol, 3-octanol, benzyl alcohol, and phenethyl alcohol. Aldehydes ((E)-2-hexenal, benzaldehyde) were also determined in *Folia mentha piperitae* even in low contents. While (E)-2-hexenal and (Z)-3-hexenol compounds were not detected in the source product in our study, it was found in the WHICD, CD, and IRD drying methods. (E)-2-Hexenal and (Z)-3-hexenol are the compounds responsible for the "fresh green" odor by leaves. They are composed due to a defense mechanism against mechanical damage (Silva & Camara, 2013).

In the present study, the predominant compounds in *Herba origani tytthanthi* are as follows (Table 2): Carvacrol (16.49-24,41%), thymol (13.15-24.07%), menthone (15.39-21.45%), *p*-cymene (5.43-25.30%), L-menthol (6.27-13.51%), eucalyptol (3.04-3.98%), isomenthone (2.71-3.86%),  $\beta$ -caryophyllene (0.98-1.25%), 1-octen-3-ol (0.99-1.72), piperitone (0.62-1.30%), and  $\alpha$ -terpinene (0.32-1.15%).

In previous studies, thymol, carvacrol, and p-cymene were found as the principal compounds of origani samples (Figiel et al., 2010; Tepe et al., 2016; Ozer et al.; 2018). In our study, p-cymene is the dominant compound in the source product and in the sample dried using the WHICD method. Thymol and carvacrol compounds are the predominant compounds in samples dried by the CD and IRD method. Similarly, Yousif et al. (2000) found that concentration of p-cymene in fresh oregano was significantly reduced in all three drying methods.

The aroma of oregano is mainly determined by one of the thymol and carvacrol or both of its compounds (Yousif et al., 2000; Di Cesare et al., 2004). Venskutonis (1997) reported that when thyme (*Thymus vulgaris L.*) and sage (*Salvia officinalis L.*) dried at 60 °C rather than 30 °C, significant decreases were observed in the concentrations of the aroma compounds of these herbs. In addition, Di Cesare et al. (2004) reported that the highest contents of thymol and carvacrol were found in unblanched oregano that was dried at 35°C. Furthermore, Calvo-Irabien et al. (2009) found that carvacrol has a higher rate when drying under the shade in the Mexican oregano. However, the effect of a certain drying technique on the retention of the aroma compound is unpredictable as it varies with the plant (Di Cesare et al., 2004).

As determined in previous studies, some compounds such as 1-octen-3-ol, (*E*)-2-hexenal, L-menthol,  $\beta$ caryophyllene,  $\alpha$ -terpineol, piperitone,  $\alpha$ -terpinene, linalool, limonene, camphene,  $\alpha$ -phellandrene,  $\beta$ -myrcene, carvone, and bisabolene are present in small amounts in our samples (Dzumayev et al., 1999; Yousif et al., 2000; Di Cesare et al., 2004, Figiel et al., 2010).

### CONCLUSIONS

In the present study, the impact of three different drying methods on volatile compounds of dried mentha and



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origani samples originating from Uzbekistan were studied for the first time in this investigation. Very limited work has been available in this context, particularly on the volatile compounds of Uzbekistan's medicinal and aromatic plants. Drying is an effective method for the preservation of these plants. A total of 36 and 48 volatiles were determined in the *Herba origani tytthianthi* and *Folia mentha piperitae* samples, respectively.

The WHICD (water heating infrared convective drying) process provided the shortest drying time with the lowest drying temperature and similar volatile compounds with the source product compared to other methods. In the *Folia mentha piperitae*, the amount of menthone, eucalyptol, and limonene were determined to be approximately the same with the main product by drying with the WHICD method. At the same time, it was determined that the amounts of *p*-cymene, thymol, carvacrol,  $\alpha$ -terpinene and terpinolene obtained by this drying method in H*erba origami tytthanthi* were the same as the source product. According to the findings, the WHICD method resulted in better preservation of volatile compounds followed by the IRD and CD methods. The shorter drying time and lowest drying temperature of the WHICD method makes it a better choice compared to the other two studied drying methods.

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# Elucidation of Retro-and Orthonasal Aroma Differences of Biscuits (*panis biscoctus*) Using Artificial Masticator

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### ABSTRACT

The present study aimed to define the interest in using a mastication simulator to understand the release of aroma compounds of biscuits in the mouth. The development of aroma compounds during artificial, human mastication and without masticated samples of veritable biscuits was determined. Then, the evaluations of aroma compounds in different types of biscuits were studied. A total of 32 compounds were identified in the veritable sample while 36 were identified in both sugar-free and organic samples. The aldehydes and pyrazines, were the main chemical groups in the samples. The number of aroma compounds in the biscuit samples revealed essential changes during mastication and increased about 2,5 times. Additionally, the presence of artificial and human saliva showed a similar effect. They increased the number of volatiles while influencing some volatile groups either by increasing the quantity of ketones or decreasing acids, alcohols, and aldehydes.

Keywords: Biscuits, Orthonasal/retronasal aroma, Artificial mouth, HS-SPME, GC-MS

#### **INTRODUCTION**

Biscuits has been a traditional and an important food across the world contributing to food consumption. The word 'biscuit' derives from *panis biscoctus* that has the Latin meaning 'twice cooked' and refers to hard dry bread produced to be stored and consumed at sea. The roots of the biscuit can be traced back as long ago as the Romans. Early biscuits were made from just flour, water, and salt and were not tasty as a food source (Manley, 2000). However today, modern biscuits have developed both in terms of the types, tastes, and the times of day we consume them. Looking at the markets, the product emerges as a staple food, snacks, luxury gifts, dietary products, infant food, as well as dog and cat food. Also, health professionals recommend that they be eaten in small quantities as part of various diets such as infant nutrition, high energy diets, food avoidance for religious beliefs, and vegetarianism or veganism (Smith, 2007; Purlis, 2010).

Like many bakery products, biscuits are high in fats and carbohydrates. These properties contribute to improvement of its technological and organoleptic character. In the luxury or snack food industry, customers must really like the flavors and textures otherwise they will turn to other food products (Smith, 2007). The aroma of biscuits originates from different resources and reactions. One of them is the ingredients of the product. The others come from lipid oxidation, fermentation and cooking (Manley, 2000; Pasqualone et al., 2015). It is worth noting that the main aromas are related to the Maillard reaction between amino acids and reducing sugars (Poinot, Arvisenet, Grua-Priol, Fillonneau, & Prost, 2009; Pasqualone et al., 2015).

A complex mixture of chemical groups is mainly responsible in bakery products such as those found in biscuits, breads, crackers, and cakes that consist of pyrazines, pyrroles, ketones, lactones, furans, aldehydes, alcohols, esters, and acids (Purlis, 2010; Pasqualone et al., 2015; Poinot, Arvisenet, Grua-



Priol, Fillonneau, & Prost, 2009; Dury-Brun, Fournier, Pernin, & Guichard, 2007; Rega, Guerard, Delarue, Maire, & Giampaoli, 2009) . However, the release of aroma compounds through the consumption of food depends on volatile composition, structure, and in-mouth phenomena. The first perception of food intake occurs through inhalation when sniffing as odorants enter the nasal cavity through the external nares (orthonasal aroma) (Van Ruth, & Roozen, 2000; Genovese, Piombino, Gambuti, & Moio, 2009). Food aromas are perceived during consumption when the odorants interact with receptors by traveling from the mouth to the nasal cavity via the nasopharynx and the lungs (retronasal aroma) (Ruijschop, Boelrijk, Burgering, de Graaf, & Westerterp-Plantenga, 2009; Welge-Lüssen, Husner, & Wolfensberger, 2009). The sensation of the retronasal aroma is different from the orthonasal aroma, even though they involve the same mechanisms. These orthonasal/retronasal differences are specific to each odorant or subject and the differences are due to the act of mastication (Linforth, Martin, Carey, Davidson, & Taylor, 2002).

The mechanisms of mastication are complex and link both the orofacial muscles and the tongue. The processes are an important part of the intake of food solids and can be considered as a pre-digestion step. During mastication, food can be subjected to substantial changes and generally the surface area of the food increases, interacts with saliva, and is exposed to air in the mouth, thus enhances the release of the flavors (Burdach, & Doty, 1987; Haring, 1990; Van Ruth, Roozen, Cozijnsen, 1994).

Some instrumental methods have been developed to study volatile compounds released from food during consumption and are called either "artificial mouths" or "mastication simulators". This equipment is considered a useful way to determine individual key odorants present in samples(Arvisenet et al., 2008; Roberts, & Acree, 1995, Van Ruth, & Buhr, 2004). Different model mouth systems have been invented and used by several authors to study liquid (Van Ruth, Buhr, 2004) or viscous (Odake, Roozen, Burger, 2000) model products and also 'semi-solid' natural food stuffs such as vegetables (Van Ruth, & Roozen 2000; Farneti et al., 2013; Van Ruth, Roozen, & Cozijnsen, 1995;) and bakery products (Poinot, Arvisenet, Grua-Priol, Fillonneau, & Prost, 2009; Onishi, Inoue, Araki, Iwabuchi, & Sagara, 2012). Finally, to the best of our knowledge, no data is present in the extant literature about the effect of saliva on the release of aromas on dry and starch-rich food such as biscuits using mastication simulators.

Therefore the main objectives of the present article were: i) to determine the conditions of the human mouth while mastication of biscuit and duplicate it with an artificial mouth device, ii) to define the flavor development during mastication in the artificial and human mouth to verify if the artificial mouth reflects human mouth conditions iii) to characterize and compare the volatile compounds in the different types of biscuits Furthermore, the HS-SPME method to obtain an aroma extract was optimized.

#### MATERIALS AND METHODS

**Biscuit samples.** Three types of commercial biscuits (veritable/VR, sugar-free/SF, and organic/OR) produced by different companies were purchased in Nantes/France. The biscuits were the most preferred type of 'petit beurre' by customers according to Rannou et al. (2017). Ingredients in the VR were wheat flour 73.4%, sugar, butter 13.6%, skimmed milk powder 1.3%, salt, baking powder, oleic sunflower oil, and acid corrector; in the SF were wheat flour 69%, sweetener, butter, sunflower oil, wheat starch, baking powders, egg yolks, salt, and semi-skimmed milk; and in the OR: whole wheat flour 78%, refined cane sugar, oleic sunflower oil 8.5%, raising powder, and rosemary extract. From each type, several samples were purchased, and they were randomly selected for the experiments performed. The samples were coded with three-digit codes.

**Chemicals.** Artificial saliva was prepared according to van Ruth et al. (2010) by dissolving in 500 mL of water (purified by a Milli-Q system, Millipore Corp., Molsheim, France), 2,604 g of NaHCO<sub>3</sub> (Merck, Darmstadt, Germany), 0,4385 g of NaCl (Fluka, Steinheim, Germany), 0,2385 g of KCl (Merck, Darmstadt, Germany), 0,2205 g of CaCl<sub>2</sub>, 2H<sub>2</sub>O (Merck, Darmstadt, Germany), 0,6845 g of K<sub>2</sub>HPO<sub>4</sub>, 3H<sub>2</sub>O (Panreac, Barcelona, Spain), 1,08 g of mucin (Sigma, Saint Louis, USA), and 6,25 g of porcin 50000 units  $\alpha$ -amylase



(Sigma, Saint Louis, USA) (pH adjusted to 7) (Van Ruth, Roozen, & Cozijnsen, 1995). NaN<sub>3</sub> (Sigma, Saint Louis, USA) was also added at 0.5%. This preservative allowed the use of a single solution of artificial saliva for each part of the study. The standards of aroma compounds were purchased from Sigma-Aldrich (Steinheim, Germany, St Louis, MO, USA). All chemicals and solvents used in this study were of analytical and chromatography grade purity.

In vivo analysis. *Quantity of biscuit*. The procedure of the in vivo analyses were performed according to Arvisenet et al. (2008). Each measurement was carried out in triplicate for each subject, and a ratio was calculated between the average amount of water (*i.e.* the volume of the mouth) and the average weight of the biscuit pieces. This ratio was then used to determine the biscuit quantity to introduce into the artificial mouth container for each experiment.

*Biscuit/saliva ratio*. Saliva quantity was determined as follows: saliva weight= total weight - biscuit weight - rinsing water weight.

*Collection of bolus.* Six people (two males and four females aged between 22 and 50) were asked to eat a piece of biscuit, chew it ordinarily, and then spit out the bolus at the point when swallowing would normally have been triggered. The experiment was carried out 15 minutes before analysis was undertaken.

**In vitro analysis.** The artificial mouth comprised a sample container (600 mL), a notched plunger and variable-speed motors which controlled precisely the speed of compression and the rotation movements. The notched plunger comprised 20 lines of sharp teeth, 3 mm in height. The container was maintained at  $37^{\circ}$ C. While food was broken down, N<sub>2</sub> flowed through the device at a sequential rate to reproduce the phenomenon of breathing. The gas flowed in at 0.2 L s<sup>-1</sup> and stopped for a few seconds at regular intervals. To respect the weight of biscuit / mouth volume measured in the humans, 20 g of biscuits were placed into the container. In the human mouth, the average ratio of biscuits to artificial saliva had previously been found to be 2/1. Therefore, 15 mL of artificial saliva was applied to the biscuits pieces in the container through the artificial masticator. At the end of the mastication, 5 ml of the artificial saliva was taken back to replicate the swallowing phenomenon.

**Image analysis.** Analyses were performed to check the similarity of the bolus after both experiments in the human and the artificial mouth. The image of the resulting biscuits from the imaging system was obtained with a CCD camera (Canon G10 with 6,1-30,5mm, 1:2 8-4,5 optic lens) equipped with Rundock (Cleaver Scientific). The experiment was carried out according to Arvisenet et al. (2008).

**Extraction of aroma compounds.** Headspace solid phase micro-extraction (HS-SPME) was used to extract volatile compounds from the biscuit samples. A 660 mg sample was weighed in a 20 mL vial. Volatile compounds were then extracted on a CAR/PDMS SPME fiber (10 mm long, 75 µm film thickness; Supelco, Bellefonte, PA, USA) and placed in the headspace of the vial for 30 min at 37 °C. Five aroma compounds were used to assess the optimization ofhe HS-SPME method and to check the ability of repeating the method. They were chosen because they belong to different chemical groups. They are presented in Table 1. For optimization, all extractions were carried out in duplicate using the VR sample masticated in the simulator with saliva.

To evaluate the development of the aroma through mastication, five different implementations were carried out prior to the extraction of the aroma using the VR sample. At first, the aroma was extracted from the whole sample uncrushed (UN), secondly, the crushed sample was extracting using a mortar (CRH), thirdly, the sample was artificially masticated in the presence of water (W), fourthly, the sample was artificially masticated in the presence of artificial saliva (SAL), and finally, the human masticated sample was used for extraction (HM).

**GC-FID, GC-MS analyses of aroma compounds.** HS-SPME extracts of the biscuits were analyzed by gas chromatography (GC; Agilent Technologies 7890N, Wilmington, DE, USA) coupled with a mass spectrometer (MS; Agilent Technologies, 5973 Network, Wilmington, DE, USA) and a flame ionization detector (FID). Volatile compounds were desorbed into the injection port of the chromatograph (splitless mode for 6 min at a temperature of 230 °C) and separated on a DB-Wax column (length 30 m, internal diameter 0.25



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mm, film thickness 0.5  $\mu$ m). Helium was used as the carrier gas at constant pressure (124 kPa). The oven temperature was programmed for 35 (5 min) at 50 °C at 5°C·min<sup>-1</sup>, then for 50 at 210 °C (5 min) at 5°C·min<sup>-1</sup>. Effluent from the end of the GC column was split 1:2 between the MS and the FID, Peak areas were integrated using MSD Chemstation software (Agilent Technologies). Mass spectra were recorded in electron impact mode (70 eV) between a mass range of 33 to 300 m/z at a scan rate of 2.7 scan·s<sup>-1</sup>.

**Statistical analyses.** The data of the present study was subjected to analysis of variance and principal component analysis using the XLStat for Windows (Addinsoft, New York, New York, USA).

#### **RESULTS AND DISCUSSION**

In vivo and image analysis. The mean ratio measured between mouth volume and biscuit weight was 0.033 mg mL<sup>-1</sup> while between saliva and masticated biscuit mass it was 0.5 according to the in vivo analysis. The analysis is essential to obtain the representative masticated matrix with similar characteristics to those of human mastication (Burdach, & Doty 1987). As for image analysis, the samples used were those obtained after in vitro and artificial mouth mastication. Triplicate images (512x512 pixels and 5x5 cm) were acquired for each sample. Then the images were converted to gray scale. One of the most appropriate methods, grey level occurrence matrix was used to evaluate the textural descriptors; the energy, entropy, and contrast (Arvisenet et al., 2008). According to the results obtained, no statistical differences were found among the samples for the descriptors (p<0.05).

**Optimization of HS-SPME condition.** HS-SPME conditions were determined based on literature with some modification (Pasqualone et al., 2014). The *Y*-axis was given as relative peak area based on sample amount. The extraction temperature was set to 37°C during the entire experiment to avoid degradation in substances by saliva and to mimic the temperature of the mouth. An increase in the peak areas with extraction time was detected. An extraction timeof 30 minutes was set to avoid any sample changes during the testing time under the presence of saliva (Lecanu, Ducruet, Jouquand, Gratadoux, & Feigenbaum, 2002). After the extraction time was fixed, the amount of the extracted sample was evaluated. As could be observed the peak areas steadily decreased with the increasing sample amount. The best relative area/sample ratio was obtained with the extraction carried out with the 1g sample. On the basis of the results, the optimal condition was set to 1g of sample with 30 minutes of extraction time at 37°C. The extraction conditions showed that the method allowed volatile extraction with acceptable repeatability.

#### Volatile compounds of biscuits.

Retro-and orthonasal aroma composition of VR sample. The aroma analysis of the biscuit samples by HS-SPME using GC-MS and profound research of the chromatograms resulted in standard and tentative identification of numerous aroma substances. To define the development of the aroma through artificial and human mastication, a VR sample was subjected to five different implementations (UN, CRH, W, SAL, and HM) prior to extraction. The evaluation made without mastication (UN, CRH) would be closer to the orthonasal aroma and masticated samples (W, SAL, and HM) would be represented as the retronasal aroma. With the artificial masticator, it is possible to evaluate the retronasal aroma in an orthonasal manner (Arvisenet, Billy, Royer, & Prost, 2006). A total of 41 compounds were identified in the VR sample. The release of the aroma compounds differed quantitatively and qualitatively with implementations. Previous studies have also exposed the effect of crushing and mastication on aroma release (Van Ruth, & Buhr, 2004). After thefficiency of the artificial mastication was determined, the biscuits samples obtained using the artificial masticator with saliva were analyzed. A total of 32 compounds were determined in the VR while 36 compounds were found in both SF and OR samples. The volatiles were categorized according to their chemical structure: acids, alcohols, aldehydes, benzene, ester, furans, hydrocarbons, ketones, lactones, phenol, pyrazines, pyrrole, and terpenes. The Maillard reaction contributed several volatile compounds: Strecker aldehydes, furan compounds, and pyrazines. All the chemical classes showed statistical differences (p<0.05).



With regard to the effect of the implementation, to assess the possibility of differentiating the mastication and crushing process taking into account the aromatic profile, we applied a multivariate statistical analysis using the individual percentage of aroma compounds in each of the chemical groups. The variables were designated for the PCAs and the clarified variance was 83,85% (factor 1, 64,27%, factor 2, 19,58%). The application of the PCA algorithms provided two distinct groups in the sample. The first group represented the result of the UN and CRH sample and the second group was characterized by W, HM, and SAL. The first group positively associated with PC1 (hydrocarbons, acids, lactones, and alcohols), whereas the second was associated with PC2 (aldehydes, furans, pyrroles, pyrazines, and ketones) in the VR sample. When the implementations were individually checked, only HM and SAL appeared to be located in the same quadrant and very close to each other. The result showed that human mastication was successfully duplicated by the artificial mastication with saliva.

The profiles of the CRH and UN sample were very similar although the raw data showed the expected trend of more volatiles from CRH. It appeared to affect only the amount of volatiles released not the pattern of the profile. The number of detected compounds was seven and that increased to 13 with the crushing implementation. Despite the increase in the number of the sample, normalized profiles showed less variation within the chemical groups. Acids, alcohols, aldehydes and lactones were the main chemical groups and consisted of 99% of the total area of the compounds in both the UN and CRH sample. The increase in the surface number of the sample was due to the increase in the area of the samples (Ingham, Linforth, & Taylor 1995). The cellular structure may also have been destroyed as a result of the crushing conditions releasing even more volatiles (Arvisenet, Billy, Rover, & Prost 2006).

The number of aroma compounds in the sample revealed essential changes during mastication and increased about 2.5 times. The breakdown of the food matrix during mastication enhances flavor release as well as retronasal odor perception.<sup>14-16</sup> Additionally, the retronasal aroma of some food while in the mouth has been determined to be very different from the orthonasal perception (Baldy, 1995). Also, as opposed to liquids, dilution and hydration are significant aspects of the retronasal flavor release from low moisture foods (Clawson, Linforth, Ingham, & Taylor 1996). The result showed that the addition of water and the associated change in the physical form of the biscuit did affect the release of volatiles. The polarity of the aroma compounds may explain this formation; the least of the polar compounds were released from the water first and the volatiles with the highest polarity were released last resulting in an increase in non-polar compounds in the composition and a decrease in polar aroma compounds (Ingham, Linforth, & Taylor 1995). However, SAL and HM had similar and greater composition than the W sample. Van Ruth et al. (2000) depicted that the alteration of artificial saliva composition regarding replacement of human  $\alpha$ amylase by porcine  $\alpha$ -amylase did not alter the aroma release significantly. The presence of artificial and human saliva increased the number of the volatiles and influenced volatile groups either by increasing their quantity (ketones) or decreasing it (acids, alcohols, and aldehydes). As a mucus, proteinaceous and enzymatic solution, saliva could also transform aroma by emulsification or by breaking down starch or esters via the action of mucin and  $\alpha$ -amylase (Hussein, Kachikian, & Pidel 1983). These enzymatic reactions accompanied by oxidation may be accelerated when mastication mixes parts of the food and combines in the presence of air. The influencing effect would be much better for biscuits that had to be broken up to allow the release of the flavor compound (Roberts, Acree, 1995). However, the decrease could be due to the fact that  $\alpha$ -amylase can affect aroma release as protein as well as having enzymatic effects. Many studies have shown that proteins diminish aroma release (Le Thanh, Thibeaudeau, Thibaut, Voilley, 1992; O'Keefe, & Wilson, 1991). Additionally, in the research investigating the saliva flow effect on the release of aroma, it has been shown that the level of aldehyde content decreases with an increasing saliva flow (Harrison, 1998). Thus, in the present study, the decrease in the aldehyde content could be due to contact between the sample and saliva during the extraction period. Poinot et al. (2009) examined the aroma compounds of bread samples using an artificial mouth system. The researchers determined that additions of saliva cause changes in the aroma profile of the sample. Similar to our research, the acetic acid and ethyl alcohol contents were reduced by the presence of



saliva. In addition, no significant differences was observed in pyrazines with the addition of saliva, similar to Lasekan (2013). The author determined that after incubation of the samples with saliva there had been no change in the amount of pyrazines.

Aroma composition of three types of biscuits. The VR sample is a classic 'petit beurre' biscuit. A total of 32 compounds were identified in the sample. Of all aroma compounds detected in the VR sample, the main group both qualitatively and constitutively were the aldehydes. The compounds have low odor threshold values, therefore, they have a potential significant effect on the aroma characterization of biscuits (Pasqualone et al 2015). A total of eight aldehyde compounds were detected representing 40% of all the aroma compounds. The main formation pathways of aldehydes are lipid oxidation and the Maillard reaction. The reaction mainly occurs during the baking of biscuits. Many volatile compounds are formed during the reaction; pyrazine, furans, pyrroles, and aldehydes. Strecker degradation occurring during the Maillard reaction allows the formation of volatile aldehydes from amino acids. Strecker aldehydes arise from isoleucine, leucine, and phenylalanine (Pasqualone et al., 2014). Isovaleraldehyde, 2-methylbutanal and furfural were the main aldehyde compounds but hexanal was also the other important compound in biscuits. Hexanal is generated at ambient temperature from oxidation of linoleic acid in biscuits (Cognat, Shepherd, Verrall, Stewart, 2014) However, it is also the most abundant volatile found in the rancid profile of bakery products (Purcaro, Moret, & Conte 2008). Pyrazines quantitatively were the other important The compounds mainly odors chemical group. arising from cooking, roasting, and burning (Schieberle, & Grosch.<sup>39</sup> A total of nine pyrazine compounds were detected. Methyl pyrazine was the featured compound followed by pyrazine and ethyl pyrazine. The compounds constituted 24% of the total aroma compounds from the sample. With six compounds, ketones were the other chemical group. These compounds are generally responsible for the perception of off-odors and accumulate with the peroxidation of unsaturated fatty acids to form intermediate hydroperoxides from morestable secondary products that are subsequently derived (Heiniö, Oksman-Caldentey, Latva-Kala, Lehtinen, & Poutanen 2001).

The sugar-free biscuit sample is the other type of 'petit beurre'. A total of 36 aroma compounds were detected in the SF sample. Different from the VR, the ratio of the pyrazine compounds (17%) was less than the other samples in SF. Regarding its recipe, the SF sample has a lower sugar content than the other sample leading to a lesser amount of the precursor for the Maillard reaction. All the chemical groups detected in the VR sample were also identified in SF, however some new compounds were identified in SF such as ester, terpene, and phenol. Concerning ester, only one compound was detected: the acetic acid ethyl ester. The primary formation pathway of the compound is Fischer esterification. Within this reaction, ethanol and acetic acid react to generate the compound. It can be obtained at room temperature but increasing temperature also increases the yield of formation (Schieberle, 2005; Abuilaiwi, A.; Laoui, Al-Harthi, Atieh, 2010). When the ingredients of the SF sample were checked, it was not possible to detect any of the precursors. It was thought that the compounds reacted to generate the formation of acetic acid ethyl ester and cleavages. The terpene compound, dL-limonene, was detected in the sample. The compound is not one of the typical volatiles of biscuits. The compound is a monoterpene with a citrusy odor and is a major component of the oil extracted from citrus peels. The compounds are widely used in the food industry to give the product its aroma (Selli, & Kelebek, 2011).

The OR sample was the last type studied of the 'petit beurre' group. A total of 36 compounds were detected in the sample. Among all the aroma compounds, aldehydes were the main chemical groups. Nine aldehydes were detected and hexanal was overwhelmingly the main compound (20%). In the OR sample, the ratio of the aldehydes (45%) was the highest among the samples. It could be due to the fact that only sunflower oil was used in the OR samples, whereas a mix of mostly butter and sunflower oils were used as ingredients in the VR and SF. Oils could be the main source of the aldehydes due to the high content of the oleic and linoleic acid contents. Decomposition of hydroperoxides produced by lipoxygenase-catalysed oxidation of unsaturated fatty acids is known to be a major pathway for aldehydes (Smith, King, & Min, 2007; Wardlaw, & Snook, 1990). Pyrazines were the other main chemical groups. A total of 10 pyrazines were detected comprising about



21% of the sample. The ratio of the pyrazines was also higher than in the other samples. The used wheat bran in the sample is about seven-times richer in proteins than wheat flour (Peterson, Johnson, Mattern 1986). As previously mentioned, the substitute is one of the main precursors of the pyrazines (Schieberle 2005). It was thought that this could be the reason for the increase in the quantity of pyrazines. Additionally,  $\alpha$ -pinene compound was identified only in the OR sample. The compound is a monoterpene and the rosemary herb (*Rosmarinus officinalis*), one of the ingredients in the sample, rich in this moneterpene (Szumny, Figiel, Gutiérrez-Ortíz, & Carbonell-Barrachina 2010).

#### CONCLUSION

The results obtained in this study showed that the overall aroma compositions of the three types of 'petit beurre' were similar to each other but differences were due to the ingredients that were used. The products of the Maillard reaction, especially aldehydes and pyrazines, were the main chemical compounds in the samples. The number of aroma compounds in the biscuit samples revealed essential changes during mastication and increased about 2.5 times. Additionally, the result showed that human mastication was successfully duplicated by the artificial masticator using saliva, and they increased the number of volatiles while influencing some of the volatile groups either by increasing their quantity (ketones) or decreasing it (acids, alcohols, and aldehydes). Thus, the artificial mouth would be particularly useful to study whether there is a difference between orthonasal and retronasal perception of dry and starch-rich foods.

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# Investigation of Fatty Acid Composition Including Trans Fatty Acids and Erucic Acid in Selected Salty Snack Foods

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#### ABSTRACT



The objectives of the present study were to determine and evaluate fatty acid composition of a selection of salty snacks present on markets in Turkey with a reliable chromatographic method; to evaluate trans fatty acid (TFA) and erucic acid contents in terms of food safety, and to check "TFA-free" statement on the label with current legislation. Based on the method verification study, the performance criteria of the analytical methods used was found acceptable. Even though the highest level of total fat was found in the potato chips, puff and popcorn category, the saturated fatty acids were higher (p < 0.05) in crackers category. Palmitic acid was predominant for cracker and grissini groups, whereas oleic acid showed higher prevalence for the last group. None of the sample prevailed food safety risk regarding erucic acid and TFA. Moreover, the declaration on the label for TFA was verified with the analytical results.

Keywords: Fatty acid composition, GC-FID, trans fatty acid, snack foods



### The Use of Arugula and Barberry Extracts as Nitrite Alternative in Heat-treated Fermented Sausages

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#### ABSTRACT

The aim of this study was to evaluate the effect of arugula and barberry extracts as nitrite source on color parameters, nitrosomyoglobin, total pigment, residual nitrite and nitrate content in heat treated fermented sausages. For this purpose, heat treated fermented sausages were formulated with 150 ppm nitrite (N), 2.73 g/kg arugula extract (AE) or 2.73 g/kg arugula extract plus 4.83g/kg barberry extract (ABE). Control sample without nitrite or extracts was also prepared (C). Thereby, five different formulations were prepared with three replications. ABE samples had the highest nitrosomyoglobin content followed by N and AE. Total pigment contents recorded as 109.03, 152.23, 122.80 and 139.65 for C, N, AE, and ABE treatments, respectively. While the lightness values of the C, N and AE samples increased in the third month, L\* values ABE samples decreased. L\* values of samples ranged between 34.52-41.81 throughout 3 months of storage and storage had an increasing effect on L\* values. It was observed that the L\* values C and N treatments increased in the third month of storage. On day 0 and at the end of the storage the highest a\* value was measured in N treatment (Table 1). The lowest a\* values were recorded in C samples formulated without nitrite or nitrite alternatives. No significant changes were observed in a\* values of ABE during storage. Yellowness value (b\*) of N treatment was lower than other counterparts. N treatment has the highest residual nitrite content initially as expected. Residual nitrate concentration of samples was highest in ABE samples and the lowest in C samples. As conclusion both arugula and barberry extracts exhibit an opportunity to use as nitrite alternative, however, addition of pre-conversion nitrate in arugula extract to nitrite should be studied. The authors are thankful for the financial support of Ege University Scientific Research Projects Coordination under project number FGA-2020-22148.

#### Keywords: Arugula, barberry, nitrite alternative, fermented sausage

			Sam	ples	
		С	N	AE	ABE
Nitrozomy	oglobin (ppm)	13.24 ±1.29 <sup>d</sup>	53.83 ±2.14 <sup>a</sup>	32.76 ±1.86°	80.12 ±0.80
Total pig	ment (ppm)	109.03 ±1.55 <sup>d</sup>	152.23 ±1.11ª	122.80 ±2.44°	139.65 ±3.43
Residual 1	nitrite (ppm)	8.45±0.13 <sup>b</sup>	22.59±1.09ª	9.5±1.15 <sup>b</sup>	8.59±0.01 <sup>b</sup>
Residual r	itrate (ppm)	15.16±0.72 <sup>d</sup>	30.76±1.04°	88.91±1.92 <sup>b</sup>	100.02±0.8
L*	0	34.52±0.90 <sup>b,t</sup>	33.10±0.63c,y	37.30±0.10 <sup>a,z</sup>	37.73±0.67
	1 <sup>st</sup> month	39.93±0.41 <sup>b,y</sup>	36.36±1.38 <sup>c,x</sup>	44.01±0.88 <sup>a,x</sup>	36.34±0.84°
	2 <sup>nd</sup> month	36.47±0.16 <sup>b,z</sup>	38.48±1.07 <sup>ab,x</sup>	40.71±1.29 <sup>a,y</sup>	36.85±2.12
	3 <sup>rd</sup> month	41.81±0.81 <sup>a,x</sup>	38.11±1.42 <sup>b,x</sup>	40.95±1.07ª,y	34.34±0.73
a*	0	11.76±0.77 <sup>bc,x</sup>	13.74±0.54 <sup>a,x</sup>	10.48±0.06 <sup>c,x</sup>	12.45±1.07ª
	1 <sup>st</sup> month	8.74±0.38 <sup>d,y</sup>	11.05±0.16 <sup>b,y</sup>	10.11±0.34 <sup>c,x</sup>	12.52±0.65
	2 <sup>nd</sup> month	8.52±0.45 <sup>b,y</sup>	11.95±1.21 <sup>a,y</sup>	7.17±0.69 <sup>b,y</sup>	11.81±0.66
	3 <sup>rd</sup> month	6.82±0.47 <sup>c,z</sup>	13.43±0.38 <sup>a,x</sup>	7.45±0.58°,y	11.22±0.39
b*	0	14.95±1.11 <sup>a,x</sup>	10.81±0.47 <sup>b,y</sup>	13.37±0.72 <sup>ab,x</sup>	12.7±2.98ab
	1 <sup>st</sup> month	10.39±1.32bc,y	12.54±1.25 <sup>b,x</sup>	9.72±0.57 <sup>c,x</sup>	16.86±1.54ª
	2 <sup>nd</sup> month	13.2±1.03ª,x	12.38±0.59 <sup>a,x</sup>	12.99±0.75 <sup>a,x</sup>	14.77±2.16 <sup>a</sup>
	3 <sup>rd</sup> month	11.03±0.12 <sup>b,y</sup>	11.19±0.34 <sup>b,xy</sup>	11.34±0.56 <sup>b,y</sup>	17.68±1.96

abcd: Different superscripts in the same row indicate statistically significant differences (p < 0.05)



# A comparative study on physicochemical properties and in vitro bioaccessibility of bioactive compounds in rosehip (*Rosa canina* L.) infusions treated by nonthermal and thermal treatments

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#### ABSTRACT

The aim of this present study was to compare the effects of non-thermal food processing technologies with thermal treatment on physicochemical properties, in vitro bioaccessibility and antioxidant activity of bioactive compounds in rosehip (*Rosa canina* L.) infusions.

For this purpose, high pressure processing (HPP) at 600 MPa for 5 min, pulsed electric field (PEF) with 5 and 15 kJ/kg specific energy inputs at 1 and 3 kV/cm electric field strength and thermal treatment (TT) at 85 °C/10 min were applied. According to the results, processing method significantly affected the content and bioaccessibility of phenolic compounds, and antioxidant activity in rosehip extracts. It appears that the highest retention of total phenolics (TPC) and flavonoids (TFC) were achieved by the TT (2278 mg GAE/100 g dw and 3728 mg RE/100 g dw, respectively) and HPP treatment with a pressure of 600 MPa for 15 min (2268 mg GAE/100 g dw and 3695 mg RE/100 g, respectively). These findings are in line with the results of antioxidant activities of the samples measured by CUPRAC and DPPH assays. On the other hand, TT, HPP treatment at 600 MPa/5 min and PEF treatment with 5 and 15 kJ/kg energy input at 1 kV/cm electric field strength resulted with a higher recovery of TPC, TFC and antioxidant activity after gastrointestinal digestion.

Overall, a glance at the results reveals that HPP and PEF technologies can be used as alternative processing methods in the production of functional foods with enhanced nutritional value.



# Development of mucilage powder from *Basella rubra* and elderly products application

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#### ABSTRACT

This research aims at developing mucilage powder from *Basella rubra* as a new choice of elderly product. Factorial design used for investigate the effect of maltodextrin to soy protein ratio and inlet temperature. The optimal spray dried condition was 38.5: 1.5 maltodextrin to soy protein ratio at 130 °C of inlet temperature. The mucilage powder had 0.323 of water activity,  $3.94\pm0.32\%$  moisture content, the total phenolic content  $6.26\pm0.01$  mgGAE/g and  $26.34\pm2.87\%$  DPPH inhibition. Then, the mucilage powder was applied by mixed 50% w/v with three beverages, namely of drinking water, orange juice and tea. The results shown that *Basella rubra* mucilage powder increased beverage viscosity in level 2 (Nectar-like; 51-350 cP). The total phenolic content was  $2.02\pm0.10$ ,  $2.72\pm0.08$  and  $2.76\pm0.10$  mgGAE/mL and antioxidant scavenging activity were  $45.18\pm0.85$ ,  $43.57\pm1.52$  and  $46.19\pm0.92$  %DPPH inhibition for drinking water, orange juice and tea, respectively. According to these results, the *Basella rubra* mucilage powder could be offering choices for dysphagia elderly who require modified for textures with antioxidant.

Keywords: Antioxidant activity, Basella rubra, Elderly, Mucilage, Phenolic compound, Spray dried



# Changes in the Bioaccessibility of Polyphenols in Fruits and Vegetables Subjected to Freezing Process

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#### ABSTRACT

Recently, reduced access to markets and restaurants due to the COVID-19 outbreak has led to increased consumption of frozen foods while people stay safe at home and seek healthy products that do not deteriorate for a long time. Freezing is an effective method to preserve nutritional and physical properties of fruits and vegetables while extending their shelf life. A diet rich in fruits and vegetables has been attributed to lower incidence of chronic diseases, which has been linked to the presence of bioactive compounds, particularly polyphenols. The availability of polyphenols following digestion is critical as several studies have pointed out that the bioaccessibility of polyphenols is food processing. Considering the above, in this paper the changes in the bioaccessibility of polyphenols in fruits and vegetables that are subjected to freezing process were discussed. Although the findings of the literature were contradictory, majority of the studies showed that bioaccessibility of polyphenols from fruits and vegetables were similar or higher than that of fresh fruits and vegetables. Overall, the current paper emphasized that freezing process may be an effective approach for preservation of bioaccessible polyphenols in fruits and vegetables.

Keywords: In Vitro Digestion, Colonic Fermentation, Industrial Freezing, Flavonoids, Phenolic Acids

#### 1. INTRODUCTION

Consumption of fruits and vegetables appears to be inversely related to the incidence of chronic diseases such as cardiovascular disease, obesity, type 2 diabetes, and neurodegenerative diseases. The potential health beneficial effects of fruits and vegetables has been linked to the presence of bioactive compounds, particularly polyphenols (Costa et al., 2017; Cory et al., 2018; Serino and Salazar, 2019). Polyphenols are plant secondary metabolites that possess antioxidant, anti-inflammatory (Zhang and Tsao, 2016), antimicrobial (Daglia, 2012), and anticancer (Niedzwiecki et al., 2016) properties. Taking into account the number of phenol rings and the structural elements binding these rings to each other, polyphenols can be classified as (i) flavonoids, (ii) phenolic acids, (iii) stilbenes, and (iv) lignans (D'Archivio et al., 2007; Grootaert et al., 2015) (Figure 1).

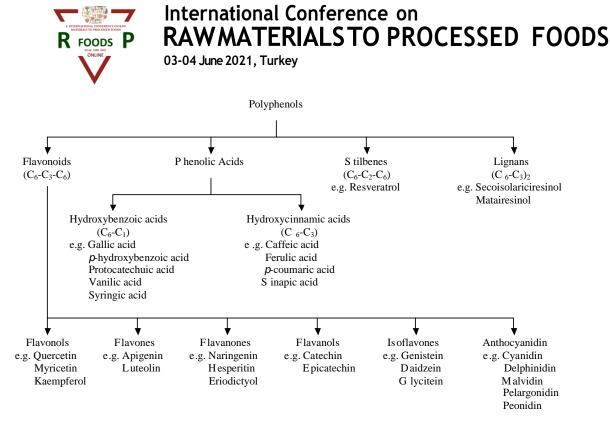


Figure 2: Classification of Major Classes of Dietary Polyphenols (Grootaert et al., 2015)

As fruits and vegetables are perishable, an efficient preservation method is necessary to extend the shelf life and protect the nutritional and physical characteristics of these commodities. Freezing is one of the most widely utilized food processing methods that reduces the deterioration rate of fruits and vegetables by decreasing enzymatic and microbial activities, respiration and oxidation (Bilbao-Sainz et al., 2019). On the other hand, previous studies in the literature have indicated that freezing process may affect the polyphenol content of fruits and vegetables (Mazzeo et al., 2015; Paciulli et al., 2015).

The term "bioaccessibility" refer to the potential portion or fraction of a nutrient that is released from the food matrix and available for absorption in the intestine in relation to the total initial content (Montiel-Sánchez et al., 2021). Only polyphenols liberated from the food matrix by digestive enzymes and microbiota are bioaccessible in the intestine and thus possibly bioavailable (Kamiloglu, 2019a). To investigate the gastrointestinal behavior of dietary components, *in vitro* methods mimicking digestion processes are commonly employed. Even though clinical trials are the "gold standard" for answering diet related issues, *in vitro* gastrointestinal digestion models have benefits of being fast, cheap, and less labor intensive, as well as being free of ethical constraints (Alminger et al., 2014).

Considering the above, the aim of this paper was to provide an overview of the findings on the changes in the bioaccessibility of polyphenols in fruits and vegetables subjected to freezing process. Below, the effect of freezing process on the contents of total phenolics, flavonoids and anthocyanins as well as individual flavonoids and phenolic acids in fruits and vegetables are discussed in detail.

#### 2. POLYPHENOLS IN FRUITS AND VEGETABLES

#### 2.1. Total Phenolics, Flavonoids and Anthocyanins

The bioaccessibility of total phenolics in apples (Dalmau et al., 2017), beetroot (Dalmau et al., 2019) and strawberry (Kamiloglu, 2019a) was reduced after freezing (up to 67%), whereas frozen green beans (Kamiloglu, 2019c) and spinach (Kamiloglu, 2020) contained significantly higher bioaccessible total phenolics compared to fresh vegetables (23-58%) (p<0.05). The differences observed among different commodities might be related to the fact that the studies on the bioaccessibility of total phenolics in apples and beetroot were determined only after gastric digestion, while for green beans and spinach phenolic bioaccessibility was calculated after intestinal digestion. For the measurement of total phenolic content, majority of the studies in the literature employed Folin-Ciocalteu method, which has the benefits of being simple, robust, and reproducible, as well as not requiring a specific equipment. On the other hand, this assay also has some drawbacks. Along with phenolic compounds, other reducing agents such as citric acid, ascorbic acid, simple sugars, or certain amino acids can interfere with the assay, and consequently the results might be overestimated.



Moreover, the assay is carried out in an aqueous medium, therefore the measurement of lipophilic phenolics could be limited (Capanoglu et al., 2021). Therefore, it is recommended to also perform chromatographic analysis before drawing conclusions.

The total flavonoid content of frozen green beans (Kamiloglu, 2019c), spinach (Kamiloglu, 2020) and strawberry (Kamiloglu, 2019a) were found to be higher than that of fresh fruits and vegetables after *in vitro* gastrointestinal digestion (28-149%) (p<0.05), while the bioaccessibility of total flavonoids in apples was reduced after individual quick freezing (IQF) process (20%), which was statistically not significant (Kamiloglu, 2019b). Aluminium chloride assay, which is carried out to determine the total flavonoid content of fruit and vegetables, is not fully specific to flavonoids. In addition to flavonoids, phenolic acids could also react in the assay, whereas majority of the flavonoids excluding flavanols, react poorly in the assay (Ho et al., 2012). Hence, as indicated above, flavonoids should be analysed using chromatographic methods in order to obtain more reliable results.

The total monomeric anthocyanin content of frozen strawberries was reported to be significantly higher than that of fresh ones after *in vitro* gastrointestinal digestion (65-86%) (p<0.05) (Kamiloglu, 2019a). Freezing disrupts the food matrix due to the development of ice crystals, and these alterations in the matrix could result in cell wall maceration and enhance the extraction of bioactives during digestion (Oliveira et al., 2016).

#### 2.2. Flavonoids

Flavonoids are low molecular weight compounds, composed of 15 carbon atoms, placed in a  $C_6-C_3-C_6$  configuration. The structure is comprised of two aromatic rings, connected by a three–carbon bridge, often in a heterocyclic ring form. Six distinct subclasses, namely flavonols, flavanols, flavanoes, flavanoes, isoflavones and anthocyanidins, arise from alterations in the substitution model of the heterocyclic ring (Tennant et al., 2014) (Figure 1).

The most common subgroup of the flavonoids are the flavonols. The main dietary flavonol aglycones are quercetin and kaempferol, which often occur as O-glycosides. Berries, apples, onion, broccoli, kale, cabbage, tea, and red wine are among the major dietary sources of flavonol aglycones and O-glycosides (Kamiloglu et al., 2019). A recent study investigating the effect of different freezing methods on the bioaccessibility of strawberry polyphenols using an *in vitro* gastrointestinal digestion model showed that both conventional and IQF processes decreased the amount of bioaccessible quercetin 3-glucuronide (40-54%) (p<0.05) (Kamiloglu, 2019a). On the other hand, in another study evaluating the bioaccessibility of polyphenols in fresh and frozen strawberries (Balasooriya et al., 2020), conflicting results were reported. While the bioaccessible quercetin was increased by 38% for fasted state of *in vitro* gastrointestinal digestion, in fed state quercetin was not detected in frozen strawberries that were grown under elevated carbon dioxide and temperature (Balasooriya et al., 2020). Similarly, a study evaluating the bioaccessibility of polyphenols in fresh and frozen apples using an in vitro gastrointestinal digestion model, also reported that bioaccessible quercetin glycosides were diminished after industrial freezing processes, probably due to the removal of apple peels that contains the majority of flavonols (Kamiloglu, 2019b). Although statistically not significant, the amount of bioaccessible kaempferol 3-glucoside increased (18-28%) after both conventional and industrial freezing of strawberries. However, freezing reduced the bioaccessible kaempferol 3-coumaroylglucoside significantly (59-64%) (p < 0.05). It is possible that kaempferol 3-coumaroylglucoside might be cleaved to kaempferol 3-glucoside during digestion process (Kamiloglu, 2019a).

Anthocyanins are water-soluble pigments that give several fruits, vegetables, roots, cereals, and legumes their red, purple, and blue color. Anthocyanins are present as glycosides of their respective aglycone anthocyanidins. The most abundant anthocyanidins are cyanidin, malvidin, delphinidin, peonidin, pelargonidin, and petunidin (Kamiloglu et al., 2015). Kamiloglu (2019a) and Balasooriya et al. (2020) investigated the bioaccessibility of anthocyanins from strawberry after different freezing processes. As a result, Kamiloglu (2019) reported increases in the bioaccessibility of pelargonidin 3-glucoside (86-129%) (p<0.05), the major anthocyanin present in strawberries, whereas Balasooriya et al. (2020) reported decreased bioaccessibility of pelargonidin 3-glucoside both at fed and fasted states (16-25 and 5-13%, respectively). Balasooriya et al. (2020) further applied a colonic fermentation model following gastrointestinal digestion, which revealed up to 55% reduced amount of bioaccessible pelargonidin 3-glucoside (p<0.05). On the other hand, the bioaccessibility of tyrosol, which has been reported to be the major microbial metabolite derived from pelargonidin 3-glucoside in strawberry after colonic fermentation (López de las Hazas et al., 2017) increased (16-36%) in frozen strawberries (Balasooriya et al., 2020). The trend observed for other minor anthocyanins present in strawberries



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varied. Conventional freezing increased the bioaccessibility of cyanidin 3-glucoside (13%) and pelargonidin 3-rutinoside (100%) (p<0.05). However, after IQF process, bioaccessible cyanidin 3-glucoside was reduced (44%) and pelargonidin 3-rutinoside did not change significantly. Furthermore, pelargonidin 3-malonylglucoside diminished after both conventional freezing and IQF processes. It is possible that freezing might have caused the cleavage of pelargonidin 3-malonylglucoside and to pelargonidin 3-glucoside (Kamiloglu, 2019a).

Epicatechin and catechin are abundant flavanols in fruits, chocolate, red wine, and tea (Manach et al., 2004). Studies investigating the effect of freezing processes on the bioaccessibility of flavanols are very limited. According to the study of Kamiloglu (2019b), IQF process increases the bioaccessible amount of epicatechin by 47% (p<0.05). Similarly, another study investigating the effect of industrial freezing process on the bioaccessibility of spinach polyphenols concluded that, IQF process enhanced the bioaccessibility of individual flavonoids by 15% (Kamiloglu, 2020). In addition to the compounds discussed above, the bioaccessibility phlorizin present in apples was reported to reduce by 71% as a result of industrial freezing (p<0.05).

#### 2.3. Phenolic Acids

Phenolic acids include hydroxycinnamic acids with a three-carbon side chain (caffeic, *p*-coumaric, ferulic and sinapic) and hydroxybenzoic acids with a  $C_6$ - $C_1$  structure (gallic, protocatechuic, 4-hydroxybenzoic, vanillic, and syringic) (Balasundram et al., 2006). Hydroxycinnamic acids are present in high concentrations in berries and coffee, while tea and many fruits are rich sources of hydroxybenzoic acids (D'Archivio et al., 2007). The total phenolic acid content of frozen spinach was found to be 16% higher than that of fresh spinach after in vitro gastrointestinal digestion (Kamiloglu, 2020). However, chlorogenic acid, a well-known hydroxycinnamic acid with quinic acid found in apples, was reported to be significantly less bioaccessible when apples are subjected to freezing process (66%) (p<0.05). Similarly, the bioaccessibility of caffeic acid was also reduced significantly after industrial freezing process of apples (42%) (p<0.05) (Kamiloglu, 2019b). On the other hand, the majority of the findings reporting the bioaccessibility of *p*-coumaric acid derivates in strawberries revealed drastic reductions after freezing process. Although the results obtained regarding the bioaccessibility of ferulic acid derivatives in frozen strawberries varied, majority of the findings suggested reduced bioaccessibility, which is in parallel with the results obtained for *p*-coumaric acid derivates (Kamiloglu, 2019a; Balasooriya et al., 2020).

The changes in the bioaccessibility of polyphenols in fruits and vegetables subjected to freezing process are summarized in Table 1.



Table 1: Changes in the bioaccessibility of polyphenols in fruits and vegetables subjected to freezing process

Product	Variety	Treatment	Model	Result(s)	Reference
Apple (Malus domestica)	Granny Smith	Freezing in liquid nitrogen at -196°C	<i>In vitro</i> gastric digestion at 37°C for 3 h	Total Phenolic Content: 67% ↓*	Dalmau et al., 2017
		Industrial freezing (IQF) at -22°C	<i>In vitro</i> gastrointestinal digestion at 37°C for 2 h for each stage	Total Phenolic Content: $6\% \uparrow$ Total Flavonoid Content: $20\% \downarrow$ Epicatechin: $47\% \uparrow^*$ Quercetin 3-galactoside: $100\% \downarrow^*$ Quercetin 3-glucoside: $100\% \downarrow^*$ Quercetin 3-xyloside: $100\% \downarrow^*$ Quercetin 3-arabinoside: $100\% \downarrow^*$ Quercetin 3-rhamnoside: $100\% \downarrow^*$ Phlorizin: $71\% \downarrow^*$ Chlorogenic acid: $66\% \downarrow^*$ Caffeic acid: $42\% \downarrow^*$	Kamiloglu, 2019b
BeetrootConditivaFreezing in liquid nitrogen at -196°C, -80°C and -20°C		<i>In vitro</i> gastric digestion at 37°C for 3 h	<u>-196°C:</u> Total Phenolic Content: 57% ↓* <u>-80°C:</u> Total Phenolic Content: 44% ↓* <u>-20°C:</u>	Dalmau et al., 2019	
Green beans	Gina	Industrial freezing (IQF)	In vitro gastrointestinal	Total Phenolic Content: 46% ↓* Total Phenolic Content: 23% ↑*	Kamiloglu,
(Phaseolus vulgaris)		at -22°C	digestion at 37°C for 2 h for each stage	Total Flavonoid Content: 149% ↑*	2019c
Spinach (Spinacia oleracea L.)	El real	Industrial freezing (IQF) at -22°C	<i>In vitro</i> gastrointestinal digestion at 37°C for 2 h for each stage	Total Phenolic Content: 58% ↑* Total Flavonoid Content: 28% ↑* Individual flavonoids: 15% ↑ Phenolic acids: 16% ↑	Kamiloglu, 2020



Table 1 (continues): Changes in the bioaccessibility of polyphenols in fruits and vegetables subjected to freezing process

Product	Variety	Treatment	Model	Result(s)	Reference
Strawberry (Fragaria x ananassa Duch.)	Tiago	Conventional freezing at -20°C and industrial freezing (IQF) at -22°C	In vitro gastrointestinal digestion at 37°C for 2 h for each stage	<b>Conventional Freezing:</b> Total Phenolic Content: $5\% \downarrow$ Total Flavonoid Content: $33\% \uparrow^*$ Total Monomeric Anthocyanin Content: $86\% \uparrow^*$ Quercetin 3-glucuronide: $54\% \downarrow^*$ Kaempferol 3-glucoside: $18\% \uparrow$ Kaempferol 3-coumaroylglucoside: $64\% \downarrow^*$ Cyanidin 3-glucoside: $129\% \uparrow^*$ Pelargonidin 3-glucoside: $100\% \uparrow^*$ Pelargonidin 3-malonylglucoside: $100\% \downarrow^*$ <i>p</i> -Coumaroylhexose: $41\% \downarrow$ Ferulic acid hexose derivative: $100\% \uparrow$ Ellagic acid deoxyhexoside: $14\% \uparrow$ <b>Industrial Freezing (IOF):</b> Total Phenolic Content: $1\% \downarrow$ Total Phenolic Content: $46\% \uparrow^*$ Total Monomeric Anthocyanin Content: $65\% \uparrow^*$ Quercetin 3-glucoside: $28\% \uparrow$ Kaempferol 3-coumaroylglucoside: $59\% \downarrow^*$ Cyanidin 3-glucoside: $44\% \downarrow$ Pelargonidin 3-glucoside: $86\% \uparrow^*$ Pelargonidin 3-malonylglucoside: $100\% \downarrow^*$ <i>p</i> -Coumaroylhexose: $36\% \downarrow$ Ferulic acid hexose derivative: - Ellagic acid deoxyhexoside: $5\% \downarrow$	Kamiloglu, 2019a



Table 1 (continues): Changes in the bioaccessibility of polyphenols in fruits and vegetables subjected to freezing process

Product	Variety	Treatment	Model	Result(s)	Reference
Strawberry ( <i>Fragaria</i> x <i>ananassa</i> Duch.)	San Andreas	Conventional freezing at -20°C	<i>In vitro</i> gastrointestinal digestion at fed and fasted state at 37°C for 2 and 3 h for gastric and intestinal digestion + colonic fermentation at 37°C for 24 h	Gastrointestinal digestion:Fed state:Quercetin: 3-100% $\downarrow$ *Pelargonidin 3-glucoside: 16-25% $\downarrow$ Pelargonidin 3-rutinoside: 55% $\uparrow$ * to 2% $\downarrow$ p-Coumaric: 4-12% $\downarrow$ Ferulic acid: 3-100% $\downarrow$ *Ferulic acid: 3-100% $\downarrow$ *P-Coumaroyl: 30-100% $\downarrow$ *Pasted state:Quercetin: 38% $\uparrow$ to 100% $\downarrow$ *Pelargonidin 3-glucoside: 5-13% $\downarrow$ Pelargonidin 3-glucoside: 5-13% $\downarrow$ Pelargonidin 3-glucoside: 11.76% $\uparrow$ *p-Coumaric acid: 11% $\downarrow$ to 12% $\uparrow$ Ferulic acid: 3-16% $\uparrow$ p-Coumaroyl: 8% $\downarrow$ to 13% $\uparrow$ Colonic fermentation:Fed state:Pelargonidin 3-glucoside: 53-55% $\downarrow$ *Tyrosol: 36% $\uparrow$ to 49% $\downarrow$ *p-Coumaric acid: 36% $\uparrow$ to 52% $\downarrow$ *Ferulic acid: 29-53% $\downarrow$ *Ferulic acid: 29-53% $\downarrow$ *Fasted state:Pelargonidin 3-glucoside: 5-54% $\downarrow$ *Tyrosol: 16-35% $\uparrow$ *p-Coumaric acid: 56% $\uparrow$ * to 15% $\downarrow$ Ferulic acid: 13-33% $\downarrow$ *	Balasooriya et al., 2020

 $\uparrow$ : increase;  $\downarrow$ : decrease; -: no change; \*: significantly different (p < 0.05)



#### CONCLUSION

Although the findings of the literature were contradictory, majority of the studies showed that bioaccessibility of polyphenols from fruits and vegetables were similar or higher than that of fresh fruits and vegetables. Therefore, freezing may be a good approach to preserve the bioaccessible polyphenols in fruits and vegetables. Nevertheless, in this paper only the influence of freezing process is discussed, the variations in the polyphenol content during frozen storage should be covered further to establish the optimized conditions for enhanced bioaccessibility of polyphenols in fruits and vegetables. In addition, in vivo studies are also required to understand the bioaccessibility polyphenols from frozen fruits and vegetables.

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# Study on the Ozone Treatment Process on the Bacteria Damaging Nutritive Fruits

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#### ABSTRACT

The objective of the study is to evaluate the effect of ozone treatment (OTRE) process on the bacteria affecting fruits, apple and pomegranate in particular. In this study, ozone was given continuously for 60 min and after every 10 min of interval. Secondly, the use of ozone in cycle, where the ozonized sample was taken after 30 min of continuous exposure followed by a 5 min break, which constituted an exposure time for 60 min. The bacterial cell count affecting the apple was decreased from  $1.5 \times 108$  to  $1.2 \times 103$  CFU.mL-1 after 30 min and from  $1.5 \times 108$  to 80 CFU.mL-1 after 60 min. The bacterial cell count affecting the apple was decreased from  $1.5 \times 108$  to  $0.9 \times 102$  CFU.mL-1 after 60 min. OTRE process is a rapid, better solution to preserve them from diseases causing bacteria and enhance their durability against spoilage.

Keywords: Ozonation, Apples, Pomegranates, Bacteria, Colony Forming Unit



# Effect of Industrial Freezing Process on the Bioaccessibility of Carotenoids in Organic Butternut Squash (*Cucurbita moschata*)

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#### ABSTRACT

Butternut squash (*Cucurbita moschata*) is recognized as a functional food owing to its diverse nutritional composition, in particular carotenoids that promote health beyond nutrition. Considering the perishable nature of butternut squash, an effective preservation method is required to maintain the physical and nutritional characteristics and extend the shelf life of this product. Freezing is one of the most widely used preservation methods that reduces the deterioration rate of fruits and vegetables. For industrial trade, fruits and vegetables are generally frozen on a belt in a low-temperature, high air-velocity blast freezer to produce individually quick frozen (IQF) products. During IQF processing of butternut squash, a number of byproducts including the skin, pulp and seeds are generated. Disposal of byproducts is both a cost to the manufacturer and a potential negative impact on the environment. On the other hand, in this case, the industrial byproducts could serve as sources of carotenoids. Considering the above, the aim of this study was to evaluate the bioaccessibility of carotenoids in the samples taken from different steps of the IQF processing of butternut squash. An *in vitro* digestion model simulating the digestion in the mouth, stomach, and intestine was applied and after each stage of the digestion samples were collected and analyzed for their carotenoid content using HPLC-PDA. The results revealed  $\alpha$ - and  $\beta$ -carotene as the major carotenoids present in butternut squash. The bioaccessibility of both  $\alpha$ - and  $\beta$ -carotene were found to be significantly higher in pulp and seed by product compared to the raw material (64-72%) (p<0.05). On the other hand, IQF process caused significant decreases in the bioaccessibility of carotenoids (47% on average) (p < 0.05). Overall, the current study highlighted byproducts from butternut squash processing as substantial sources of bioaccessible carotenoids.

**Keywords:** β-carotene, HPLC-PDA, *in vitro* digestion, individual quick freezing (IQF)

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# **Research on Solar Water Heating Drying Plant for Drying Medicinal Plants**

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#### ABSTRACT

Scientists at TSTU have developed a solar water heating dryer. The developed solar water heating dryer is designed for food and pharmaceutical companies to produce high-quality products - dried and concentrated extracts of vegetables, fruits and herbs, as well as syrups, mashed potatoes and dry concentrates with the preservation of biologically active substances and healthy ingredients based on local raw materials. Experimental studies were performed on a laboratory solar-water convective heating unit and curves were obtained for the duration and temperature of the drying process of medicinal plants. Based on the results of experimental studies of the dehydration process in a laboratory unit, an engineering methodology has been developed for calculating the design and technological parameters of an industrial solar-water convective installation. A comparative analysis of the behavior of the constituent extractives of medicinal plants, as well as the ash content and final moisture content of medicinal plants under the implementation of various drying methods, was carried out.

Keywords: solar water heating dryer, medicinal plants, drying



# Investigation of Gluten-free Cake Production from Poppy Seed (*Papaver* somniferum L.) Pulp: TOPSIS Application

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#### ABSTRACT

Poppy seed (*Papaver somniferum* L.) is a widely used product whose oil is extracted by the cold press method since seeds has high oil content. After oil extraction, poppy pulp is obtained as a waste with a high protein content but not utilized in any way. In this study, the possibilities of using poppy seed pulp in gluten-free cake production were investigated. For this purpose, rice flour and poppy pulp combinations (contain of 25, 50, 75 and 100% poppy seed pulp) were prepared in the cake formulation and 4 different cakes were produced as gluten-free. Color values, the amount of ash, dry matter and protein, volume and symmetry index, cooking efficiency, textural and sensory properties of gluten-free cakes were determined. As a result of the analysis, it was determined that the addition of poppy pulp decreased the  $L^*$  value in the cake samples and the cakes were darker, but addition poppy seed pulp was occurred softer cakes. On the other hand, it was determined that the volume index values were 28, 30, 28 and 26 mm respectively, and the cooking efficiency decreases with the addition of poppy pulp. TOPSIS method, one of the Multi-criteria Decision Making Techniques, was applied in order to determine the most preferred cake for sensory analysis. According to the TOPSIS model result, the most preferred sample was the cake containing 25% poppy seed pulp. Poppy seed pulp can be used as an alternative food ingredient for food preparation.

Keywords: cake, gluten-free, poppy pulp, sensory, texture, TOPSIS

#### 1. INTRODUCTION

Celiac patients are the individuals, who are intolerant to gluten which is a wheat protein, and they should not consume wheat-containing products (Sicherer and Sampson 2014). The disease develops as damage in the mucosa of the small intestine (De Arcangelis et al., 2020).

In celiac patients, the villi in the structure of intestine do not fulfill their function and the intestinal absorption does not occur appropriately. On the other hand, in presence of gluten, also the flatulence and digestion problems that are called gluten intolerance and develop as a result of consuming gluten-containing foods can be seen (Y1lmaz and Koca, 2020). The individuals struggling and diagnosed with celiac disease constitute 0.6-1% of the global population (Fasano et al., 2003; Hamdani et al., 2020). Gluten is responsible for the viscoelastic properties (Gujral & amp; Rosell, 2004; Hamdani et al., 2020) Besides, it is emphasized that the main structure of the products made of wheat flour is formed by gluten (Ammar et al., 2021).

Among the wheat products, the cakes consumed as a snack are among the popular products. The market size of cakes, which are mainly composed of flour, egg, sugar, and oil, is believed to reach 75 billion USD in year 2023 (Xu et al., 2020). The cakes are classified according to content, shape, and baking methods. In recent periods, producers started to produce gluten-free cakes for people with gluten sensitivity In general, the production of gluten-free cakes is made using rice flour and the protein network doesn't form due to gluten deficiency (Roman et al., 2019). Studies are carried out on ensuring the optimal product formulation and shape because cake quality problems occur in gluten-free cake production due to the lack of gluten



(Tsatsaragkou et al., 2015; Saeidi ve al., 2018; Tuta Şimşek, 2019)

Poppy (*Papaver somniferum* L.) plant is an industrial plant that is widely used in the food industry (Cibulková et al., 2014). Poppy seeds are generally used in bakery food, sauce, cake, and dessert production and oil production (López et al., 2018). On the other hand, besides their anti-cancer and anti-tumor effects, it is known that poopy and its oil have positive effects on diabetes (Higashi and Setoguchi, 2000). Moreover, thanks to its high protein content, poppy recently became a material for products that are enriched with herbal proteins. Poppy seeds are in form of oil, filling (bakery products), and paste (breakfast). Poppy pulp is obtained as a result of oil production. Poopy pulp is rich in protein and dietary fiber. Its high protein content drew interest from the aspect of the potential effects of poppy pulp on the product formulations.

Within the scope of the present study, gluten-free cake formulation development studies were carried out for celiac patients and individuals preferring to consume nutrient-rich products. In this study, four cakes containing 25%, 50%, 75%, and 100% poppy pulp in their rice flour - poppy pulp mixture were prepared and the product characteristics were determined using physicochemical, textural, and sensorial analyses. Moreover, TOPSIS which is one of the multi-criteria decision-making methods was used in interpreting the analysis results.

#### 2. MATERIAL and METHODS

#### 2.1. Material

Gluten-free rice flour, herbal oil, sugar, baking powder, egg, and vanilla used in the production of glutenfree cake were obtained from the local stores. Poppy pulp was purchased from the companies producing opium oil and used after grinding.

#### 2.2. Gluten-free Cake Production

In producing the gluten-free cakes, the poppy pulp obtained as a result of cold-pressed opium oil was mixed with rice flour at 25%, 50%, 75%, and 100% concentrations, and four different cake formulations were prepared. In preparing the cake mixture, the cake formulation obtained from the preliminary studies was used. For this purpose, 270 g poppy pulp and rice flour mixtures, 260 g sugar, 120 ml sunflower oil, 300 ml milk, 110 g egg, 2.5 g baking powder, and 2.5 g vanilla were used. The cake mixture was baked in muffin mold at 180°C temperature for 25 minutes. After the baking process, the cakes were cooled and removed from the molds.

#### 2.3. Physicochemical Analyses

Since the poppy pulp added to the formula has high protein and dietary fiber content, ash (AOAC, 1995), moisture (AOAC, 2005), and protein (AOAC, 1990) contents of the gluten-free cakes were determined using the relevant formulas. Color values of the gluten-free cakes containing poppy pulp at different concentrations were determined using a color-meter (Konica Minolta, model CR-400, Mississauga, ON, Canada) expressed as of  $L^*$ ,  $a^*$ , and  $b^*$  values. Volume index, symmetry index, and uniformity index values of gluten-free cakes were calculated using AACC (2000) methods.

#### 2.4. Textural Analyses

The textural profiles of gluten-free cakes prepared using four different formulations were determined using a textural analysis device (T.A.HD Plus Stable Micro Systems, İngiltere) with P/36R cylindrical probe. The test configuration was set as follows: pretest speed=1 mm/s, test speed= 5 mm/s, strain rate =75%, and strain force= 5 g. Hardness (N), cohesiveness, springiness (g.cm), gumminess and chewiness values of gluten-free muffin cake samples were determined.

#### **2.5. Sensorial Analyses**



In analyzing the consumer preferences for gluten-free cakes, the crumb and crust color, taste, and chewability values were asked to the panelists. In sensorial analyses, a rating scale ranging between 1 and 9 points was used. In interpreting the criteria, 1 refers to the lowest score and 9 refers to the highest score.

#### 2.6. TOPSIS Application

TOPSIS is one of the multi-criteria decision-making methods. Within the scope of this study, all the criteria were used in determining the most preferred product in the sensorial analyses (Figure 1).

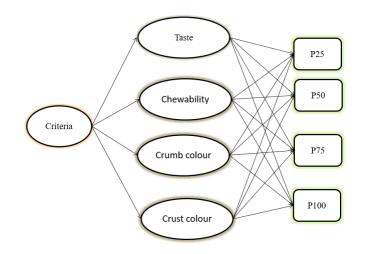


Figure 1: Decision hierarchy process of the gluten-free muffin cake sensory properties

(1)

1. First, the normalized decision matrix is established for TOPSIS application:

$$r_{ij} = \frac{a_{ij}}{\sqrt{\sum_{k=1}^{m} a_{kj}^2}}$$

2. Normalized decision matrix is weighted;

$$Y_{ij} = \begin{bmatrix} w_1 x_{11} & w_2 x_{12} & \dots & w_n x_{1n} \\ w_1 x_{21} & w_2 x_{22} & \dots & w_n x_{2n} \\ \vdots & & & \vdots \\ \vdots & & & & \vdots \\ w_1 x_{m1} & w_2 x_{m2} & \dots & w_n x_{mn} \end{bmatrix}$$
(2)

5. Positive  $(A^{-})$  and negative  $(A^{-})$  ideal solution determination

$$A^{*} = \{v_{1}^{*}, v_{2}^{*}, ..., v_{n}^{*}\}$$
(Maximum values)  

$$A^{-} = \{v_{1}^{-}, v_{2}^{-}, ..., v_{n}^{-}\}$$
(Minimum values) (3)  
(4)

vis the weighted normalised values.

4. Calculation of the distance of the alternatives from positive and negative ideal solution

$$D_{i}^{*} = \sqrt{\sum_{j=1}^{n} (v_{ij} - v_{j}^{*})^{2}}$$
(5)



(6)

(7)

$$D_{i}^{-} = \sqrt{\sum_{j=1}^{n} (v_{ij} - v_{j}^{-})^{2}}$$

5. For each alternative, the distance to ideal solution is calculated using the formula;

$$R_i^* = rac{D_i^-}{D_i^- + D_i^*}$$

6. R values are sorted in descending order and the most preferred product is determined (Sadeghzadeh and Salehi, 2011).

#### 2.7. Statistical Analyses

At the end of the study, the One-Way variance analysis (ANOVA) and Tukey's HSD test were used in SPSS Statistics 17.0 package software in order to interpret the results.

#### 3. RESULTS AND DISCUSSION

#### **3.1.** Physicochemical Analysis

Moisture, protein, and ash contents of gluten-free muffin cakes added with cold-pressed poppy pulp are presented in Table 1. It was determined that the addition of pulp had a significant effect on the moisture, protein, and ash contents of cake samples (P < 0.05). Among the cupcakes, the highest moisture content was found in Sample P25 and it suggests that the increasing moisture content might be because of the water retention by high pectin and starch content in the rice flour. Protein and ash contents of cake samples were found to range between 9.00-16.52 and 1.56-3.47, respectively, and the highest protein and ash contents were found in sample P100. It was found that the protein and ash contents of muffin cake samples significantly increased with the increase in the amount of poppy pulp addition. The increase in protein and ash contents is related with the high protein and ash content of the poppy pulp. Grasso et al. (2020) reported that the protein contents of cake samples added with cold-pressed aniseed flour at different concentrations significantly increased (Goksen and Ekiz, 2021). In a study, in which the hydrosol that is the residuum of mint distillation was added into the cake samples at different concentrations, the increase in hydrosol concentration in the cake increased the dry matter values (Berktas and Cam, 2020).

Calculated by dividing the post-baking weight of cake dough by the pre-baking weight, the baking efficiency values of samples P25, P50, P75, and P100 were found to be 93, 94, 90 and 85%, respectively. Baking efficiencies decreased with the increase in the addition of cold-pressed poppy pulp. Increasing amount of poppy pulp caused significant changes in volume and symmetry indices of cake samples (P < 0.05) and decreases were observed with the increase in the amount of pulp addition.

Samples	Protein Content	Moisture	Ash	Volume Index	Symmetry Index
-	(%)	(%)	(%)	( <b>mm</b> )	(mm)
P25	$9.00^{d} \pm 0.25$	$27.91^{\circ} \pm 0.01$	$1.56^{d} \pm 0.11$	28 <sup>b</sup> ±0.12	13.00 <sup>a</sup> ±0.04
P50	$11.01^{\circ} \pm 0.30$	$24.88^{b} \pm 0.00$	$2.02^{\circ} \pm 0.13$	30ª±0.12	13.50ª±0.50
P75	$13.63^{b} \pm 0.01$	$25.06^{ab} \pm 0.00$	$2.79^{b} \pm 0.08$	28 <sup>b</sup> ±0.00	11.00 <sup>b</sup> ±0.01
P100	$16.52^{a} \pm 0.71$	$23.70^{a} \pm 0.01$	$3.47^{a} \pm 0.05$	26°±0.05	10.75 <sup>b</sup> ±0.35

Table 1: Physicochemical analysis of gluten-free cakes with the addition of poppy pulp

Different letters in the same column mean significantly different (p < 0.05).



#### 3.2. Color Analysis

Table 2: Crumb and crust color values of muffin gluten-free cakes

Samples		Crumb Color		Crust Color				
	L*	a*	b*	L*	a*	b*		
P25	$51.60^{a} \pm 0.04$	$7.94^{a} \pm 0.37$	$23.83^{a} \pm 0.53$	57.13 <sup>a</sup> ±4.73	$2.47^{d} \pm 0.25$	$17.42^{b} \pm 0.60$		
P50	$44.92^{ab} \pm 0.82$	$8.14^{a} \pm 0.22$	$23.72^{a} \pm 0.52$	$53.01^{a} \pm 6.98$	$5.92^{\circ} \pm 0.44$	23.16 <sup>a</sup> ±0.29		
P75	$44.46^{ab} \pm 0.67$	$9.03^{a} \pm 0.96$	$23.05^{a} \pm 0.47$	$51.22^{a} \pm 0.60$	$7.34^{b} \pm 0.60$	$22.58^{a} \pm 0.55$		
P100	$40.00^{b} \pm 0.69$	9.39 <sup>a</sup> ±0.54	$22.54^{a} \pm 0.58$	$47.19^{a} \pm 0.54$	$9.54^{a}\pm 0.22$	25.13 <sup>a</sup> ±0.29		

Different letters in the same column mean significantly different (p < 0.05).

Color is a characteristic that increases the food quality and contributes to attracting consumers (Lucas et al., 2018). The color of cakes is directly related with materials used and Maillard and caramelization reactions during the baking (Yalcin et al.,2020). Gluten-free muffin samples' crumb and crust color parameters ( $L^*$ ,  $a^*$  ve  $b^*$ ) are presented in Table 2. With increasing addition of poppy pulp in cake samples,  $L^*$  and  $b^*$  values of crumb and crust decreased and  $a^*$  values increased. The crumb and crust colors were observed to become darker with increasing amount of poppy pulp addition (Figure 2). Similarly, it was reported that the cakes' color parameters changed in cakes enriched with pulp obtained from the extraction of oils of chia and aniseed seeds (Aranibar et al., 2019; Goksen ve Ekiz, 2021).



Figure 2. Picture of prepared gluten free muffin cakes

#### **3.3. Textural Analysis**

The textural characteristics of gluten-free muffin cake samples such as hardness (N), adhesiveness (g.sec), springiness (cm), cohesiveness, gumminess ve chewiness are presented in Table 3. The highest hardness, gumminess, and chewiness values were observed in sample P100, whereas the lowest hardness, gumminess, and chewiness values were observed in sample P25. Significant differences were found between hardness, gumminess, and chewiness values of all samples (p < 0.05) and these values were observed to decrease with increases in the amount of poppy pulp addition.

Hardness (N)	Adhesiveness (g.sec)	Springiness (cm)	Cohesiveness	Gumminess	Chewiness
104.26ª±12.69	-104.85ª±35.92	$0.78^{a}\pm0.10$	0.27ª±0.03	2866.2ª±62.26	2264.1ª±69.75
83.33 <sup>ab</sup> ±7.71	-170.01 <sup>b</sup> ±22.72	0.86ª±0.09	0.31ª±0.02	2636.5ª±36.58	2277.6 <sup>a</sup> ±47.60
78.66 <sup>b</sup> ±14.73	-207.34 <sup>b</sup> ±41.22	$0.80^{a} \pm 0.08$	0.28ª±0.02	2155.1 <sup>ab</sup> ±50.24	1750.5 <sup>ab</sup> ±57.36
47.54°±6.16	-232.00 <sup>b</sup> ±31.26	0.75ª±0.16	0.27ª±0.03	1299.4 <sup>b</sup> ±25.86	996.8 <sup>b</sup> ±35.44
	(N) 104.26 <sup>a</sup> ±12.69 83.33 <sup>ab</sup> ±7.71 78.66 <sup>b</sup> ±14.73	(N)(g.sec)104.26a±12.69-104.85a±35.9283.33ab±7.71-170.01b±22.7278.66b±14.73-207.34b±41.22	(N)(g.sec)(cm) $104.26^{a}\pm12.69$ $-104.85^{a}\pm35.92$ $0.78^{a}\pm0.10$ $83.33^{ab}\pm7.71$ $-170.01^{b}\pm22.72$ $0.86^{a}\pm0.09$ $78.66^{b}\pm14.73$ $-207.34^{b}\pm41.22$ $0.80^{a}\pm0.08$	(N)(g.sec)(cm)Cohesiveness $104.26^{a}\pm 12.69$ $-104.85^{a}\pm 35.92$ $0.78^{a}\pm 0.10$ $0.27^{a}\pm 0.03$ $83.33^{ab}\pm 7.71$ $-170.01^{b}\pm 22.72$ $0.86^{a}\pm 0.09$ $0.31^{a}\pm 0.02$ $78.66^{b}\pm 14.73$ $-207.34^{b}\pm 41.22$ $0.80^{a}\pm 0.08$ $0.28^{a}\pm 0.02$	(N)(g.sec)CohesivenessCohesivenessGumminess $104.26^{a}\pm12.69$ $-104.85^{a}\pm35.92$ $0.78^{a}\pm0.10$ $0.27^{a}\pm0.03$ $2866.2^{a}\pm62.26$ $83.33^{ab}\pm7.71$ $-170.01^{b}\pm22.72$ $0.86^{a}\pm0.09$ $0.31^{a}\pm0.02$ $2636.5^{a}\pm36.58$ $78.66^{b}\pm14.73$ $-207.34^{b}\pm41.22$ $0.80^{a}\pm0.08$ $0.28^{a}\pm0.02$ $2155.1^{ab}\pm50.24$

Table 3: Texture properties of gluten-free cupcakes prepared with different poppy pulp ratio



Different letters in the same column mean significantly different (p < 0.05).

#### 3.4. Sensorial Analysis

The sensorial analysis results of gluten-free muffin cake samples' taste, chewability, and crumb and crust colors are illustrated in Figure 3 using spider-web diagram. With an increase in the amount of poppy pulp addition, the most preferred sample in terms of crumb and crust color and taste was found to be sample P100. From the aspect of chewability, samples P25 and P50 were found to be the most preferred samples. Increasing amount of pulp addition caused a decrease in chewability. According to the sensorial analysis results, it was proven that added functional muffins were preferable in terms of crust color and taste.

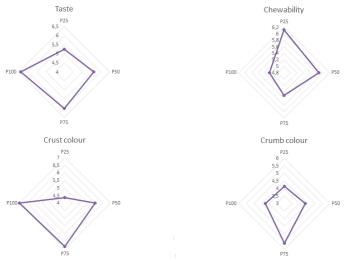


Figure 3: Sensory attributes of muffins

#### **3.5. TOPSIS Application**

Obtained from the TOPSIS application used for sensorial analyses of gluten-free cakes, the positive and negative distance and the R values used in sorting are presented in Table 4.

Table 4: Distance from positive (D+), negative (D-) and ratio values of each alternative for technique for order preference by similarity to ideal solution (TOPSIS) techniques

Alternative	$D^+$	$D^{-}$	R
P25	0.01	0.09	0.86
P50	0.06	0.04	0.36
P75	0.09	0.02	0.16
P100	0.09	0.02	0.15

In TOPSIS analysis, the highest R value refers to the most preferred product. Considering all the criteria, the most preferred product after the weighting was found to be the sample P25, followed by P50, P75, and P100.



#### 4. CONCLUSION

In this study, poppy pulp was added into rice flour, which is used in gluten-free cake production, at different concentrations. Poppy pulp is a product that has high protein content and is rich in nutrients. Protein content of gluten-free cakes increased with the increase in the amount of pulp. Since the poppy pulp is a dark-colored material, the color values of cakes changed with increasing amount of poppy pulp addition. On the other hand, softer cakes were obtained with increasing amount of pulp addition and volume and symmetry indices were observed to decrease. As a result of TOPSIS assessment after the sensorial analysis, the most preferred sample was found to be P25. Especially for their high protein content, the poppy pulps used in the present study was found to be an important additive for supporting the foods consumed by individuals having special nutrition preferences, especially the celiac patients. Poppy pulp can be used in production of different gluten-free products together with alternative product raw materials and the effects can be investigated by experimentally investigating the product formulations of other food groups.

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# Investigation of Sensorial and Physicochemical Properties of Yoghurt Colored with Phycocyanin of *Spirulina platensis*

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#### ABSTRACT

Pigments obtained from microalgae, which can be used as food and food additives thanks to their rich nutritional content, are used as colorants in many foods. In this study, the sensorial and physicochemical properties of yoghurt colored with phycocyanin from *Spirulina platensis* were investigated. *Spirulina* in three different concentrations (0.5%, 1.0% and 1.5% and a control without *Spirulina*) was added to the milk used for yoghurt, and phycocyanin pigment has been transferred to the milk. Then, the sensorial properties (appearance, color, flavor and general acceptability), general composition (moisture, protein, fat, carbohydrate and ash content) and color values ( $L^*$ ,  $a^*$ ,  $b^*$ ,  $\Delta E$ , Chroma,  $h^\circ$ ) of yoghurt produced were measured. The highest sensory score of preference was obtained by 0.5% *Spirulina* added colored yoghurt. Protein content of colored yoghurt increased in proportionally with added *Spirulina* powder and was measured as 29.25 g/100g DM in 0.5% *Spirulina* added yogurt. Increase in *Spirulina* content, it caused decrease in L\*, a\* and b\* values and increase in  $\Delta E$  value. L\*, a\*, b\*,  $\Delta E$ , Chroma and h° values of yoghurt colored with 0.5% *Spirulina* can be used to enrich yoghurt as a natural functional ingredient due to its high nutrient and pigment content.

Keywords: Spirulina platensis, Yoghurt, Phycocyanin, Physicochemical properties, Sensory properties



# **Purification and Charecterisation of Polyphenoloxidase from Myrtle Berries**

(Myrtus Communis L.)

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#### ABSTRACT

Myrtle (*Myrtus communis* L.) is belonging to the family of Myrtaceae and is mostly distributed in Mediterranean Region and is also called as "Murt", "Hambeles" or "adi mersin" in Turkey. While fruit is white and pulpy at the beginning of maturity ripened fruit can turn into blue-black color. Browning reaction because of polyphenol oxidase (PPO) can be observed in plant that is rich in phenolic substances. In this study, optimum temperature, optimum pH, substrate spesifity, thermal stability of polyphenol oxidase partially purified from white myrtle berries and effect of inhibitors on enzyme activity was investigated. Cathechol was used as substrate. Optimum pH and temperature were found as 6.8 and 30 °C respectively for myrtle PPO while Km and Vmax values were calculated as 3.34 mM and 4.1 unit. In thermal inactivation studies at 60, 70, and 80 °C reaction rate constants (k) were found as 0.0282, 0.526 and 0.1117 minute<sup>-1</sup>, thermal half-life times ( $t_{1/2}$ ) were calculated as 24.6, 13.2 and 6.2 minute, and decimal reduction time (D-value) were calculated as 81.6, 43.8 and 20.6 minute, respectively. Activation energy (Ea) and Z-value were calculated as 67.2 kj.mol<sup>-1</sup> and 33.4°C, respectively. Similar inactivation ratios from 40 % to 100 % were observed for both inhibitors of ascorbic acid and disulphite at 0.01-10 mM concentration.

Keywords: Myrtus Communis L., Polyphenol Oxidase, Kinetic Parameters, Thermal Stability, Purification

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# The Effects of Ultraviolet Light Application on the Quality of Kaymak (Clotted Cream) During Storage Period

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#### ABSTRACT

Ultraviolet light (UV-C) application, one of the non-thermal technology, finds widespread use in the dairy industry to provide safety and extend shelf life. It can be considered as an effective method for controlling post-processing contamination by surface application in dairy products. Kaymak, a traditional Turkish cream, can be contaminated with undesirable microorganisms especially during the hardening process carried out at ambient conditions. This study aimed to determine the effects of UV-C light application on the quality changes of kaymak during the storage period. The surfaces of samples were exposed to UV-C light from 20 cm height and at 0.8, 1.6 and 3.2 m/min band speeds (equaled to 1.419, 2.797, 5.697 kJ/m<sup>2</sup> doses, respectively) using a specially designed continuous UV-C disinfection system. Also, control sample not treated with light was used for comparison. pH values of samples decreased whereas titratable acidity increased during the storage period which could be due to the lactic acid fermentation. The slight changes in the color parameters (L\*, a\* and b\* values) affected by UV-C light application and storage period were obtained, which were not noticeable by the eye. The degree of lipid oxidation (both peroxide and thiobarbituric acid reactive substances) significantly increased with the application of high UV-C light doses, as well as the storage time. UV-C application decreased the visible mold and yeast growth on the surface of kaymak samples compared to the control samples. The off-flavour perceived in the highest UV light dose did not change during storage. As a result, UV-C light can be used to reduce the mold and yeast growth and extend the shelf life of kaymak, but it accelerates the oxidative potential and the off-flavor perception. Therefore, applied light doses should be selected considering the sensorial and oxidative aspects of high-fat dairy products before the industrial application.

Keywords: UV-C Light, Kaymak, Lipid Oxidation, Sensory Aspects

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# A Sensory Observation for Cold Stored Beef Steak and Norway Salmon

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#### ABSTRACT

Cold preservation is one of the conventional methods that protect red meat and fish meat for longer shelf life. In this regard, storage in refrigeration temperature is a critical cold preservation way for meat hygiene and safety. Some quality parameters such as total mesophilic bacteria count, pH, total volatile basic nitrogen, or trimethylamine values can be investigated to estimate the shelf life of meat during cold storage. On the other, after purchasing the meat products, consumers can also evaluate sensory properties like odor, color, and texture in a short time to observe physical quality changes. For this reason, this study, it is aimed to determine sensory changes of beefsteak and Norway salmon products every 6 hours from the initial hour at refrigerated temperature ( $4\pm2^{\circ}$ C). According to overall acceptability scores, beefsteak and Norway salmon samples were defined as unacceptable at 72nd and 60th hours which are supported by visual photographs. Results of this study showed that consumers may gain knowledge regarding color, odor, and textural changes in course of time and average storage period of these two cold stored products.

Keywords: Beef Steak, Norway Salmon, Sensory Analysis, Odor, Color, Texture

#### **INTRODUCTION**

Meat and meat products have an important place in health and nutrition. With an increase in the human population, demand for this kind of product has been also increasing. In terms of popular meat products, consumption of red meat and fish meat is of great importance to ensure a healthy and balanced diet (Tosun and Demirbas, 2012). Steak is generally sliced boneless piece of red meat with high protein, which often grilled or pan-fried. On the other hand, salmon is a healthy fish with polyunsaturated fatty acids, special carotenoid pigment astaxanthin, and vitamin D. (Haq et al. 2017). Although fish meat can spoil more quickly than red meat regardless of the type of meat, food safety must be ensured from slaughtering or harvesting to the fork. Food safety can be defined as a set of measures taken to prevent physical, chemical, biological, and all kinds of damages to food (Anonymous, 2004; Ceylan et al., 2021). To ensure food safety for raw food, different preservation methods are generally applied. In these methods, cold preservation is one of the most widely used methods (Maddock, 2012; Xiong 2017; Ceylan et al. 2021). Although cold preservation increases the shelf life, it also has limited time to protect the meat. When meat shelf-life is going to be expired, spoilage is going to have occurred. The spoilage is caused by the microbial growth in meat by bacteria, resulting in the release of metabolites. Such as volatile organic compounds alcohols, aldehydes, ketones, fatty acids, esters, and sulfur compounds. Volatile organic compounds formed as a result of microbial growth during meat storage cause odor and when their concentration increases significantly, which can draw the product below the sensory acceptable level (Soncin et al. 2007; Casaburi et al., 2015; Ceylan et al. 2021). In this respect, sensory evaluation of meat products is a significant parameter to understand freshness and estimate if it should be consumed. For his reason, in this study characteristic change of two raw products (beef steak and Norway salmon) in terms of odor, texture, color, and overall acceptability scores has been



investigated to estimate shelf life.

#### MATERIAL AND METHODS

Beefsteak and Norway salmon samples were obtained from an international supermarket in Istanbul. They were stored at refrigerated conditions  $(4\pm2^{\circ}C)$  and were analyzed per 6 hours until the sensory scores reached low values. To reveal consumer awareness at home conditions, sensory properties were investigated in terms of odor, texture, color, and overall acceptance. Sensory scores were applied between 0-9 points and 4 points were accepted as the limit value for the rejection (Ceylan et al., 2018). Furthermore, changes in course of time were supported by using visual photographic images. Collected data were subjected to analysis of variance (ANOVA) to determine the sensory quality changes. GraphPad Prism software (California Corporation, USA) was applied to reveal significant differences between groups by ANOVA. Once a significant (p<0.05) main effect was obtained, the mean values of the samples were further analyzed using Tukey's multiple range comparison tests.

#### **RESULT AND DISCUSSION**

#### **Beef Steak**

One of the most important parameters in determining the quality of red meat is the initial quality of the raw material. In Figure 1, the overall acceptability evaluation of steak samples every 6 hours can be seen from statistical results. On the initial day of the experimental period odor, color, texture scores were found to be 8.94, 8.98, and 8.96, respectively (all data not shown). Furthermore, the overall acceptability on the initial day was 8.98. The overall score values were >8 in the first 24 hours (p>0.05). Although there is a slight decrease, high scores showed the freshness of the product. After the 30th hour, overall scores values decreased faster, and statistically significant differences were observed (p<0.05). On hours 60th, 66th, 72nd, and 78th overall scores at the 72nd hour of the storage period (p<0.05). On the other hand, odor, color, and texture were defined as 4.62, 3.46, and 3.86 at the 72nd hour, respectively.

Conr	nec	ti	ng	1 L	et	te	rs	Re	port	
Level									Mean	
0	A								8,9800000	
6	A	В							8,8200000	
12	А	В							8,7200000	
18	A	В							8,4600000	
24	A	В							8,4000000	
30		В	С						8,1200000	
36			С	D					7,4200000	
42				D	Е				6,9400000	
48					Е				6,5000000	
54						F			5,7600000	
60							G		4,6200000	
66							G	н	4,0600000	
72								Н	3,8400000	
78								н	3,4600000	

Levels not connected by same letter are significantly different.

Figure 1: Statistical overall acceptability score results of steak in every 6 hours.

All Pairs Tukey-Kramer test results and visual photographs of steak with initial, and 78th-hour photographs are presented in Figure 2. As could be seen from the Figure steak had bright, shining pinky color with perfect



structure in the initial photo. The color of the meat is may directly affect the purchasing decision of the consumer. Myoglobin is a protein macromolecule that is significantly responsible for flesh color. Physicochemical and biochemical interactions between myoglobin and chemical compounds, light, and other elements in muscle structure can sometimes cause the abnormal color/appearance of fresh meat (Ranken, 2000; Faustman and Suman, 2017). As can be noticed from Figure 2 pinky color changed to dark brown color at 78th hour. Ceylan et al. (2021) mentioned that color defects such as darkening; browning can be seen in meat products with spoilage. As bacterial growth increases, characteristics such as rapid breakdown of connective tissue can be expected (Zagorec and Champomier-Vergès, 2017; Ceylan 2021). After 78 hours, especially on the front part surface drying and harder structure were observed. These changes are attributed to water and oil loss which leads to lower texture scores. On the other, on the bottom side changes in connective tissue such as softening noticed. Although it was higher than other scores, especially on the bottom side, odor loss was also noticed. Odor, color, and texture scores were 4.14, 2.9, and 3.6, respectively at the 78th hour. These scores can be used as a guide for the evaluation of the shelf life of steak samples for consumers as <4 are accepted as unconsumable.

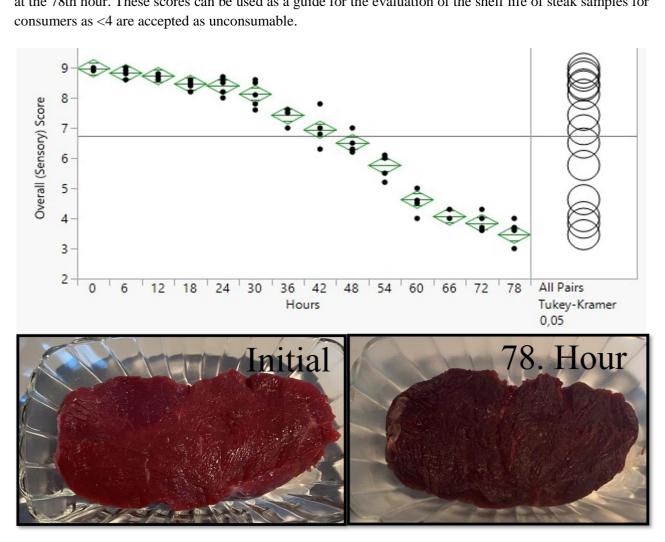


Figure 2: All Pairs Tukey-Kramer test results and visual photographs

#### Norway Salmon

According to the annual seafood consumer index study of the Norwegian Seafood Council, the demand for salmon has increased in the last few years. Furthermore, it ranks first in imports of aquaculture products in



Turkey. Therefore, consumers should pay attention to shelf life during storage. Cold preservation is the most simple and common way to increase shelf life (Gram and Huss, 1996). On the initial day of the experimental period odor, color, texture scores were found to be 8.94, 8.96, and 8.94, respectively (all data not shown). The initial overall acceptability score found as 8.92 and changes every 6 hours can be seen in Figure 3. During cold preservation storage, overall scores decreased slowly in the first 30 hours (p<0.05). However, from the 36th hour to the 60th hour there was a  $\sim$ 3.4 point decrease (p>0.05). Norway salmon was observed as unacceptable with 3.82 scores at the 60th hour of the storage period (p<0.05). Compared to beef steak, there was a 12-hour difference in terms of acceptability. On the other hand, odor, color, and texture scores were defined as 4.22, 3.94, and 3.9 at the 60th hour.

Conr	ect	ing Lett	er	s Report	
Level				Mean	
0	Α			8,9200000	
6	Α			8,8400000	
12	AB			8,6600000	
18	AB			8,3800000	
24	AB			8,1800000	
30	B			8,0200000	
36		С		7,2200000	
42		D		6,3800000	
48		D		5,7600000	
54		E		4,8400000	
60			F	3,8200000	
66			F	3,3200000	

Levels not connected by same letter are significantly different.

Figure 3: Statistical overall acceptability score results of Norway salmon in every 6 hours

All Pairs Tukey-Kramer test results and visual photographs of Norway salmon with initial, and 66th-hour photographs are presented in Figure 4. The skin color of fish is an important quality indicator and affects purchase decisions, because consumers initially accept or reject food based on its color (Unal Sengör et al., 2019). Shining color with fat streaks in the initial photo showed that the sample was fresh and had a high color score (8.96). Color changes of the skin of the fish can be evaluated to identify the freshness of the product (Unal Sengör et al., 2019). With a closer look at the image at the right, a color change is clearly noticeable after the 60th hour. This showed that there is a color deterioration with decreasing freshness.



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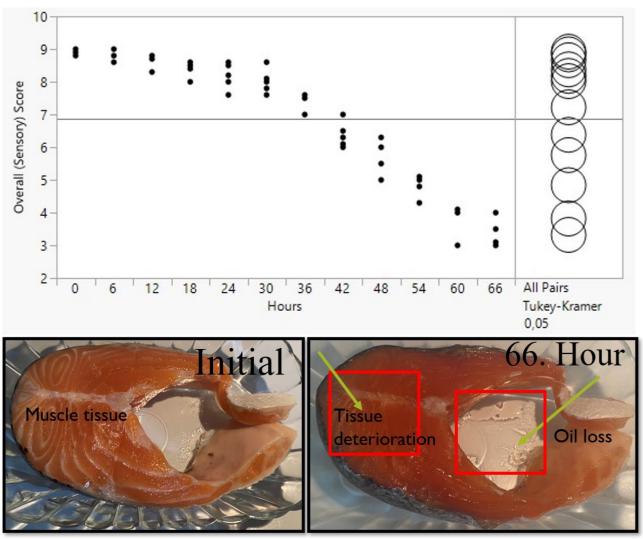


Figure 4: All Pairs Tukey-Kramer test results and visual photographs

Salmon oil contains 75% triglycerides as the main ingredient, 17% diglycerides, and monoglycerides as minor ingredients. Overall, monounsaturated fatty acids (MUFA) and polyunsaturated fatty acids (PUFA) are reported to be 44% and 34%, respectively (Cascant et al. 2018). So Norway salmon can be considered with a high rate of fat. After 60 hours oil loss noticed which also resulted in drying on the skin on the front side. Texture scores significantly decreased with drying. Odor loss and defects have also occurred with degradation. This may result from the presence of various volatile compounds and their combinations. When biogenic amine production and gas formation starts to increase, the formation of bad odor can be observed (Zagorec and Champomier-Vergès, 2017; Ceylan 2021). Odor, color, and texture scores were 3.62, 3.42, and 3.4, respectively at the 66th hour.

#### CONCLUSION

Odor, color, and texture parameters for the consumers come to the fore to estimate the shelf life of the product. Microbial growth may lead to the sensory deterioration of steak and salmon products. In this case, it affects the consumer's liking and may also create some defects in terms of food safety. Therefore, the importance of household applications for beefsteak and Norway salmon presented in this study. Sensory evaluation of these products is examined by odor, color, texture scores at refrigeration temperature. From the



initial day, samples were scored and photographed until they reach the unacceptable score of 4. Results showed that beefsteak had higher scores compared to Norway salmon and was accepted as consumable at 66th hour. However, Norway salmon was evaluated as inconsumable on the 60th hour. Color change, oil sealing, drying on the surface can be seen clearly by visual photography at the end of storing time. Furthermore, a bad odor (3.62 on 66th hour) has been observed lower side of Norway salmon. For the steak drying on surface and color darkening observed on the surface of the product when compared to the initial time. The results of this study provided an important idea about the shelf life of these two product products.

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## Optimization of Tray Dryer Drying Parameters of Hacıhaliloğlu Apricot Using Response Surface Methodology

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#### ABSTRACT

Drying of fruits and vegetables reduces storage, packaging, and transport costs, making them microbiologically and chemically more stable. This study aimed to investigate the effects of drying temperature and air velocity on some quality characteristics of apricot. The drying parameters were optimized response surface methodology. Pretreatments were performed at temperatures of 60, 70, and 80°C and air velocity of 1, 1.5, and 2 m/s with a face-centered composite design. Total phenolic content, total carotenoid content, pH, total titratable acidity, bulk density, and color values of dried apricot were determined. Changes in the drying rate, rehydration capacity and L\* values in response to independent variable parameters were evaluated. The optimum drying conditions (75.18°C and 2.0 m/s) were experimentally verified. As a result, the drying rate and rehydration capacity of apricot increased depending on the increase in temperature and air velocity. In addition, low temperature and low air velocity values caused a significant decrease in lightness. The optimum conditions provided better preservation of the parameters (drying rate, rehydration, L\*) studied to commercialize dried apricots.

Keywords: Dried apricot, Lightness, Drying rate, Response surface methodology, Tray dryer



## Production of Orange Juice Concentrate Using Conventional and Microwave Vacuum Evaporation: Thermal Degradation Kinetics of Bioactive Compounds and Color Values

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#### ABSTRACT

This study aimed to concentrate orange juice using the microwave vacuum evaporation and the rotary evaporation at different absolute pressures (20, 31, and 47 kPa) and investigate the effects of different concentration techniques on degradation kinetics of vitamin C, total phenolic content, total carotenoid content, and color values. The microwave vacuum evaporation significantly raised the evaporation rate compared to the rotary evaporation. It was determined that the microwave vacuum evaporation and the rotary evaporation at 80°C required 21 and 42 min, respectively. Total soluble solid changes during the microwave vacuum evaporation and the rotary evaporation processes were fitted to six different empirical models with  $R^2$  range between 0.915 and 0.998. The results showed that degradation rate constants for vitamin C, total phenolic content, and total carotenoid content of orange juices concentrated using the rotary evaporation were higher significantly than the microwave vacuum evaporation. It was determined that the degradation rate constants of the L\*, a\*, b\*, and  $\Delta E$  orange juices concentrated using the microwave vacuum evaporation were lower than the rotary evaporation.

Keywords: Orange juice, Microwave heating, Thermal processing, Kinetics, Quality



## Pollen Content in Raw Spanish Rosemary Honey: Influence of the Geographical Origin

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#### ABSTRACT

The highly appreciated rosemary honey is very light in color, with a weak intensity of odor and aroma, and fresh, floral, and vegetal tones. In general, it is considered monofloral if the pollen from Rosmarinus officinalis is not lower than 10%, assessed by melissopalynological analysis. Commercial transactions tend to agree with this percentage, since it is a honey with the pollen underrepresented and there is not an official value established. This honey stems mainly from wild areas, hence the vast presence of abundant pollen types. At the reception of the honey packaging process, the analyst classifying this raw honey, must also consider in the pollen count the presence of all these accompanying pollens. The objective of this work was to evaluate the influence of the geographical area on the pollen content of raw rosemary honey. To this aim, 50 honeys provided by beekeepers were analyzed: 25 from Valencian coast and 25 from the interior (Albacete). The pollen quantification revealed that practically all the samples analyzed could be classified as rosemary monofloral, since their pollen content of *Rosmarinus officinalis* was higher than 10-15%. Many characteristic pollens of the Mediterranean flora were observed common to both geographic zones (Brassicaceae, Tipo Genista, Hypecoum sp., Ceratonia siliqua, Quercus sp., Oleaceae, Helianthus annuus, Thymus sp., Echium sp.). However, abundant pollen from Prunus dulcis stood out as a differentiating element in the samples from the interior zone (exceeding 40% in some cases) and from *Erica* sp. in those from the coast. The pollen content indicates that these honeys must be considered as monofloral rosemary for their commercialization having the expected organoleptic characteristics of this type of honey. It also shows that the geographical area influences the accompanying flora which in turn reflects the contribution of nectar from other plants and in fact conditions its peculiar sensory-nuances.

Keywords: geographical origin, honey packaging process, monofloral honey, pollen analysis, rosemary honey

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# Volatile profile of Spanish raw citrus honey: the best strategy for its correct labelling

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#### ABSTRACT

Among monoflorals, the citrus honey is highly valued due to its delicate flavour reminiscent to the orange blossom. Classifying a honey as monofloral is a tedious and time-consuming procedure and requires an expert analyst in the identification/quantification of the pollen morphologies. For citrus honey, this difficulty is even bigger considering the low pollen content of the new hybrid varieties of fruits without seeds. Therefore, the identification of volatile compounds is increasingly being considered as a good option in the classification of this type of honey. The proper classification of citrus honey before entering as a raw material in the honey packaging process would ensure its correct labeling hence benefiting the consumer and the beekeepers that produce this type of honey. Among the few compounds that are accepted as a true marker, methylanthranilate (MA) stands out in the case of citrus honey because it is only present in the citrus blossom nectar. The objective of this study was to evaluate the presence of specific volatile compounds in Spanish citrus honeys and to correlate their abundance with the MA level. Citrus honey samples (25) previously classified by pollen (more than 10% Citrus spp.) and MA (more than 2 mg/kg), collected in the Valencian Region (Spain), and other 25 types of honey (collected around Spain) were used in this study. Their volatile fraction was analyzed by GC-MS/MS with previous extraction by SPME. It can be concluded that, in addition to MA (measured by HPLC), there were certain volatile compounds such as the four lilac aldehydes (A, B, C, and D) always present together in citrus honey and being their abundance ratio relatively stable. Furthermore, linalool, ethyl linalool, dill-ether, 2(3H)-furanone, p-mentha-1,8-dien-7-ol (limonene) and 1-p-menthene-9al, were found exclusively in this type of honey. The amount of all these compounds was positively correlated with the abundance of MA.

Keywords: citrus honey, monofloral honey, volatile profile, GC-MS/MS, SPME

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## **DNA Degradation from Raw Material to Canned Products**

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#### ABSTRACT

Food traceability has become an important issue for the stakeholders of the food industry. Seafood products, especially processed fish products are known in the list of top 10 food products which are subjected to fraudulent actions. Due to the market size, being highly profitable products and difficulties of identification of species in processed food products, processed fish products are highly vulnerable to fraudulent actions. unearned gain and life-threatening health problems such as toxicity and allergenicity are the main consequences of these fraudulent actions. Therefore, the traceability of fish products has gained importance and DNA-based techniques are promising approaches for species identification over the last decades. The quality of extracted DNA is the main factor, which has great impacts on the achievements of steps in the species identification procedure. Thermal processing, high pressure or treatment with acidic or alkali solutions reduce the DNA quality in terms of purity and yield. this research was aimed at the determination of DNA degradation during canned tuna production. For this aim, the DNA was extracted from prior and post stages of canned tuna sterilization; raw tuna, the thermal process applied tuna, tuna with brine medium. Additionally, for determination of filling medium effect on DNA degradation; DNA extracted from canned tuna filled with sunflower oil and olive oil as most commonly used filling medium in canning tuna industry. The degradation level and quality of DNA from all processed tuna groups compared with un-processed tuna with nanodrop and Real-Time PCR results. The results revealed that thermal processing, treating with salt and canning process cause to DNA degradation. While the DNA yield was determined as 806.1 ug/ul in raw material, this value determined as 485.20 ug/ul and 301.9 ug/ul from thermally processed and soaked in a on, respectively. Within the canning process, the DNA yield reduced to 15.4 ug/ul and 20.6 brine soluti ug/ul for olive oil and sunflower oil canned tuna respectively. The optimal purity range of DNA was determined from cooked tuna and tuna soaked in a brine solution prior to the canning procedure as 1.85. The canning process reduced the A260/A230 ratio, which used as an indicator of organic contaminants.

# KEYWORDS: CANNED FOOD, DNA DEGRADATION, FOOD FRAUD, REAL TIME PCR, THERMAL PROCESS

#### **1 INTRODUCTION**

Seafood products are known one of the important parts of human diet caused by their ambitious benefits to human health such as preventing cancer or heart issues. Fatty acids especially, omega 3 and omega 6 essential and amino acid content make seafood products are one of the most consumed products in human diets. Therefore, the consumption of seafood has increased and the market size of seafood industry has become one of the most traded industries over the world. (FAO, 2018;Aksun Tümerkan and Emiroğlu, 2020). Global seafood consumption has been rising from 16.1 kg/per to 20.2 kg/per capita between 2001 and 2015 and estimated reached to approximately 21 kg/per increasing in last decade(FAO., 2019.; Fernandes et al., 2020). Recently, the size of seafood trade has reported as higher than the size of total pork and poultry trade (Asche et al., 2015; Do et al., 2019). Food fraud generally known as placing of food components illegally for unfair financial gain (FAO, 2018). Intentional fraud activities cause not only damage financially to consumer, but also some types of fraud such as mislabelling or species substation also cause to important health problem(Giusti et al., 2019). Threatening of biodiversity and fishing is an another serious issue caused by these fraudulent actions by is illegal, unregulated and/or unreported fishing (Giusti et al., 2019).Due to the difficulties of monitoring the processed seafood, and increasing profitable potential in the relevant



industry, these products have commonly committed to the fraudulent (Willette et al., 2017). While intentional fraudulent activities have reported most commonly, unintentional mislabelling or substitution of species can observed due to difficulties of identification of cryptic or closely related species (Barendse et al., 2019). The species-specific morphological properties such as size or colour can used for species identification, however this identification sometimes does not meet the correct classification demand in close species or processed food. While some chromatographic and spectroscopic methods utilized for species identification, the achievements of protein based methods such as Isoelectric focusing (IEF) or Sodium Dodecyl Sulfate Polyacrylamide Gel Electrophoresis (SDS-PAGE) had reported formerly (Ho et al., 2020). Usage of molecular based techniques for species identification has increased steadily recently. Owing to the reliability and sensitivity of molecular based methods for identification of cryptic species, these methods have also used in species authentication in food products.

Especially, following to detection of horsemeat substitution of beef and mislabelling of seafood in last decade, the importance of species traceability in food products have take attention over the world (Mataet al., 2020). Therefore, some developments have observed in the process of molecular-based methods from DNA extraction to bioinformatics. While all steps of molecular based methods are important for the achievements of correct identification of species, the purity and quality of extracted DNA is an essential initial requirement for following processes. Whereas the species authentication based on extraction methods, selected gene region and applied PCR-based techniques when the material as un-processed, the achievements of this authentication can vary depending on processing methods, packaging conditions additionally in processed food products. Especially thermal processing or treatment of raw material with acidic or alkali solution or pressure application cause to DNA degradation and thereof differences in identification of species. Canning of different fish species is one of the commonly applied processing methods used by the fist world war (Martin, 1990). Tuna known as the main specie used for canning process due to its trading and profitable potential. The tuna market is listed in top ten trading food commodities list with \$6 billion financial value (Garrett and Adam, 2010; Gordoa et al., 2017).

Therefore, the fraudulent activities especially mislabelling and species substitution are frequently reported in canned tuna industry. A wide range of research revealed that the fraudulent of tuna species in industrial scale and catering scale such as restaurant local retailer; Tunas are among the most desirable marine fish worldwide, with a global tuna species catch that peaked at 7.7 million tones in 2014. Skipjack (Katsuwonus pelamis) and Yellowfin (Thunnus albacares) were the tuna species most captured with about 3 and 1.5 million tonnes, respectively. In contrast, the captures of Bluefin tuna through the same period did not exceed 40,000 tonnes (FAO., 2012; Sotelo et al., 2018). Thereof, the ecological and economic value of tuna species differ among tuna-sub species (Abdullah and Rehbein, 2016). The over-consumption of the most desirable species cause to unstable price and uncontrolled fisheries actions are considered the two main reasons for the increasing number of substitution and mislabelling cases in the canned tuna industry (Sotelo et al., 2018). These fraudulent actions differ significantly depending on socio-economic situation of countries, consumer preference and traceability awareness and geographical tuna availability. For instance, mislabelling and substitution rate reported between 37% and 48% in Spanish tuna chain, this rate was reported as 95% in Bruxelles restaurants (Christiansen et al., 2018; Gordoa et al., 2017). Most of the processing techniques damage to biochemical composition of raw material. Processing techniques especially include thermal process and acid treatments impact to the yield and quality of DNA. Due to canning process including different thermal procedure and pressure application, this process cause to DNA degradation. Filling medium is also accepted as primary reason of DNA degradation in canned food products (Chapela et al., 2007).Due to central role played by the quality of DNA and its yield in the DNA-based species identification and therefore traceability of food products, these properties have considered prior to PCR-amplification. A wellpurified, non-degraded and non-contaminated DNA is the main requirements for the achievements of DNA-



based analyses. In the light of this, the aim of present study was to assess the DNA degradation at the different steps of tuna canning process. DNA quality and yield are assessed from tuna processing. Two different filling mediums are also compared. The amplificability of raw, processed and canned tuna products through a Real-Time PCR approach.

#### 2 MATERIAL AND METHOD

#### 2.1 Products Sampling

Both raw and processed tuna sample obtained from Turkish tuna processing plant. All the sample chosen from the same origin in terms of catching season and area for avoiding any variation originated from fish sample. Raw tuna sample used as control (G1), thermal processed tuna prior to canning process (pre-cooked at 70 °C (G2), soaked in brine solution prior to canning process (G3), canned tuna with olive oil medium (G4), canned tuna with sunflower oil medium (G5). Canning procedure performed according to commercial protocol as sealing of tuna can with industrial sealing machine, then sterilization step carried out 121°C for 117 minutes, finally cooled under top water immediately. Raw (G1) and processed samples (G2, G3) were stored at -80° C. Canned tuna samples (G4 and G5) stored at ambient temperature until analyses.

#### 2.2 DNA extraction

Raw, processed and canned tuna samples were pre-treated removing oils, spices and sauces by blotting with sterile filter paper and each sample was then subjected to extraction method according to commercial kit. The DNA extraction from canned tuna was carried out through the manufacturer's procedure and the following minor modifications. Initially, 20 mg sample,250  $\mu$ l Buffer ATL and 20  $\mu$ l Proteinase K were mixed and heated at 56°C until the tissue was entirely lysed. Then, the mixture was centrifuged at 12000 g for 30 second and supernatant transferred to another tube. 250  $\mu$ l extraction buffer and heated at 56 °C for 10 minutes. Afterwards, the mixture was mixed with 250  $\mu$ l binding buffer and vortexed for 15 second and transferred to spin columns. Then the spin column washed with AW1 (650  $\mu$ l) and AW2 (500  $\mu$ l). Finally, the purified DNA was eluted with 200  $\mu$ L pre-heated Buffer AE (37°C). The purified DNA was stored at -20°C until for further experiments

#### 2.3 Determination of DNA quality and amplificability

The quality (in terms of 260/230 and 260/280 ratios) and the concentration of DNA (ug/mL) were measured by means of a NanoDrop 1000 spectrophotometer (Thermo Scientific, Pittsburgh, PA, USA). The integrity and the amplificability of the gDNA was then evaluated by amplifying a 100 bp region from the 12S rRNA was targeted teleo primer (Teleo F: 5'ACACCGCCCGTCACTCT 3' and Teleo R: 3'CTTCCGGTACACTTACCATG 5') (Valentini et al., 2016).The reaction mixtures contained 2  $\mu$ L of template DNA, 10  $\mu$ L Master Mix(Thermo Scientific<sup>TM</sup> Maxima SYBR Green/ROX qPCR Master Mix (2X), 2  $\mu$ L of each primer, and 6  $\mu$ l Dna free water. The PCR was conducted using a StepOnePlus Real-Time PCR System (Applied Biosystems, Foster City, CA, USA) with the following program: an initial denaturation step at 95°C for 2 min, 35 cycles of 30 s at 94 °C, 30 s at 53 °C, and 60 s at 72 °C, followed by the final extension at 72°C for 10 min . Following to amplification, the melting curve analysis (MCA) was performed by cooling amplification products at 65°C for 95 s and then heating from 65°C to 95°C.

#### 2.4. Statistical analysis

All statistical analyses were performed using Software IBM SPSS version 16.0.For comparison of the yield of DNA extracted from all tuna sample (raw, processed and canned), a two-way cross-classification analysis of variance (ANOVA) was performed. Differences were considered statistically significant at a level of 5% (P< 0.05) All the DNA quality analyses were conducted in triplicate assays for each tuna sample.



#### **3 RESULTS AND DISCUSSION**

#### **3.1. Determination of DNA quality**

The results of extracted DNA from raw, processed and canned tuna samples are summarized in Figure 1 and Table 1. The DNA yield was calculated based on DNA concentration, initial tuna muscle weight and obtained the final volume. There were significant differences found among samples (P<0.05) in terms of DNA yield (Table 1, Figure 1)., DNA yield was significantly higher in raw (unprocessed) tuna when compared to processed and canned tuna groups. Similar results reported by (Piskatá and Pospí-šilová, 2016) who found the higher DNA yield from raw fish than processed fish sample. Cooking and soaking in brine solution cause to degradation of DNA from tuna sample. DNA yield was determined as 806.1 ug/uL in raw material, following the cooking process, the DNA yield reduced to 485.20 ug/uL and after soaking in brine solution prior to canning process, the DNA yield was determined as 301.8 ug/uL. Canning process reduced the DNA yield dramatically. The DNA yield was determined as 15.4 ug/uL and 20.6 ug/uL from canned tuna with olive oil and sunflower oil filling medium, respectively. This significant decline can be explained by the canning process include pressure and thermal steps which effect the DNA quality. Johny and Bhat (2017) highlighted that canning process involves mechanical, thermal and chemical treatments that have detrimental effects on the quality and the integrity of DNA, and, in turns, its amplificability. It is known that most of the processing methods cause to degradation of DNA, for instance, thermal processing of meat to at 121°C for 10 min, reduce to DNA length up to 300 bp (Levin et al., 2018). In the canned tuna sample groups, there were no significant differences from DNA yields based on can-filling mediums.

Tuna type		Quality assesment of muscle	
	DNA Yield (ug/uL)	Purity (A260/A280)	Chemical contamination (A260/A230)
G1	$806.1 \pm 0.13^{\circ}$	2.13±0.11 <sup>b</sup>	2.13±0.11 <sup>b</sup>
G2	$485.2\pm0.21^{\circ}$	$1.75{\pm}0.05^{\rm ab}$	$2.10{\pm}0.07^{b}$
G3	$301.8\pm0.12^{\mathrm{b}}$	$1.85{\pm}0.01^{a}$	$2.21{\pm}0.09^{a}$
G4	$15.4\pm0.00^{\rm a}$	1.00±0.03ª	$0.47{\pm}0.04^{b}$
G5	$20.6\pm0.00^{\rm a}$	$1.33{\pm}0.02^{a}$	$0.44{\pm}0.05^{\circ}$

Table 5: Quality differences among the tuna groups

Data are expressed as mean value  $\pm$  standard deviation of triplicates. Values followed by different letters indicate significant differences (P<0.05) Values in a same column followed by different numbers indicate significant differences of the parameter with respect to Groups: G1: Raw tuna muscle, G2:pre-cooked tuna sample, G3: soaked in brine solution prior to canning process, G4: canned tuna with olive oil medium, G5: canned tuna with sunflower oil



# International Conference on RAWMATERIALSTO PROCESSED FOODS

03-04 June 2021, Turkey

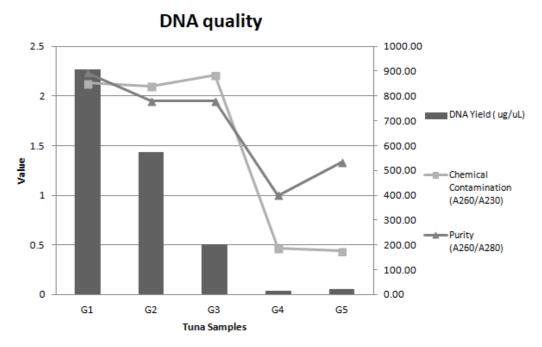


Figure 3: Variation of DNA Quality Parameters During The Canning Process

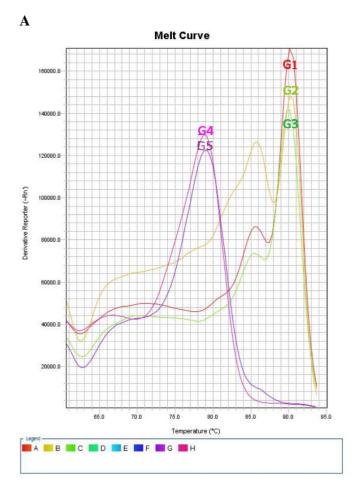
The purity of the gDNA is another parameter for the achievements of PCR amplification and sequencing processes. The purity of DNA evaluated with A260/A280 ratio. There were some significantly differences were observed among groups (Table 1). While the highest and the lowest values obtained in raw tuna muscle and canned tuna with sunflower oil with 2.13 and 1.00., respectively, the optimal purity value was detected from tuna soaked in brine solution with 1.85. The commonly accepted optimal range for purity ratio is between 1.8-2.0 (Piskata et al., 2017). Canning process also reduce to purity rate of DNA; 1.00 to 1.33. Canning process and treating with oil changed the quality of DNA.

Another important parameter for the achievements of DNA based analyses is the A260/A230 ratio, which accepted a sign of organic contaminants such as carbohydrates and salts. The highest contamination rate determined from tuna soaked in brine solution prior to canning process. These differences could be explained by treatment with salt. This rate should be between from 2.0 to 2.2 (Lucena-Aguilar et al., 2016). The lowest contaminant values observed from canned tuna samples as 0.47 and 0.44 for canned tuna in olive oil and sunflower oil, respectively. This significantly decline can be explained by to the thermal process during canning

#### 3.2. Determination of DNA degradation

DNA degradation among to raw, processed and canned tuna samples detected by threshold cycle (Ct) value. As seen in Figure.2; the significant differences in terms of melting curve and Ct values were determined. These differences could be explained by thermal processing of raw material. Findings DNA fragmentation and amplicability of DNA resulted by autoclaving (Ballari and Martin, 2013). The degradation of DNA is one of the issues that reduce the reliability of analyses. With changed demand and technological advance, several kind of processed food products have become more popular than fresh, especially canned food. The achievements of food tracebility driven by the DNA quality, yield and degradation level.





B

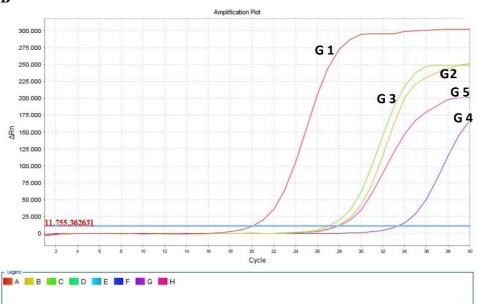


Figure 2: Melting Curve (A) and Amplicon plots(B) of Tuna Sample during The Canning Process

#### **4** CONCLUSION

The results of this research clearly show that how the quality, yield and degradation level of DNA change



depending on thermal processing, treatment with salt and canning process with different can-filling medium. Besides canning of food items is very popular in food industries, several processing methods include thermal and/or pressure application, these findings could be other canned products, useful for future research and industry in relevant area.

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## Production of Cocoa Powder with Low Protein Content

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#### ABSTRACT

Phenylketonuria (PKU) is an inherited metabolic disease caused by the lack or absence of phenylalanine hydroxylase (PAH) enzyme, enabling the conversion of phenylalanine to tyrosine. Since the PAH enzyme is not active in PKU patients, the accumulation of phenylalanine and its derivatives in the body causes mental and neurological development retardation. The aim of this study was to produce low-protein cocoa powder as an alternative low-protein food for PKU patients who need to follow a strict diet. First of all, defatted cocoa powder was suspended in distilled water (1:10, w/w). To increase the solubility of the cocoa solution, amylase was added in 1:10 enzyme: substrate ratio and the reaction continued for 3 hours after the temperature was adjusted to 50°C. Heat shock treatment was applied by freezing the suspension followed by contact with a hot aqueous solution at 95°C. The heat shock was allowed to continue until the temperature stabilized. Then, the suspension was boiled under the condenser for 3 hours and centrifuged at 25°C for 15 min to remove the insoluble part. The degrading enzymes, flavourzyme and alcalase, were added to the soluble part, respectively and enzymatic treatment proceeded 3 hours for each enzyme. The reaction was stopped by heating at 95°C for 5 min. After centrifugation, the pH of the solution was adjusted to 3.5 using 2M  $H_3PO_4$  solution to precipitate proteins, followed by centrifugation at 13,130 g for 10 min. The pH increased back to 7.0 using 2M Ca(OH)<sub>2</sub>. After the solution was dried, the solubility and protein content was detected. According to the results, the solubility of cocoa increased by more than 50% and the protein content decreased by 40%. Although the protein content was highly reduced with this study, different methods can also be tried for PKU patients to be fed with low protein-based foods.

Keywords: Cocoa, Low protein, Phenylketonuria



## FTIR based Chemometric Analysis of Bioactive Compounds of Peach Juice during Thermal Treatment

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#### ABSTRACT

Fruit juices contain many bioactive compounds such as phenolic compounds and vitamin C that have positive effects on health. Peach (*Prunus persica* L.) is one of the most popular fruits due to its flavor and nutritional value and it has been ranked the first among fruits that are processed into the nectar in Turkey. Thermal treatment is the main technique to obtain a safe product in the fruit juice industry for inactivating microorganisms and enzymes. However, bioactive components of fruit juices have been adversely affected by the heat. Fourier-transform infrared (FTIR) spectroscopy with chemometrics has been used as a new, rapid and efficient method to estimate the concentrations of the bioactive compounds and antioxidant activity of foods in recent years. In this study, FTIR spectroscopy with chemometrics was used to evaluate the bioactive compounds and antioxidant activity of peach juices during thermal treatment. The control and thermal treatment were differentiated by using principle component analysis (PCA), and models that correlated total phenolic content, antioxidant activity and vitamin C with infrared spectra were constructed by using partial least square (PLS). FTIR can be used as a reliable method to determine the bioactive compounds and antioxidant activity in peach juice during thermal treatment.

Keywords: FTIR, Peach Juice, PCA, PLS

#### INTRODUCTION

Fruit juices contain many bioactive compounds such as phenolic compounds, carotenoids and vitamin C. Fruit juices have been consumed in increasing amounts all over the world. Peach (*Prunus persica* L.) is one of the most important fruits which is processed to pure in the juice industry in Turkey (Jordão, 2018). Peach has been reported as a good source of bioactive compounds such as ascorbic acid and phenolic compounds which are well known for their health-promoting effect (Liu et al., 2015).

In the fruit juice industry, thermal treatment is generally applied to products for inactivating microorganisms and enzymes. However, heat has negative effects on the nutritional quality of fruit juices. In many research, the effect of thermal treatment on bioactive compounds in peaches have been studied and it was reported that heat may cause changes in the phenolic composition (Oliveira et al., 2012; Oliveira et al., 2014; Oliveira et al., 2015; Oliveira et al., 2016).

Fourier transform infrared (FTIR) spectroscopy has already been used as an easy, rapid and efficient method to estimate changes in bioactive compounds and antioxidant activity of food extracts. Moreover, FTIR spectroscopy coupled with chemometrics was used to build a relationship between chemical changes with infrared data (Baltacıoğlu et al., 2021).

In this study, peach juice bioactive compounds and antioxidant activity were investigated during thermal treatment by using FTIR spectroscopy. Principle component analysis (PCA) was used to differentiate the control and thermal treated juice sample and partial least square (PLS) was used to construct models that correlated bioactive compounds and antioxidant activity with infrared spectra.



#### MATERIAL AND METHOD

#### Material

Freshly collected peaches (*Prunus persica* L. cv. Monroe), that were from Niğde Ömer Halisdemir University, Faculty of Agricultural Sciences and Technologies, Research and Application Fields, were washed and dried. After that the seeds were discarded by cutting fruits in two pieces. Fruit juices were obtained by using juice extractor (K 1579 Y, Arcelik, Turkey). Extracted juice was separated into small volumes and one of them was left as a control.

#### **Thermal Treatment at Ambient Pressure**

Thermal treatment of peach juice was performed at 70 °C for 10 min. 200 ml of peach juice was placed in a glass vessel (7 cm diameter and 9.5 cm long) were heated in a water bath (Yüksel Kaya Makine, YKM-AS209, Turkey) that was temperature controlled. Laboratory type mechanical stirrer (Isolab, Germany) was used to stir juice during the thermal treatment to provide uniform distribution of the temperature in the sample. The temperature of samples was controlled by thermocouple during the thermal treatment and the time was started when the set temperature was reached. The samples were immediately transferred to an ice water to stop thermal inactivation instantaneously. All the experiments and measurements were repeated in triplicate.

#### **Determination of Total Phenolic Content**

The Folin–Ciocalteu colorimetric method was used to measure total phenolic content of samples (Singleton and Rossi, 1965). For this purpose, 100  $\mu$ L of extract (phenolic compounds were extracted from samples with methanol solution (80%, v/v) containing 1% hydrochloric acid (37%) at a ratio 1/5) was mixed with 0.75 mL of Folin–Ciocalteu reagent (10%) and kept at room temperature for 5 min. After that 0.75 mL of sodium bicarbonate (75 g/L) was added and the mixture was hold on at room temperature for 60 min. Absorbance of the samples was measured at 725 nm. Gallic acid was used as a standard and different concentrations of gallic acid solutions were prepared for the calibration curve. All determinations were carried out in triplicate, and results were represented as milligrams of gallic acid equivalent (GAE) per kilogram of juice.

#### **Determination of Antioxidant Activity**

Antioxidant activity of the extracts was determined according to the method of Blois (1958) by using DPPH (2,2-diphenyl-1-picrylhydrazyl). The daily prepared 3.9 mL of DPPH solution (0.1 mM) in methanol (80%) was mixed with 100  $\mu$ L of fruit juice extracts, prepared at different concentrations. After incubated for 30 min at room temperature, absorbance was recorded at 517 nm. For the blank sample, 100  $\mu$ L of methanol (80%) was mixed with the DPPH solution and its absorbance was measured at the same nm. The experiment was carried out in triplicate. Radical scavenging activity was calculated using the following formula:

% inhibition = [(Absorbance of the blank sample - Absorbance of the fruit juice extract)/ (1)  
Absorbance of the blank sample] 
$$\times$$
 100

Concentrations of the extracts were plotted against the % inhibition values in order to obtain the  $EC_{50}$ , defined as the amount of antioxidant needed to decrease the initial DPPH concentration by 50%.



The ascorbic acid contents were determined by using HPLC (Shimadzu LC-20A/Prominence), equipped with a 20- $\mu$ L injection loop (Shimadzu CBM-20Alite System Controller), UV-VIS detector (Shimadzu SPD-20A), a column oven (Shimadzu CTO-10AS VP) at 25 °C, and an operating system (LC-Solution) according to the method reported by Abid et al. (2014) with slight modifications. For the extraction of ascorbic acid, 5.0 mL of juice sample was mixed with 5 ml of orthophosphoric acid (2.5%) in a tube. After the mixture was centrifuged (6500 rpm for 5 min at 4°C), 0.5 ml aliquot of the supernatant was transferred to a 10 ml of centrifuge tube and completed to volume with orthophosphoric acid. The tube was shaken and filtered through syringe filters (0.45  $\mu$ m diameter). The sample (20  $\mu$ L) was injected into a C18 column (Supelco, 4.6 mm x 250 mm, 5  $\mu$ m). HPLC grade methanol (30%) was chosen as mobile phase and its flow rate was 1.0 mL/min. Eluate was detected at 280 nm. Standard ascorbic acid solutions were used to prepare a calibration curve and the results were shown as mg/L of peach juice.

#### Fourier-Transformed Infrared (FTIR) spectroscopy

After a few milligrams of lyophilized powder of peach juices were put on the attenuated total reflection (ATR) crystal, spectra of samples were recorded at room temperature (ca. 20 °C) over the frequency range of 4000–400 cm<sup>-1</sup> by using FTIR (Vertex 70, Bruker Optics, Germany) spectroscopy. It was taken 128 scans at a resolution of 2 cm<sup>-1</sup> for each spectrum. OPUS (v.8.1, Bruker Optics, Germany) software was used to collect and analyse the spectra. The spectrum of air was recorded as background and subtracted automatically by using the same software.

#### **Chemometric analyses**

FTIR spectra of control and thermal treated peach juice samples were discriminated by using Principal component analysis (PCA). Minitab 17 (Minitab Inc., State College, PA, USA) software was used to analyses the data. The total phenolic content, antioxidant activity and ascorbic acid of samples were predicted by using FTIR spectral data with Partial least squares (PLS) regression analysis. PLS analyses were performed by using Minitab 17 (Minitab Inc.) software. The FTIR spectral data were used as X variables (predictors), and each of the total phenolic content, antioxidant activity and ascorbic acid of samples was used as a Y variable (responses) to construct PLS models. Construction of the PLS models was done described by the method of Baltacioğlu et al. (2021). Chemometric models were established in the wavenumbers between 4000 and 400 cm<sup>-1</sup>.

#### **Statistical Analysis**

The data were analysed using Minitab 17 (Minitab Inc., State College, PA, USA) at 95% confidence interval, and the general linear model was used in the analysis of the data. Tukey's multiple comparison test was conducted to determine the differences between applications. Each experiment was repeated at least three times.

#### **RESULTS AND DISCUSSION**

#### **FTIR** analysis

As shown in Figure 1, the band observed at  $3270 \text{ cm}^{-1}$  was due to the O–H stretching of polysaccharides and N–H stretching of Amide A groups of proteins for FTIR spectra of samples (Baltacıoğlu et al., 2021b). The band located at 2924 cm<sup>-1</sup> corresponded to asymmetric stretching vibrations of CH<sub>2</sub> groups and the band determined after that at 2885 cm<sup>-1</sup> was related to symmetric stretching vibrations of CH<sub>3</sub> groups (Sun, 2008). It was thought that both of these bands were mainly related to unsaturated lipids while proteins, carbohydrates, and nucleic acids had little contribution to these bands. The absorption bands, observed in the region between 1800 and 1500 cm<sup>-1</sup> were reported as the carboxylic acid and the carboxylic ester groups of the pectin molecules (Sun, 2008). On the other hand, the contribution of proteins was observed in this region



because the bands observed between 1700 and 1600 cm–1 were assigned as the Amide I band and 1600–1500 cm–1 were related to the Amide II bands due to proteins (Baltacıoğlu et al., 2021b). The peaks, which were seen from the graph in the region from 1500 to 1200 cm<sup>-1</sup>, could be due to CH<sub>2</sub>, C-C-H and H-C-O deformations. Moreover, phenols, carboxylic acids and carbohydrates also contributed to this region due to their C-O stretching absorption and to the C-O-H bending (Coelho et al., 2020). The bands observed in the region between 1200 to 900 cm<sup>-1</sup> were assigned to the stretching vibrations of C-O and C-C bonds that were associated with sugars and organic acids (Miaw et al., 2018).

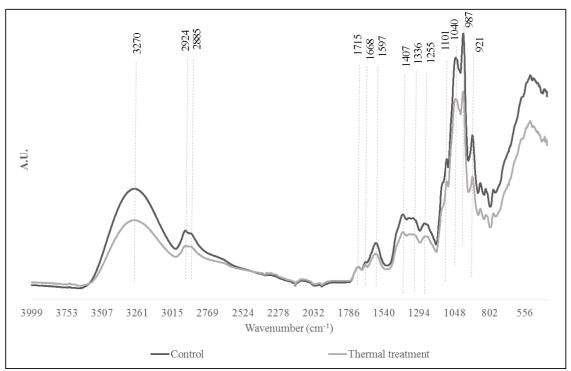


Figure 1: FTIR spectra of thermal treated peach juice samples

#### Chemometric analyses

PCA was performed to discriminate control and thermal treated peach juice by the help of IR spectra. The PCA plot was demonstrated in Figure 2 with two principal components accounting for 100% of the total variance. PC1 and PC2 explained 98.7% and 1.3% of the total variance, respectively. According to plots, it was easily observed differences between the control and thermal treated peach juice. It could be seen that control sample was differentiated from thermal treated samples according to PC1. In the same manner, PCA was used to differentiate between apple juice samples on the basis of applied heat treatment by using infrared data (Reid et al., 2005).



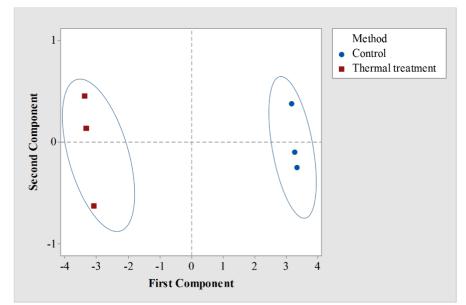


Figure 2: PCA score plot (PC1–PC2) of the FTIR spectra of thermal treated peach juice samples

PLS regression analysis results for the prediction of total phenolic content, antioxidant activity and ascorbic acid of peach juice using FTIR spectral data were given in Table 1. As shown in Table 1, rCV and rPre values were higher than 0.95, indicated good correlation within the calculated and reference values for the models. Additionally, the RMSEC and RMSEP values within each model had small values, and the differences between these values were also small. It can be said that the PLS models provided satisfactory predictions (Baltacioğlu et al., 2021). PLS regression plot of actual versus predicted total phenolic content analyses for juices using FTIR spectra was shown in Fig. 3A. PLS regression plot of actual versus predicted antioxidant activity analyses for juices using FTIR spectra was shown in Fig. 3C. High correlations between reference and IR predicted values in total phenolic content, antioxidant activity and ascorbic acid content could also be easily seen in the plots. Antioxidant activity and total phenolic content of tomato extracts has been successfully quantified using IR spectra combined with PLS regression (Baltacioğlu et al., 2021).

Parameters	Components	r <sub>CV</sub>	r <sub>Pre</sub>	RMSEC	RMSEP
Total phenolic content	4	0.9997	0.9798	1.366	1.222
Antioxidant activity	3	0.9999	0.9995	0.016	0.014
Ascorbic acid	1	0.9994	0.9959	0.591	0.529

Table1: PLS regression analysis for the prediction of bioactive compounds and antioxidant activity of thermal treated peach juices using FTIR spectral data



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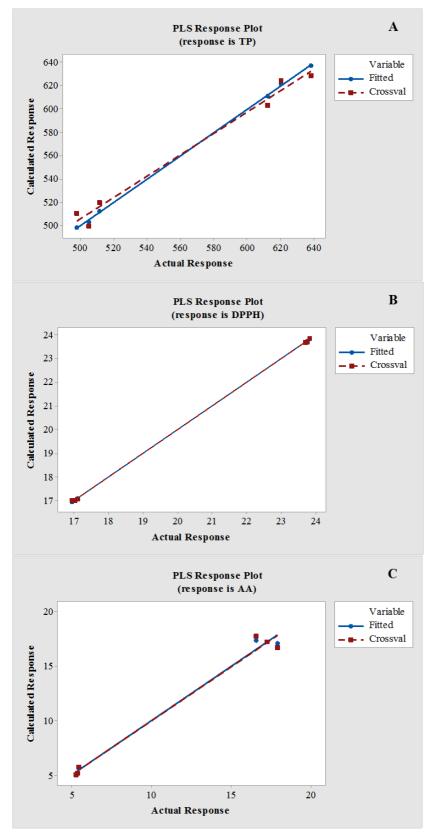


Figure 3: (A) PLS regression plot of actual versus predicted for total phenolic content using FTIR spectra (B) PLS regression plot of actual versus predicted for antioxidant activity using FTIR spectra (C) PLS regression plot of actual versus predicted for ascorbic acid using FTIR spectra



#### CONCLUSION

FTIR was firstly used to identify changes in functional groups in the thermal treated peach juices. FTIR based chemometric analyses was easily used to evaluate the bioactive compounds and antioxidant activity of peach juices during thermal treatment. PCA was used to discriminate the methods and models were constructed for determining bioactive contents and antioxidant activity of peach juices during thermal treatment as a function of the spectra by using PLS.

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## Optimization of Extraction Parameters to improve Cottonseed milk yield and reduce gossypol levels using Response Surface Methodology (RSM)

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#### ABSTRACT

The consumption of plant based milk products is gaining importance as a healthy alternative to dairy based milk for lactose intolerance people. Also, the nutraceutical functional ingredients of plant based milk like cottonseed milk confer potential health and functional benefits. This study aimed to optimize the extraction condition for extracting cottonseed milk by using an enzymatic assisted aqueous extraction technique (EAAE) and access the interaction of the gossypol-protein molecules by electrophoresis. The influence of enzymatic treatments and other parameters included protease enzyme concentration (0.5 to 1%), incubation temperature (30 to 55°C), pH (4.5 to 7.0) and hydrolysis time (120 to 360 min) on extraction yield, protein and gossypol content was studied by RSM. The optimized enzymatic treatment conditions were enzyme concentration of 0.50%, temperature 30°C, pH 7 and hydrolysis time 165.31 min with the desirability value of 0.825. The optimized EAAE cottonseed milk had a significant increase in extraction yield by 11.10% and protein content by 17.94%, while the gossypol content decreased by 45.61% with conventional extracted cottonseed milk (CECM). The less intensity of the protein bonds appeared in CECM with EAAE cottonseed milk through electrophoresis. The gossypol content in the optimized cottonseed milk met the permissible intake level of gossypol content as set by the USFDA is 450 mg/kg and FAO/WHO is 600 mg/kg.

Keywords: Cottonseed milk, Enzyme assisted extraction, Gossypol, Gossypol-protein interaction



## A study on the determination of various quality characteristics of astragalus honey obtained from different altitudes of Adana-Turkey

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#### ABSTRACT

In this study, the effects of altitude on the chemical and physicochemical properties and aroma compounds of astragalus honey from the different parts of Adana, Turkey, were investigated. As the altitude increased, the moisture content of honey samples decreased. Similarly, as the altitude increased, the acidity and the diastase activity of samples increased. These compounds were identified and quantified using the GC-MS-FID. It was determined, that with the increasing altitude, the total amount of the alcohol compounds decreased, and the total amount of the lactone compounds and the norisoprenoid compounds increased with the decreasing altitude. Moreover, 11 different aroma compounds with an odor activity value above 1 were detected. From these compounds, Limonene, (E)-Linalool oxide, Z-Linalool oxide, and acetic acid were determined as the odor active compounds in all of the honey samples. With this study, the altitude effect of honey on aroma compounds was revealed for the first time.

Keywords: Honey, Astragalus, Altitude, Aroma, GS-MS-FID.

#### **Pratical Applications**

Honey, produced by honey bees using many different herbal resources, is an important food item with its economic and nutritional value worldwide. Aroma compounds play an important role in the quality of honey, which is named according to the plant origin from which it is obtained. Although many scientific studies have been conducted on the aroma compounds of honey, no studies have been found on the effect of altitude on these compounds. The influence of altitude on astragalus honey was presented in this study, and thus the gap in the relationship between aroma compounds and altitude was scientifically filled for the first time.

#### **INTRODUCTION**

Honey is a natural, sweet, and viscous product produces by honey bees. Honey is an important food substance with an economic value around the world (Kaçaroğlu, 2011). According to FAO data, 1850868 tonnes of honey was produced in 2019, worldwide. After China, Turkey is the second producer country with a total of 109330 tonnes of annual honey production (Anonymous, 2021). Astragalus honey is being produced in numerous parts of Turkey where is rich in the means of endemic plant diversity. Astragalus plant is deployed in Anatolia up to approximately 3500 meters altitude (Kadioglu vd., 2008). Adana is an important region for its production of astragalus honey. Approximately 10% of the high-quality honey of Turkey is produced in Adana (Anonymous, 2019b).

When it comes to quality, properties such as color, taste, texture, and aroma are mentioned. The effect of the aroma on the quality of honey is crucial among these characteristics. In many studies on astragalus honey and the other honey types to date, aroma groups such as terpenes, norisoprenoids, lactones, alcohols, esters, aldehydes, and ketones were detected (ChangWei vd., 2018; Costa vd., 2019; Karabagias vd., 2018, 2020; Makowicz vd., 2018; Tanleque-Alberto vd., 2019; Uçkun, 2011). However, no studies focusing on the effect



of altitude on aroma compounds of any kind of honey have been encountered so far. Only a few studies focus on the effect of altitude on the chemical and physicochemical properties (Bouhala vd., 2020; Mohammed vd., 2017).

In the presented study, it is aimed to determine the effect of the altitude on aroma compounds as well as the chemical and physicochemical properties of astragalus honey obtained from the different altitudes (620 meters, 1050 meters, and 1700 meters) of the Adana region.

#### MATERIALS AND METHOD

#### Materials

Honey samples were obtained from honey producers as centrifugal honey (1 kg each) from the county of Feke (620 meters), Saimbeyli (1050 meters), and Tufanbeyli (1700 meters) of Adana province in 2017. All the chemical solvents and standard compounds were acquired from Sigma Aldrich (St. Louis, Missouri, USA) and Merck (Darmstadt, Germany).

#### Chemical and Physico-Chemical Analyses

The pollen analyses of honey samples were performed according to DIN 10760 (Anonymous, 2002a). For dry materials analyses, honey samples were weighed as 0.5-1 gr in aluminum crucibles. After that, the samples were dried at 60 °C until they reached the constant weight (Kurt & Yamankaradeniz, 1982). The electrical conductivity test was carried out according to DIN 10753 method for honey samples (Anonymous, 2000). The pH analyses were made by directly using a pH meter. Then, 5 gr of honey were dissolved in water and they were titrated with 0.1 N NaOH until the pH reached 8.2. Total acidity calculations were made by using the amount of NaOH used (Manzanares vd., 2011). The proline amount in the honey samples was determined quantitively by using the spectrophotometric technique (Horwitz, 2000). Diastase numbers were determined with the TS-3036 method. The water bath that was used for this purpose was set to 48 °C (Anonymous, 2002b).

#### **HMF** Analyses

HMF content in honey samples was determined by using Agilent 1100A model HPLC and Agilent 1100A model diode array detector (Zappalà vd., 2005). For HMF analysis ACE C18 column was used. The column flow rate was 0,5 ml/min, and the column temperature was 30 °C. As the mobile phase, MeOH/Acetic Acid/Water (20/2/78) was preferred. The injection volume was 20 µL, elution time was 20 min and the wavelength was 285 nm. The external standard method was used to calculate the amount of HMF in honey samples.

#### **Sugar Component Analyses**

The sugar (glucose, fructose, and saccharose) analyses in honey samples were made by using Shimadzu 10A model HPLC, Shimadzu RID-10A reactive index detector, and aminex column (Rudnitskaya vd., 2006). The column temperature was set to 30 °C and the flow rate was set to 1,3 ml/min. As the mobile phase, Acetonitrile/Water (80/20) was used. The injection volume was 10  $\mu$ L. The external standard method was used to calculate the amount of sugar component (glucose, fructose, and saccharose) in honey samples.

#### **Carbon Isotope and C4 Sugar Ratio**

13C/12C ratio and from this value C4 sugar content was determined by mass spectrometry. The honey samples were weighed 1 mg and put in tinnen capsules and closed tightly. As a standard, 1 gr of saccharose was weighed and put in tinnen capsules. To prepare a protein precipitate, 10-12 gr of honey samples were put in 50 ml centrifuge tubes and mixed with 4ml of distilled water. In another tube, 2 ml of 10% Na<sub>2</sub>WO<sub>4</sub> (Sodium tungstate) and 2 ml of 0.335 M H<sub>2</sub>SO<sub>4</sub> were mixed and added to the honey solution. To form a clouding and a precipitate afterward, they were put in the water bath at 80 °C. In case of no clouding, 2 ml more of 0.335 M H<sub>2</sub>SO<sub>4</sub> was added. After the precipitation, distilled water was added to the tubes until the total amount reached 50 ml. After that, the samples were centrifuged at 1500 rpm for 5 minutes and the clear



part was poured out. The remaining precipitate was diluted to 50 ml and centrifuged again 5 times. The precipitate was dried in the oven at 75 °C for 3 hours and 1 mg of dried protein was put into the tinnen capsule. Saccharose standard in the tinnen capsule, honey sample, and the protein precipitate was put in the THERMO IRMS (infrared-mass spectroscopy) and 13C/12C ratios were determined and from the acquired values, C4 sugar ratio was calculated (Çınar, 2010).

#### **Isolation of the Aroma Compounds**

The aroma compounds were isolated by using the liquid-liquid extraction method in the honey samples. The extraction processes were repeated for each sample at least three times with dichloromethane (CH<sub>2</sub>Cl<sub>2</sub>) solvent. 40 g of honey sample was diluted with 22 ml of water, then 40 ml of dichloromethane and 40  $\mu$ g 4-nonalol (internal standard) were added. After that, the mixture was poured into the 500 ml Erlenmeyer flask. This mixture was stirred at 4-5 °C for 40 minutes under the nitrogen atmosphere. Then the mixture was centrifuged at 9000 rpm for 15 minutes at 0 °C. At the end of this procedure, the organic phase consisting of the aroma compounds was taken and concentrated at 45 °C in Vigreux concentrator until 0,5 ml left. The concentrated extract was injected into the GC-MS-FID (Jerković vd., 2010; Uçkun, 2011).

#### **GS-MS-FID** Analyses

Agilent 6890N gas chromatography integrated with flame ionization detector (FID) and Agilent 5975B VL MSD mass spectrometry was used for identification and quantification of the aroma compounds. The separation of the aroma compounds was carried out by using a DB-WAX capillary column (30 m x 0.25 mm x 0.25 µm). The injector temperature was set to 220 °C and the detector temperature was set to 250 °C. Helium was selected as the carrier gas. The flow rate of helium was 3,3 ml/min. The injection volume was 3 µl. The ion source temperature of the mass spectrometry was kept at 250°C, ionization energy was kept at 70 eV, quadrupole temperature was kept at 120 °C and scanned with 1-second intervals between 29-350 mass/charge (m/e) The column temperature was increased from 40 °C for 4 minutes to 220 °C with a rate of 2 °C per minute, and then to 245 °C with a rate of 3 °C per minute and kept there for 20 minutes (Schneider vd., 1998, 2001). The identification of the peaks was conducted using the mass spectrometry device library (Wiley 7.0, NIST 98, and Flavor 2L), standards, and retention index of the aroma compounds. All the retention index values of the aroma compounds were determined by injecting a solution that consists of every alkane between C8-C36, under the aforementioned column and gas chromatography circumstances. Concentrations of the aroma compounds were calculated by the method of internal standards (Van Den Dool & Kratz, 1963).

#### **Calculation of Odor Activity Value**

Odor active compounds give food its characteristic fragrance. Various methods can be used to determine the odor active compounds. One of these methods is to determine the "Odor Activity Value" of the aroma compounds that have been determined in GC-MS. This value is calculated by dividing the concentration of the compound in the honey by the odor threshold value of that compound in water. The odor threshold values of aroma compounds in water were determined as a result of literature research. If the odor activity value is greater than 1, the compound is considered as an active compound, and the activity increases with the increasing value.

#### **Sensory Evaluation of the Honey Samples**

The sensory evaluation of the astragalus honey was performed with the help of a pre-made 10 cm scale conducted by a 9 people specialist panelist group, in two different methods (Uçkun, 2011). As a taste profile analysis, firstly, the panelists were asked to evaluate the samples in manners of color, viscosity, flavor, taste balance, crystallization, and general impressions. Later, they were asked to do an aroma profile analysis, evaluating the odor in honey samples if it is flowery, caramel, fresh, fruity, and foreign odor (medication,



etc.)

#### **Statistical Analyses**

In the statistical evaluation of the research results, XLStat (2020) (Addinsoft, New York, NY, USA) package software was used. Concentrations and the standard deviations (SD) were determined by using MS Office Excel. To estimate the differences, variance analysis Duncan's multiple comparison test was used. Meaningful differences are accepted as p < 0.05. Also, a principal component analysis (PCA) was made.

#### **RESULTS AND DISCUSSION**

#### The Chemical and Psycho-chemical Composition of Honeys

The physical and chemical analysis results of the astragalus honey samples were given in Table 1.

	620 meters	1050 meters	1700 meters
Pollen Amount %	67.47 Astragalus	63.46 Astragalus	49.29 Astragalus
	5.26 Scorzonera	12.50 Terebinth	28.45 Eucalyptus
	5.26 Plantago	8.33 Onobrychis	12.12 Linaria
	5.26 Onobrychis	8.33 Centaurea	5.45 Sunflower
	5.26 Maple	7.38 Trifolium repens	4.69 Other
	5.26 Cedrus		
	6.23 Other		
рН	3.99ª	3.98ª	3.35 <sup>b</sup>
Total Acidity (meq/kg)	18.21 <sup>c</sup>	22.14 <sup>b</sup>	26.00 <sup>a</sup>
Dry Material (%)	82.94ª	83.06 <sup>a</sup>	84.13 <sup>a</sup>
Humidity (%)	17.06 <sup>a</sup>	16.94ª	15.87ª
Conductivity (mS/cm)	0.34 <sup>c</sup>	0.51ª	0.39 <sup>b</sup>
Proline (mg/kg)	434.33°	802.00 <sup>a</sup>	644.67 <sup>b</sup>
Fructose + Glucose (g/100g)	66.20 <sup>a</sup>	66.89 ª	67.42 <sup>a</sup>
Fructose / Glucose	1.17 <sup>a</sup>	1.18 <sup>a</sup>	1.19 <sup> a</sup>
Saccharose (g/100g)	0	0	0.82 <sup>a</sup>

Table 1. Chemical and Physico-chemical Properties of the Astragalus Honey Samples



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HMF (mg/kg)	18.32°	32.9 <sup>b</sup>	38.6ª
Diastase Number	13.04 <sup>a</sup>	13.14 <sup>a</sup>	13.33 °
δ <sup>13</sup> Cprotein - δ <sup>13</sup> Choney	0.53 <sup>a</sup>	0.13 <sup>b</sup>	0.20 <sup>b</sup>
C4 %	0	0	0

a, b, c: The difference between the values shown with different letters in the same column is statistically significant (p < 0.05).

The moisture contents of the honey samples were determined between 15.87% and 17.06%. It is stated in Codex Alimentarius that the moisture level should not be more than 20% in the flower honey (Anonymous, 2019a). On the other hand, the moisture level slightly decreased in the astragalus honey samples as the altitude increases. Batu et al. stated in one of their studies that the kinds of honey gathered from the higher altitudes have a lesser level of moisture (Batu vd., 2013). In the honey samples, pH was measured between 3.35 and 3.99, and the total acidity is determined between 18.21 meq/kg and 26 meq/kg. The acidity level of the astragalus honey increased according to the increasing altitude. These results are supported by different studies (Bouhala vd., 2020; Korkutal & Bahar, 2016; Mohammed vd., 2017). Also, addition, pH and total acidity levels of all three samples are in agreement with the literature data (Tanleque-Alberto vd., 2019; Tornuk vd., 2013). When the pollen contents of the three samples were analyzed, it was seen that the dominant pollen belongs to the astragalus. In the astragalus honey that was gathered from 1700 meters' altitude, the seconder pollen was determined to be eucalyptus with a rate of 28.45%. Eucalyptus trees do not grow at an altitude of 1700 meters. Hence, it is thought that the eucalyptus as seconder pollen comes from the hive before the beekeepers carried it to the altitude of 1700 meters so that the bees do not starve in the process of carrying. It is stated in Codex Alimentarius that the electrical conductivity of the kinds of honey can be 0.8 mS/cm at most (Anonymous, 2019a). According to this, the electrical conductivities of the samples are found to be between 0.34 mS/cm and 0.51 mS/cm and are lower than the mentioned value. The proline values in the honey samples are found to be between 434,33 mg/kg and 802,00 mg/kg and these values are agreed with the literature (Manzanares vd., 2011; Meda vd., 2005; Terrab vd., 2003). According to Codex Alimentarius, the invert sugar content of honey should be at least 60g/100g (Anonymous, 2019a). It is detected that the invert sugar level in astragalus honey samples changes between 66.20 g/100 g and 67.42 g/100 g (p>0.05) and these values are higher than the values mentioned in Codex Alimentarius. The Saccharose content of honey in Codex Alimentarius is given as a maximum of 5g/100 g (Anonymous, 2019a). While there was no saccharose in the samples from the altitudes of 620 and 1050 meters, there is a rate of 0.82 g/100 g saccharose in the honey sample gathered from the altitude of 1700 meters. After these values, the saccharose level was found to be lower than the permitted level. The HMF amount is a limited maximum of 40 mg/kg in flower honeys in Codex Alimentarius (Anonymous, 2019a). The HMF amount in the astragalus honeys (18.32 - 38.6 mg/kg) are determined to be lower than the values that are in the codex. Also, all the HMF levels of the samples are determined to be in harmony with the other data in the literature (Kaçaroğlu, 2011; Uçkun, 2011). It is reported that the diastase activity of the honeys should be at least 8 in Codex Alimentarius (Anonymous, 2019a). In general, the diastase activity of the astragalus honey samples (13.04-13.33) are found to be more than that is in the codex and that these values are agreed with the literature (Bergamo vd., 2019; Gangwar & Gebremariam, 2010; Gomes vd., 2010; Kaçaroğlu, 2011). In a former study it is concluded that the value difference between the values of protein in honey and values of delta C13 in



crude honey should be -1.0 or higher. In another study it is said that the C4 sugar content, that is calculated from the levels of protein in honey and of delta C13 in crude honey, can be maximum 7% (Padovan vd., 2003, 2007). In all the samples of astragalus honey, mentioned values are met; this means that sugar cane or corn syrup was not used to make the honeys.

#### The Aroma Compounds of Astragalus Honeys

The aroma compounds of the astragalus honey samples are given in Table 2. The sum of the aroma compounds of the astragalus honey samples gathered from altitudes of 620, 1050, and 1700 meters were 14600.50  $\mu$ g/kg, 29882.90  $\mu$ g/kg, 16372.89  $\mu$ g/kg, respectively. In the honey samples obtained from altitudes of 620, 1050, and 1700 meters; 53, 50, and 49 different aroma compounds identified, respectively; and these compounds are gathered under 10 different groups, such as aldehydes and ketones, alcohols, esters, furans and pyrans, lactones, norisoprenoids, terpenes and terpenoids, volatile acids, volatile phenol compounds, and volatile sulfur compounds. Costa et al., in one of their studies on unifloral Melipona honey, determined 42 different aroma compounds and gathered them in 12 groups (Costa vd., 2019).

#### **Aldehydes and Ketones**

Even though aldehydes and ketones are present in the honey with low concentrations, they can be characterized by their strong odors (Tian vd., 2016). Among the aldehydes and ketones, 4-hydroxy4-methyl-2-pentanone was the dominant compound in the honey samples gathered from altitudes of 620 and 1700 meters. Whereas 1- hydroxy-2-propanone was the dominant compound in the honey sample obtained from the altitude of 1050 meters. The ratio of the 4-hydroxy-4-methyl-2-pentanone compound was 53.5%, 23.8%, and 22.4%, respectively with the increasing altitude and the amount of these compounds decreases with the increasing altitude (p<0.05). Similarly, the amount of benzaldehyde lowers with the increasing altitude (p<0.05).

Generally, the sum amount of the aldehydes and ketones did not change with the increasing altitude. Although the amount of 3-hydroxy-2-butanone and 1-hydroxy-2-butanone increased with increasing altitude, the amount of 4-hydroxy-4-methyl-2-pentanone and benzaldehyde decreased.

#### Alcohols

The alcohol compounds in the honey come from the reductase enzymes catalyzing the aldehydes by the bees and the contaminant microorganisms or the oxidative decomposition of the lipids (Moreira vd., 2010). The alcohol content in the astragalus honey obtained from the altitude of 1050 meters was found to be more than the other samples. Remarkably, the proportional decrease in the alcohol compounds got higher with the increasing altitude.

In a former study, it is concluded that the 2-methyl-2-butanol, which was characterized by the fragrance of camphor tree has a 1200  $\mu$ g/kg odor threshold value (Forero vd., 2015). The amount of this compound in the honey samples is 1147.22  $\mu$ g/kg, 2090.64  $\mu$ g/kg, and 1251.46  $\mu$ g/kg, respectively to the altitude. The odor activity values of the 2-methyl-2-butanol were 2.05 for the honey sample obtained from the altitude of 1050 meters and 1.23 for the honey sample gathered from the altitude of 1700 meters. However, the odor activity values of this compound were less than 1 for the honey sample obtained from the altitude of 620 meters. According to these values, it could be said that with the increasing altitude, the influence of 2-methyl-2-butanol on the odor characteristics of the astragalus honey also increased concerning honey sample obtained from the altitude of 620 meters.



The odor threshold value for 2-phenyl-ethanol, characterized by the fragrance of rose or honey (Forero vd., 2015), is reported as 1000  $\mu$ g/kg (Leffingwell & Leffingwell, 1991). The amounts of 2phenyl-ethanol were respectively detected as 122.29  $\mu$ g/kg, 130.19  $\mu$ g/kg, and 117.76  $\mu$ g/kg, concerning the increasing altitude (p>0.05). The odor activity value of 2-phenyl-ethanol was determined to be less than 1 in each of the three samples.

It was established that with the increasing altitude, the content of the alcohol compounds in the sum of all the aroma compounds proportionally decreased. However, it was recorded that the 3-methyl- 1-butanol and benzyl alcohol compounds increased with the altitude.



Table 2. Aroma Compounds of Astragalus Honey Samples

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	Aroma Compounds	LRI	Odor Threshold Value (µg/kg)	References	Quantity 620 m (µg/kg)	Odor Activity Value	Quantity 1050 m (μg/kg)	Odor Active Value	Quantity 1700 m (μg/kg)	Odor Active Value	Odor Description	References	References Identification
	Aldehydes and Ketones												
1	3-Hydroxy-3-Methyl-2- Butanone	1236			ри		$61.9 \pm 3.04^{a}$		$40.33 \pm 3.51^{b}$				LRI,MS,Std
5	3-Hydroxy-2-Butanone	1275	800 I	(Leffingwell & Leffingwell, 1991)	$122.58\pm5.29^{b}$	$\overline{\vee}$	$130.01\pm9.7^{\text{b}}$	$\overline{\vee}$	$479\pm42.88^{a}$	$\overline{\vee}$	Fatty	(Niu vd., 2011)	LRI,MS,Std
б	1-Hydroxy-2-Propanone	1288			$273.36\pm14.31^{b}$		$524.82\pm 26.05^{a}$		$197.01 \pm 17.63^{\circ}$		Alcoholic, Fruity, malty	(Niu vd., 2011)	LRI,MS,Tent
4	4-Hydroxy-4-Methyl-2- Pentanone	1356			$624.13 \pm 29.5^{a}$		$285.87\pm26.82^{b}$		$256.6\pm20.66^{\flat}$				LRI,MS,Tent
ŝ	1-Hydroxy-2-Butanone	1363			$73.99 \pm 3.97$		$90.43\pm6.48^{\rm b}$		$127.03 \pm 11.32^{a}$				LRI,MS,Std
9	Benzaldehyde	1507	350	(Bonvehí, 2005)	$34.19\pm2.3^{\mathrm{a}}$	$\overline{\vee}$	$28.29\pm1.24^{b}$	$\overline{\vee}$	$20.57 \pm 1.98$	$\overline{\vee}$	Fruity, Almond	(Niu vd., 2011)	LRI,MS,Std
L	3-Methyl-1,2- cyclopentanedione	1824			$37.26\pm0.2^{\rm a}$		ри		$24.37\pm1.21^{b}$				LRI,MS,Tent
8	6,10,14-trimethyl-2- pentadecanone	2130			ри		$79.4\pm1.98^{\rm a}$		рп				LRI,MS,Tent
	Total				1165.51		1200.72		1144.91				



(Conti	(Continued Table 2)												
	Aroma Compounds LRI	LRI	Odor Threshold Value (µg/kg)	References	Quantity 620 m (µg/kg)	Odor Active Value	Quantity 1050 m (µg/kg)	Odor Active Value	Quantity 1700 m (μg/kg)	Odor Active Value	Odor Description	References	
	Alcohols												
1	2-Methyl-2-Butanol 1018	1018	1200	(Buttery vd., 1988) 1147.22 $\pm$ 111.9 <sup>b</sup>	1147.22 ± 111.9 <sup>b</sup>	$\overline{\vee}$	$2090.64 \pm 146.41^{\rm a}$	2.05	$1251.46 \pm 124.92^{b}$	1.23	Woody, camphor tree	(Lewis, 2016)	LRI,MS,Std
2	3-Penten-2-ol	1171	400	(Guclu vd., 2016)	$74.45\pm4.17^{\mathrm{b}}$	$\overline{\vee}$	$150.35\pm9.74^{\mathrm{a}}$	$\overline{\vee}$	$80.01\pm2.43^{\rm b}$	$\overline{\vee}$	perfume, woody	(Guclu vd., 2016)	LRI,MS,Std
3	3-Methyl-1-Butanol	1210	250	(Fariña vd., 2015)	$63.75\pm4.06^{\text{b}}$	$\overline{\vee}$	$82.66\pm6.58^{a}$	$\overline{\vee}$	$91.34 \pm 9.08^{a}$	$\overline{\vee}$	fruity, wine	(Pino & Febles, 2013)	LRI,MS,Std
4	4-Methyl-2-pentanol 1303	1303			$136.99\pm11.28^{\text{b}}$		$222.94 \pm 18.8^{\mathrm{a}}$		$136.11\pm13.08^{b}$				LRI,MS,Std
283	58 28 28 28	1321			$55.54\pm3.29^a$		ри		ри				LRI,MS,Std
6	1-Penten-3-ol	1618			$71.81\pm2.95^{b}$		pu		$90.89\pm5.72^{\mathrm{a}}$				LRI,MS,Tent
Γ	1,2-Ethanediol	1626			$1605.88 \pm 96.24^{b}$		$2691.63 \pm 187.28^{a}$		$1057.07 \pm 79.76^{\circ}$		Sweet	(Ruth, 1986)	LRI,MS,Std
8	2- (2-butoxyethoxy) -ethanol	1794			ри		$33.31\pm2.42^{\rm a}$		$16.24\pm1.38^{b}$				LRI,MS,Tent
6	Benzyl alcohol	1871	10000	(Fariña vd., 2015)	$35.11 \pm 1.87^\circ$	$\overline{\vee}$	$66.55\pm3.91^{b}$	$\overline{\vee}$	$246.36 \pm 3.5^{a}$	$\overline{\vee}$	Flower, sweet	(Niu vd., 2011)	LRI,MS,Std
10	2-Phenyl-ethanol	1908	1000	(Leffingwell & Leffingwell, 1991)	$122.29\pm1.26^{\rm ab}$	$\overline{\vee}$	$130.19\pm 6.51^{a}$	$\overrightarrow{\vee}$	$117.76\pm6.11^{b}$	$\overline{\vee}$	Rose, honey	(Forero vd., 2015)	LRI,MS,Std
	Toatal				3313,04		5468,27		3087,24				



(Continued Table 2)												
Aroma Compounds	LRI	Odor Threshold Value (μg/kg)	References	Quantity 620 m (µg/kg)	Odor Activity Value	Quantity 1050 m (µg/kg)	Odor Activity Value	Quantity 1700 m / (µg/kg)	Odor Activity Value	Odor Description		
Esters												
1 Methyl-2-furoate	2001			$374.22 \pm 6.54$		$984.27 \pm 27.49^{a}$		$602.93\pm22.09^{b}$		fruity (U	(Uçkun & Selli,	LRI,MS,Std
2 Ethylhexadecanoate	2260	>2000	(Fariña vd., 2015)	$45.58\pm0.92^{b}$	$\overline{\vee}$	$53.41\pm3.66^{a}$	$\overline{\vee}$	$16.78\pm0.15^{\rm e}$	$\overline{\vee}$			LRI,MS,Std
Total				419.8		1037.68		619.71				
Furans and Pyrans												
1 Furfural	1451	720	(Fariña vd., 2015)	$186.99\pm10.9\mathbf{\hat{z}}$	$\overline{\vee}$	$739.51 \pm 33.57^{a}$	1.02	$288.61\pm28.28^{b}$	$\overrightarrow{\vee}$	Sweet, fusel ( alcohol, toasted bread	(Fariña vd., 2015)	LRI,MS,Std
2 1- (2-Furanyl) - Etanone	1491			$71.84\pm0.84^{\text{b}}$		$116.32 \pm 7.62^{a}$		$46.02\pm4.34$				LRI,MS,Tent
38 5-Methyl-2- 38 Furancarboxaldehyde	1561			$42.72\pm2.4^{b}$		$80.43\pm4.87^{a}$		$38.04\pm3.77^{\mathrm{b}}$				LRI,MS,Tent
4 Furfuryl Alcohol	1654	2000	(Escudero vd., 2007)	$148.48\pm4.68^{b}$	$\overline{\vee}$	$190.96 \pm 11.37^{\rm a}$	$\overline{\vee}$	$79.41\pm6.95$	$\overline{\nabla}$	Caramel, sweet (Niu vd., 2011)		LRI,MS,Std
5 2,5- Furan dicarboxaldehyde	1970			ри		$164.97\pm8.14^{\mathrm{a}}$		$56.37\pm4.91^{b}$				LRI,MS,Std
2,3-dihydro-3,5- dihydroxy-6-methyl- 4H-pyran-4-one-4H- piran-4-on	2269	35000	(Buttery vd., 1999)	$75.88 \pm 5.73^{b}$	$\overline{\vee}$	$169.45 \pm 7.88^{a}$	$\overline{\vee}$	ри				LRI,MS, Tent
7 5-Hydroxy-methyl- furfural	2498	10000	(Escudero vd., 2 2007)	$2882.23 \pm 148.07^{b}$	$\overline{\vee}$	$8733.63 \pm 729.54^{a}$	$\overline{\vee}$	$3084.24 \pm 159.78^{b}$	$\overline{\vee}$			LRI,MS,Std
Total				3408.14		10195.27		3592.69				



	LRI,MS,Std	LRI,MS,Std	LRI,MS,Std	LRI,MS,Tent	LRI,MS,Tent	LRI,MS,Tent			LRI,MS,Tent	LRI,MS,Std	LRI,MS,Std	LRU,MS,Std	
по	d (Fariña vd., 2015)		(Fariña vd., 2015)								(Fariña vd., 2015)		
Odor Description	Toasted burned		Toasted bread, smoked								Honey, apricots		
Odor Activity Value	$\overline{\vee}$		$\overline{\vee}$										
Quantity 1700 m (µg/kg)	$90.24 \pm 4.71^{\circ}$	pu	$151.22\pm0.94^{\circ}$	pu	$69.76\pm6.6^{a}$	$44.76 \pm 3.99^{a}$	355.98		$47.25 \pm 4.59$	$101.76\pm7.06^{b}$	$221.2 \pm 11.96^{a}$	pu	370.21
Odor Activity Value	$\overline{\nabla}$		$\overline{\vee}$										
Quantity 1050 m (µg/kg)	$111.06 \pm 1.2^{\mathrm{a}}$	pu	$209.45 \pm 9.15^{a}$	$33.11\pm2.47^{\text{b}}$	pu	pu	353.62		$81.17 \pm 3.52^{b}$	pu	pu	pu	81.17
Odor Activity Value	$\overline{\vee}$		$\overline{\vee}$										
Quantity 620 m (μg/kg)	$96.76\pm2.62^{b}$	$47.66\pm1.08^{a}$	$179.87 \pm 10.15^{b}$	$94.64\pm3.18^{\mathrm{a}}$	$51.72\pm1.91^{b}$	$37\pm2.75^{b}$	507.65		$121.66 \pm 0.25^{a}$	$334.57\pm 31.71^{\rm a}$	pu	$301.01 \pm 10.62^{a}$	757.24
Sources	(Fariña vd., 2015)		(Fariña vd., 2015)										7
Odor Threshold Value (µg/Kg)	1000		2200 (										
LRI <sup>1</sup>	1613	1786	2027	2048	2331	2385			- 2453	2489	2651	- 2694	
Aroma Compounds	Lactones 1 y-Butyrolactone	ô-Hexalactone	Pantolactone	a-Butyrolactam	4- (1-hydroxy-ethyl) - <sup>v</sup> - butanolactone	dihydro-5- (1- 6 chydroxyethyl) -2- (3H) - Guranone (sherry lactone)	Total	Norisoprenoids	9-Hydroxy -megastigm-7- 2453 en-3-one	3-Hydroxy-β-damascone	3-Oxo-a-ionol	3-Hydroxy-7,8-dihydro-β- 2694 ionol	Total
	-	5	3	4	S L	285			1	7	3	4	

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LRI,MS,Std LRI,MS,Std LRI,MS,Std LRI,MS,Tent LRI,MS,Tent LRI,MS,Std LRI,MS,Std LRI,MS,Std LRI,MS,Std (Selli & Kelebek, 2011) (Selli & Kelebek, 2011) Fresh,sweet,floral Va'zquez vd., 2007) (Castro-Fresh,sweet,floral Va'zquez vd., 2007) Fresh, sweet, floral Va'zquez vd., (Amoore & Venstrom, (Castro-(Castro-2007) 1966) Orange-like, fruity Floral,green Description Minty Odor Odor Activity Value 13.35 3.05  $\overline{\vee}$  $\overline{\vee}$  $199,17 \pm 19,84^{b}$  $38.51\pm3.73^{\rm o}$  $80.14\pm7.84^{\circ}$  $18.33\pm1.83^{\rm o}$  $56.07\pm4.38^{b}$  $19.77 \pm 1.72$  $86.02 \pm 6.62$ Quantity 1700 m (µg/kg) pu pu 498,01 Odor Activity Value 13.97 1.0053.01 3.301.94 $212.99 \pm 10.83^{a}$  $318.08\pm 3.91^{a}$  $274,09 \pm 18,62^{a}$  $60.27\pm0.98^{\rm b}$  $83.82\pm1.74^{\mathrm{a}}$  $206.41 \pm 3.32^{a}$  $82.48 \pm 4.86^{a}$  $531 \pm 48.68^{a}$ Quantity 1050 m (µg/kg) pu 1769,14 Odor Activity Value 17.871.18 7.75 2.10Va'zquez vd.,  $107.23 \pm 7.09^{b}$ Va'zquez vd.,  $46.52 \pm 2.77^{b}$ 2007)  $145.02 \pm 6.48^{b}$ (Selli & Kelebek,  $70.84 \pm 5.32^{n}$ 2011) (Selli & Kelebek, 52.58  $\pm$  4.29<sup>b</sup> 2011)  $76,63 \pm 4,36$  $25.67\pm0.3^{a}$  $46.3 \pm 0.97^{b}$ Quantity 620 m (µg/kg) pu 570,79 (Castro-2007) (Castro-(Castro-Va'zquez vd., 2007) References Odor Threshol d Value (µg/kg) 110 60 25 9 9 LRI 1193 1469 1773 1944 2308 1442 547 607 l 642 ((E) - 2,6-Dimethylocta-3,7-diene-2,6-diol Aroma Compounds Terpenes And Terpenoids (Z) -8-Hydroxy-linalool Dihydro oxophorone (E)- Linalool oxide (Z)- Linalool oxide L-Menthol Limonene Hotrienol Linalool Total -2 ŝ 286 5 ~ 4 9  $\infty$ 6

(Continued Table 2)



	Aroma Compounds	LRI	Odor Threshold Value	References	Quantity 620 m (µg/kg)	Odor Activity Value	Quantity 1050 m (µg/kg)	Odor Activity Value	Quantity 1700 m (µg/kg)	Odor Activity Value	Odor References Description	References	
1	Volattile Acids												
-	Acetic acid	1443	33	(Bonvehí, 2005)	$207.27\pm2.96^{b}$	6.28	$255.41 \pm 23.28^{a}$	7.74	$190.92\pm15.56^{b}$	5.78	Strong, purgent	(Bonvehí, 2005)	LRI,MS,Std
5	Propanoic acid	1531	20000	(Bonvehí, 2005)	$55.91\pm2.43^{\rm b}$	$\overline{\vee}$	$66.38\pm5.22^a$	$\overline{\vee}$	$62.02\pm5.41^{ab}$	$\overline{\lor}$	Purgent, rancid	(Bonvehí, 2005)	LRI,MS,Std
3	3-Methyl-Butanoic acid	1664	700	(Bonvehí, 2005)	$92.05\pm5.38$	$\overline{\vee}$	$157.11 \pm 7.36^{a}$	$\overline{\vee}$	$133.37\pm10.4^{b}$	$\overline{\lor}$	Rncid, cheesy, faecal,	(Bonvehí, 2005)	LRI,MS,Tent
4	Hexanoic acid	1839	420	(Fariña vd., 2015)	$71.56\pm5.41^{b}$	$\overline{\vee}$	$120.31 \pm 3.27^{\rm a}$	$\overline{\vee}$	$74.52\pm1.79^{\text{b}}$	$\overline{\vee}$	Fatty, cheese (Fariña vd., 2015)	(Fariña vd., 2015)	LRI,MS,Std
2	Octanoic acid	2054	500	(Fariña vd., 2015)	$277.09 \pm 15.12$	$\overline{\vee}$	$769.98 \pm 70.67^{a}$	1.54	$366.04\pm15.17^{b}$	$\overline{\lor}$	Fatty	(Fariña vd., 2015)	LRI,MS,Std
9	Nonanoic acid	2162	3000	(Bonvehí, 2005)	$85.88 \pm 3.18^{\circ}$	$\overline{\vee}$	$182.36 \pm 16.13^{a}$	$\overline{\vee}$	$133.18\pm7.46^{b}$	$\overline{\vee}$			LRI,MS,Std
5	Benzoic acid	2433	1000	(Escudero vd., 2007)	$110.46\pm2.9^{a}$	$\overline{\vee}$	$104.93 \pm 3.62^{b}$	$\overline{\vee}$	pu		Faint balsamic	(Bonvehí, 2005)	LRI,MS,Std
8	Hexadecanoic acid	2825	10000	(Leffingwell & Leffingwell, 1991)	$2303.39 \pm 84.64$	$\overline{\vee}$	$5489.02 \pm 459.43^{a}$	$\overline{\vee}$	$4295.59 \pm 418.76^{b}$	$\overline{\lor}$			LRI,MS,Std
6	Octadecanoic acid	3107		20000 (Fariña vd., 2015)	$830.33\pm59.67$	$\overline{\vee}$	$2312.92 \pm 197.72^{a}$	$\overline{\vee}$	$1300.56\pm 63.79^{b}$	$\overline{\vee}$			LRI,MS,Std
	Total			4	4033,94		9458,42		6556,20				
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$\sqrt{vanice} (vanice)\frac{0.001}{(vanice)}$	(Contir	(Continued Table 2)												
L & 991) Malturni,		Aroma Compounds	LRI	Odor Threshold Value (µg/kg)	References	Quamtity 620 (µg/kg)	Odor Activity Value	Quantity 1050 <sup>(µg/kg)</sup>	Odor Activity Value	~	Odor Activity Value	Odor Description	References	
les, 1 & 991)		Volatile Phenols												
les, (& 991) MS);	1	Orcinol	1973			$201.58\pm 5.33^{a}$		$153.18\pm9.65^{b}$		pu				LRI,MS,Std
991) MS);	2	2-Methoxy-4-vinyl-phene	ol 2192	S	(Pino & Febles, 2013)	$11.85\pm1.15^{b}$	2.37	pu		$48.55\pm3.99^{a}$		Intense spicy	(Pino & Febles, 2013)	LRI,MS,Std
991) Maltimiti, MS);	б	2,3,5-Trimethyl-phenol	2219			$23.76\pm0.5^{\rm a}$		ри		pu				LRI,MS,Std
991) Multumi; MS);	288	3,4,5-Trimethyl-phenol	2367			$129.68 \pm 1.88^{a}$		$16.02\pm1.5^\circ$		$31.38\pm3.12^{b}$				LRI,MS,Std
991) 991) MSN;;		Total				366,87		169,20		79,93				
991) 991) MS);		Volatile Sulfurs												
olumn; MS);	Н	Benzothiazole	1955	80	(Leffingwell & Leffingwell, 1991)	$57.52\pm5.09^{b}$	$\overline{\vee}$		1.67	$68.01 \pm 3.24^{b}$	$\overline{\vee}$		(Xie vd., 2008)	LRI,MS,Std
olumn; MS);		Total				57.52		149.41		68.01				
MS);		General Total				14600,50		29882,90		16372,89				
	LRI: Li library) differer	inear retention index was c ), Std (Standard substance) at letters in the same colum	alculated , MS tent. m is statis	on a DB -W . (Tentative i stically signif	MS);	Concentration: Avera 1d not detected; Stan	ige of 3 differ dar d deviatio	ent injection results n values of aroma c	t in µg / kg; ompounds	Identification: I are below 10%.	.RI (Linear a, b, c: Th	r retenti on ir e difference b	ndex), MS (Mass etween the value	spectrometer s shown with

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### Esters

Two ester compounds, methyl-2-furoate and ethyl hexadecanoate, were found in all astragalus honey samples. The ratios of these compounds in the sum of all the aroma compounds for honey samples obtained from the altitudes of 620, 1050 and 1700 meters were 2.8%, 3.4%, and 3.7%, respectively. Most of the ester compounds, which occur with the esterification of acidic and alcoholic compounds, can be characterized by fruit and flower odors (Tian vd., 2016). Ethyl hexadecanoate in the astragalus honey samples was found to be 45.58  $\mu$ g/kg, 53.41  $\mu$ g/kg, and 16.78  $\mu$ g/kg, respectively to the altitude. On the other hand, amounts of the methyl-2-furoate compound were determined to be 374.22  $\mu$ g/kg, 984.27  $\mu$ g/kg, and 602.93  $\mu$ g/kg respectively to the altitude. In an overall look, the amount of the ester compounds increased in the higher altitudes, among all the aroma compounds.

### **Furans and Pyrans**

The ratios of furan and pyran compounds in the sum of all the aroma compounds were found to be 23%, 34%, and 21%, respectively to the altitude. Furan compounds could be present in kinds of honey as a result of insufficient heat treatment and storing conditions. Furan compounds could also be associated with the smoke that is being used by the beekeepers to drive the bees out of the hive and reduce their aggression. (Tanleque-Alberto vd., 2019).

Furfural is a product of the Maillard reaction and occurs from the cyclization and dehydration of a dehydroreductor from a pentose (Moreira vd., 2010). The odor threshold value of the furfural compound, which was characterized by alcoholic and burnt smell, is 720  $\mu$ g/kg (Buttery vd., 1988). The odor activity value of the furfural compound was only determined as 1.02 in the honey sample obtained from the altitude of 1050 meters. The amount of this compound is 186.99  $\mu$ g/kg, 739.51  $\mu$ g/kg, and 288.61  $\mu$ g/kg, respectively to the altitude.

The 5-hydroxy-methyl-furfural compound may increase during storage. Also, it is known that this compound may be formed as a result of the Maillard reaction that took place with the cyclization and hydration of a dehydroreductor from a hexose (glucose and fructose) (Moreira vd., 2010). The amounts of the 5-hydroxy-methyl-furfural compound were found to be 2882.23  $\mu$ g/kg, 8733.63  $\mu$ g/kg, and 3084.24  $\mu$ g/kg in honey samples obtained from altitudes of 620, 1050, and 1700 meters, respectively.

Amounts of furfuryl alcohol in the honey samples were 148.84  $\mu$ g/kg, 190.96  $\mu$ g/kg, and 79.41  $\mu$ g/kg, respectively (p<0.05). 5-methyl-2-furancarboxaldehyde compound was found to be 42.72  $\mu$ g/kg, 80.43  $\mu$ g/kg and 38.04  $\mu$ g/kg, respectively (p<0.05). Generally, it was observed that the furan and pyran compounds are not affected by the change of altitude.

### Lactones

 $\gamma$ - and  $\delta$ -lactones are important lactone groups that are present naturally in some fruits and some fermented foods, and they have a pleasant odor characteristic. It is also reported that most of the lactone compounds have a fruity fragrance (peach, coconut, anise, etc.) (Yılmaztekin & Erten, 2008).

The amount of the  $\gamma$ -butyrolactone compound is 96.76 µg/kg, 111.06 µg/kg, and 90.24 µg/kg, respective to the altitude. In a prior study, the aroma profiles of different kinds of honey were investigated and it is found that  $\gamma$ -butyrolactone compound, which is characterized by the fragrance of honey, is present in 9 different kinds of honey (Seisonen vd., 2015).

Pantolactone compound is characterized by the fragrance of burnt bread. (Forero vd., 2015). The ratios of this compound in the sum of the lactone compounds was found to be 36%, 59%, and 42% in the honey samples respectively to altitudes (p<0.05)

While dihydro-5-(1-hydroxyethyl)-2-(3H)-furanone, also known as sherry lactone, was not detected in the



honey sample from 1050 meters' altitude, the amount of this compound in the honey sample obtained from the altitude of 620 meters was found as 37  $\mu$ g/kg. Moreover, the amount of sherry lactone in the honey sample obtained from the altitude of 1700 meters was also determined as 44.76  $\mu$ g/kg (p<0.05).

If it is concluded in terms of the altitude effect on the lactone compounds, it could be said that the total amount of lactone compounds was higher in the low altitude honey samples. On the other hand, while the amount of the  $\alpha$ -butyrolactam compound decreased with the increasing altitude, the amount of  $\gamma$ -butyrolactone increased.

#### Norisoprenoids

Norisoprenoids are an important class in the aroma compounds, that is defined in nature. Norisoprenoids, which are present in most plants, especially tobacco, occur as a result of the degradation of carotenoids (Cabaroğlu vd., 1997). They can also be isolated from honey since the source of most of them are flowers (Guyot-declerck vd., 2000).

Among the norisoprenoids, 3-oxo- $\alpha$ -ionol is characterized by the honey odor (Forero vd., 2015). Also, this compound was found in the honey sample obtained from the altitude of 1700 meters and its amount was calculated as 221.2 µg/kg (p<0.05). It is believed that the presence of the 3-oxo- $\alpha$ -ionol compound in the honey sample gathered from the altitude of 1700 meters is related to the presence of eucalyptus in the pollen analysis. This point of view is supported by two different studies that found 3-oxo- $\alpha$ -ionol in the eucalyptus honey (Alissandrakis vd., 2011; Vázquez vd., 2006).

The presence of the 9-hydroxy-megastigm-7-en-3-on compound in the honey samples is 121.66  $\mu$ g/kg, 81.17  $\mu$ g/kg, and 47.25  $\mu$ g/kg, respectively concerning the altitude. Another important norisoprenoid compound 3-hydroxy- $\beta$ -damascone amount was determined as 334.57  $\mu$ g/kg in the honey sample obtained from the altitude of 620 meters and 101.76  $\mu$ g/kg in honey sample gathered from the altitude of 1700 meters (p<0.05). The amount of the norisoprenoid compounds was detected to be high in the lower altitudes. It is known that the source of these compounds has flower origins (Guyot-declerck vd., 2000). It may be thought that the reason for the lower amount of the norisoprenoid compounds in the honey samples obtained from high altitudes may be connected to the decrease of the variety of flowers and plants in these altitudes.

### **Terpene and Terpenoids**

The origin of the terpenes and their derivatives in the honey is related to the gathered nectar by the bees and the honey essence (Soria vd., 2008). The ratios of the terpenes and terpenoids in the sum of all aroma compounds were 3.9%, 5.8%, and 3%, respectively concerning altitudes. It was determined that some of the terpene and terpenoid compounds have a significant role in the aroma of the astragalus honey. It was reported that the odor threshold value of (E)-Linalool oxide and (Z)-Linalool oxide, which is characterized by the fragrance of fresh and flowery, is 6 µg/kg (Castro-Vazquez vd., 2007). The amount of (E)-Linalool oxide in the honey samples was 107.23  $\mu$ g/kg, 318.08  $\mu$ g/kg, and 80.14  $\mu$ g/kg; and the odor activity values were calculated as 17.87, 53.01, and 13.35, respectively to the increasing altitude. The amount of (Z)-Linalool oxide in the honey samples was 46.52  $\mu$ g/kg, 83.82  $\mu$ g/kg, and 18.33  $\mu$ g/; and the odor activity values were calculated as 7.75, 13.97, and 3.05, respectively to the increasing altitude. It could be seen from the results, that both (E)-Linalool oxide and (Z)-Linalool oxide compounds influence significantly the flavour characteristics of the astragalus honey. It is reported that the hotrienol compound is present in most kinds of flower honey, especially lavender honey (Marijanovic vd., 2009). This compound can occur either in the maturity process of the honey or during the thermal degradation of 1-terpendiol or 8-hydroxylinalool compounds. Also, hotrienol may occur in the later stage oxidation of lilac aldehydes, as well as a result of isomerization of (E)-8-hydroxylinalool to lilac alcohols from lilac aldehydes (Jerkovic vd., 2013). The odor threshold value of the hotrienol, which is characterized by the flowery fragrance, is 110 µg/kg (Castro-Va'zquez vd., 2007). The amount of this compound was determined to be 212.99 µg/kg in the honey sample



obtained from 1050 meters' altitude and 56.07  $\mu$ g/kg in the honey sample gathered from the altitude of 1700 meters. However, it was not found in the honey sample obtained from 620 meters' altitude (p<0.05). The odor activity value of hotrienol was calculated as lower than 1 in the honey sample gathered from 1700 meters' altitude and as 1.94 in the honey sample obtained from the altitude of 1050 meters. It was reported that the hotrienol compound is the characteristic aroma compound of different citrus and lavender honey which are characterized by the flowery and fruity odor (Gianelli Barra vd., 2010). The odor threshold value of the linalool, which is characterized by a flowery and green-like fragrance, is 25 µg/kg (Selli & Kelebek, 2011). The amount of this compound was determined as 52.58  $\mu$ g/kg in the honey sample obtained from the altitude of 620 meters and 82.48 µg/kg in the honey sample gathered from the altitude of 1050 meters, yet there is no trace of it in the honey sample obtained from 1700 meters altitude. The odor activity value of linalool was calculated as 2.10 in the honey sample gathered from 620 meters altitude and 3.30 in the honey sample obtained from 1050 meters altitude. It was seen from these results that linalool plays an important role in the characteristic aroma of astragalus honey gathered from 620 and 1050 meters altitudes. The odor threshold value of the limonene compound, which is characterized by orange-like and fruity fragrance, is 60  $\mu$ g/kg (Selli & Kelebek, 2011). The amounts of limonene compound in the honey samples were 70.84  $\mu$ g/kg,  $60.27 \ \mu g/kg$ , and  $38.51 \ \mu g/kg$ , respectively to the altitudes. The odor activity values of limonene were respectively found as 1.18 and 1 in the honey samples obtained from 620 meters and 1050 meters altitudes. The reason for the absence of some terpene and terpenoid compounds such as linalool and limonene in the honey sample gathered from 1700 meters altitude may be the lack of diversity in higher altitudes. While the amount of (E)-Linalool oxide, (Z)-Linalool oxide, L-menthol, and linalool decreased with the increase of altitude, the amount of (Z)-8-hydroxy linalool increased. The amount of limonene compound also decreased with increasing the altitude.

### **Volatile Acids**

It is reported in a previous study, that the odor threshold value of acetic acid, which is characterized by the spicy, vinegar fragrance, is 33  $\mu$ g/kg (Bonvehí, 2005). The amount of acetic acid in the honey samples is 207.27  $\mu$ g/kg, 255.41  $\mu$ g/kg, and 190.92  $\mu$ g/k, respectively to the increasing altitude. The odor activity values of this compound were respectively calculated as 6.28, 7.74, and 5.78 in the honey samples obtained from altitudes of 620, 1050, and 1700 meters, and it is concluded that the acetic acid influences the characteristic aroma of the astragalus honey. It was reported in former studies, that the carboxylic acids, such as acetic acid, give a pleasant and spicy odor to the kinds of honey, whereas butanoic acid and hexanoic acid, which are present in butter, give an odor, which may be unpleasant, to honey (Tian vd., 2016).

#### **Volatile Phenols**

The odor threshold value of 2-methoxy-4-vinyl-phenol characterized the spicy fragrance is  $5\mu g/kg$  in the literature (Forero vd., 2015). This compound was determined as 11.85  $\mu g/kg$  in the honey sample obtained from 620 meters altitude and 48.55  $\mu g/kg$  in the honey sample gathered from 1700 meters altitude. However, it was not found in the honey sample obtained from 1050 meters. Odor activity values of this compound were calculated as 2.37 in the honey sample obtained from 620 meters altitude and as 9.71 in the honey sample gathered the altitude of 1700 meters. It was detected that the 2-methoxy-4-vinyl-phenol has a significant influence on the formation of the characteristic odor of the two astragalus honey samples.

#### **Volatile Sulphurs**

The odor threshold value of benzothiazole, which is characterized by a moss-like, roasted fragrance is 80  $\mu$ g/kg (Leffingwell & Leffingwell, 1991; Okabe vd., 2019). The amount of benzothiazole in the honey samples is 57.52  $\mu$ g/kg, 149.41  $\mu$ g/kg, and 68.01  $\mu$ g/kg, respectively to the altitudes. The odor activity value of the benzothiazole was calculated less than 1 for the honey samples obtained from altitudes of 620 and 1700 meters. On the other hand, this value was calculated as 1.67 in the honey sample gathered from the



altitude of 1050 meters. It was determined that benzothiazole was effective on the characteristic odor of only the honey sample obtained from 1050 meters altitude.

In order to create a model to classify the honey samples in manners of the aroma groups, principal component analysis (PCA) was applied. As could be seen in the biplots (Figure 1.), the PCA model formed of two main components that explain 100% of the total variance (F1: 80.84%, F2: 19.16%). According to the biplot of the aroma groups, the honey samples obtained from the altitudes of 620 and 1700 meters are located on the left-hand side and the honey sample gathered from the altitude of 1050 meters is located on the right-hand side of the plot. The correlation between the location of the honey samples and the aroma groups on the coordinate system was found to be crucial. The honey sample obtained from the altitude of 620 meters showed a dominant character concerning volatile phenol compounds, lactone compounds, and norisoprenoid compounds. Similarly, the honey sample gathered from the altitude of 1050 meters was found to be a dominant character concerning aldehyde and ketone compounds, alcoholic compounds, volatile sulfur compounds. On the other hand, the honey sample obtained from the altitude of 1700 meters demonstrated a weaker character in terms of aroma compounds, relative to the other samples.

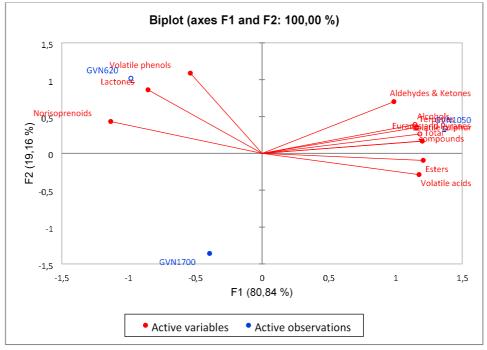


Figure 1. PCA biplot of astragalus honey samples

### The Sensory Properties of Honey Samples

The results of the general sensorial profile analyses and the aroma profile analyses are given in the spider web graphics in Figure 2 and Figure 3. In general, the panelists liked the honey sample obtained from the 620 meters altitude more in the general sensorial profile analysis, followed by the honey samples gathered from altitudes of 1050 and 1700 meters. According to the sensory evaluation of the panelists, a decrease occurred in the scores, such as color, aroma, taste balance, with the increasing altitude. However, the crystallization score increased proportionally to the altitude.

According to the results of the aroma profile analysis, the honey sample of 1050 meters' altitude got the highest score (7.6) in terms of floral odor. It was followed by the honey sample of 620 meters' altitude (6.24) and finally the honey sample of 1700 meters' altitude (4.6) (p> 0.05). It is known that the source of



terpene and terpenoid compounds, which are characterized by flowery fragrances, is flowers (Soria vd., 2008). As stated before, a high amount of terpene and terpenoid compounds were found in the honey sample obtained from1050 meters altitude. The reason why the panelists gave a higher score to the honey sample obtained from1050 meters altitude was considered to be related to the amount of these compounds in the honey sample. The panelists gave approximately the same score for each sample concerning refreshing odor (p>0.05). L-menthol, (E),(Z) linalool oxide and hotrienol are known to be responsible for the refreshing odor (Amoore & Venstrom, 1966). The scores of the panelists concerning refreshing odor were the honey samples obtained from 1050-, 620-, and 1700-meters altitudes, from high to low. It is thought that the odor of L-menthol, hotrienol, (E)- and (Z)-linalool oxide compounds were effective in the scoring.

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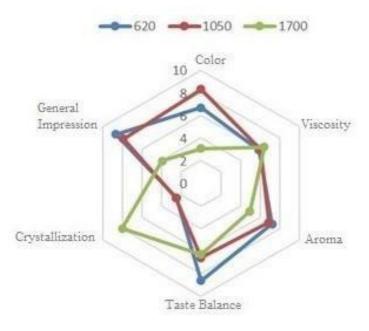


Figure 2. The general sensorial profile of astragalus honey samples

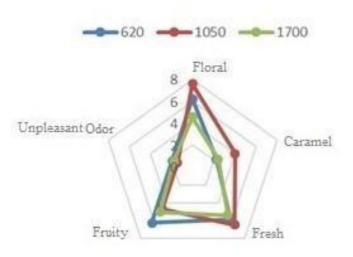


Figure 3. The aroma profile of astragalus honey samples

### CONCLUSION



The general properties of all astragalus honey samples were found to be adequate concerning the national and international standards. A decrease occurred in the amount of the dominant pollen with the increasing altitude. In comparison with this decrease, an increase in acidity and diastase activity was observed with the increasing altitude. With the increasing altitude, a slight increase in the amount of sum of fructose and glucose, the ratio of fructose to glucose, and the amount of HMF were observed, as well. Proline amount was not affected by the change of altitude.

When the characteristic aroma of the honey samples was taken into the consideration, it was seen that the different compounds in different altitudes have effects on the characteristic aroma of the honey samples. Among all the compounds, especially (E)-Linalool oxide and (Z)-Linalool oxide compounds had remarkably high amounts, as well as their odor activity values. On the other hand linalool oxide, 2-methyl-2-butanol, furfural, hotrienol, linalool, limonene, acetic acid, and 2-methoxy-4-vinyl phenol compounds had also odor activity values greater than 1. According to these results, it could be said that the astragalus honey could be characterized by fresh, sweetish, flowery, orange-like fruity, camphor-like, spicy, lightly burnt, and lightly pungent. Nevertheless, the odor characteristics of the astragalus honey can be exactly determined only with the GC-O technique.

In recent years, aroma compounds are considered to be the marker compounds that characterized the food. There are many studies on kinds of honey, which is an important nutrient for humans. However, any study concerning the aroma compounds of kinds of honey obtained from different altitudes was not encountered in the literature. The influence of the altitude on the astragalus honey was presented with this study. This study is also the first study to fill the gap in the relationship between the aroma compounds and the altitude. However, it may be beneficial to conduct similar studies with other kinds of honey and to investigate this subject in depth.

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Ethical Review - Ethical approval was not required for this research.

**Data availability statement -** The data that support the findings of this study are available from the corresponding author upon reasonable request.

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# Drying Treatments Change the Composition of Aromatic Compounds from Fresh to Dried Centennial Seedless Grapes

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#### ABSTRACT

Raisin aroma is a vital sensory characteristic that determines consumers' acceptance. Volatile organic compounds (VOCs) in fresh grapes, air-dried (AD), pre-treated air-dried (PAD), sun-dried (SD), and pre-treated sun-dried (PSD) raisins were analyzed, with 99 and 77 free- and bound-form compounds identified in centennial seedless grapes, respectively. The hexenal, (E)-2-hexenal, 1-hexanol, ethyl alcohol, and ethyl acetate in free-form while benzyl alcohol,  $\beta$ -damascenone, gerenic acid in bound-form were the leading compounds. Overall, the concentration of aldehydes, alcohols, esters, acids, terpenoids, ketones, benzene, and phenols were abundant in fresh grapes but pyrazine and furan were identified in raisin. Out of 99 VOCs, 30 compounds had an odour active value above 1. The intensity of green, floral, and fruity aromas were quite higher in fresh grapes followed by AD-raisins, PAD-raisins, SD-raisins, and PSD-raisins. The intense roasted aroma was found in SD-raisins due to 2,6-diethylpyrazine and 3-ethyl-2,5-dimethylpyrazine. Among raisins, the concentration of unsaturated fatty acid oxidized and Maillard reaction volatiles were higher in SD-raisins and mainly contributed green, fruity and floral, and roasted aromas, respectively.

**Keywords:** free-form volatile compounds; glycosidically bound-form volatile compound; centennial seedless; aromatic profile; SPME-GS/MS



# Development of Value-Added Functional Food by Fusion of Colored Potato and Buckwheat Flour Through Hot Melt Extrusion

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### ABSTRACT

Thermal sensitivity of anthocyanin and low-water solubility of rutin limit their stability in processed foods and biological activity, respectively. Therefore, we aimed to develop a value-added food composite (VAFC) by mixing of anthocyanin-enriched potato (P) and rutin-enriched buckwheat (B) flour using soy lecithin (LCT) and vitamin E (Vita E) by hot-melt extrusion (HME). The VAFC was formulated using P 50% + B 50% (F1), P 45%+B 45%+LCT 10% (F2), and P 45%+B 45%+LCT 8%+Vita E 2% (F3). Results revealed that the solubility of the VAFC was significantly increased in F3 formulation. Likewise, total phenolic content, including single phenolic acids, total flavonoid including rutin and quercetin, total anthocyanin including single anthocyanins, and antioxidant capacity of the VAFC were significantly increased in F3 formulation. This study successfully demonstrated the approach of developing biopolymermediated functional VAFC from potato and buckwheat by HME.



### Investigation of the Use of Pekmez in Orange Nectar Production

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### ABSTRACT

Sugar syrups have been used in many foods such as fruit nectars for many years since they positively affect many characteristics of foods and provide the sensory quality and shelf life desired by the producer. Sugar syrups are clear, colorless and viscous syrups obtained from starch by hydrolysis. However, it has been found that sugar syrups have many negative effects on health such as various pathological changes, oxidative stress, glucose intolerance, insulin resistance, type 2 diabetes, obesity, hypertension and cardiovascular diseases. Pekmez (molasses) is a traditional Turkish food that is generally produced from grapes. Pekmez contains organic acids, minerals and vitamins so it is very important for nutrition. Pekmez meets most of the daily calcium, iron, potassium and magnesium needs. In this study, the use of pekmez in orange nectar instead of sugar syrup has been investigated and the effects of different pekmez concentrations on various quality characteristics of orange nectar such as Brix, titratable acidity, color and total phenolic content have been determined and consumer preferences have been demonstrated by sensory evaluation. With the addition of pekmez, it was determined that the water-soluble solid content, titratable acidity and total phenolic content of the samples increased. On the other hand, the color of the sample became darker as the pekmez concentration increased. Similarly, as the concentration of pekmez in nectar increased, it was less appreciated. Although pekmez, which was used in a high concentration, in orange nectar had a negative effect on color and sensory properties, the addition of pekmez improved the nutritional value of orange nectar so a new functional product rich in phenolic compounds, vitamins and minerals can be developed.

Keywords: Orange Nectar, Pekmez, Sugar Syrup, Total Phenolic Content

### **INTRODUCTION**

Pekmez (molasses) is a traditional Turkish food and it has been widely produced from grapes in Turkey. According to the Turkish Food Codex, grape molasses is defined as "the viscous product obtained by reducing the acidity of unfermented fresh grape or raisin extract with appropriate methods and then thickening it under vacuum or in the open in accordance with the technique." (TFC, 2017). Grape molasses is a good source of nutrient and energy because it contains vitamin and mineral such as iron, calcium and high sugar (Kıran et al., 2019). Pekmez is used in making blood in the body. It has positive effects on stomach, intestines and kidneys. It is good for arteriosclerosis and facilitates blood circulation (Evrendilek, 2017). One of the most consumed and produced products in the fruit juice industry in the world is orange juice. Orange is one of the six main fruits that are processed into fruit juices and similar products in our country. Orange ranks first in nectar production in many countries around the world (Jordão, 2018). Orange juice, rich in vitamin C, carotenoids, flavonoids and minerals, is an important daily nutritional source (Topuz et al., 2005). It is available in the market in the form of orange juice (contains 100% orange juice), orange nectar.

### MATERIAL AND METHOD

### Material

In this study, fresh orange (Citrus sinensis), fresh lemon (Citrus limoni), powdered sugar (Balküpü) and



grape molasses (Gülsan) used for the production of orange nectar were obtained from local markets (Niğde). The fruits were stored at 4 °C before the juice was obtained. 50 mL of orange juice, 12 g of sugar, 0.2 g of lemon juice were mixed and completed with 100 mL of water while preparing orange nectar as a control sample. Orange nectar containing different concentrations (5, 10 and 15%) of grape molasses instead of sugar syrup was prepared to examine the effect of molasses on its quality properties.

### **Determination of Color**

L\*, a\* and b\* values were determined using the Konica-Minolta (CR400, Osaka, Japan) color reader.

### **Determination of Water Soluble Solid Content**

Total water soluble solid content was determined using an abbe refractometer (A. Krüss Optronic GmbH, AR4, Hamburg). Results were expressed in °Brix.

### **Determination of Titratable Acidity**

0.5 mL of phenol phthalein reagent was added to 10 mL sample and it was titrated with 0.1N NaOH. The NaOH spent in the titration was recorded and the results were calculated according to AOAC (2000).

### **Determination of Total Phenolic Content**

The total phenolic content was determined according to the Folin–Ciocalteu colorimetric method (Singleton and Rossi, 1965). 100  $\mu$ L of sample was mixed with 0.75 mL of Folin–Ciocalteu reagent (diluted 10-fold in deionized water) and kept at room temperature for 5 min. After adding 0.75 mL of sodium bicarbonate (75 g L<sup>-1</sup>), the mixture was allowed to stand at room temperature for 60 min and absorbance was measured at 725 nm. Gallic acid was used as a standard, and the same procedures were applied to gallic acid solutions at different concentrations prepared for the calibration curve. All determinations were carried out in triplicate, and results were represented as milligrams of gallic acid equivalent (GAE) per kilogram of juice.

### Sensory analysis

The taste, odor, color and appearance properties of fruit juice samples as sensory quality parameters were evaluated by semi-trained panelists from Niğde Ömer Halisdemir University, Department of Food Engineering. In scoring, the best was numbered as 10 and the worst was numbered as 1. 13 panelists took part in the evaluations. The sample evaluation form is shown in Table 1.

Sa	ample	Taste	Odor	Color-Appearance
1				
2				
3				
4				

Table	1:	Sensorv	evaluation	ı form
1 uore	т.	Demotry	e a u u u u u u	1 IOIIII

### **Statistical Analysis**

The data were analysed using Minitab (17 version, Minitab Inc., State College, PA, USA) at 95% confidence interval, and the general linear model was used in the analysis of the data. Tukey's multiple comparison test was conducted to determine the differences between applications. Each experiment was repeated at least three times.



### **RESULTS AND DISCUSSION**

Color, °Brix, titratable acidity, total phenolic content and sensory analyses were performed in the orange nectar samples. The data obtained as a result of color analysis were shown in Table 2. Orange nectar samples that were added different amount of grape molasses (pekmez) was shown in Figure 1.

	Control	5%	10%	15%
L*	$43.13\pm0.94^{\rm a}$	$37.486 \pm$	$33.366 \pm$	$32.308 \pm 1.23^{\circ}$
		1.17 <sup>b</sup>	0.92 <sup>c</sup>	
a*	$-5.5\pm0.38^{d}$	$-2.69 \pm 0.13^{\circ}$	$-1.37 \pm 0.15^{b}$	$\textbf{-0.39}\pm0.13^{a}$
b*	$22.57\pm1.69^{\mathrm{a}}$	$16.33\pm0.75^{\mathrm{b}}$	$13.86 \pm 1.27^{\circ}$	$13.15\pm0.84^{\rm c}$

Table 2: Result of color analysis

Means that do not share a letter in the same line were significantly different.



Figure 1: Orange nectar samples

As stated in the Table 2, as the pekmez concentration increased, L\* (Lightness) values decreased (p<0.05). Accordingly, it was said that as the molasses concentration increased in the fruit nectar samples, the color of the sample became darker (Figure 1). It was determined that the higher the molasses concentration in the samples, the higher a\* values (p<0.05) and the lower the b\* values (p<0.05). Thus, it was observed that the red color increased and the yellow color decreased in the samples. Similarly, it was determined that the addition of molasses affected the color values in yoghurt, cake and ice cream (Ertaş and Çoklar, 2008; Temiz and Yeşilsu, 2010; Karaca et al., 2012). With the addition of molasses, the crust color values of the cakes generally decreased (Bilgiçli and Akbulut, 2009) and the redness value increased (Akbulut and Bilgiçli, 2010). °Brix, titratable acidity and total phenolic content of the samples were shown in Table 3.

Sample	°Brix (%)	Titratable	Total phenolic content
		acidity (%)	(mg GAE/kg)
Control	$17.92\pm0.29^{\rm a}$	$0.070 \pm$	$381.47 \pm 19.41^{d}$
		0.003 <sup>b</sup>	
5%	$10.08\pm0.14^{\text{d}}$	$0.077 \pm$	$513.84 \pm 38.31^{\circ}$
		0.001 <sup>a</sup>	
10%	$13.17\pm0.18^{\rm c}$	$0.077 \pm$	$621.84\pm9.18^{\text{b}}$
		$0.002^{a}$	

Table 3: °Brix, titratable acidity and total phenolic content of the samples



15%	$15.75\pm0^{b}$	$0.076 \pm$	$695.69 \pm 35.42^{a}$
		0.003ª	

Means that do not share a letter in the same column were significantly different.

When the °Brix values in the samples were examined, it was seen that the addition of grape molasses increases the amount of water soluble solid content (p<0.05). It has been reported that the addition of molasses also increased the total soluble solid content in ice cream samples (Temiz and Yeşilsu, 2010). The lowest brix value was obtained in orange nectar containing 5% molasses. This value did not meet the minimum brix value specified in the "Turkish Food Codex, Regulation of Fruit Juice and Similar Products" (TFC, 2014). However, the °Brix values of the other samples were found to be more than 11.2, which was the minimum brix value specified in the Regulation.

Titratable acidity values of the samples increased with the addition of molasses compared to the control sample (p<0.05). Similarly, it has been determined that the addition of pekmez increased the titratable acidity value in yoghurt, cake and ice cream (Ertaş and Çoklar, 2008; Temiz and Yeşilsu, 2010; Karaca et al., 2012). On the other hand, it was said that the increase in the pekmez concentration did not make a significant difference ( $p\geq0.05$ ) on the titratable acidity of the samples.

The total phenolic content of the samples increased compared to the control sample (p<0.05). In addition, depending on the grape molasses concentration, the total phenolic content of orange nectars increased (p<0.05). the result of sensory analysis were shown in Table 4.

Sample	Taste	Odor	Color-Appearance
Control	$8.69 \pm 1.18^{\rm a}$	$9.15\pm1.07^{\rm a}$	$8.31 \pm 1.25^{a}$
5%	$7.09 \pm 1.97^{\text{b}}$	$7.55 \pm 1.44^{b}$	$8.00\pm1.49^{\text{b}}$
10%	$6.85\pm2.34^{\mathrm{b}}$	$7.00\pm1.58^{\circ}$	$5.92 \pm 1.89^{\rm c}$
15%	$4.85 \pm 2.64^{\circ}$	$5.62\pm2.57^{\rm d}$	$4.92\pm2.60^{d}$

Table 4: Sensory analysis

Means that do not share a letter in the same column were significantly different.

Sensory analysis showed that the effect of using different amounts of molasses on the taste, odor, color and appearance scores of the samples was statistically significant (p<0.05). The sensory analysis results of the sample containing 5% molasses were closest to the control sample. As the molasses concentration increased, the rate of acceptability decreased (p<0.05). In a study examining the addition of molasses to ice cream samples, it was determined as a result of the sensory analysis that the addition of molasses up to 7.5%, improved the total acceptability scores of the samples (Temiz and Yeşilsu, 2010). It has been reported that the addition of molasses improves the sensory properties of the cake, and as a result, a nutritional and acceptable new product can be developed by using molasses as natural sugar in the cake formulation (Akbulut and Bilgiçli, 2010). As a result of the sensory analysis of molasses addition in yoghurt, increasing the molasses ratio negatively affected the appearance and color scores of the samples due to the decrease in whiteness. However, it was reported that the panelists gave the highest sensory scores to yoghurts produced with grape molasses and preferred 10% concentration for molasses (Karaca et al., 2012).

### CONCLUSION

Sugar syrups have been widely used in various products in the food industry due to their beneficial properties such as providing viscosity, improving flavour, providing texture and moisture-retaining properties, giving



sweetness and increasing resistance to color loss. In the production of soft drinks and fruit juice, fructose syrup has been widely preferred as sugar syrup. It is claimed that fructose is associated with chronic diseases such as obesity, hypertension, insulin resistance and gout. Consumption of sugary drinks, which is widespread among children and youth, increases the risk from fructose syrup. For this reason, the use of corn syrups, especially fructose syrups, is limited in various countries and serious restrictions have been imposed on the sale of products with rich fructose content (Zargaraan et al., 2016).

In this study, the possibilities of using molasses in orange nectar instead of sugar and sugar syrup were investigated. According to the results, it was determined that as the molasses concentration increased, the color of the sample darkened, the red color increased and the yellow color decreased in the samples. However, it was found that the addition of molasses increased the amount of water-soluble solid content and more than 5% molasses should be used to provide the minimum value (11.2 °Brix) specified in the "Turkish Food Codex, Regulation of Fruit Juice and Similar Products". Moreover, as a result of the addition of molasses, the titratable acidity and total phenolic content of orange nectars increased compared to the control sample. According to the results of the sensory analysis, as the molasses concentration increased, acceptability of the samples decreased. Although the use of molasses instead of sugar and sugar syrup in orange nectar has a negative effect on color and sensory properties, the addition of molasses improves the nutritional value of orange nectar. Thus, it has been seen that a new functional product rich in phenolic compounds, vitamins and minerals can be developed by using molasses as a natural sugar.

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# **Determination of Honey Adulteration with High Fructose Wheat Syrup**

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#### ABSTRACT

Honey is a natural source of sugars along with several health promoting minor compounds. Commercial value of honey is quite high which makes it attractive for food frauds.

The most commonly used starch-based sugar source for honey adulteration is high fructose corn syrup (HFCS). Nevertheless, this type of adulteration could easily be detected by a simple IR-MS analysis as HFCS is obtained from a C4 type plant.

In this study, it was aimed to determine the adulteration made by using high fructose wheat syrup (HFWS). The sugar syrup was obtained by hydrolyzing wheat starch to glucose syrup followed by converting it into 42% fructose syrup. Honeys of different botanical and geographic origins were collected and several parameters including moisture, electrical conductivity, ash ratio, water insoluble matter, free acidity, diastase activity, HMF and proline contents were determined. Adulterated samples were prepared by adding different amounts (%10, 20 and 40) of HFWS to the authentic honeys. Major sugars (sucrose, glucose and fructose) were quantified by using high-performance liquid chromatography (HPLC) and minor sugars (Trehalose, turanose, cellobiose, panose, erlose, nigerose, raffinose, kojibiose, maltose, maltotriose, isomaltose, isomaltotriose, maltotetraose) were quantified by using high-performance ion chromatography coupled with pulsed amperometric detection (HPAE-PAD).

It has been observed that the rates of major sugars are within the legal limits in both authentic and adulterated samples. Moreover, carbon isotope ratio analysis has revealed that even 40% adulterated samples were classified as authentic according to the current legislation limits. However, by evaluation of minor sugar analysis results with chemometric techniques, it was seen that adulterated honeys could successfully be distinguished from authentic ones.

Keywords: Honey, Adulteration, Wheat Starch, HPAE-PAD

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# UV-C Irradiation for Inactivation of *Listeria monocytogenes* on Frozen Sweet Cherry

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### ABSTRACT

This study evaluated inactivation efficiency of short wave ultraviolet (UV-C) light at 254 nm on Listeria monocytogenes or Listeria innocua inoculated on the surface of frozen sweet cherry (Prunus avium L.). Frozen sweet cherries inoculated with Listeria species were exposed to UV-C radiation treatments between 0-4 kj/m<sup>2</sup> in an enclosed chamber at room temperature with radiation doses using varying exposure times and the UV-C light intensity of radiation as measured by a portable digital radiometer at the fruit surface. Frozen sweet cherries were both obtained from local producer and purchased from supermarket the day before each experiment and stored at +4°C until use. 10 g of fruits (7-10 frozen sweet cherries) were surface spot inoculated separately with two strains of L. monocytogenes and L. innocua then placed in sterile empty petri plates and placed under germicidal UV lamp. Fruits were irradiated at a distance of 8 cm to obtain specific doses. Experiments were repeated three times. After UV-C treatment samples were transferred into a 90 ml of buffered peptone water and then homogenized samples were serially diluted. Each dilution of the samples was surface plated on selective and differential medium (ALOA) and non-selective TSA agar plates for Listeria counts. After UV-C treatment with 4.81 kJ/cm<sup>2</sup> (96 s) 4 log cfu/g reductions of L. monocytogenes counts were obtained. Results showed that L. monocytogenes counts decreased with increasing UV-C dose. The reductions in the number of two L. monocytogenes strains were different due to irradiation and Weibull model estimates UV-C inactivation of the strains.

Keywords: Inactivation, Listeria monocytogenes, sweet cherry, UV-C



# Modelling the Effect of Ultrasonic Washing on the Amount of Sulfur and Quality Characteristics of Dried Apricots Using Artificial Neural Networks (ANN)

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### ABSTRACT

Apricot is of great importance in nutrition with minerals, vitamins and dietary fibers that regulate mental activities, reduce stress, improve teeth and bones in children, ensure the regular functioning of the stomach and intestinal system, prevent constipation, increase blood production. Sulfur treatment is applied to protect the apricot from microbiological deterioration and to improve its characteristic color properties. However, utilization of excessive amount of sulfur dioxide in foods causes many health problems including asthma and allergenic inflammation. For this reason, the present study was aimed to investigate the use of ultrasound as an innovative technology to reduce the amount of sulfur in dried apricots and to determine the required ultrasonic washing time with artificial neural networks (ANN). With this purpose, dried apricots with an initial moisture content of  $25 \pm 2\%$  and containing  $458 \pm 14$  ppm sulfur were sonicated for 120 minutes using an ultrasonic bath ( $32 \pm 5$  kHz). It has been found that ultrasonic washing causes more color change and higher sulfur removal in apricots compared to traditional washing. The model with the lowest RMSE and the highest R<sup>2</sup> was selected with the "Levenberg-Marquardt" (trainlm) algorithm.

Keywords: ANN, apricot, modelling, sulfur, ultrasound



## Color Quality, Ascorbic acid and Total Carotenoid Contents of Dried Orange Slices as Influenced by Packaging Methods and Storage Conditions

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### ABSTRACT

Dried orange slices can be produced using different drying techniques. It is considered as a snack food or as an additive in formulations of some foods such as patisserie products and juices in powder form, due to its high amount of bioactive and aromatic compounds. The package affects the quality of foods by controlling the factors connected with their storage and handling. Thus, the influence of packaging methods and storage conditions on colour, ascorbic acid content and total carotenoid value of dried orange slice samples were investigated.

The total amount of ascorbic acid of dried orange slices was determined as 366.75 mg/100g at initial day. Significant reductions in the ascorbic acid amounts were observed during the 3 months of storage in connection with the applied packaging techniques. While the maximum reduction was observed with a rate of about 43% in the package containing normal atmosphere and exposed to light (CL), the least decrease was observed in the nitrogen containing package and stored in dark conditions (N<sub>2</sub>D) with a rate of about %17. The total amount of carotenoids in dried orange slices (initial day) was determined as 2.59 mg/100g. After 3 months of storage under light conditions, about 80% carotenoid loss was observed for CL while only 38% was computed for N<sub>2</sub>D samples. Notably, the total carotenoid content decreased more rapidly under light storage as compared to dark conditions, findings revealed that nitrogen containing package which stored dark conditions yielded a better preservation of colour, ascorbic acid and total carotenoid content.

Keywords: Dried orange slice, ascorbic acid, packaging, total carotenoid

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# The Improvement of Rheological Properties, Emulsion and Oxidative Stability of Low Fat Salad Dressing by Cold Pressed Hot Pepper Seed Oil By-product

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### ABSTRACT

This study aimed to investigate the potential use of hot pepper seed oil by-product (HPOB) in a low-fat salad dressing to improve rheological properties, emulsion, and oxidative stability. After the cold pressing process of hot pepper seeds, a by-product rich in protein, carbohydrate, phenolic component, and carotene, especially capsaicin, was obtained. This by-product has potential use in the low-fat emulsion as a natural fat replacer and antioxidant agent. The rheological properties (steady shear, frequency sweep, and 3-ITT), emulsion stability, and oxidative stability of low-fat salad dressing prepared by different concentrations of HPOB were compared with low fat and full-fat control salad dressing samples. All emulsions showed the shear-thinning characters. K value of the samples ranged from 4.10 to 7.79 and increased with increasing HPOB concentration, indicating that shear thinning characters could be improved by using HPOB. The G' value was higher than G" for all samples, indicating that all samples showed solid-like viscoelastic character. G' value was significantly increased as increasing HPOB concentration, and the low-fat samples prepared with 5% HBOB showed similar behavior to full-fat samples. The 3-ITT test was modeled with the second-order structural kinetic model, and the model parameters, namely, Go', Ge', and Ge'/Go' were calculated. Ge'/Go' increased with increasing HPOB concentration, indicating that recoverable characters improved by HPOB. Physical stability of emulsion was characterized by zeta potential, particle size, thermal loop test, and light microscope images. Oxidative stability was tested by OXITEST and IP values of samples enriched by HPOB compared to control samples. The oxidation rate was modeled by zero, first, and second-order kinetic models, and the oxidation kinetics constant (k) value was estimated. This study recommended that HPOB could be successfully used as natural fat replacers and antioxidant agents.

Keywords: Antioxidant, capsaicin, fat replacers, oxidation kinetic, rheology



## **Current Trends in Encapsulation: Applications in Food Science**

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### ABSTRACT

Encapsulation has been used as an efficient technology used in scientific and industrial areas for many years. Encapsulation in food science area can be defined as getting core material surrounded by a wall material often referred to shell, carrier, coating, matrix or encapsulant with the aim to protect the entrapped constituents from severe environments and release them at controlled rates over prolonged periods of time or at specific target sites (e.g., in the gastrointestinal track). Different encapsulation methods (spray drying, freeze drying, fluidized bed drying, emulsion and extrusion) have been usually used to encapsulate different bioactive agents, such as, vitamins, essential oils, peptides and enzymes. Recently, liposome technology, hydrogel based microgel particles and molecular encapsulation have gained great attention for food encapsulation. This review aims to present all of these current trend methods and usage of them in the encapsulation of bioactive compounds applied in food.

Keywords: encapsulation, liposome, hydrogel, cyclodextrin

### INTRODUCTION

Incorporating bioactive components as active agents as far as food packaging is concerned or their inclusion as nutraceuticals in functional food substances has gained significant attention in the area of food research. Unfortunately, use of bioactive ingredients is mostly limited by their poor solubility, stability, and flavour during food processing due to oxygen, temperature, and light. This is in addition to their low bioavailability and uncontrolled release pattern in the gastro-intestinal tract (GIT). As a result, their potential biological benefits can be affected significantly. As a way to overcome such challenges, scientists have decided to design suitable carriers. One popular method that is increasingly being applied is encapsulation. It is a procedure designed to entrap bioactive compounds and protect them from the GIT (for example, the stomach acid) and adverse environmental conditions (Dias et al., 2015).

In the field of food science, encapsulation is defined as the packaging technology of materials, solid, liquid or gaseous, into small, sealed capsules capable of releasing the contents at a controlled rate. The coating material is called capsule wall material, membrane carrier or shell. Capsule wall structures are often composed of food grade polymers and lipids, such as proteins (gelatin, whey protein, etc.) and polysaccharides (gum arabic, alginate, pectin, chitosan, cellulose, starch derivatives, etc.) (Fang and Bhandari, 2010).

The encapsule wall material protects the core material from environmental influences (oxygen, light, humidity, etc.) and improves stability handling conditions and overall acceptability. It also extends the shelf life of products, improves the functionality of additives and expands the range of nutritionally important food ingredients, including omega-3 fatty acids. (Kaushik et al., 2015).

Spray drying, freeze drying, spray cooling-chilling, spinning disk and centrifugal co-extrusion, extrusion, fluidized bed, and coecervation are some of the technologies used in the food industry to manufacture a variety of capsules through the encapsulation process (Jyothi et al., 2010). The review's goal is to describe the fundamental and advanced technologies to encapsulate bioactive compounds with liposomes, hydrogel



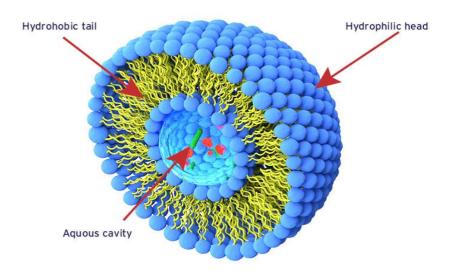
based microgel and cyclodextrins, and recent advancements in researches.

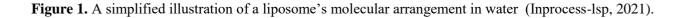
#### LIPOSOMES

A liposome is an artificial lipid vesicle with a bilayer phospholipids arrangement with the head groups directed towards the interior of the bilayer and the acyl group facing water on the outside of the membrane (Figure 1). Liposomes have the unique property of delivering their content to specific parts of food. Liposomes can also be used to deliver the encapsulated ingredient at a specific and well-defined temperature (Ajeeshkumar et al., 2021).

Liposomes are usually made up of phosphatidylcholine (lipid) molecules, although they can also be made up of phospholipid mixes. Major phospholipids used in liposomes are soy lecithin, egg lecithin, marine lecithin, and milk phospholipid. Thin-film formation and the proliposome method are two of the most frequent and up-scalable liposome manufacturing processes, resulting in liposomes of various sizes and degrees of lamellarity (multilamellar vesicles). The proliposome approach has been used to encapsulate food bioactives with liposomes that have the correct properties, such as high encapsulation efficiency, good stability, narrow particle size, and controlled release (Ajeeshkumar et al., 2021; González-Ortega et al., 2021).

Liposomes have been widely used as carrier's systems for pharmaceutical, food and nutraceutical applications. Li and co workers (2018) studied stability and bioaccessibility of curcumin liposomes (cur-Lps) by using different pluronics (triblock copolymers with a central hydrophobic poly (propylene oxide) (PPO) chain with two hydrophilic poly (ethylene oxide) (PEO) on each side) as modifiers. Thin film evaporation combined with dynamic high pressure microfluidization were used to make pluronics modified curcumin liposomes (cur-pluronic-Lps). Chitosan-coated nanoliposomes which prepared using thin-film hydration method, as a practical delivery system for encapsulation of caffeine were studied by Seyedabadi et al. (2021). González-Ortega et al. (2020) encapsulated oleuropein-rich olive leaf extract in soy phosphatidycholine and incorparated in model and commercial drinks. They demonstrated that in food systems such as drinks, lipid encapsulation does indeed provide a good carrier for oleuropein.





#### HYDROGEL BASED MICROGEL PARTICLES



Hydrogels are three-dimensional solid networks made up of physically or chemically cross-linked hydrophilic polymeric structures that can entangle a large amount of water or other biological fluids. Microparticles, nanoparticles, coatings, and films are all examples of physical states in which hydrogels can be produced (Abaee et al., 2017).

They are receiving a lot of attention in the field of encapsulated delivery systems, which are used to transport therapeutic medications and bioactive components to the location of interest, and have a lot of applications in biotechnology, medicine, and food technology (Zhang et al, 2016).

Because of their biodegradability, biocompatibility, and nontoxicity, many hydrogels, mainly from proteins and polysaccharides such rice starch, alginate, chitosan, and pectin as food grade polymers, have been employed as delivery matrix for active agents in recent years. Furthermore, the combined use of protein and polysaccharides is of great importance for the development of innovative gel systems, as different polysaccharides interact differently with the protein network, allowing for greater control over the release rates of such gels (Abaee et al., 2017).

Pedrali et al. (2020) extracted phenolics from winemaking byproducts and encapsulated by a a new hydrogelbased system. Their study lead to alginate and chitosan hydrogels can be used for encapsulation of phenolics extracted from grape seeds with encapsulation efficiency up to 92%. In another study, Zhang et al. (2016) incorporated b-carotene into three different delivery systems: free lipid droplets; filled hydrogel beads formed using 0.5% alginate ("0.5% beads"); and, filled hydrogel beads formed using 1% alginate ("1% beads"). Using an extrusion apparatus, they created hydrogel beads by inserting an alginate solution into a calcium ion solution (Encapsulator).

### MOLECULAR ENCAPSULATION

Molecular encapsulation is a process during which a single molecule is incorporated as guest in another host molecule that contains a cavity in its structure (Parbuntari et al., 2021).

Most common host molecules with applications in foods are cyclodextrins (CDs), which are cyclic nonreducing oligosaccharides with a torus-like structure. Most common host molecules with applications in foods are cyclodextrins (CDs), which are cyclic nonreducing oligosaccharides with a toruslike structure; the most common native CDs are composed of 6, 7, and 8 glucosyl units interconnected via  $\alpha$ -(1  $\rightarrow$  4) glycosidic linkages, and called  $\alpha$ -,  $\beta$ -, and  $\gamma$ -CD, respectively (Figure 2) (Hu et al., 2021).

An active nonpolar molecule (guest), with the right size and co-solubilized or dispersed with the cyclodextrin (host) in an aqueous solution, can be reversibly entrapped into the cavity to form an inclusion complex. The driving forces of host-guest binding are mainly due to the release of enthalpyrich water molecules from the cavity and noncovalent interactions including van der Waals interactions, hydrogen bonding, and hydrophobic interactions. The greater the affinity for the CDs, the higher the hydrophobicity and the smaller the guest molecule (Del Valle, 2004).



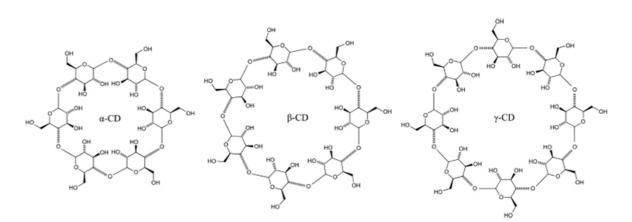


Figure 2. Different forms of cyclodextrins (CDs)

In the applications of molecular encapsulation in food systems, polyphenols from fruits and plants can be encapsulated in cyclodextrins because polyphenols' benzene ring makes cyclodextrins an excellent guest. Encapsulation of gallic acid, hydroxycinnamic acids such as caffeic acid, p-coumaric acid, ferulic acid, and ellagic acid, an important antioxidant polyphenol found in many fruits such as pomegranates, raspberries, strawberries, and blackberries, are just a few examples (Aytac et al., 2016; Kurkow and Loftsson, 2013).

Olive leaf extracts (rich in oleuropein) were also encapsulated in  $\beta$ -CD. Inclusion complex formation with polyphenols was found to increase their antioxidant activity, which may be attributed to an increase in their solubility (Fang and Bhandari, 2010).

Moreover, several food flavors have been encapsulated with cylodextrins (anethole, thymol and geraniol, citral, citronellal, menthol, linalool—two enantiomers of a naturally occurring terpene alcohol—and a number of other essential oils including anise, sage, cinnamon, jasmine, bergamot, orange, lemon, lime, onion, garlic, mustard, and marjoram oils), in order to decrease their volatility during thermal processing of the food product and to improve their chemical stability when exposed to air, light, moisture, and heat (Marques, 2010).

### CONCLUSION

Apart from its traditional uses in food as a carrier for natural colorants and preservatives, liposome, hydrogel and molecular encapsulation technology is gaining popularity in food fortification to add nutritional and health-promoting constituents in the development of functional foods, particularly in terms of controlled release and enhanced stability of target bioactive compounds.

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# Effect of Roasting and Drying on Phenolic Compounds and Color Properties of Domat Variety Olive Seeds

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### ABSTRACT

The olive, precious fruit, is eaten for centuries with its unique nutritional properties. The cultivation of olive was particularly concentrated in countries surrounding the Mediterranean Sea. Olive seed (inner kernel) represents approximately 2-4% of olive fruit and is a by-product of the production of stuffed and sliced olives. The research represents that olive seeds contain bioactive components close to olive and olive oil. Market demand for biological products rich in phenolic compounds, have many positive health effects such as preventing bad cholesterol on human health, reducing the risk of heart disease and cancer, has been increasing for decades. In this study, the effect of hot air drying and roasting on the color properties and phenolic compounds of olive seed seed including gallic acid, 3-hydroxytyrosol, vanillic acid, verbascoside and oleuropein has been investigated. Total phenolic compounds of olive seed were increased both in hot air drying and roasting processes compared to raw olive seeds. L\* values were found 47.15, 38.14, 47.14 and 40.93 olive seeds roasted for 25 min., roasted for 35 min., dried at 60 °C and dried at 70 °C, respectively.

Keywords: Drying, Roasting, Olive Seed, Phenolic Compounds, Color Properties



### **Bioavalibility of Olive and Olive oil Phenolic Compounds**

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#### ABSTRACT

Phenolic compounds are an important class of natural antioxidants. In small amounts in food, phenolic compounds are capable of preventing/retarding and/or reducing the oxidation witch oxidation can lead to a decrease in both nutritional value and sensory quality. Phenolic compounds are intensively studied in order to evaluate their effects on health, including antioxidant, antiallergic, antimicrobial, antithrombotic, antiatherogenic, hypoglycemic, antiinflammatory, antitumor, cytostatic, and immunosuppressive properties, and protective activities. The increase interest in extra virgin olive oil (EVOO) is mainly attributed to its high oxidative stability due to its chemical composition that includes an unsaponifiable fraction, containing important phenolic compounds. The antioxidant properties of phenolic compounds in olives and olive oil are mainly based to their reduction - oxidation (redox) properties, which allow them to act as reducing agents, hydrogen donators, and singlet oxygen quenchers. Accumulating evidence suggests that virgin olive oil may have health benefits; it can be considered as an example of a functional food containing a variety of compounds of olive oil have numerous antioxidant effects, however little is known about their bioavailability at real-life doses. The purpose of this presentation is to discuss the importance and the bioavailability of olives and olive oil phenolic compounds.

Keywords: Olives, Olive Oil, Phenolic Compounds, Antioxidants, Bioavailability

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### Evaluation of Plant Ingredients Obtained from Cannabis Species for Use as Food Additives

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### ABSTRACT

Cannabis, whose growing area has recently been expanded with legal permission in our country, is an important plant material that, attracts the attention of the pharmaceutical industry due to its content of secondary metabolites such as cannabinoids, terpenes, and phenolic compounds. Nowadays, the active substance is also used in the food industry as cannabis infused foods in many countries where cannabis is legal. However, these kinds of products have been prohibited by legal authorities in many countries, including Turkey. According to "Plant List" declared by Republic of Turkey Ministry of Agriculture and Forest, only the seed part of the Cannabis can be used, and total cannabinoid content should be less than 5 mg/kg. In this case, seeds of cannabis, whose cannabinoid contents are lower than the permitted level, can be used for food products like edible oil, dietary fiber, protein powder and snacks.

Generally, cannabis seeds contain 20–25% protein, 20–30% carbohydrates, 25–35% oil (50-70% linoleic, 15-25%,  $\alpha$ -linoleic, 10-16% oleic), 10–15% insoluble fiber and variety of minerals (P, K, Mg, Ca, Na, Fe). Cannabis oil is particularly high in polyunsaturated fatty acids, particularly omega-3 and omega-6, which account for approximately 80% of the fatty acids, while the protein contains a high amount of essential amino acids and arginine. The seed protein's high arginine content makes it particularly useful as a dietary component in foods that promote cardiovascular health. In addition, fiber content of the seed is beneficial for maintaining a healthy colon, where it acts as a prebiotic to encourage probiotic development.

In conclusion, although the cannabis vegetative plants have been restricted for using as food products in many countries due to their cannabinoid contents, some parts of the plant especially seeds could be used for production of valuable food materials. These materials should be checked for the cannabinoid contents before use as food ingredients.

Keywords: Cannabis, Food additives, Plant ingredients, Products.



# Volatiles Compositions of Strawberry Fruit During Shelf Life Using Pre and Postharvest Hexanal Treatment

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### ABSTRACT

Changes in volatile compositions were determined in 'Rubygem' strawberry varieties releated to pre and postharvest hexanal application during shelf life. Fort his concern 'Rubygem' varieties were treated with hexanal vapor and sprey applications which were (%0, %0.01 %0.02), doses before and after harvest and strawberry fruits were stored at 2°C and RH %90 conditions. Effects of hexanal sprey and vapor application of strawberry fruit volatile profiles were analyzed with HS-SPME/GC-MS tecnique. Differences among treatments were identified in volatile compositions at three days intervals during 15 days of storage. Results showed that, hexanal application type and concentration effected in the amount of volatile and composition of esters, ketones, alcohols, acids, aldehydes during shelf life. The hexanal application time and concentrations were affected on volatiles composition and amount 'Rubygem' strawberry varieties. For this reason, hexanal application has important effects on volatiles of strawberries during shelf life.

Keywords: strawberry, hexanal, volatile profile, pre-harvest, postharvest



# Efficacy of Pre-Harvest Sprey Application on Strawberry Fruit Postharvest Quality

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#### ABSTRACT

Synthetic fungicides use for prevention of fungal decay however hexanal is known as GRAS which is organic volatile compound that sprey applicaion uses for antifungal protection instead. Aimed to evaluate the efficacy of "Fortuna" cultivars on physical, chemical and microbial qualities. To this end, "Fortuna" strawberry varieties were spreyed with hexanal in three different doses (0%, 0.01% 0.02%), before harvest. After spreying strawberry fruits were stored under cold-stored conditions. Efficacy of hexanal sprey application on fruit color, firmness, soluble solids, titratable acidity total phenolic content, total flavonoid content, organic acids and soluble sugars, microbial quality in stored at 2 °C for 15 days were evaluated. Results indicated that hexanal sprey treatment positively effects to 'Fortuna' strawberry firmness, microbial qualities furthermore maintained physical quality, chemical composition and postharvest quality and the shelf life.

Keywords: hexanal, sprey, strawberry, sugar, shelf life



# The Effect of Different Drying Methods on Drying Kinetic, Bioactive and Color Properties of Cape Gooseberry Fruit

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#### ABSTRACT

In this study, the effects of freeze-drying (FD), ultrasound-assisted vacuum drying (UAVD), vacuum drying (VD) and hot air drying (HAD) methods on the drying kinetic, total bioactive content, phenolic profile, carotenoid content, and color and microstructural properties of Cape Gooseberry (*Physalis peruviana* L.) samples were evaluated. The effects of different drying methods were investigated and the drying methods significantly affected all selected parameters of dried Cape gooseberry (P<0.05). At 50 °C, the drying time of the Cape Gooseberry samples was 780 min, 960 min, and 1180 min for UAVD, VD, and HAD, respectively. According to the phenolic profile, total phenolic content (TPC), antioxidant activity (DPPH method), and total carotenoid analysis, FD retained more bioactive compounds than other methods, followed by UAVD. Compared to other dried samples, FD had less shrinkage and highest color quality. UAVD showed lower shrinkage and less drying times than VD and HAD. Thanks to higher bioactive compounds retention, better color and surface quality, and higher recovery of bioactive compounds, UAVD and VD methods should be used as alternative methods to HAD method for drying of Cape Gooseberry fruit.

Keywords: Antioxidant, carotenoid, color, SEM



# Some Rheological Characteristics of Kefiran Biopolymer Isolated from Kefir

**Grains Biomass** 

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#### ABSTRACT

Kefiran is an extracellular polysaccharide constituted by glucose and galactose produced by *Lactobacillus kefiranofaciens*. It is included into kefir grains has several health promoting properties. In the present work, kefiran was produced from kefir grains, using UHT skimmed milk as a raw material. The some rheological, physico-chemical and morphological characteristics of this polysaccharide were assessed by means of a series of techniques. The flow properties of kefiran solutions with concentrations ranging from 1.1 to 3.3 % (w/v) were examined. Kefiran showed a Newtonian behaviour at low concentrations and a pseudoplastic or shear thinning flow at the higher concentrations. Apparent viscosity decreased with the increased of shear rate according to pseudoplastic behaviour. High-performance liquid chromatography (HPLC) analysis of monosaccharides suggested that kefiran was composed of glucose and galactose in an approximate ratio of 1.0:0.4.) Spectral analysis of kefiran (FTIR) revealed the presence of carboxyl, hydroxyl, and amide groups, which corresponded to a typical heteropolymeric polysaccharide, which indicated a purified structure of isolated kefiran from kefir grains biomass. The scanning electron microscopy (SEM) images of polysaccharide kefiran showed homogeneous morphology with porous and a sponge-like structure. This polysaccharide brings a perspective for its use as gelling agent in foods.

Keywords: Exopolysaccharide, Kefiran, Rheology



# **Properties and Applications of a Branched Polysaccharide Kefiran**

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#### ABSTRACT

The synthetic polymers lead to serious environmental problems due to their non-biodegradation. Considering increased concerns about the synthetic polymers and harmful effects of the environmental pollution, biopolymers have recently attracted mounting interest among researchers and industrialists. Rising interesting in the use of eco-friendly materials as green alternatives for fossil-based biopolymers has shifted the research focus towards biopolymers. Kefiran has been introduced as a biodegradable polymer due to its nontoxicity. Kefiran, an extracellular polysaccharide with many hydroxyl groups, obtained from the microorganisms present in kefir grain. This edible biopolymer is a water-soluble branched glucogalactan with equal amounts of D-glucose and D-galactose. This review aims at presenting an overview on recent advances in the use of kefiran biopolymer with special reference to their antioxidant and antimicrobial applications, biologic activities, film-forming ability in various fields.

Keywords: Biopolymer, Exopolysaccharide, Kefiran



# The effect of Activated Carbon Obtained from Hazelnut Shell by KOH on the Removal of Aqueous Methylene Blue Solutions

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#### ABSTRACT

Activated carbon is widely used for water and gas purification, removal of metal ions and dyes, and production of high purity substances. Activated carbon can be produced from almost any carbon-rich and inexpensive biomass with low inorganic content. In this study, chemical activation method was used in the production of activated carbon from hazelnut shell with high carbon content. Chemical activation consists of a series of reactions that result in the aromatization of the carbon skeleton and formation of the porous structure as a result of dehydration of the cellulosic components of the raw material. In this study, potassium hydroxide (KOH) was used as the activating agent. In order to achieve optimum activated carbon production conditions from hazelnut shell, hazelnut shells in different mesh range (50-70-150) are impregnated with different raw material / KOH ratios  $(1/1, \frac{1}{2}, 1/3)$  and nitrogen atmosphere (flow) at 600 oC. (flow rate 200 mL / min) and heat treatment rate 10 ° C / min for 2 hours. The optimum application conditions were determined by determining the moisture content, ash ratio, when all the results obtained at the end of the study were evaluated, it was seen that the sample obtained from the study with a range of 70 mesh and 1/3ratio had the highest adsorption capacity. Due to its high surface area, low moisture and ash content, KOH was thought to be a suitable chemical in the activation of activated carbon produced from hazelnut shell. Volatile and fixed carbon amount, carbonization efficiency, iodine number, methylene blue number of the produced activated carbon samples. In this study, in order to understand the methylene blue adsorption mechanism, a series of instrumentation techniques were performed for the characterization of the adsorbent using SEM / EDS, X-Ray and FT-IR analysis.

Keywords: Activated Carbon, Hazelnut shell, KOH, Methylene Blue



# Assessment of the aroma profiles of *Spirulina Platensis* using HS-SPME–GC-MS and Determination of Antioxidant Capacity

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#### ABSTRACT

Increased population, increased nutritional needs, and the risk of diseases caused by malnutrition have prompted humanity to improve existing resources and take advantage of novel new food sources. Microalgae are microbial resources capable of generating a variety of commercially valuable chemical and biological compounds. The use of *Spirulina platensis* as human food dates back to the Aztec civilization period. The impact of extraction solvents (methanol, ethanol, and water) on the total phenolic composition and antioxidant activity of commercial *Spirulina platensis* was investigated in this study. The antioxidant capacities of extracts were assessed via the DPPH methods. GC and HS-SPME-GC / MS were used to evaluate the fatty acid profile and aroma compounds of *Spirulina platensis*, respectively. The total phenolic content (TPC) and antioxidant potential of *Spirulina* extract obtained with water were clearly higher than methanol, ethanol, and acetone extracts. The most abundant fatty acids in commercial *Spirulina* samples were determined as elaidic acid (C18: 1 trans) and arachidic acid (C20:0). The high amount of hexadecane and heptadecane as aroma compounds in *Spirulina platensis* and were identified, and  $\beta$ -ionone has also been identified, which is considered a volatile fragrance compound and is used in the food industry as an artificial sweetening ingredient.

Keywords: Spirulina platensis, aroma compounds, antioxidant potential

#### **INTRODUCTION**

Cyanobacteria, also described as blue-green algae (*Cyanophyta*), are gram-negative, photosynthetic, and prokaryotic organisms. Their habitats are mostly around the world, from aquatic to land, and salt lakes, deserts, polar regions, and hot springs (Borowitzka, 2018). Spirulina platensis is a filamentous, multicellular, survival in alkaline environments micro-algae which is commonly cultivated the worldwide as a source both of nutritious and of phycocyanin (blue pigment) utilized in food and cosmetics (Belay, 2013). Concurrently, the earliest records were showed that the Aztecs collected "Arthrospira platensis" a nutrient-rich (particularly in terms of vitamin B12 and protein) from Lake Texcoco in Mexico and consumed it as food for many years. (Ciferri, 1983; Fox, 1996). Spirulina platensis has been collected from by the local people in Lake Chad for many years and used as a nutritional supplement known as "dihe" (Siva Kiran et al., 2015). Among the Spirulina species, Spirulina fusiformis (Arthrospira fusiformis), Spirulina platensis (Arthrospira platensis), and Spirulina maxima (Arthrospira maxima) have been investigated the most extensively. They have high nutritional and potentially medicinal properties (Deng & Chow, 2010). The World Health Organization (WHO) has approved Spirulina platensis as a rich natural source of valuable secondary metabolites with potential economic applications and therapeutic characteristis that are commonly utilized to supplement human and animal food (Hadizadeh et al., 2019). Spirulina contains high amounts of protein (60-70 percent of dry weight), as well as essential fatty acids, minerals (iron, magnesium, calcium,



potassium), and vitamins (Deng & Chow, 2010). Both the National Aeronautics and Space Administration (NASA) and the European Space Agency (ESA) it is suggested as one of the primary food in long-term space missions due to its remarkably high nutritional values (Deng & Chow, 2010). The cell wall is easy to be digested by humans as it consists of 86% digestible polysaccharides (Li & Qi, 1997). *Spirulina* includes about 15% carbohydrate, 70% protein, 7% mineral, 5% fat, and 3% moisture. Unlike other plant-based proteins, it contains both essential and non-essential amino acids in a balanced way (Colla et al., 2007; Saranraj & Sivasakthi, 2014). It includes abundant the essential fatty acids such as linoleic acid,  $\gamma$ -linolenic acid (36% of total PUFAs), eicosapentaenoic acid, stearidonic acid, arachidonic acid, and docosahexaenoic acid. Furthermore, it has been stated that gamma-linoleic acid is a significant component of the *Spirulina* cell membrane (Diraman et al., 2009; Mühling et al., 2005).

Spirulina and its components have been reported to offer beneficial effects as a 'full' protein supply in several human health indications, ranging from malnutrition to antioxidant capabilities. (Ravi et al., 2010). Spirulina contains active phytochemicals such as chlorophyll (green pigment), zeaxanthin (yellow pigment), xanthophylls (myxantophyll, zeazanthin, cryptozantine, echinone), and carotenes ( $\alpha$ -carotene,  $\beta$ -carotene, euglenanone, lutein), in addition to C-phycocyanin (blue pigment) and allo phycocyanin, which are the phycobiliproteins that make up about 20% of the dry weight and have the highest economic value (El-Baz et al., 2013).

The interest in micro-algae aroma compounds has grown in recent years, owing primarily to the various structural properties and intriguing pharmacological functions of volatile compounds (Andrade et al., 2018). Dimethyl sulfide, organohalogens, and unsaturated aldehydes aremong the aroma compounds known to affect the flavor or aroma of micro-algae (Steinke et al., 2002). Furthermore, micro-algae aroma compounds are not usually associated with musty, fishy, or mud-like odors (Milovanović et al., 2015).

*Spirulina*'s functional and nutritional potential has piqued researcher's interest in recent years. The total phenolic contents, antioxidant potentials, fatty acids, and aroma compounds of *Spirulina platensis*, as well as other important quality parameters, were thoroughly investigated in this study. The fatty acid profile and aroma compounds were analyzed by GC and GC/MS, respectively.

#### MATERIAL AND METHOD

#### Chemicals

Folin-Ciocalteu reagent were bought from Merck (Darmstadt, Germany) and Trolox and 2,2-Diphenyl-1picrylhydrazyl (DPPH) were also procured from Sigma-Aldrich Chemical Co. (St. Louis, USA). All of the solvents and chemicals utilized in this research were chromatographic and analytical grade.

#### Spirulina platensis extract preparation

Egert (S1) and Algopyhco (S2) commercial *Spirulina* samples were procured from local markets in powder form in Turkey. *Spirulina* extracts were prepared with various solvents such as water, ethanol, acetone, and methanol at a 80% ratio. In this study, the reason for using different solvents is to determine if there is any difference in antioxidant and total phenolic analyzed because of solvents. In a 50 ml erlenmeyer flask, one gram of *Spirulina* was weighed, 20 ml of water was put in, and the mixture was mixed at room temperature for one night in the dark. Identical conditions were also used for extracts of acetone, ethanol, and methanol. The mixture was then centrifuged for 15 minutes at 4°C at 5.500 rpm.

#### Chemical Composition of *Spirulina platensis* The total protein content analysis



Protein analysis is required to determine the amount of a specific protein in a mixture, the amount of nonprotein hydrogen, and the nutritional value of nutrients. The basic principles of the methods are the determination of nitrogen and peptide bonds. The total protein content was analyzed using the Kjeldahl method (AOAC, 2007; Cemerolu, 2014) in this study.

#### The moisture content analysis

Moisture content was determined by drying 2 g of sample for 16 hours in a 105 ° C drying oven in fixed weighed petri dishes. The samples cooled in a glass desiccator were weighed and Official Methods of Analysis were used to calculate the moisture content (AOAC, 2007).

#### The fatty acid methyl esters (FAME) analysis

Using the Soxhlet extraction device, *Spirulina platensis* was extracted with an appropriate solvent. Gas chromatography was used in coupled with a flame ionization detector(FID) and split injection (1:50) to assess the fatty acid profile of *Spirulina*. Cold transmethylation was used to prepare fatty acid methyl esters (IOOC, 2001COI/T. 20/Doc. no. 24). A 60 m capillary column (DB23; Agilent Inc.) with a 0.25 mm I.D. and 0.25 µm film thickness was used to separate the samples (AOAC, 2007; Christie, 1989; David et al., 2005).

#### Antioxidant activity

*DPPH assay* The antioxidant activity was evaluated using the DPPH method, which was modified (Sanchez-Moreno et al.,1998) from Brand-Williams et al., (1995). In a brief, 100µl of each *Spirulina* extract was mixed with 3.9 ml of DPPH solution and incubated at room temperature for 1 hour in the dark. Using a UV–visible spectrophotometer, the absorbance was measured at 515 nm (Carry 60, Agilent, Malaysia). The antioxidant activity was determined using a calibration curve and expressed as Trolox equivalents per gram.

#### Total phenolic content analyses

The total phenolic content were detected utilizing a Folin–Ciocalteu reagent previously expounded by Cemeroğlu (2014) and Shahidi & Ambigaipalan (2015). In briefly, 7.5 ml of distilled water was added to 100  $\mu$ l of extract or standard, then mixed for 3 minutes with 0.5 ml of Folin reagent, and added 1 ml of 20% (w/v) Na<sub>2</sub>CO<sub>3</sub> (sodium carbonate) and 0.9 ml of distilled water. The absorbance was measured using a UV-VIS spectrophotomeer at 765 nm after the mixture was left to proceed for 1 hour at room temperature. TPC was measured in milligrams per gram of extract and expressed as gallic acid equivalents (GAE).

#### Aroma compounds analysis

The headspace solid phase micro-extraction (HS-SPME) method was used to extract aroma compounds in *Spirulina platensis* powders for gas chromatography-mass spectrometry (GC-MS) analysis (Agustini vd. 2019). On the "Agilent 6890N" brand gas chromatography and the associated "Agilent 5975B VL MSD" mass spectrometer, the amount, identification, and determination of aroma compounds were all performed concurrently. With the aid of a special separator, the column output is split into two and sent similarly to the FID and MSD in this method (Dean switch-Agilent). As a result of the simultaneous quantification and determined by "Agilent 7890A" brand flame ionization detector (FID) gas chromatography. A DB-WAX capillary column (60 m x 0.25 mm x 0.4 m) was used to separate aroma compounds. "Agilent 5975B VL MSD" brand mass spectrometer based on gas chromatography was used for the identification of its. Identification of peaks was made by injecting the standard solution for compounds with the standard, matching the mass spectrum of non-standard compounds with mass spectra from flavor libraries stored in computer memory (Wiley 7.0, NIST-98, and Flavor.2L). The concentrations of aroma were



determined using the internal standard method after identification (Selli et al., 2008). Each analysis was carried out three times. In the calculation, the response factor of each compound was taken into account.

#### Statistical Analysis

SPSS 22.0 with ANOVA (SPSS Inc) was used to for the statistical data analysis. Duncan's test measured the variations in the content levels of the results. Means with p-values below 0.05 have been found to be statistically important.

#### **RESULTS AND DISCUSSION**

#### The moisture and Total protein content of Spirulina samples

Protein accounts for 60-70% of the dry weight of *Spirulina*, depending on the source (Phang et al., 2000). *Spirulina* is "a full protein source" that contains essential amino acids, albeit it has less methionine, cysteine, and lysine than meat, eggs, and milk proteins. Recently, there has been increasing relevance in gaining some peptides and biofunctional proteins from micro-algae. Algal proteins have been found to be of high quality with extensive examination and nutritional studies (Becker, 2007; Samarakoon & Jeon, 2012). There are studies on the effects of proteins and peptides obtained from micro-algae such as antihypertensive, antioxidant, and anti-inflammatory (Samarakoon & Jeon, 2012; Raposo et al., 2013). The total protein and moisture content varied significantly depending on commercial samples (p<0.05). The physicochemical compositions of commercial *Spirulina platensis* in dry basis were moisture contents 8.78 (SP1) and 9.24(SP2), protein content 42.14 (SP2) and 43.90 (SP1) in percentage.

In a study, antimicrobial effect and the nutraceutical properties of Moroccan *Spirulina* were investigated, and it was stated that this Moroccan strain contained a significant amount of protein (76.65%) (Seghiri et al., 2019).

*Spirulina* protein concentration was reported to be 58.2% by Alvarenga et al. (2011), but Mbaguinan et al. (2006) was observed a higher protein content (69.2 %) in *Spirulina* obtained from Kanem Lake Chad (Mbaguinan et al., 2006).

Moisture is a crucial component in determining the quality of micro-algae. The content of dry matter of commercial *Spirulina* samples were average 90.99%. In fact, companies incorporate moisture standards in their nutritional data, with a limit of less than 9% (WHO, 2004). It's possible that the difference in value is attributable to the drying processes and time (Show et al., 2013). The packaging and storage circumstances may influence indirectly the humidity rate. Extreme drying of microalgae is not recommended since it may alter the structure of living cells, reducing their physicochemical qualities. As a result, their overall quality will be subpar (Show et al., 2013).

#### The fatty acid profile of Spirulina samples

Microalgae can accumulate lipids till 90% of their dry weight, and their mean lipid composition ranges between 1-70%. Microalgae lipids are organisms rich in long-chain fatty acids (PUFA) such as gamma linoleic acid (GLA), eicosapentaenoic acid (EPA), and arachidonic acid (ARA) (Spolaore et al., 2006; El-Baky & El-Baroty, 2013). The most abundant fatty acids for SP1 and SP2 samples were determined as arachidic acid (C20:0) and elaidic acid (C18: 1 trans), respectively. *Spirulina*'s long chain fatty acids, provide a variety of benefits and functions in dietetic and medicinal applications. The synthesis of prostaglandin hormone requires polyunsaturated fatty acids are also in field of in the food and agriculture to make omega-3 fatty acid-enriched foods (El-Baky & El-Baroty, 2013; Spolaore et al., 2006).



*Spirulina* is rich in  $\gamma$ -linolenic acid, which accounts for 36 % of total PUFAs, and also contains arachidonic acid, linoleic acid, docosahexaenoic acid, eicosapentaenoic acid, arachidonic acid, and stearidonic acid, according to Habib et al. (2008). Ten unsaturated fatty acids (palmitoleic acid, palmitoleic acid, sapienic acid, vaccenic acid, linolelaidic acid, linoleic acid, elaidic acid,  $\gamma$ -linolenic acid, oleic acid, dihomo-gamma-linolenic acid, and eicosenoic acid) and three saturated fatty acids (eicosadienoic acid were determined in the study with commercial *Spirulina* by Al-Dhabi& Valan Arasu (2016).

#### Antioxidant activity and total phenolic content and of *Spirulina* samples

*Spirulina* is considered a rich source of nutritional phenolic and flavonoid compounds due to its higher production capacity than traditional plant-based sources. The easy participation of phenolic compounds in oxidation-reduction processes occurring in internal and exterior cells results in their antioxidant action (Atouia et al., 2005). The antioxidant capacity and TPC of extracts prepared from *Spirulina* using various solvents were calculated using the gallic acid calibration curve in milligrams of gallic acid equivalents per gram of dry weight (mg GAE/g dw). A calibration curve was used to calculate the antioxidant capacity, that was expressed as a mM Trolox equivalent per gram. The commercial *Spirulina* samples extracted using various solvents have DPPH values ranging from 499 to 1746 mmol Trolox/100 g DW, and TPC values ranging from 123 to 998 mg GAE/100 g DW. Antioxidant potential and TPC of *Spirulina* extract obtained with water were clearly higher than methanol, ethanol, and acetone extracts (p<0.05). The highest TPC and DPPH value between commercial *Spirulina* samples (998 mg GAE/ 100 g DW and 1746 µmol Trolox/100 g DW, rrespectively) were determined in SP2 sample extracted with water.

The TPC of commercial *Spirulina* products ranged from 2.4 mg/g (21st Century HealthCare, Inc., Arizona) to to 24.4 mg/g (source Naturals, Inc., Santa Cruz, California), according to Al-Dhabi & Valan Arasu (2016). The existence of total phenolic components and other metabolites is linked to antioxidant capabilities, according to Wu et al. (2005).

Antioxidants are chemicals that neutralize free radicals or their effects and help to protect the body from free radicals or their actions (Wanasundara& Shahidi, 2005). Spirulina protects against lipid peroxidation and DNA damage by warn antioxidant enzyme activity and neutralizing free radicals (Abdelkhalek et al., 2015). In a study, the DPPH value of Spirulina maxima samples was determined between 23.22 to 35.62 µg/ml (Abd El-Baky et al., 2009). The micro-algae are regarded a promising therapy for fighting cardiovascular disorders due to their high antioxidant activity (Deng& Chow, 2010). Antioxidant activity was determined to be higher in the methanolic fraction (23 mg TE/g dw)(DPPH) in a study assessing the nutritional biological and properties, well as the functional composition as Moroccan Spirulina. Plants and Spirulina both a large number of antioxidant contain metabolites. Metabolites containing the phenolic functional group in their chemical structure have been discovered to have enzyme inhibition, anti-inflammatory activity, antiallergic activity, oestrogenic activity, antioxidant activity, cytotoxic anticancer activity, and vascular activity (Shukla et al., 2009).

#### Aroma compounds of Spirulina samples

Microalgae naturally produce aroma compounds such as alcohols, esters, carbonyls, terpenes, and aldehydes. In the food industry, these chemicals are utilized as flavoring additives.

There were a total of 53 aroma compounds identified and quantified. *Spirulina platensis* contains a high amount of heptadecane (80-82%), hexadecane (4-4.7%), and  $\beta$ -ionone has been widely utilized in the food industry as an artificial flavoring ingredient. Pentadecane, hexadecane, and heptadecane between the most common hydrocarbons found in *Spirulina platensis*. Hydrocarbons are produced as a result of the



decarboxylation of stearic and palmitic acids (Andrade et al., 2018; Cuellar-Bermdez et al., 2017). Although it has a high odour threshold, the presence of this compound may cause to the off-flavor of algae linked with crude fish notes (Cuellar-Bermúdez et al., 2017)

Milovanovic et al. (2015), detected odorous compounds such as 2-pentylfuran, 2-methylisoborneol,  $\beta$ -ionone and  $\beta$ -cyclochitral and hydrocarbons (alkenes and medium-length alkanes alkenes) in small amounts in *Spirulina*.

In a study explored the role of *Spirulina* on growth and chemical composition in different geographical regions, volatile compounds such as 5-hepten-2-one, 5-methyl-2-hexanone,  $\alpha$ -ionone, 6 methyl-, and epoxy- $\beta$ -ionone were determined (De Jesus et al., 2018).

In a study conducted at Ege University, volatile components were examined in the extracts of *Spirulina* platensis obtained by using various solvents (methanol, dichloromethane petroleum ether, ethyl acetate) and heptadecane and tetradecane were determined as the main components (Ozdemir et al., 2004).

#### Conclusion

The total phenolic content (TPC) and antioxidant potential of *Spirulina* extracts prepared with water were clearly higher than extracts obtained with methanol, ethanol, or acetone. The most abundant fatty acids for *Spirulina* samples were determined as arachidic acid (C20:0) and elaidic acid (C18: 1 trans). The HS-SPME-GC/MS was used to evaluate the aroma compounds of commercial *Spirulina* samples. A total of 53 aroma compounds were identified and quantified. The high amount of heptadecane (%80-82) and hexadecane (%4-4.7) were determined as aroma compounds in *Spirulina platensis*. *Spirulina* has incredible characteristics and can be regarded a "Super Food" in many ways. It has the highest concentration of nutrients of any plant, food, herb, or grain ever discovered. It includes 60% highly digestible vegetable protein, as well as particularly high levels of vitamin B-12, beta-carotene, iron, trace minerals, and important fatty acids. It has a balanced spectrum of amino acids, chlorophyll, and the blue pigment, phycocyanin, and due to its high nutrient and protein content is accepted as an ideal food supplement and an immune booster.

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# **Optimization of Low Fat High Protein Cookies Formulation: Effects of Using Butter and Composite Flour on Nutritional, Physical and Sensory Properties**

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#### ABSTRACT

In the recent century, consumers demand in the area of food products have changed significantly with lifestyle modifications related to change in the eating habit. Based on the consumers' demand, the food industry and scientists focus on low fat and calorie functional foods that prohibit nutrition related with disease and develop mental well-being and physical. This study, therefore, is aimed at improvement a low fat-high calorie functional cookie fortified with composite flour (chickpea 50%, whole grain wheat 25% and oat 25%), butter, almond, dried mulberry, egg powder and whey powder. The statistical analyses were carried out using response surface methodology (RSM). The nutritional (moisture, ash, fat, protein, carbohydrate and energy content), physical (diameter and thickness measurement, spread ratio) and sensory properties (color, appearance, taste-odor, texture, overall acceptability and affordability) of produced cookies were evaluated. The results indicate that the protein content of cookies increased from 13 g/ 100g to 24.38 g/ 100g with reduced-fat and calorie nearly 9 % in cookies. Cookie containing 15% butter and 15% composite flour has the highest score for overall acceptability and affordability among the cookie samples. The research demonstrated that low fat-high protein cookies fortified by composite flour, highly acceptable and nutrition composition can be produced.

Keywords: butter, high protein, low fat cookie, reduced calorie, response surface methodology

#### **INTRODUCTION**

Obesity and overweight have been increasing in many parts of the world (Tchang et al., 2021; Pinche et al., 2018). These increases are related with many chronic diseases such as high blood pressure, type 2 diabetes, cardiovascular disease (Seidu et al., 2021; Sun et al., 2018; Hu et al., 2016; Pergola and Silvestris., 2013). Many studies present that rising obesity and overweight are associated with eating habits like increasing fat and caloric intake (Neuhouser et al., 2012; Philipson et al., 2008; Cutler et al., 2003). Fat is a necessary nutritious for humans and also one of the essential food substances. However, a high fat diet may cause an increasing risk of many health problems (Pergola et al., 2013; Kafatos et al., 2000). In the recent century, health and wellness are crucial factors so consumers are growing aware that food impacts health conditions because of the consumption of high fat foods, adequate nutrition, sedentary lifestyle, increasing costs of medicine (Aljunid and Syed, 2021). This increasing awareness of consumers leads to understand that their food choices may have consequences for their health and are paying more attention to the health benefits of food to maintain a healthy lifestyle. The convenience food consumed by the majority of consumers is high fat intake and a lack of minerals, vitamins, dietary fiber and proteins. For these reasons, consumers tend to consume low fat, reduced fat, fat free and functional foods during the last decade (Betoret et al., 2011; Incles and Matt, 2011; Annunziata et al., 2011). One of the biggest challenges today is to improve cheap foods which have a high nutritional value and mostly acceptable to mean customers (Pimentel et al., 2021). Functional food promotes health benefits above normal nutrition. The functional food sector is one of the fastest increasing markets of the food sector all over the world. This situation can lead to different areas of



science provides factories with opportunities to improve various new functional productions (Santos et al., 2014).

Bakery productions are one of the excellent tools for fortification, value addition and feeding at a mass scale. Currently, the fortification of cookies has evolved to develop its functional and nutritional properties because of the healthy eating awareness of consumers (Awolu and Olugbenga, 2017; Iora et al., 2015). Cookies are extensively consumed ready to eat and high fat bakery production due to the cheapness, acceptable taste, availability, long shelf life and quick release of energy (Panghal et al., 2018). Fat is a basic ingredient in cookies because it is responsible for flavour, mouthfeel, texture, nutritional and sensory properties (Devi et al., 2016). However, cookies are low in, proteins, vitamins, minerals, fiber and rich in undesirable fats, carbohydrates and calories. Therefore, it is necessary to improve low fat and high nutritional value cookies. In the last decades, researchers and the food industry focus on the development of the fortification of cookies. It was reported that extruded bean flour was used in cookies in order to reduce fat, improve nutritional value and sensory properties in the final product (Moriano et al., 2019). Fortified cookie by vitamins, prebiotic fibre and reducing fat was accepted by the consumers in terms of colour, flavour and eating quality (Mudgil et al., 2017; Struck et al., 2016; Boobier et al., 2006). Moreover, cookies can be readily fortified with legume flour in order to increase protein and fibre. The using of mixed flour in cookies improvement to develop nutritional values has been reported by several studies (Bello et al., 2021; Adegunwa et al., 2020; Eyenga et al., 2020; Vieira et al., 2020; Mas et al., 2020; Wirawan et al., 2020).

Oat recently is attracted by researchers because of a high amount of beta glucan content composites of antioxidant activity and lipid fraction which has a significant impact on the nutritional and technological quality (Wirawan et al., 2020; Wang et al., 2014; Wu et al., 2011; Gray et al., 2000). Oats have a good source of beta glucan which reduce the risk of some disease like diabetes, hypertension, cardiovascular diseases and obesity (Chen et al., 2021; Loskutov et al., 2021; Zurbau et al., 2021; Wolever et al., 2019; Weickert et al., 2018; Mackie et al., 2016; Liu et al., 2014). Compare to other cereals, oats involve much more fat which is rich in polyunsaturated fatty acids. Such fat is unstable, because of the rapid oxidation process so oat productions for example, oat flakes and oat flour can be used in bakery productions like cookies in order to improve shelf-life (Forsido et al., 2021; Sagar et al., 2021). The addition of oat flour in the cookie can lead to improve protein quality, dietary fibre, shelf life and sensory properties in final production (Sudha et al., 2007).

Chickpea which is characterised by the highest nutritional value among all legumes consists of 50% carbohydrates, 17-20% protein, 5-6% of fat and 3-4% crude fiber (Alajaji et al., 2006). Moreover, chickpea seed is a good source of carotenoids, folic acid, sterols, tocopherols, B-group vitamins, microelements; magnesium, potassium, selenium, zinc, phosphorous. In addition, chickpea has great digestible protein, a complex carbohydrate with a low glycemic index and dietary fiber which can protect against cardiovascular disease. Using chickpea flour to making bakery products not only rising mineral and protein content but also contributed to lower glycemic response in consumers (Faridy et al., 2020; Gon~i et al., 2003). In recent years, studies about the applications of chickpea in bakery productions have been increasing (Dhankhar et al., 2021; Han et aş., 2021; Torra et al., 2021; Sibian et al., 2020; Benkadri et al., 2018; Mieszkowska et al., 2016; Thongram et al., 2016).

Wheat (*Triticum aestivum*) is a nutritionally substantial cereal and staple food for humans all over the world (Inyang et al., 2018; Akhtar et al., 2008). It is widely used in bakery productions such as pasta, cookies, extruded snacks and crackers due to the valuable properties of the protein (gluten) that compound elasticity and strength to get a desirable flavour and texture (Beta et al., 2020; Tropping and David, 2007; Potter and Hotchkiss, 2006). Consumption of whole grain wheat productions is known to have beneficial impacts on the human body related with their high substance of bioactive phytochemicals, minerals, vitamins, protein and dietary fiber (Ktenioudaki et al., 2015). It is reported that the consumption of whole grain wheat is



positive effects on type 2 diabetes, cancer, obesity and cardiovascular disease (Aune et al., 2016; Aune et al., 2013; Kumar et al., 2011). For this reason, whole grain wheat is attracted by customers and the food industry in recent years (Chen et al., 2017; Singh et al., 2017). Using whole grain or refined varieties can contribute significant nutritional and functional variation (Babiker et al., 2021; Sagar et al., 2021; Bakke et al., 2014: Ndife et al., 2014; Bakke and Vickers, 2007). Keep in this view, the purpose of this study to improve functional cookies that increased protein quality and quantity, soluble, insoluble dietary fiber and reduced fat, and calorie intake by prepared composite flour: oat, chickpea and whole grain wheat flour.

### **MATERIAL- METHOD**

#### Materials

Pre-cooked chickpea flour, whole grain wheat flour, oat flour, corn starch, rice flour, butter, almond, dried mulberry, egg powder, whey powder, salt, guar gum, sodium bicarbonate, and ammonium bicarbonate were purchased commercially. All chemicals and reagents were analytical grades.

#### Method

#### Determination of the Suitable Mixture and Parameter Ranges for Cookie Production

It is planned to obtain a biscuit formulation with increased protein quality and quantity by preparing a mixture consisting of pre-cooked chickpea flour, whole wheat flour, oat flour, corn starch and rice flour. As a result of preliminary studies, the mixing ratio of chickpeas, whole wheat grain and oat flour (CWO) was determined as 2:1:1. As a result of preliminary trials, CWO ratio was determined as 25-100 g / 100 g flour and 10-25 g butter as independent variables. Corn starch: rice flour at the ratio of 1:1 was used in the productions with a CWO mixture ratio of less than 100 g.

#### **Experimental Design for Cookies Formulation**

It is designed according to Response Surface Method, which is an experimental design method. For the optimization of rich protein and low-fat cookies, experiments were conducted according to a central composite design containing two independent variables which dictated 13 experimental runs. The experiments at a central point for five in order to calculate to the repeatability of the method. Independent variables used to determine optimum cookie formulations were CWO and butter content. The low and high parameters were 25-100g for. CWO and 10-25g for butter. The level of different variables is shown in Table 1.

#### **Cookie Preparation**

The production of cookies was carried out by making some modifications in the method specified in AACC Method No: 10-54. Cookies were produced by adding rice flour and corn starch (1:1) in the ratio of 0%, 37.5% and 75% to the C:W:O flour mixture. First of all, mixing flours and all powder components were made homogeneous by mixing in a mixer (Kitchenaid, U.S.) for 2 minutes. Then the specified amount of butter was added and stripped every 30 seconds and mixed for 3 more minutes. After that, different amounts of water were added according to the amount of butter and flour specified in the experimental design and the kneading process was completed by mixing for 2 more minutes by stripping every 30 seconds. The obtained dough was rested for 20 minutes and then shaped into discs of approximately 50 mm diameter and 5 mm thickness. The cookies were transferred to the oven (M4256, Simfer, Kayseri, Turkey) and baked at 180°C for 20 minutes. After baking, the cookies were cooled at room temperature nearly 30 minutes and then necessary measurements were made, and the rest of the cookies were ground in a grinder (Premier PRG 259, Istanbul Turkey) and stored in polyethylene containers for further analyses.



Independent variables	Code	-1	0	+1	
Butter content (g/100g flour)	<b>X</b> 1	10	17.50	25	
CWO (g/100g flour)	$\mathbf{X}_2$	25	62.50	100	
Production	X1		X2		
1	-1.00		-1.00		
2	1.00		1.00		
3	-1.00		1.00		
4	0.00		0.00		
5	0.00		-1.00		
6	0.00		0.00		
7	0.00		0.00		
8	-1.00		0.00		
9	1.00		-1.00		
10	0.00		0.00 0.00		
11	0.00		0.00		
12	0.00		1.00		
13	1.00		0.00		

Table 1: Experiment design of independent variables of cookie samples

#### **Proximate and Nutritional Composition**

Crude protein, crude fat, total ash, moisture was determined by employing a standard method of analysis AOAC, 1990. The total carbohydrate and energy content of cookies was calculated by using the following equations (1) and (2) (Gibson, 1990):

#### **Physical Analyses**

#### **Diameter, Thickness and Spread ratio**

The diameter (D) and thickness (T) values of the cookie samples were measured using a vernier caliper as specified in AACC Standard Method No: 10-54 (AOAC, 1990). After the diameter (mm) and thickness (mm) values of the biscuits are determined spread ratio. The spread ratio of the cookies was determined by calculating the ratio of diameter to thickness for each sample.

(2)

#### **Colour Analyses**

Colour parameters of cookie samples were measured with a Hunterlab MiniScan EZ (Reston, Virginia, USA), and the values were expressed based on the CIALAB measurement system. White and black calibration tiles were used to standardize the device before analysis. In HunterLab colour scale,  $L^*$  (lightness factor 0=black, 100 white); a\* (-*a* green, +*a* red); *b*\* (-*b* blue, +*b*\* yellow) values were recorded at the daylight (D65/10°) setting.

#### **Sensory Analyses**



The sensory evaluation of the cookies was conducted using 10 trained panelists from Hatay Mustafa Kemal University Food Engineering Department. Cookies were coded with three-digit numbers and positioned randomly. The sensory evaluation sheet was provided to the panelist and was asked to assess the colour, appearance, flavour, texture, overall acceptability and affordability according to their preferences on a 1-5 hedonic scale. According to the scale; 1: bad, 2: not enough, 3: acceptable, 4: good, 5: very good. All sensory evaluations were conducted at room temperature and water was served to panelists for mouth cleaning between the evaluations of the samples (Martinez-Flores et al., 2005; Meilgaard et al., 1999).

#### **Statistical Analyses**

In determining the effects of independent variables on dependent variables, the Central Composite Design of the Response Surface Method was used for variance analysis. As a result of the variance analysis, significant differences between group means were determined using the SPSS package program. Chemical and physical analyses were performed in triplicate and two replications for sensory evaluation.

#### **RESULT AND DISCUSSION**

#### **Proximate and Nutritional Composition**

The result of moisture, ash, fat, protein, carbohydrate and energy content of cookies are summarized in Table 2.

Sample	Moisture	Ash Fat		Protein	Carbohydrate	Energy	
Number	(%)	(%)	(%)	(%)	(%)	(kcal/100g)	
1	10,11±0,01 <sup>a</sup>	2,50±0,00 <sup>d</sup>	11,22±0,05ª	13,93±0,08 <sup>b</sup>	62,24±0,12 <sup>g</sup>	406±0,22 <sup>b</sup>	
2	13,00±0,01 <sup>i</sup>	2,94±0,00 <sup>i</sup>	19,23±0,06 <sup>i</sup>	24,17±0,50 <sup>f</sup>	40,66±0,55ª	432±0,27 <sup>h</sup>	
3	13,30±0,08 <sup>j</sup>	3,07±0,02 <sup>j</sup>	12,70±0,02°	24,38±0,68 <sup>f</sup>	46,55±0,72°	398±0,30ª	
4	12,83±0,01 <sup>h</sup>	2,59±0,00 <sup>f</sup>	14,26±0,02 <sup>d</sup>	18,40±0,15 <sup>d</sup>	51,92±0,12 <sup>e</sup>	410±0,05 <sup>d</sup>	
5	10,70±0,03°	2,26±0,00 <sup>b</sup>	14,25±0,06 <sup>d</sup>	15,01±0,55°	57,77±0,59 <sup>f</sup>	419±0,37 <sup>g</sup>	
6	12,23±0,05 <sup>e</sup>	2,55±0,00 <sup>e</sup>	14,85±0,05 <sup>e</sup>	18,35±0,07 <sup>d</sup>	52,02±0,07°	415±0,46 <sup>e</sup>	
7	$12,45\pm0,04^{f}$	2,68±0,01 <sup>g</sup>	15,55±0,08 <sup>f</sup>	18,28±0,54 <sup>d</sup>	51,04±0,64 <sup>d</sup>	417±0,27 <sup>f</sup>	
8	12,32±0,07 <sup>e</sup>	2,56±0,02 <sup>e</sup>	11,51±0,10 <sup>b</sup>	15,60±0,11°	57,99±0,26 <sup>f</sup>	398±0,29ª	
9	10,26±0,01 <sup>b</sup>	2,18±0,00ª	17,02±0,08 <sup>g</sup>	13,00±0,50ª	57,55±0,56 <sup>f</sup>	435±0,39 <sup>j</sup>	
10	12,89±0,03 <sup>h</sup>	2,43±0,01°	14,20±0,00 <sup>d</sup>	18,09±0,47 <sup>d</sup>	52,40±0,49°	410±0,09 <sup>d</sup>	
11	12,71±0,01 <sup>g</sup>	2,43±0,00°	14,20±0,01 <sup>d</sup>	18,60±0,26 <sup>d</sup>	52,05±0,25 <sup>e</sup>	410±0,01 <sup>d</sup>	
12	13,29±0,08 <sup>j</sup>	2,79±0,01 <sup>h</sup>	14,20±0,04 <sup>d</sup>	24,27±0,04 <sup>f</sup>	45,45±0,02 <sup>b</sup>	407±0,50°	
13	12,00±0,13 <sup>d</sup>	2,49±0,01 <sup>d</sup>	18,28±0,04 <sup>h</sup>	20,23±0,38°	47,00±0,49°	433±0,73 <sup>i</sup>	

Table 2: Nutritional properties of low-fat high protein cookies

<sup>a-j</sup> For each parameter, different superscript letters indicate a significant difference (p<0.01) among cookie samples

#### **Moisture Content**



The result of moisture content of cookies ranged between 10.11% and 13.30%. The moisture content of a production effect on the quality of foods (Bugusu et al., 2001). The effect of variables on the moisture content is shown in Fig. 1 the 3D plot. The raising of the ratio of CWO in cookies lead to increase moisture content and this increasing significantly important p<0.01.

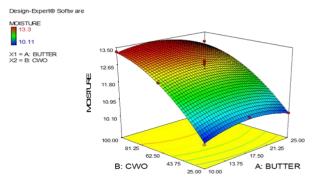


Figure 1: Response plot showing the effect of CWO concentration and butter on the moisture of cookie samples

#### Ash Content

The value of ash content of cookies ranged from 2.18% to 3.07%. While sample number 3 has the highest ash value, sample number 9 has the lowest ash content. The effect of variables on the ash content is shown in Fig. 2 the 3D plot. The ash values of cookies increased significantly with the rising of CWO ratio in the cookies p<0.01. A similar result was found that using composite flour increase the ash content of cookies (Sharma et al., 2013).

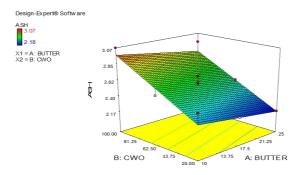


Figure 2: Response plot showing the effect of CWO concentration and butter on ash of cookie samples

#### **Fat Content**

Sample number 2 has the highest fat value (19.23%) and sample number 1 has the lowest fat value (11.22%). The response surface plot (Fig. 3) showed that increased the ratio of CWO and butter led to an increase in fat content in cookies. The fat values of cookies increased slightly with the increasing of CWO ratio in the cookies p<0.05. The fat content of mixed flour was found similarly 18-22% (Sibian et al., 2020), 15.75% (Awolu and Olugbenga, 2017), 14. 1% (Olagunju et al., 2013). Moreover, the increasing ratio of using butter significantly increases the fat content of cookies p<0.01.



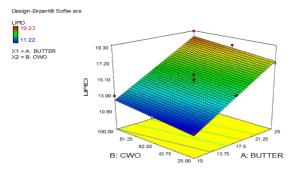


Figure 3: Response plot showing the effect of CWO concentration and butter on lipid of cookie samples

#### **Protein Content**

The protein content values varied from 13 to 24.38 g/100 g. The high protein content may be attributed to the presence of chickpea flours. Protein is a significant component that improves the nutrient properties of composite flours (Gruss and Teri, 2009). The response surface plot (Fig.4) shows the effects of variables on protein content in cookies. The increasing in the percentage of using CWO in cookies can lead to increase significantly protein content in cookies p<0.01. The results of protein content obtained in this study in close agreement with to rise in protein content using composite flour reported by several studies (Giri et al., 2019; Awolu et al., 2016; Izembaeva et al., 2013; Sharma et al., 2013; Peter et al., 2012; Gupta, 2001; Sathe, 1983).

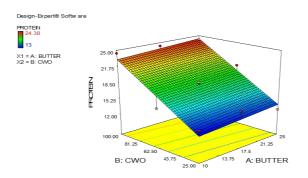
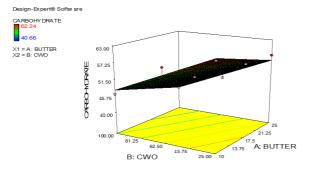


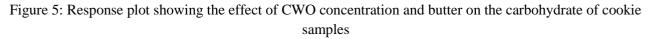
Figure 4: Response plot showing the effect of CWO concentration and butter on the protein of cookie samples

#### **Carbohydrate Content**

The results of the carbohydrate content in cookies ranged from 40.66% to 62.24%. The response surface plot (Fig.5) shows that the rising percentage of using CWO and butter leads to a decrease significantly in carbohydrate content in cookies p<0.01. Similarly, the result was found in the study which fortified cookies using chickpea and wheat flour, the carbohydrate content of cookies ranged from 47.30% - 50.03% (Sharma et al., 2013). Another study reported that increasing of using chickpea flour can decrease the carbohydrate content (Torra et al., 2021). The comparable, outcome of the study which using wheat flour in cookies shows that the content of carbohydrates was found 44- 46% (Giri et al., 2019).







#### **Energy Content**

As it can be seen in Table 2 the energy content of cookies ranged between 398 and 435 kcal. As the percent of CWO increases in cookies, there was a significant decrease in energy content p<0.05. However, the raising of using butter ratio in cookies can lead to increase significantly energy content in cookies p<0.01. The effects of the percentage of using CWO and butter on the energy content are shown in Fig. 6 the 3D plot. This finding corresponded to previous studies which show using mixing flours leads to increase energy content (Dhankhar et al., 2021; Kulthe et al., 2014; Sharma et al., 2013).

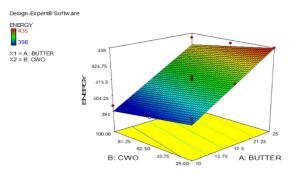


Figure 6: Response plot showing the effect of CWO and butter concentration on energy content of cookie samples

#### **Diameter, Thickness and Spread Ratio**

Diameter values of cookie samples ranged from 4.40 to 4.70. The effect of variables on the diameter content is shown in Fig.7 in the 3D plot. It can be seen in Fig.7 that the diameters of cookie samples are not affected significantly by the ratio of CWO and butter. The effects of the percentage of using CWO and butter on the thickness are shown in Fig. 8 the 3D plot. While the percentage of CWO in cookie samples affect significantly thickness of cookie samples, there is no relationship between the concentration of butter in cookie samples and thickness. The value of thickness in cookie samples is decreasing from 0.82 to 0.52 with an increasing CWO percentage in cookie samples. As it can be seen in Fig. 8 the increasing in the percentage of CWO in cookie samples can lead to a decrease significantly in the value of thickness p<0.05. Spread ratio values of cookie samples ranged from 5.78 to 8.46. The response surface pilot Fig. 9 shows that there is a significant effect of the percentage of using CWO on spread ratio in cookies. Nevertheless, the percentage of using butter in cookies does not affect the spread ratio statistically. Spread factor results showed that as the concentration of incorporated treatments of whole grain wheat increased, the spread ratio increased significantly p<0.05. Higher protein content impacts negatively on the spread ratio in cookies (Gaines et al.,



1989). However, cookies developed by a high percentage of chickpea flour, despite having high protein content demonstrated a higher spread ratio. This anomalous behaviour could be attributed to the reduced viscosity of chickpea dough and it causes a higher spread ratio. A previous study indicates a decrease in the viscosity of dough with the addition of chickpea flour (Barron et al., 1993). A similar result found that lower is the viscosity of dough, faster is the spreading rate of cookies (Hoseney et al., 1994).

#### **Physical Analyses**

The result of diameter, thickness, spread ratio, and colour of cookie samples are shown in Table 3. and Table 4. respectively.

Sample Number	Diameter	Thickness	Spread Ratio
1	4,74±0,05	$0,82{\pm}0,04^{\rm h}$	5,78±0,33ª
2	4,40±0,00	0,52±0,04ª	$8,46\pm0,66^{f}$
3	4,48±0,04	0,62±0,04 <sup>b,c</sup>	7,23±0,54 <sup>d,e</sup>
4	4,64±0,09	0,7±0,07 <sup>d,e,f</sup>	6,63±0,62 <sup>b,c,d</sup>
5	4,58±0,04	$0,78{\pm}0,04^{ m g,h}$	5,87±0,30ª
6	4,48±0,04	$0,60{\pm}0,00^{b}$	$7,47\pm0,07^{e}$
7	4,70±0,07		6,91±0,51 <sup>c,d,e</sup>
8	4,64±0,05		6,27±0,45 <sup>a,b,c</sup>
9	4,60±0,00	$0,78{\pm}0,04^{ m g,h}$	5,90±0,37ª
10	4,62±0,08	0,66±0,05 <sup>b,c,d</sup>	7,00±0,59 <sup>d,e</sup>
11	4,66±0,05	0,76±0,05 <sup>f,g,h</sup>	6,13±0,51 <sup>a,b</sup>
12	4,70±0,07	0,68±0,04 <sup>c,d,e</sup>	6,91±0,59 <sup>c,d,e</sup>
13	4,6±0,07	$0,78{\pm}0,04^{ m g,h}$	$5,90{\pm}0,45^{a}$

Table 3: Diameter, thickness and spread ratio values of low fat high protein cookie samples

<sup>a-h</sup>For each parameter, different superscript letters indicate a significant difference (p<0.01) among cookie samples

Table 4: L, a, b values of cookie samples.

Sample Number	L value	a value	b value
1	57,83±0,06°	12,32±0,03 <sup>j</sup>	$28,03{\pm}0,08^{ m c,d}$
2	56,78±0,04 <sup>b</sup>	12,72±0,01 <sup>k</sup>	28,22±0,04°
3	56,67±0,02 <sup>b</sup>	11,55±0,07 <sup>i</sup>	26,68±0,03ª
4	$64,11\pm0,08^{j}$	9,15±0,00 <sup>a</sup>	$27,42\pm0,02^{b}$
5	65,07±0,03 <sup>1</sup>	10,64±0,05 <sup>d</sup>	29,95±0,06 <sup>i</sup>
6	63,81±0,02 <sup>i</sup>	9,80±0,01 <sup>b</sup>	28,31±0,05°
7	62,52±0,01 <sup>h</sup>	9,96±0,17°	28,55±0,10 <sup>f</sup>
8	59,97±0,17 <sup>d</sup>	10,75±0,01e	28,17±0,24 <sup>d,e</sup>
9	64,93±0,05 <sup>k</sup>	10,73±0,02 <sup>d,e</sup>	30,85±0,06 <sup>j</sup>
10	61,13±0,09 <sup>f</sup>	11,03±0,01 <sup>g</sup>	28,91±0,01 <sup>g</sup>
11	60,95±0,04 <sup>e</sup>	10,94±0,01 <sup>f,g</sup>	29,06±0,04 <sup>h</sup>
12	56,41±0,10 <sup>a</sup>	10,92±0,02 <sup>f</sup>	28,01±0,01°
13	62,01±0,04 <sup>g</sup>	11,33±0,04 <sup>h</sup>	30,04±0,07 <sup>i</sup>

<sup>a-j</sup> For each parameter, different superscript letters indicate a significant difference (p<0.05) among cookie samples



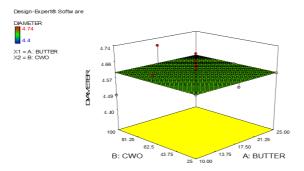


Figure 7: Response plot showing the effect of CWO concentration and butter on the diameter of cookie samples

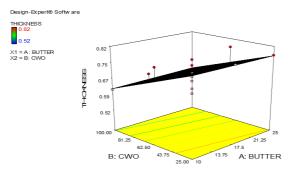


Figure 8: Response plot showing the effect of CWO concentration and butter on the thickness of cookie samples

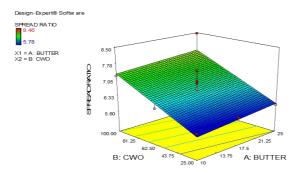


Figure 9: Response plot showing the effect of CWO concentration and butter on spread ratio of cookie samples

#### Colour

The effect of variables on the L value is shown in Fig.10 in the 3D plot. The L values of cookies are affected significantly by the CWO concentration in cookies p<0.01. Moreover, the interaction between butter and CWO concentration can impact on the L values of cookies statistically p<0.05. As it can be seen from Fig. 11 the butter affect significantly on a value of cookies samples p<0.05. The response surface plot Fig. 12 shows that increasing the ratio of CWO in cookies led to decrease significantly in the b values of cookie samples p<0.05. However, the b values of cookie samples increases significantly with the rising of the



percentage of butter in cookie samples p<0.01.

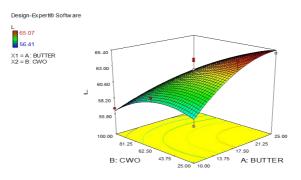


Figure 10: Response plot showing the effect of CWO concentration and butter on L value of cookie samples

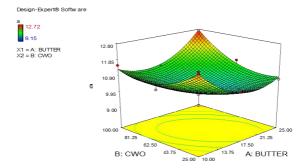
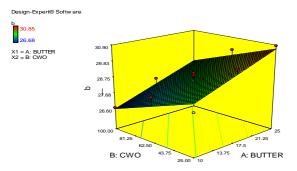
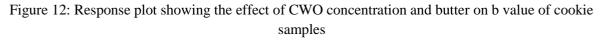


Figure 11: Response plot showing the effect of CWO concentration and butter on a value of cookie samples





#### **Sensory Evaluation of Cookies**

Sensory evaluation of cookie samples is summarized in Table 5.



Sample Number	Colour	Apperance	Flavour	Texture	Overall Acceptability	Affordability
1	3,89±0,60 <sup>a,b</sup>	4,00±1,22	4,22±0,97 <sup>e,f</sup>	4,56±0,73 <sup>d</sup>	4,11±0,78 <sup>d,e</sup>	4,00±1,00 <sup>c,d</sup>
2	2,89±1,05ª	3,78±0,83	2,67±1,12 <sup>a,b</sup>	2,22±0,83 <sup>a,b</sup>	2,56±0,53ª	2,22±0,83ª
3	2,89±1,17 <sup>a</sup>	3,22±0,97	2,33±0,71ª	2,00±0,87ª	2,44±0,73ª	2,22±0,83ª
4	4,33±0,87 <sup>b</sup>	4,33±0,71	3,33±0,71 <sup>b,c,d,e</sup>	3,00±0,71 <sup>b,c</sup>	$3,44\pm0,88^{b,c,d}$	2,78±1,20 <sup>a,b</sup>
5	4,11±1,36 <sup>b</sup>	3,89±1,36	4,11±0,78 <sup>d,e,f</sup>	4,44±0,73 <sup>d</sup>	4,11±0,78 <sup>d,e</sup>	4,00±1,00 <sup>c,d</sup>
6	4,00±0,71 <sup>b</sup>	4,22±0,67	3,67±0,87 <sup>c,d,e,f</sup>	3,00±0,71 <sup>b,c</sup>	3,44±0,73 <sup>b,c,d</sup>	$3,11\pm0,78^{a,b,c}$
7	3,56±0,88 <sup>a,b</sup>	3,44±0,88	3,78±0,97 <sup>c,d,e,f</sup>	4,11±0,78 <sup>d</sup>	3,56±0,53 <sup>b,c,d,e</sup>	3,44±0,73 <sup>b,c,d</sup>
8	3,89±0,93 <sup>a,b</sup>	3,89±1,27	3,11±1,17 <sup>a,b,c</sup>	2,89±0,93 <sup>b,c</sup>	3,22±0,97 <sup>a,b,c</sup>	2,89±1,27 <sup>a,b</sup>
9	4,11±0,93 <sup>b</sup>	4,11±0,93	4,33±0,71 <sup>f</sup>	$4,44{\pm}0,88^{d}$	4,33±0,87°	4,22±1,09 <sup>d</sup>
10	3,44±1,13 <sup>a,b</sup>	3,44±0,73	3,22±0,44 <sup>a,b,c,d</sup>	2,67±0,50 <sup>a,b,c</sup>	3,00±0,71 <sup>a,b</sup>	2,78±0,67 <sup>a,b</sup>
11	4,11±0,60 <sup>b</sup>	3,56±0,88	3,89±1,05 <sup>c,d,e,f</sup>	3,11±1,05°	3,67±0,71 <sup>b,c,d,e</sup>	3,56±0,88 <sup>b,c,d</sup>
12	3,56±1,13 <sup>a,b</sup>	3,56±1,01	3,11±0,78 <sup>a,b,c</sup>	2,44±0,73 <sup>a,b,c</sup>	2,89±0,78 <sup>a,b</sup>	2,78±1,09 <sup>a,b</sup>
13	3,78±1,09 <sup>a,b</sup>	3,56±1,24	4,11±0,78 <sup>d,e,f</sup>	$4,00{\pm}0,87^{d}$	4,00±0,87 <sup>c,d,e</sup>	4,11±0,78 <sup>c,d</sup>

Table 5: Sensory scores for low-fat high-protein cookie samples

<sup>a-f</sup> For each parameter, different superscript letters indicate a significant difference (p<0.05) among cookie samples

The scores for colour, appearance, flavour, texture, overall acceptability and affordability were ranged from 2.89 to 4.33, 3.22 to 4.22, 2.33 to 4.33, 2.00 to 4.56, 2.44 to 4.33 and 2.22 to 4.22 respectively based on the panelist assigned for each parameter using a 5- point hedonic scale. There were significant differences between the treatment of fortification of the ratio of CWO and butter in cookie samples in terms of colour, flavour, texture, overall acceptability and affordability. However, the statistical results indicate that no differences in appearance were found between cookie samples in terms of the ratio of CWO and butter. As it can be seen from Fig. 13, CWO ratio increased, the score given to colour in the sensory evaluation was decreased and this ratio was found to be statistically significant p < 0.05. The response surface plot Fig. 14 shows that the increase in the percentage of CWO in cookie samples can lead to decrease significantly in the value of the score given to flavour p < 0.01. It can be seen in Fig.15 that the scores given to the texture of cookie samples were decreased significantly by the ratio of CWO increased. Similarly, the increasing in the CWO ratio in cookie samples affect negatively the score given to overall acceptability and this ratio was found to be statistically significant p < 0.01. Moreover, the effect of variables on overall acceptability is shown in Fig. 16 the ratio of CWO in cookies affects the affordability of the products. This effect was found statistically significant. As the CWO ratio increased, the score given to the affordability by the panelists decreased p < 0.01. The effect of variables on affordability is shown in Fig.17 Sample number 9 the highest score for overall acceptability and affordability.

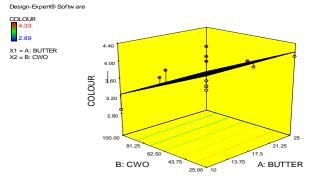


Figure 13: Response plot showing the effect of CWO concentration and butter on the score given



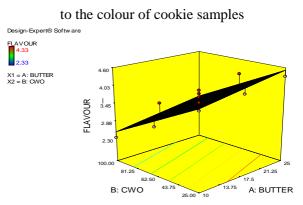


Figure 14: Response plot showing the effect of CWO concentration and butter on the score given to the flavour of cookie samples

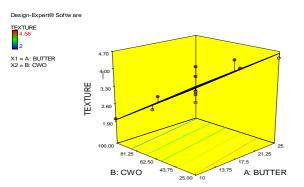


Figure 15: Response plot showing the effect of CWO concentration and butter on the score given to the texture of cookie samples

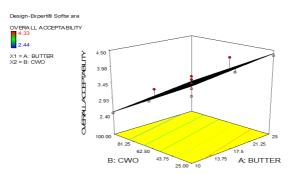


Figure 16: Response plot showing the effect of CWO concentration and butter on the score given to the overall acceptability of cookie samples



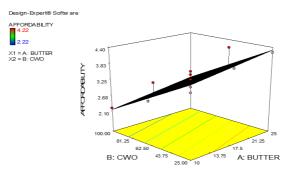


Figure 17: Response plot showing the effect of CWO concentration and butter on the score given to the affordability of cookie samples

#### CONCLUSION

It can be concluded from the above research that composite flour consisting of 32% chickpea, 16% whole grain wheat, and 16% oat flours can be used successfully to replace 100% of the refined wheat flour in order to formulate healthy low fat high protein cookies having the additional benefit of daily nutrition. Thus from the results, it may be concluded that cookies high in proteins (nearly 100% increase) and low in calories (nearly 9 % decrease) could be prepared with composite flour. Also from the physical analyses, it may be concluded that cookies can be acceptable for sensory quality. The optimized cookie production chosen by the software was 25% chickpea flour, 12.9 % whole grain wheat flour, 12.9% oat flour and 15% butter that gives the value of protein %20. The formulated functional cookies had higher protein content than cookies in the literature. The research demonstrated that highly acceptable reduced-calorie with high protein cookies which are fortified by composite flour, almond, butter, dried mulberry highly acceptable and nutrition composition, can be produced.

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# Optimization of Gluten Free Cookies Produced with Nutritious Ingredients: Evaluating a New Food Product

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#### ABSTRACT

Especially; in order to reduce the risk of many diseases like diabetes mellitus, hypertension, celiac, cardiovascular disease, hypertension, the need for new bakery products with low sugar and increased protein amount is increasing day by day. Nutritionally balanced gluten-free cookies were prepared from composite flour (chickpea, buckwheat, corn, almond milk waste flour) with high protein and low sugar were developed using the central composite design of the response surface. ANOVA of models showed that the moisture, ash, protein, fat, carbohydrate, L\*, a\*, b\*, hardness, overall acceptability were significant (P<0.01); and energy, diameter, thickness, spread ratio and mouthful were significant (p<05). The result of the study determined the optimization of gluten free cookies with protein content and spread ratio, overall acceptability maximum are 66 g composite flour, 20 g powder sugar, 15 g protein and 7.70 spread ratio.

Keywords: nutritional composition, low sugar, gluten free cookie, response surface methodology

#### INTRODUCTION

Biscuits are an easy-to-carry snack product, inexpensive, ready to eat, with a long shelf life consumed by all age groups. Biscuits are generally formulated from flour, oil and sugar. The percentage of sugar is approximately between 30 and 40% (Nagi et al., 2012, Van der Sman et al., 2019) Sucrose is used because of its significant effect on the formation of a dough, structure and texture as well as on the quality of the final product (Devi et al., 2016, Zoulias et al., 2002, Laguna et al., 2013). However, it is well known that high sugar intake is related with a several of health issues such as diabetes mellitus, obesity, tooth decay, cardiovascular disease(Yin et al., 2021, Milner et al., 2020). Recently, consumers' demand for healthier products with increasing nutritional awareness; the interest in low-sugar bakery products is increasing significantly. This situation leads the food industry to develop new products with high nutritional value (high protein and dietary fibre, low fat, sugar and salt, etc.) or to reformulate existing products.

Celiac disease (CD) is a chronic systemic autoimmune disorder where the body cannot handle the effect of gluten. If left untreated CD can lead to serious complications, such as intestinal cancers, osteoporosis, anaemia and infertility (Elli et al., 2019, Dimidi et al., 2021, Grogan et al., 2021). Celiac patients have several nutritional deficiencies like protein, mineral (iron, calcium and magnesium), vitamins, minerals and dietary fibre. The only treatments that lifelong gluten-free diet eliminates these deficiencies (Hallert et al., 2002, Barton et al., 2007, Kinsey et al., 2008, Saturni et al., 2010).

In recent years, healthy eating-conscious celiac patients prefer gluten-free grain-based foods more due to the raising symptoms and of gluten intolerance like weight fluctuation, fatigue, bone and joint pain, bloating (Houben et al., 2012, Zaharkova et al., 2019, Hu et al., 2020). Moreover, people who have not been diagnosed with celiac disease and gluten sensitivity continue to adopt this diet as lifestyle choice. (Stantiall and Serventi, 2017, Arslain et al., 2021). Gluten-free (GF) products have a very poor nutritional profile, as they are mostly made from refined flours, sugar, fat and starches (usually rice flour and corn or potato starch) (Šarić et al., 2019). The use of legumes, pseudo cereals, cereals and nuts may improve the nutritional quality of gluten-



free baked goods (Ibanoglu et al., 2006). Currently, the production amount and variety of gluten-free food products has been gradually increased. Pseudo grains (Gallagher, 2004, Lee et al, 2009), nuts, legumes, dried fruits and vegetables were included to improve the nutritional value of GF breads, cookies, pastas, snack cereals, crackers (Ibanoglu et al., 2006, Dimidi et al., 2021, Mosca and Pellegrini 2021, Hu et al., 2020). For instance, functional cookie was developed with enhancing flavonoids, protein, dietary fibre, antioxidants, polyunsaturated fatty acids, minerals, phenolic, with highly acceptable (Goyat et al., 2018). Addition, the study conducted by Sionons and Holl (2018) showed that biscuits are produced by pinto beans and tiger nut flour 40% were acceptable sensory properties at the same value as cookies produced by pre-treated pinto beans. Valitutti et al., (2017) indicated that CD patients mostly prefer biscuits and crackers among the bakery products.

For these reasons, the nutritional content of GF cookies needs to be balanced and made more nutritious especially for celiac patients and gluten intolerance people. In the light of this information, the aim of this study is to develop reduced sugar, high nutritional value cookie with acceptable sensory and texture attributes from composite flour which consist of chickpea, buckwheat, corn, and almond milk waste flour.

#### MATERIAL AND METHODS

#### Materials

Buckwheat flour, corn flour, chickpea flour, almond milk waste flour, rice flour, flax seed, powdered sugar, butter, egg powder, instant skim milk powder, salt, guar gum, sodium bicarbonate, and ammonium bicarbonate were commercially purchased. All chemicals and reagents were analytical grade.

#### Methods

#### Experimental design, modelling and optimization

Response surface methodology was used to determine the impacts of two independent variables, composite flour (A) and powdered sugar (B) on nutritional colour, physical properties composition, and sensory attributes (dependent variables) of GF cookies. Range of upper and lower values of independent variables were 25-75 g/100 g and 20-40 g respectively was used in order to optimize and evaluate the impact of independent variables on the depend variables a central composite design (CCD) (Table 1). The relationship between the independent variables were analysed by fitting a proper model, a second order polynomial model.

$$Y = \beta_0 + \sum_{i=1}^n \beta_i X_i + \sum_{i=1}^n \beta_{ii} X_i^2 + \sum_{i \neq j=1}^n \beta_{ij} X_i X_{ij} + e$$

That Y is the depend variables,  $b_0$  is the centre point of the model (0,0), bi is the regression coefficient, bii is the quadratic cofficient, bij is the the value of interaction of independent variables,  $X_i$  Xi and Xj are the independent variables of model.

Variance and regression analyses of the design were performed by Design Expert 7.0 software (Stat-Ease Inc., USA). Numerical optimization was used by the maximum values of protein content, spread ratio and overall acceptability in order to optimum values of independent variables (Myers and Montgomery, 2002).



Independent variables			
Run number	Composite flour	r Powdered sugar (g/100 g)	
	(g/100g)		
1	50.0	30.0	
2	50.0	30.0	
3	14.64	30.0	
4	50.0	30.0	
5	50.0	15.86	
6	50.0	30.0	
7	75.0	20.0	
8	50.0	30.0	
9	25.0	20.0	
10	25.0	40.0	
11	85.36	30.0	
12	75.0	40.0	
13	50.0	44.14	

#### Table 1. Experimental design of independent variables for gluten free cookies

#### **Production of GF Cookie**

The gluten free cookies were produced with modifying method AACC (Method no: 10-54, 2000). Composite flour blends prepared to by mixing chickpea, buckwheat, corn and almond milk waste flours (1:1:1:1) in different proportions. Composite flours and all cookie components were made homogeneous dough in a mixer (Kitchenaid, U.S.) for 5 minutes. All the made dough's were wrapped in polyethylene stretch film and rested at room temperature for 30 minutes. Then dough were shaped it discs of nearly 50 mm diameter and 5 mm thickness. After that, the cookies baked in oven (M4256, Simfer, Kayseri, Turkey) at 180±2 °C for 20 minutes. Then, cookies were cooled down at room temperature for 30 minutes then physical analyses such as size measurements, colour measurements, and sensorial evaluation were performed. Finally, cookies were ground in a grinder (Premier PRG 259, Istanbul Turkey) and stored in polyethylene containers until further analyses.

#### Nutritional composition of GF cookies

Moisture, ash, crude protein and crude fat were analysed (AOAC, 2005). Total carbohydrate content was determined by deduction method. The energy content was calculated by using the following formula;

Energy value = (Cal/100g) = (4 x protein) + (9 x fat) + 4 x carbohydrate)

#### Physical attributes of GF cookies

#### Diameter, thickness and spread ratio

Thickness and diameter and of GF cookies were determined by measuring with a manuel vernier calliper. Spread ratio was determined by calculating the ratio of the value of diameter to the average value of thickness (Zouliaset et al., 2000).

#### **Textural hardness**

The hardness of the GF cookies were evaluated by a Texture analyser (TA-XT2, StableMicro Systems, UK) equipped with a 3-point bending rig (HDP/M3PB). Vz<321"t 2 mm s-1 crosshead speed. Peak force (N) required to break the cookie was recorded (Hamdani et al., 2020).



#### **Colour analyses**

The colour intensity of the GF cookies samples were measured using Chroma meter Minolta CR-400 (Konica Minolta Co., Osaka, Japan). The instrument was calibrated using a standard light white calibration tile before analysis. The  $L^*$  (lightness),  $a^*$  (redness-greenness) and  $b^*$  (yellowness-blueness) values were recorded at the daylight separately (Olawoye and Gbadamosi, 2020).

#### **Sensory evaluation**

Sensory evaluation of GF cookies samples on 5-point hedonic scale (5- liked extremely and 1- disliked extremely) conducted by ten trained panellists (both male and female) from Hatay Mustafa Kemal University Food Engineering and Agriculture Engineering Department (Hejrani et al., 2017).

#### **RESULT AND DISCASSION**

#### Nutritional composition of GF cookies

The result of the nutrient composition of GF cookies are shown in Table 2 and it was found that moisture (3.34 to 4.97%), ash (1.60 to 2.44%), protein (11.68 to 15.96%), fat (21.46 to 23.19%), carbohydrate (52.84 to 63.12%), and energy value (478 to 491Cal/100 g). The moisture, ash, protein and fat content of GF cookies were found significantly increased with the increase in however, carbohydrate, and calorie content found decreased. The protein amount of gluten-free cookies increased linearly with the raising in amount of composite flour in the cookies and this increase was found to be statistically significant (p<0.01). One of the aims of this study was to increase the amount of protein. The amount of protein varies between 10.46 and 16.96. This value is considerably higher than the protein amount of GF cookies which are commercially sales on market. The implication of these results is that the composite flour consisting were good sources of protein. Similar result was found by Giri and Sakhale (2021) increase in the level of incorporation of orange fleshed sweet potato flour in GF cookies lead to increase significantly crude fibre, moisture, ash, carbohydrate content of cookies samples however, protein, fat, and carbohydrate content decreased.

Sample	Moisture	Ash	Protein	Fat	Carbohydrate	Energy
Number	(%)	(%)	(%)	(%)	(%)	(kcal/100g)
1	4.43±0,10	$2.09{\pm}0,08$	13,53±0,20	22,27±0,10	57,68±0,20	485
2	$3.95 \pm 0,11$	$2.11\pm0,08$	$13,81\pm0,09$	22,49±0,50	$57,\!64{\pm}0,\!40$	488
3	$3,36\pm0,10$	$1,60\pm0,12$	$10,46\pm0,12$	21,46±0,25	63,12±0,20	487
4	$4,18\pm0,01$	$2,15\pm0,05$	$13,82\pm0,12$	22,35±0,21	57,50±0,25	486
5	$4,79{\pm}0,04$	$2,00\pm0,03$	$13,84{\pm}0,08$	22,41±0,34	$55,96{\pm}0,40$	484
6	$3,92{\pm}0,09$	$2,00\pm0,04$	$13,83{\pm}0,54$	22,65±0,10	57,60±0,20	490
7	$4,69{\pm}0,05$	$2,36\pm0,02$	$15,25\pm0,11$	23,19±0,40	54,51±0,40	478
8	$4,62{\pm}0,10$	$1,96{\pm}0,07$	$13,50\pm0,14$	22,34±0,25	57,58±0,25	485
9	$3,56\pm0,01$	$1,82{\pm}0,06$	$11,83\pm0,10$	22,22±0,46	60,57±0,55	490
10	$3,45{\pm}0,04$	$1,84{\pm}0,01$	$11,\!68{\pm}0,\!09$	22,25±0,50	$60,78{\pm}0,25$	490
11	$4,97{\pm}0,06$	$2,44{\pm}0,08$	$15,96\pm0,10$	22,79±0,25	52,84±0,25	489
12	4,21±0,11	$2,31\pm0,11$	$15,37{\pm}0,05$	23,19±0,05	54,92±0,10	490
13	$3,34{\pm}0,18$	2,11±0,16	$13,90{\pm}0,05$	22,52±0,54	58,13±0,50	491

Table 2. Nutritional properties of GF cookies

The fitted regression model developed for moisture, ash, protein, fat, carbohydrate and energy are given in Table 3. Fit summary statistics suggested lineer model for moisture, ash, protein, carbohydrate and energy;



and suggested quadratic model for fat were generated in the regression model are present. The models for moisture, ash, protein, fat, carbohydrate (P<0.01) and energy (P<0.05) were all significant, and their correlation coefficients were all higher than 0.80. Therefore, the proposed model is accurate for predicting changes in these responses with the experimental domain.

Parameters	Model	Model R2	F Value	value Prob > F	Final equation in terms of coded factors
Moisture	lineer	0.8859	18.35	0.0500	Moisture = +4.11+0.52*A-0.33*B
Ash	lineer	0.8043	11.38	0.0500	Thickness =+0.62+6.066E-004* A+0.050* B
Protein	lineer	0.8858	7.07	0.0500	Spread Ratio =+7.20+0.095 *A-0.46 * B
Fat	Quadratic	0.9969	44.94	0.0500	Hardnes =+19.95+5.15* A+0.49* B- 0.14* A * B-1.07A <sup>2</sup> +0.046* B <sup>2</sup>
Carbohydrate	lineer	0.9719	17.32	0.0500	Carbohydrate =+57.60-3.31 *A+0.46 * B
Energy	lineer	0.8838	6.49	0.0500	Energy =+7.20+0.095 *A-0.46 * B

Table 3. Analysis of predicted model equation for the nutritional composition of GF cookies

#### Physical and textural properties of GF Cookies

The diameter, thickness, and spread ratio of the GF cookies samples are shown in Table 4. The diameter and thickness of the GF cookie samples ranged from 4.36-4.59 mm to 0.53-0.70 mm, respectively. The fitted regression equations diameter, thickness, are presented Table 5. As the amount of powder sugar on the diameter and thickness were found statistically significant (p < 0.01). The response surfaces of the effects of composite flour and powder sugar on diameter, thickness, spread ratio, hardness of cookies is presented in Figure 1. It shows the effects of composite flour and powder sugar were positively related to the diameter, spread ratio and hardness of GF cookies. As the amount of powder sugar increased, the diameter and thickness were increased. The result indicates that the powdered sugar had a significant impact on the thickness, diameter of the GF cookie. One of the important quality characteristic is spread ratio for cookies. It is known that one of the desirable quality parameters is high spread ratio (Bolarinwa et al., 2019). The spread ratio of the cookie ranged from 6.56 to 8.31. The fitted regression equations for spread ratio is presented in Table 5. Fit summary statistics suggested lineer model for spread ratio. The effect of the amount of sugar on the spreading rate was found to be significant (P < 0.01). As the amount of sugar increased, the spreading rate decreased. A study indicates that produced cookies with reduced fat and sugar content may not shrink and flow (Manley, 2011). Similar result was found that developed gluten free cookies with rice flour (Ogunbusola et al., 2020). The regression equation for spread ratio is presented in Table.5 in terms of coded levels that A, and B represent composite flour and powdered sugar, respectively. Hardness is one of the important quality parameters of cookies. Hardness of cookies ranged between 11.18 and 29.64 N (Table 4). The fitted regression model for hardness of GF cookies is shown in Table 5. Fit summary statistics suggested quadratic model for hardness; were generated in the regression model. The amount of composite



flour and powder sugar increased, the hardness of the cookies increased and it was found to be statistically significant (p < 0.01). A study reported that cookies prepared from protein rich flours to have harder structure due to the strong adherence between starch and proteins (Altındağ et al., 2015).

Sample	Diameter	Thickness	Spread Ratio	Hardness
Number	mm	mm		Ν
1	4,36±0,06	$0,62{\pm}0,06$	7,02±0,12	19,86±0,32
2	$4.42 \pm 0,01$	$0,57{\pm}0,04$	7,86±0,10	19,65±0,52
3	$4,56\pm0,04$	$0,60{\pm}0,05$	$7,44{\pm}0,25$	$11,18\pm0,62$
4	$4,54{\pm}0,08$	$0,63{\pm}0,06$	7,21±0,22	$20,20\pm0,87$
5	4,39±0,05	$0,57{\pm}0,06$	7,76±0,15	18,45±0,56
6	$4,54{\pm}0,04$	$0,60{\pm}0,02$	7,58±0,11	$19,98\pm0,70$
7	$4,40\pm0,06$	$0,53{\pm}0,04$	8,31±0,12	25,86±0,59
8	$4,45\pm0,04$	$0,65{\pm}0,06$	6,84±0,14	20,43±0,44
9	4,38±0,02	$0,60{\pm}0,06$	7,29±0,17	13,54±0,79
10	$4,59{\pm}0,08$	$0,\!68{\pm}0,\!07$	6,80±0,25	15,38±0,66
11	4,51±0,06	$0,66{\pm}0,05$	6,93±0,24	29,64±0,76
12	$4,52{\pm}0,04$	$0,\!67\!\pm\!0,\!02$	6,82±0,14	26,48±0,49
13	4,59±0,03	0,70±0,02	6,56±0,12	21,98±0,56

Table 4. Diameter, thickness, spread ratio and hardness of GF cookies

Table 5. Analysis of predicted model equation for the diameter, thickness, spread ratio and hardness of GF cookies

Parameters	Model	Model R2	F Value	value Pro>F	Final equation in terms of coded factors
Diameter	lineer	0.8829	6.98	0.0500	Diameter = +4.48-0.015*A+0.077*B
Thickness	lineer	0.8043	11.38	0.0500	Thickness =+0.62+6.066E-004* A+0.050* B
Spred ratio	lineer	0.8858	7.07	0.0500	Spread Ratio =+7.20+0.095 *A-0.46 * B
Hardness	Quadratic	0.9969	44.94	0.0500	Hardnes =+19.95+5.15* A+0.49* B- 0.14* A * B-1.07 A <sup>2</sup> +0.046* B <sup>2</sup>



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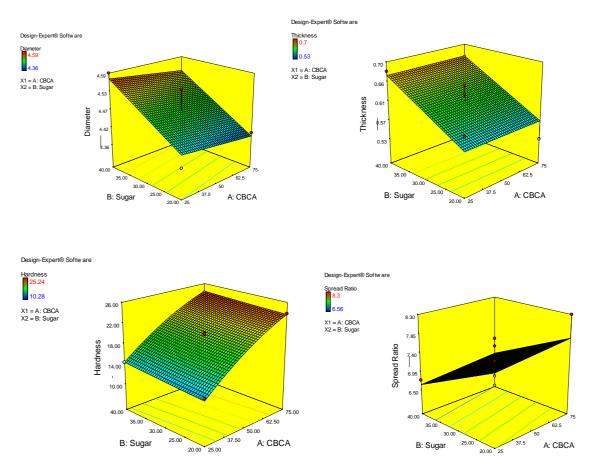


Figure 1. Response surfaces for the impact of composite flour and powder sugar on diameter, thickness, spread ratio and hardness of GF cookies

Colour is one of the crucial quality parameter that effect directly on acceptability by consumer. As seen in Table 6 the L\*, a\* and b\* values of GF cookies were found to be 38.27 to 73.20, 4.87 to 15.40, 16.56 to 29.06, respectively. The fitted regression equations for  $L^*$ ,  $a^*$  and  $b^*$  are presented Table 7.

As the amount of composite flour and sugar amount increased, the colour darkness of GF cookies increased. (Table 7). Colour characteristics reflect starch dextrination, caramelization, and Maillard reaction induced by cooking the product (Chung et al., 2014, Giuberti et al., 2018). (Figure 2). Increased levels of composite flour proportion lead to decreased the lightness (L\*), probably because of maillard reaction and raising protein content (Gomez et al., 2003). a\* value increased with increase composite flour and powder sugar. b\* value increased with degreased composite flour and increased powder sugar.

Similar results were found that using mixing flour in GF cookies lead to differences significantly in a\* and b\* values (Bolarinwa et al., 2018).



Table 6. Colour characteristics of GF cookies

Sample Number	$L^*$	<b>a</b> *	b*
1	57,33±0,18	11,02±0,04	23,25±0,11
2	56,65±0,06	$10,98{\pm}0,01$	22,27±0,06
3	73,20±0,03	4,87±0,09	16,56±0,04
4	67,32±0,11	11,50±0,0	23,45±0,02
5	59,13±0,04	9,88±0,06	22,28±0,08
6	56,98±0,03	11,06±0,01	27,58±0,06
7	45,43±0,01	13,96±0,24	23,25±0,13
8	57,31±0,24	$10,65\pm0,01$	18,87±0,33
9	67,72±0,06	6,97±0,02	$18,02{\pm}0,08$
10	64,20±0,13	$7,40\pm0,01$	28,34±0,01
11	38,27±0,06	$15,40\pm0,01$	29,06±0,06
12	42,34±0,14	14,77±0,02	26,76±0,01
13	50,32±0,06	12,30±0,06	23,02±0,09

Table 7. Analysis of predicted model e	equation for colour of GF cookies
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Parameters	Model	Model R2	F Value	value Prob > F	Final equation in terms of coded factors
L*	lineer	0.8880	39.66	0.0500	L* = +56.63-116*A-2.38*B
a*	lineer	0.97883	23.11	0.0500	a *=+10.83+3.66* A+0.05B* B
b*	lineer	0.9637	13.26	0.0500	b* =+22.74-4.26 * A+0.15 * B



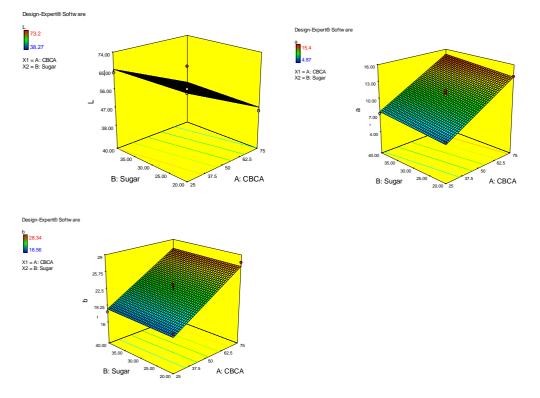


Figure 2. Response surfaces for the impact of composite flour and powder sugar on L\* value, a\* value and b\* value of GF cookies

#### Sensory analysis of GF cookies

Sensory analysis of GF cookies are summarized in Table 8. Overall acceptability is one of the important parameter for consumer acceptance of the developed product. The score to given overall acceptability for GF cookie samples ranged between 2.57 and 4.67. The regression models developed for overall acceptability is given (table 9). Both composite flour (A) and interactive effect of  $A^2$  had significant negative linear effects on overall acceptability. Powdered sugar (B) has significantly positive effect on overall acceptability. The models for overall acceptability and mouthfeel were significant (p<0.01) and (p <05) respectively. As it can be seen in Table 9 their correlation coefficients were all higher than 0.85. Thus, the proposed research model is fit for predicting changes in these responses within the experimental domain. ANOVA indicated that the mouthfeel was significantly affected by powder sugar (p<05), but the effect of the composite flour was not significantly for this attributes (p > 05); and overall acceptability was significantly influenced by composite flour and powder sugar (P<0.01). The response surfaces of the effects of composite flour and powder sugar on mouthfeel and overall acceptability of GF cookies samples is shown in Figure 3. It shows the effects of composite flour and powder sugar were positively on the given score to the mouthfeel of GF cookies.



### Table 8. Sensory properties of GF cookies

Sample Numbe	Colour	Flavour	Mouthfeel	Overall Acceptability
r				Acceptability
1	4.17±0,5	3.50±0,99	3.50±0,77	4,12±0,82
	0			
2	$3.50\pm0.8$	3.50±1,16	3.17±0,88	4.17±0,54
3	4.33±1.0 8	3.67±0,77	3.83±0,77	3.67±0,74
4	3.50±0,8	3.67±0,82	4.00±0,81	3.89±0,84
5	4.00±0.9 5	3.67±0,88	3.17±0,99	3.50±0,78
6	4.17±0,7 9	3.50±1.12	3.83±0,54	4.17±0,92
7	3.67±0,8 8	3.83±0,99	3.83±0,77	3.45±0,62
8	4.67±0,9 9	4.33±1,14	4.67±0,99	4.67±0,99
9	4.00±0,9 3	3.86±0,99	3.86±0,88	3.86±0,68
10	4.57±1,1 4	3.96±0,76	4.57±0,54	4.86±0,98
11	4.57±0,6	2.43±1,15	4.43±1,08	2.57±0,82
12	$4.00\pm1,1$	3.43±1.67	4.43±0,95	3.70±0,95
13	4 4.17±1,2 5	4.67±0,74	4.83±0,67	4.67±0,64



Table 9. Analysis of predicted model equation for the diameter, thickness, spread ratio and hardness of GF cookies

Parameters	Model	Model R2	F Value	value Prob > F	Final equation in terms of coded factors
Mouthfeel	lineer	0.9637	13.26	0.500	Mouthfeel.=+4.01+0.085* A+0.46* B
Overall	Quadratic	0.8640	8.89	0.500	overall Acceptability =+4.20-0.39*
acceptability					A+0.36* B -0.19* A * B-0.45*
					A <sup>2</sup> +0.032*

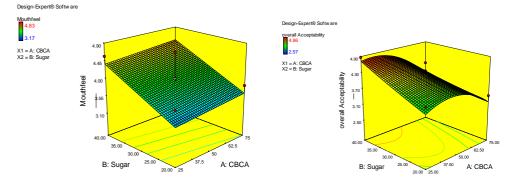


Figure 3. Response surfaces for the impact of composite flour and powder sugar on mouthfeel and overall acceptability of GF cookies

### **Optimization of GF cookies**

The suitability and adequacy of the models were evaluated by the coefficient of determination R-Squared ( $R^2$ ). Generally, models are considered good when R2 is greater than 0.75, and models less than  $R^2$  0.25 are considered unusable (Mandenius and Brundin, 2008). In this research, a model is suitable for optimizing since  $R^2$  is greater than 0.80 (Table 3, 5, 7, 9). The optimization of the responses were determined numerically with statistical software Design-expert 7. The optimal amounts of composite flour and powder sugar to determine GF cookies with the highest for spread factor, protein content and overall acceptability and their combination were selected. The optimal amounts of ingredients in GF cookie was determined maximum overall sensory acceptability, protein content and spread ratio are 66 g composite flour, 20 g powder sugar, 15 g protein and 7.70 spread ratio. These optimum parameters were obtained selected based on the highest desirability value is determined 0.71 (Figure 4).



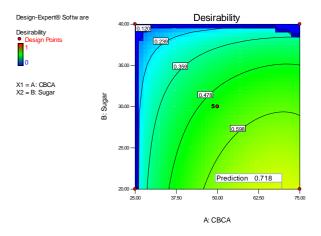


Figure 4. Response surfaces for the impact of composite flour and powder sugar on desirability

### CONCLUSION

It was determined in this study the effects of composite flour, and powder sugar on nutritional composition, physical properties, colour characteristics and sensory analysis of GF cookies with response surface experimental design. The results indicated that composite flour and powder sugar were significant for moisture, carbohydrate, energy, hardness, a\* value and overall acceptability; composite flour was mainly responsible for ash, protein, fat, L\* value and b\* value; and powder sugar was all significant for diameter, thickness, spread ratio and mouhtfeel in the cookie. Gluten free flour blends such as legumes, cereals, pseudo cereals, nuts, dried vegetable and fruits could be used to produce the gluten free cookies with increased nutritional content. So, this product will be a suitable snack food not only for celiac patients, but also for diabetics, cardiovascular patients, vegetarians and individuals with healthy eating consciousness. Thus, dietetic, functional and easy-to-carry products can be developed.

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### Nutritional Composition of Functional Snack Grain Based Mix

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### ABSTRACT

The aim of this study is; to produce extrude cereal-based snack, it is to prepare a nutrient balanced multigrain composite mix with natural ingredients and to calculate the nutritional composition of this mixture using BEBIS (Nutrition Information System) software. To balance the nutritional composition; consisting of corn flour, corn starch, oat flour, chickpea flour, carrot powder and nuts; a mixture was prepared from a combination of different food groups. The nutritional composition of the mixture was calculated using the BEBIS software program (The Nutrient Database BEBIS pro for Windows, Willstaett, Germany; Turkish Version, BEBIS 7). Multigrain composite mix had 10.0% moisture, 2.55% ash, 11.68% protein, 69.08% carbohydrate, 4.03% and crude lipid. Energy value 380 kcal/100 g. The energy of the multigrain composite mix % ratios from protein, fat and carbohydrate were determined as 12.52, 15.95 and 72.78, respectively. The protein quality score was 73.7%. 100 grams of the mixture; It has been calculated to contain 86.44 mg calcium, 4.16 mg iron, 556.8 mg potassium, 6.64 mg sodium, 8939 IU vitamin A and carotene, 0.3 mg thiamine, 0.13 mg riboflavin and 1.61 mg niacin. 100 grams of the multigrain mix; total amount of saturated fatty acids 0.89 grams; monounsaturated fatty acids were calculated as 3.34 grams and polyunsaturated fatty acids as 2.35 grams. Since the nutritional content of the product to be produced from this multigrain mix is balanced; it will be a suitable snack food especially for adolescents, pregnant and lactating people, the elderly, vegans and some patient groups (diabetes and cardiovascular patients).

Keywords: Nutritive snack formulation, BEBIS, cereals, Legumes, nutritional composition

#### 1. INTRODUCTION

Adequate and balanced nutrition is one of the basic conditions, perhaps the most important, for to be healthy and maintains of life, for its economic and social development, for its success in the society (Hanry and Chapman, 2002, Dimidi et al, 2021). Today, with the changing lifestyle (as a result of social and economic changes such as rapid population growth, increase in education level, more women entering the business life, the importance of time factor and increase in income level) lead to changes in eating habits and attracted read to eat food by people. Foods such as chips, biscuits, breakfast cereals are foods with high energy value, but low nutritional value because they are insufficient and unbalanced in terms of some nutrients. Cereal-based snack foods can cause unbalanced nutrition and excessive energy consumption when consumed frequently in between meals (Richardson, 1990; Obatolu and Cole, 2000, Dimidi et al, 2021). It creates a feeling of fullness and loss of appetite can cause reduce of the food consumption especially high nutritional components. It is known that cereal based snack can increase the risk of certain diseases such as obesity, type 2 diabetes, cardiovascular diseases, hypertension and cancer. For these reasons, currently, it is needed to develop healthier and more nutritious snack products in order to contribute adequate and sufficient diet. It will be possible to contribute to an adequate, balanced and healthy diet. Today, awareness of the relationship between nutrition and health is increasing. Especially in developed countries, the place of natural products in nutrition becomes more important as the awareness of returning to nature becomes widespread. While the food industry strives to make the products produced more reliable and nutritious, on the other hand, it tries to prevent the loss of many nutrients; it also continues its efforts to develop new products to prevent excessive intake of foods that have negative effects on health, such as sugar, salt, and fat.

It has been going on for years to make various food combinations to increase the nutritional value and quality of foods and to eliminate the deficiencies of certain nutrients. In many years, different food groups are consumed together for example, the consumption of combination of fish and rice that is staple food are



consumed in countries while, the consumption of combination of cereals and legumes in other countries in order to balance nutritional composition. Such practices are seen as the most valid way to solve some nutritional deficiencies (Richardson, 1990; Dutra-De-Oliveria and Marchini, 2007; Angeles-Agdeppa et al., 2007; Hieu et al., 2007, Carvalho et al, 2021).

In various studies showed that different new products have been with legumes and cereal mixtures that complement each other in terms of essential amino acids, and which have increased nutritional values both in terms of protein amount, quality and other nutrients (minerals, B group vitamins) (Anon, 1997; Rajahama and Sabate, 2000). Low-income groups that received insufficient animal protein consumption and vegetarian groups who do not consume animal products receive insufficient protein and several vitamins and minerals. This situation can cause nutritional and health problems. These groups are recommended to consume foods containing cereals and legumes mixtures, and to consume sufficient fruits, vegetables and nuts (Cala et al., 1981; Rajahama and Sabate, 2000).

Novel formulation of cookies from germinated legumes and triticale was produced by using central composite design of response surface methodology. Cookies prepared were good in nutritional attribute with improved protein and carbohydrates digestibility. Essential amino acid content of optimized cookies was higher due to germination and legume substitution (Sibian and Riar, 2021).

Development of new snack mix products containing nutrients from different food groups; it can also be used in the nutrition of special groups with increased nutritional requirements such as young, old, pregnant and lactating, and athletes will be able to get benefit (Baysal; 2020). When such nutrient mix products with low fat and high fibre content are developed; it may be appropriate to use in patients with cardiovascular diseases and diabetes. This kind of nutritious functional snack type mix products with when developed; it can be recommended instead of food or drinks (biscuits, chips, candies, sugary drinks, etc.) that have high energy value, but have no nutritional properties or lead to an unbalanced diet, healthy products. Keep in this view, the aim of this study; to prepare a more balanced and nutritious mixture in terms of nutrient content by combining foods from different food groups and different types of cereals (corn and oats); such as legumes (chickpeas), vegetable powder (carrots) and nuts (hazelnuts), and to calculate the nutrient composition of the mixture.

The new product to be produced from the herbal mixture to be prepared should be more balanced in terms of nutritional content (protein, fat, vitamin, mineral and dietary fibre content), especially special groups with increased nutritional requirements such as adolescents, pregnant and lactating women, the elderly, athletes, and some patient groups (cardiovascular patients, diabetics, etc.) and make it a suitable snack food for vegetarians who do not consume animal products. In summary; a nutritious (balanced in terms of nutrients), dietetic, ready-to-eat snack and easy-to-carry product may be produced.

### 2. MATERIALS AND METHODS

#### 2.1. Materials

Flour mix; it consists of chickpea flour, oat flour, cornmeal, corn starch, carrot powder and ground hazelnut components.

#### 2.2. Methods

The proportions of the components of the mixture were determined by preliminary calculations. Thus, the components and their proportions have been standardized. The mixture consisting of 20% oat flour, 20% corn flour, 15% corn starch, 30% chickpea flour, 10% carrot powder and 5% ground hazelnut was considered suitable.

Energy and nutrients, essential amino acids, minerals and vitamins and fatty acids composition determined in the mixture using the BEBIS software program (The Nutrient Database BEBIS pro for Windows, Willstaett, Germany; Turkish Version, BEBIS 7). The protein quality score of the mixture is based on the chemical calculation method (Baysal, 2020).

### 3. RESULT AND DISCUSSION

The ingredients and their amounts, as well as the amounts of energy and nutrients in the mixture determined by preliminary studies, are given in Table 3.1. The mixture is composed of 20% oat flour, 20% corn flour, 15% corn starch, 30% chickpea flour, 10% carrot and 5% hazelnut. The energy contained in 100 grams of



such a mixture is 380 Cal, protein 11.8, fat 6.74, carbohydrate 69.08, ash 2.55, moisture 10, dietary fibre 4.03 grams (Anon, 1991; Baysal, 2020). The percentages of the energy of the mixture coming from protein, fat and carbohydrates were found as 12.52, 15.95 and 72.78, respectively. In terms of protein of chickpeas in the mixture in Table 4.1; it can be seen that oat flour, chickpea flour and carrot enrich the mixture in terms of dietary fibre. In such a mixture, it can be seen that the ratio of energy coming from protein is also high.

Ingredien ts	Amo unt (g)	As h (g)	Moist ure (g)	Protei n (g)	Fat (g)	Carbohyd rate (g)	Dietary fiber (g)	Energ (Cal)
Oat flour	20,0 0	0,70	1,80	2,40	1,50	13,60	1,40	78
Chickpea flour	30,0 0	0,90	3,21	6,15	1,44	18,30	1,50	108
Corn flour	20,0 0	0,16	2,40	1,56	0,52	15,38	0,15	74
Corn starch	15,0 0	0,01 5	1,80	0,05	Trace	13,14	0,02	54
Carrot powder	10,0 0	0,64	0,50	0,89	0,16	7,81	0,81	34
Hazelnut	5,00	0,13	0,29	0,63	3,12	0,84	0,15	32
Total	100, 00	2,55	10,00	11,68	6,74	69,08	4,03	380

Table 3.1. Energy and nutrients of the mixture

The essential amino acid amounts of the mixture are given in Table 3.2. Protein quality score of the mixture was determined as 73.7%. From here it is seen that the protein quality score of the mixture is high. The protein quality scores of meat and milk proteins, which are considered to be good quality proteins, are around 80 and have been approximated. Protein quality scores of cereals are between 50-60% (Baysal, 2020). As it can be seen, both the protein amount and the protein quality score of the mixture were increased with the prepared mixture. The amino acid lysine, which is a limited essential amino acid in cereals, is balanced.

According to Yagmur et al. (2005) determined the energy and nutritional elements of biscuits and similar cereal products offered for sale in the market. On average, moisture in biscuits and similar cereal products is 5.54%; protein 6.48%; fat 18.49%; it was determined carbohydrate 68.43% ash 1.08% and energy amount as 466 Cal/100 g.



Ingredie nts	Tryptop hane	Threo nine	Isoleu cine	Leuc ine	Lysi ne	Methio nine	Cystin e (mg)	Phenylal anine	Vali ne
	(mg)	(mg)	(mg)	(mg)	(mg)	( <b>mg</b> )	× 0,	( <b>mg</b> )	(mg)
Oat flour	36,60	94,00	146,60	213, 00	104, 20	41,80	61,80	151,60	169, 00
Chickpea flour	51,00	221,70	358,50	461, 40	430, 20	82,80	88,80	303,60	307, 50
Corn flour	9,40	62.22	72,20	202, 20	45,0 0	29,00	20,20	70,80	79,6 0
Corn starch	-	-	-	-	-	-	-	-	-
Carrot powder	1,00	4,30	4,60	6,50	5,20	1,00	2,90	4,20	5,60
Hazelnut	8,75	29,45	38,35	61,4 0	22,0 0	15,30	16,00	38,35	48,7 0
Total	106,75	411,67	620,25	944, 50	606, 60	169,90	189,70	568,55	610, 40
Egg	194	596	759	1066	820	392	289	686	874

Table 3.2. Essential amino acids in 100 Grams of the mixture and egg

In a study was found that 34% of the energy in potato chips comes from carbohydrates, 60% from fat, 6% from protein on the other hand in corn chips, 46% of the energy comes from carbohydrates, 48% from fat, and 6% from protein. As it can be seen, the energy from fat are very high and from protein are very low. Such snack foods may cause unbalanced nutrition when consumed frequently in between meals. Generally, in such products, the percentage of energy from protein can vary between 5-6%, from fat 40-60, and from carbohydrates 35-50 (Uzun et al., 2006). In another study, an extruded snack product enriched with oat flour, chickpea, carrot powder and ground hazelnuts was developed using the response surface methodology (Özer et al., 2004).

The amounts of minerals and vitamins found by calculating the amounts of the ingredients in 100 grams of the mixture are shown in Table 3.3. 100 grams of the mixture contains 86.44 mg of calcium, 4.16 mg of iron, 556.8 mg of potassium, 6.74 mg of sodium; 8939 IU of vitamin A and carotene, 0.3 mg of thiamine, 0.13 mg of riboflavin and 1.61 mg of niacin.

As it can be seen in table 3.3, the sodium content of the mixture is quite low and the potassium content is high. Excess sodium consumption is associated with high blood pressure. Diets rich in sodium increase the tendency to high blood pressure. In addition, excess sodium intake increases urinary calcium excretion. This causes calcium loss from the bones. In addition, increased calcium loss from bones increases the risk of osteoporosis. In particular, foods with low sodium content should be preferred (Anon, 2010). Potassium provides the controlling blood pressure. Moreover, it reduces the risk of heart attack by balancing blood pressure and regulating heart functions. By making the kidneys work better, it accelerates the excretion of sodium from the body. In addition, it helps the muscles to contract and ensures that the nerves carry signals properly (Baysal, 2020).

It is clear that, the multigrain mixture can be a balanced product in terms of mineral content. It is known that especially chickpea flour added to the mixture in order to increases the calsium content of carrots and hazelnuts, the Fe content of oat and chickpea flour, the vitamin A content of carrots, and the vitamin E content of hazelnuts.

Daily recommended amounts for adult men and women, respectively; 4500 and 4000 IU for vitamin A (Baysal, 2020); 1.2 and 1.1 for thiamine; 1.3 and 1.1 mg for riboflavin; 16 and 14 mg for niacin respectively (Anon, 2005).

When an evaluation is made according to the recommended daily nutrient requirements (RDA) for adults, 100 grams of multigrain mixture consist of almost twice the vitamin A requirement for men and more than twice for women; for adult men and women respectively 25% and 27.27% thiamine; 10% and 13% for;



riboflavin; 10.06% and 11.5% niacin. As for the need for vitamin E, 17.73% of men and women appears to be acceptable.

Ingredients (g)	Ca (mg)	Fe (mg)	K (mg)	Na (mg)	Vit.A/ Carotene (IU)	Thiamine (mg)	Riboflavin (mg)	Niacin (mg)	Vitamin E (mg)
Oat flour	1,20	1,00	8,00	200,00	0,00	0,10	0,03	0,20	0,28
Chickpea flour	45,00	2,07	239,1 0	7,80	15,00	0,09	0,05	0,60	-
Corn flour	1,20	0,36	-	0,20	68,00	0,04	0,012	0,28	0,61
Corn starch	0,00	0,00	0,00	Trace	Trace	0,00	0,00	0,00	-
Carrot powder	29,79	0,56	274,5 0	37,80	8856,00	0,05	0,04	0,48	-
Hazelnut	10,45	0,17	35,20	0,10	-	0,02	-	0,05	1,77
Total	86,44	4,16	556,8 0	6,74	8939,00	0,30	0,13	1,61	2,66

Table 3.3. Mineral and vitamin of 100 grams of the mixture

The fatty acid composition of the mixture has been calculated and given in Table 3.4. The total amount of saturated fatty acids in 100 g of the mixture is 0.89 g; monounsaturated fatty acids were calculated as 3.34 g and polyunsaturated fatty acids as 2.35 g. 13.53% of the fat it contains is saturated, 50.76% is monounsaturated and 35.71% is polyunsaturated made up of fatty acids. Fatty acid types in the composition of the oil are as important as the amount of fat in the food (Kırbaslar and Erkmen, 2003). It is fact that the total amount of fat in the daily diet affect negatively on cardiovascular disease due to high and rich in saturated fatty acids composition in foods. As the ratio of saturated fatty acids in the diet increases, the amount of low-density lipoprotein (LDL) in the blood increases. The increase in the LDL ratio in the blood also increases the risk of cardiovascular diseases (Milner, 2000; Halsted, 2003; Chisholm et al., 2005). Monounsaturated fatty acids has a protective effect against cardiovascular diseases has been obtained (Milner, 2000). The major component of the fatty acid content of hazelnut and hazelnut oil and the one that should be preferred for health is monounsaturated fatty acids (oleic acid etc.). In scientific studies show that monounsaturated fatty acids reduce blood levels of LDL-Cholesterol (bad cholesterol) and it increases apo lipoprotein A-1 levels, which has a protective effect from cardiovascular diseases, and decreases risky apo lipoprotein B levels by 7.5%. Linoleic acid (omega-6 fatty acid), one of the polyunsaturated fatty acids found in hazelnut and hazelnut oil, has a lowering effect on blood cholesterol levels.



Table 3.4. Fatty acid composition of mixture

Ingredie nts	Amou nt (g)	Fat (g)	Satur F.A. (		Tot	Tot Monounsaturated F.A. (g)		Tot (g) Polyunsaturated F.A.			Tot		
			C16 :0	C18 :0	al	C16 :1	C18 :1	C20 :1	al	C18 :2	C18 :3	C20 :4	al
Oat flour	20,00	1,50	0,26	0,01	0,27		0,58	-	0,58	0,64	0,01	-	0,65
Chickpea flour	30,00	1,44	0,29	0,04	0,33	0,01	0,26	-	0,27	0,81	0,01	0,02	0,84
Corn flour	20,00	0,52	0,06	-	0,06	0,16			0,16	0,28	0,02	-	0,30
Corn starch	15,00	Tra ce	-	-	-	-	-	-	-	-	-	-	-
Carrot powder	10,00	0,16	-	-	-	-	-	-	-	-	-	-	-
Hazelnut	5,00	3,12	0,17	0,06	0,23	0,01	2,31	0,01	2,33	0,55	0,01	-	0,56
Total	100,0 0	6,74	0,78	0,11	0,89	0,18	3,15	0,01	3,34	2,28	0,05	0,02	2,35

Linoleic acid (omega-6 fatty acid) has a lowering effect on blood cholesterol levels. Linolenic acid (omega-3 fatty acid), one of the polyunsaturated fatty acids that found in hazelnut and hazelnut oil, reduces the production of endogenous VLDL cholesterol, lowers plasma triglyceride levels, prevents postprandial triglyceride increase. It reduces life-threatening heart rhythm disorders and the tendency of blood to clot, hence the vascular occlusion and risk of sudden death (Frank, 2003). The product to be produced from this mixture; It will have properties that are poor in saturated fatty acids, rich in monounsaturated fatty acids, sufficient in polyunsaturated fatty acids, and have high antioxidant potential. As it contains hazelnut and hazelnut oil, it will be a healthy product and may help prevent atherosclerotic cardiovascular diseases.

### 4. CONCLUSION

The aim of this study is to prepare a more balanced and nutritious mixture in terms of nutrient content by combining different types of cereals, legumes, vegetables and nuts from different food groups. For this purpose, a mixture of 20% oat flour, 20% corn flour, 15% corn starch, 30% chickpea flour, 10% carrot powder and 5% ground hazelnut was prepared with preliminary trials in this study. The nutritional composition of the multigrain mixture; moisture 10.5%; protein 13.61%; oil 6.41%; carbohydrate 67.78%; dietary fibre was found to be 12.86%; the energy value was calculated as 384 Cal/100 g. The percentages of energy in % dry matter from protein, fat and carbohydrate in the mixture were determined as 14, 15 and 71, respectively.

This research can be a guide for the production of different functional new products that are ready to eat, easy to carry, nutritious and healthy. Recently, awareness of the relationship between nutrition and health is increasing. Changes in eating habits with the modernizing lifestyle increase the consumption of ready-to-eat snacks that are easy to carry. The new product to be produced from the herbal mixture to be more balanced in terms of nutritional content (protein, fat, vitamin, mineral and dietary fibre content), especially for special groups with increased nutritional requirements such as adolescents, pregnant and lactant woman, elderly, vegetarians, athletes, and some patient groups (cardiovascular patients, type 2 diabetes, etc.).

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