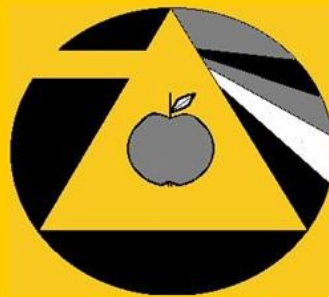


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CONTENTS

Food physics Public Foundation	3
XIIIth International Conference of Food Physicists	5
<i>Andras S. Szabo, Peter Laszlo</i>	
Is food physics the science of the XXI century?	11
<i>Taner Erkamaz, Firuze Ergin, Gizem Yildiz, Ahmet Kucukcetin</i>	
Determination of Milk Proteins in Dairy Products by Analytical Methods	17
<i>Duygu Aslan, Mahmut Dogan, Meryem Goksel Sarac</i>	
Interfacial behavior of casein-inulin interactions at the oil and water interfaces	22
<i>Adem Kaya, Cavit Aktar, Osman Kadir Topuz</i>	
Effect of fluidized bed drying on the fatty acid content of giant red shrimp (<i>Aristaeomorpha foliacea</i>) byproducts	29
Akdeniz University	35
<i>Senem Suna, Azime Ozkan-Karabacak, Canan Ece Tamer, Omer Utku Copur</i>	
Drying Kinetics and Color Properties of Lemon Balm (<i>Melissa officinalis</i>) Leaves Dried by Convective Hot Air Drying	36
Instructions for authors	47

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To publish the physical knowledge of food science and the results of research and development for the specialists of food production and R+D.

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EDITORIAL

This is the XXXI Volume of the Journal of Food Physics, and as You know the first issue was published in 1988, so 3 decades ago. Many thanks for your kind help, cooperation, support and understanding also the problems, during this period of not too easy existence. We are sure, that also this issue gives the opportunity for the readers to get interesting and useful information about some special questions of our loved and highly respected sub science, food physics.

The topics of the scientific articles in this issue cover the following fields:

- is food physics the science of the XXI century?
- determination of milk proteins in dairy products
- interfacial behaviour of casein-inulin interactions at the oil and water interfaces
- effect of fluidized bed drying on the fatty acid content of giant red shrimp
- drying kinetics and color properties of lemon leafs dried by connective hot air drying

Lert me mentiopn a little the history of the ISFP conferences As You probably know or remember the first conference we organized in Budapest, Hungary, 1994,

followed by the second one in Bucharest, Romania, 1996. The place of the third meeting was Poland, Lublin, 1998, and in 2000 we met in Turkey, Istanbul. Later we decided to organize the conference in Brno, Czech Republic, 2002, and 2 years later, in 2004 we came back again to Hungary, but the place was Pecs. The 2006 meeting we had in Serbia, in a beautiful small town, Senta, and the next one in Plovdiv, Bulgaria, 2008. The place of the 2010 conference was Nitra, Slovakia, then in 2012 Budapest, and again in Plovdiv, 2014. After Debrecen in 2016 we were really happy to have the possibility to continue the organisation of the ISFP conferences, and the last one, the 2018 ICFP was organized again in Turkey, in Antalya at the sea-side.

We are trying to continue our activity, publishing the Journal of Food Physics and organize the conferences of the International Society of Food Physicists. Please, do not miss the opportunity and come to Romania in 2020, the town is Iasi!

Dear Colleagues! Read and enjoy this issue! And please - if You are able - support the Food Physics Public Foundation! We need help and donations for existence.

<http://www.foodphysics.net>

Prof. Andras S. Szabo
president of ISFP
and
editor-in-chief of JFP

XIIIth International Conference of Food Physicists



The XIII. International Conference of Food Physicists (23-25 Oct. 2018) was organized by the Akdeniz University, Faculty of Engineering, Department of Food Engineering, Antalya, Turkey. Hotel Porto Bello at the seaside was the congress venue. Scientists and experts of 80 presentations represented 20 countries from all around the world. The conference topics were:

- Physical aspects of agronomy
- Chemistry, physical chemistry and food analysis
- Non-destructive analysis
- Rheology
- Unit operations and technology
- Quality control, quality assurance, food safety
- Health aspects
- Environmental physics

There were 8 concurrent sessions. András S. Szabó delivered a keynote presentation with the title "Is food physics the science of the XXI century?". The co-author of this presentation was Péter László, the founder of the Food Physics foundation. Besides 37 oral presentations, 38 posters were presented.

The XIII. International Conference of Food Physicists was really fruitful, useful and interesting. Participants from Europe and Asia enjoyed both active discussions and the beautiful location. The professional community is looking forward to the forthcoming conference.

After these three thought provoking days the organizers closed the event as a successful meeting and decided that the next conference of this series will be held in Romania, IASI in 2020. We hope that all the participants and their colleagues will join to us and we will have another beautiful conference, concerning the topics of our lovely science, food physics.

Ahmet Kucukcetin Ayhan Topuz Andras S. Szabo



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ORAL PRESENTATIONS

- Is food physics the science of the XXI century?
- Texture as flavor driver? - An example of “food soft matter science”
- Time-resolved fluorescence and fluorescence quenching in model food emulsion stabilized by β -lactoglobulin
- Characteristics of pumpkin seed oil powder microencapsulated by freeze-drying

XIIIth International Conference of Food Physicists

- Effect of hydro colloids and dietary fibers on different quality attributes of cheddar cheese
- Computer vision-based colour analysis: an online tool to monitor food quality and safety during processing
- Beef colour evolution from pigment concentration profiles during oxygenation
- Perspectives of non-destructive spectroscopic techniques to detect quality & safety of food
- Metagenomics and high-throughput sequencing methods: applications in food microbiology
- Physical properties of semi-refined carrageenan-potato protein gels
- Gel strength estimation for gelatin-cmc hydrogels using small amplitude oscillatory rheometry
- Rheological properties balangu seed gum/sodium caseinate stabilized emulsions and oleogels
- Durability of mycelium based food packaging materials under conditions mimicking the potential extremes
- Mathematical modeling of temperature distribution and velocity profile in toroidal cans during thermal processing with horizontal-axial rotation
- The effect of different drying processes on the drying characteristics, physical and powder properties of red pepper pulp
- The impact of ultrasound pre-treatment and oven-drying on the quality of dried pears
- SESSION 6 Quality Control & Food Safety
- Color measurement: an unbiased method for food quality control?
- Binding analysis between monomeric β -casein and hydrophobic ligands investigated by surface plasmon resonance and fluorescence spectroscopy
- Characterisation of monoglyceride-based cubosomes under the influence of flavonoids
- Assurance of poultry meat quality and safety by exploring potential of organic acids

XIIIth International Conference of Food Physicists

- Innovative thermal processing – effects of physical properties
- Preliminary studies regarding nutritional performance of quinoa crop as leaf vegetables
- A review on textural profile analysis of meat and meat products
- The effect of thermosonication on some quality parameters of watermelon juice
- Application of cold membrane filtration at pilot scale to fractionate dairybased functional ingredients from skim milk
- Neuroactive molecules production by fermenting bacteria and health
- Enrichment of d-pinitol in carob pod extract
- Recycling waste from the food industry for construction industry
- Determination of Milk Proteins in Dairy Products by Analytical Methods
- Effect of solvent polarity on the eicosapentaenoic acid (EPA) content of algal (*N. oculata*) oil
- Interfacial behavior of casein-inulin interactions at the oil and water interfaces
- Effect of fluidized bed drying on the fatty acid content of giant red shrimp (*Aristaeomorpha foliacea*) byproducts
- Regulation of AhR-XRE and Nrf2-ARE signaling pathways by dietary phytochemicals Determination of some physicochemical, microbiological and sensorial properties of the concentrated acidophilus milk produced from cow's milk and goat's milk
- with different production methods
- Drying Kinetics and Color Properties of Lemon Balm (*Melissa officinalis*) Leaves Dried by Convective Hot Air Drying
- Extraction optimization of sunflower head pectin and determination some gel properties of the pectin
- Comparing the rheological properties of emulsion and oleogel based on gum Tragacanth and sodium caseinate

POSTER PRESENTATIONS

- Increase of the stability and the functionality of commercial lactic acid bacteria starters by co microencapsulation with buckwheat flour and oat bran
- Extraction and highlighting the protein fractions from black rice flour by gel electrophoresis (SDS-PAGE)
- The effects of potassium lactate used in pastırma production on protein oxidation and some other qualitative properties
- Some physicochemical properties of turkish coffee fortified with apricot kernel powder
- Effect of osmotic drying on physicochemical aspects of dehydrofrozen sliced red pepper (*Capsicum annuum* L.)
- The presence of bisphenol A (BPA) in milk and dairy products
- The low-lactose yoghurt
- The usage of centrifuge technique in concentrated yoghurt production
- The effects of different microencapsulation methods on the viability of *Lactobacillus acidophilus* in gastrointestinal media
- Physicochemical properties some physicochemical properties of commercial protein isolates
- Effects of edible coatings before drying on some properties of dried banana
- The effect of different drying processes on the powder properties of red beet puree powders
- Drying characteristics and kinetics of lovastatin degradation of oyster mushroom (*Pleurotus ostreatus*) slices
- Improving quality and shelf-life of poultry meat through application of protein-based edible coatings
- Probing the hepatoprotective effect of camel milk on arsenic induced liver damage
- Evaluating the effectiveness of flaxseed fortified functional yoghurt against type-2 diabetes
- Non-destructive analysis of edible oil oxidation

XIIIth International Conference of Food Physicists

- Protection of microbial development through freezing technology
- Re-structured meat products
- Effect of starter cultures and addition of buffalo milk on chemical and sensory characteristics of camel milk cheese
- Mathematical modeling of infrared heating for process design
- Combination of hyperspectral imaging with complementary data mining methods for identification of microorganisms
- Antioxidant activity of glucosyl-hesperidin solutions
- Coconut proteins: alternative source of protein for retention of phenolics
- Migration of phthalate esters to seafood in PVC containers
- Interfacial rheology of gelatin with whey and skim milk powder
- The effect of ultrasound pretreatment on color properties of raisins
- Rheological properties of mellorine produced with mono-diglycerides from rendering waste oil
- Interfacial rheological properties of mono-diglycerides produced from rendering waste oil in oil/water interface
- Mechanical properties of furcellaran and furcellaran/bovine serum albumin composite films
- Performances of tomato crop under organic fertilizer
- Adsorption isotherms and isosteric heat (qst) of the flours from three edible insects: *Rhynchophorus phoenicis*, *Imbrasia truncata* and *Imbrasia epimethea*
- Effect of the pH on the topography and nanomechanics of whey protein microgel particles investigated by atomic force microscopy
- Characteristics and microbiological properties of the cakes produced by using sourdough
- The effect of stevia and isomalt on the quality of cakes as a sugar substitute
- Effect of packaging materials in composition and sensory characteristics of Romanian Telemea Cheese
- Effect of storage on textural properties of different strawberry cultivars

Andras S. Szabo, Peter Laszlo
Is food physics the science of the XXI century?

Is food physics the science of the XXI century?

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Keywords:

Food physics

Abstract. The development and modification of the science, forming and establishment of rather new fields is a normal process, carried out dominantly by 2 ways: differentiation and integration. This phenomenon of development is typical also for food physics. As integration: food science and physics and as differentiation: within food science and within applied physics.

The lecture deals with the following topics?

- the most important parts of food science and applied physics
- why is food physics a bridge between applied physics and food science?
- what are the factors, influencing the development of food physics?
- is food physics an interdisciplinary subsience? if yes, what are the connections with food analysis, measurement technique, agrophysics, biophysics, food technology, nutrition science?
- what are the development trends of food physics? (quo vadis Cibus Physicorum?)
- development in up-to-date science, problems of the future, answers from the field of food physics?
- is it true, that without high level of knowledge in physics the food engineers can not fulfill the expectations of modern food processing technologies?

INTRODUCTION

The development and modification of the science, forming and establishment of rather new fields is a normal process, carried out dominantly by 2 ways: differentiation and integration. This phenomenon of development is typical also for food physics.

As integration: food science and physics.
As differentiation: within food science and within applied physics.

TOPICS

The paper deals with the following topics:

- the most important parts of food science and applied physics

Andras S. Szabo, Peter Laszlo

Is food physics the science of the XXI century?

- why is food physics a bridge between applied physics and food science?
- factors, influencing the development of food physics, radiation methods and techniques
- is food physics an interdisciplinary subsience? if yes, what are the connections with food analysis, measurement technique, agrophysics, biophysics, food technology, nutrition science?
- what are the development trends of food physics? (Quo Vadis Cibus Physicorum?)
- development in up-to-date science, problems of the future, answers from the field of food physics
- is it true, that without high level of knowledge in physics the food engineers can not fulfill the expectations of modern food processing technologies?

PARTS OF FOOD SCIENCE AND APPLIED PHYSICS

Food science: food chemistry, food physics, food microbiology, food technology, food machinery and unit operations applied physics: agrophysics, biophysics, food physics, radiation physics, medical physics.

FOOD PHYSICS AS A BRIDGE BETWEEN APPLIED PHYSICS AND FOOD SCIENCE

Food physics has 3 main topics:

- physical parameters of foodstuffs
- physical methods for investigation of foodstuffs
- physical methods for treatment and processing of foodstuffs

RADIATION METHODS AND TECHNIQUES IN THE AGRO-FOOD SECTOR

- Ionizing radiation techniques and technologies (gamma-sources, X-ray equipments, accelerators, reactors)
- Non-ionizing radiation techniques (light-technique, IR, UV, Laser, SYNERGOLUX: UV+ozone, polarized light)
- Radiostimulation
- Radiomutation
- Food and feed irradiation
- Isotope techniques, tracer techniques
- Radio-analytical techniques (e. g. AA, XRF)
- Measurement techniques (quantity, level, thickness etc.)
- Radiometrical control of the food chain
- Radioecological measurements

INTERDISCIPLINARY SCIENCES

The term „food physics” is not known enough in spite of the fact, that the constituent words (food and physics) have been used for thousands of years in the history of mankind. Food physics is a part of applied physics, but belongs to the food sciences, as well.

Food physics is a new field of science, rather special, but typically interdisciplinary science. If we use the term in wider interpretation, food physics will cover a significant part of the R+D activity of food industry, because the base of measurement techniques, mechanisation, instrumentation, automation, regulation, control and even robot-techniques is the same: physics.

Food physics deals with the physical properties of food, food ingredients and

Andras S. Szabo, Peter Laszlo

Is food physics the science of the XXI century?

their measurement. Physical properties of food play a key role in all fields where modern technological processes are applied for the generation of food raw materials and the production and processing of food.

The determination of physical properties of food and related products is a requisite for planning, production engineering and automation processes in today's food industry, as well as in quality control activities.

CONNECTIONS

Food physics has several close connections with other sciences and subspecies, including e.g. food analysis, food quality control, food technology, nutrition.

FOOD ANALYSIS

Physical methods are suitable for determination of the composition (e.g. protein content, water content) or physical parameters (e.g. viscosity, radioactivity).

MODERN FOOD TECHNOLOGIES

- dehydration
- freezing
- lyophilization
- high pressure
- ohmic processing
- pulsing electrical fields
- magnetic fields
- nondestructive techniques (e.g. NIR-NIT, NMR, PAS)

HOW TO HELP FOR NUTRITION SCIENCE?

- food quality control, quality assurance

- determination of macro components (e.g. NIR/NIT, NMR, PAS)
- determination of micro components (e.g. INAA, XRF)
- to produce safe, sterile food (no microbial contamination) e.g. with irradiation or heat treatment or high pressure technology
- to improve the sensory properties of foodstuffs with physical treatments
- improvement of food processing technology, minimal processing, combination of different technologies, microwave, nanofiltration etc.
- development of robot technologies for food production

QUO VADIS CIBUS PHYSICORUM? TRENDS AND MODERN NONDESTRUCTIVE TECHNIQUES

- NIR-NIT spectrometry for determination of main components
- NMR techniques for rapid fat /oil measurements
- INAA techniques for determination of elements
- DSC method for study of different processes in foodstuffs (e.g. heat denaturation of proteins)
- XRF techniques for measurement of elements
- Rheometry (viscosimetry, plastometry, penetrometry, fructometry) for texture and consistence analysis
- PROBLEMS OF THE FUTURE, ANSWERS FROM THE FIELD OF FOOD PHYSICS

Andras S. Szabo, Peter Laszlo

Is food physics the science of the XXI century?

- food physics is able to solve some problems e.g. on the fields of:
 - Production of safe food with high quality
 - Water-management, purification of water (RO)
 - Waste-management, recirculation technologies (green chemistry)
 - Environmental protection, ecology (measurement – decision – action – result
- innovation, R+D activity, creation of new technological lines, even in the everyday processing of food products using up-to-date technologies.

REFERENCES:

IS IT TRUE, THAT WITHOUT HIGH LEVEL OF KNOWLEDGE IN PHYSICS THE FOOD ENGINEERS CAN NOT FULFILL THE EXPECTATIONS OF MODERN FOOD PROCESSING TECHNOLOGIES?

Yes, it is. They need good knowledge in basic physics, food physics, electrotechnics, measurement techniques, control and automatization, instrumental food analysis, experiment planning and process control. Yes, if we consider the expectations in modern food processing:

- Decrease the microbial contamination, disinfection
- Increase the storability
- Improve the sensory properties
- Apply of environment-friendly and economical technique

CONCLUSIONS

Physics is a basic subject, fundament to understand food science, unit operations, food technology, measurement technique, automation.

It is evident, that without high level of knowledge in physics the food technologists and engineers can not fulfill the expectations of modern food processing.

In other words: without this knowlege they can not take part successfully in

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Andras S. Szabo, Peter Laszlo
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Determination of Milk Proteins in Dairy Products by Analytical Methods

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Reversed-Phase HPLC

Abstract. Globally, milk is a commonly consumed food due to its high nutrient composition. Milk naturally contains a number of key nutrients, including protein, which is beneficial to humans regardless of their age. Proteins are macromolecules that play a crucial role in nutrition, growth and development. The percent of protein ranges from 3.0% to 3.6% in cow's milk. There are several methods used for the determination of milk proteins in dairy products, such as qualitative methods, determination of total organic nitrogen by Kjeldahl technique, colorimetric principles, enzymelinked immunosorbent assay (ELISA), electrophoresis, X-ray crystallography, nuclear magnetic resonance (NMR) and chromatographic methods. Milk proteins can be detected more easily by analytical instruments compatible with liquid chromatography due to polar ligands. Reversed-phase HPLC technique has become an essential technique in determination of milk proteins and peptides in dairy products. Reversed-phase HPLC combined with mass spectrometry (MS) provides a powerful technique for milk protein analysis. It is possible to determine also the animal origin of milk by detecting milk proteins. Chromatography combined mass verification technique is the leading technique for determination of milk proteins in dairy products.

INTRODUCTION

Milk proteins are the most important structures for the development, growth and self-renewal of the organism. Milk proteins are organic compounds that are essential for life in terms of their chemical composition. Milk proteins contain for the life all essential amino acids that cannot be synthesized by the human body and should be provided from everyday diet. Milk proteins mainly consist of 2 different groups; caseins and serum proteins (whey

proteins). Casein is found only in milk in nature and is the main protein of milk. Whey protein is called noncoagulant and non-casein part of milk protein.

PROTEIN ANALYSIS

Qualitative methods

Proteins are determined by colour reactions.

Millon Test; when proteins are heated with concentrated nitric acid + mercury II Milan separator, the blood forms a red colour. This reaction results from the tyrosine amino acid. Ninhydrin Reaction; blue-violet colour occurs when proteins are heated with ninhydrin solution.

Xanthoprotein Reaction; proteins give a severe yellow colour with concentrated nitric acid (HNO₃). If ammonia (NH₃) is added to the medium, the colour turns orange. These colour transformations are due to the tyrosine and tryptophan amino acids. When the nitric acid gets into the hands, the reaction of the hands is yellow.

Diacetyl Reaction; arginine is a characteristic reaction for the amino acid. A dilute protein solution is mixed with 10% KOH solution and a dark pink colour with green fluorescence is formed if 1% diacetyl solution is added dropwise.

Lead Sulphur Reaction; if the alkaline solution of the protein is boiled with lead acetate solution, the sulfuric amino acids give a black lead sulphide precipitate or a brunette colour.

Methods for determination of total organic nitrogen

Proteins are composed of C, H, O, N, S and P. The amount of nitrogen in protein molecules is approximately 16%, this ratio is different in different foodstuffs. In the determination of the total organic nitrogen, there are two methods for the foods. Methods based on the conversion of nitrogen in natural form into elemental nitrogen in food. Methods based on converting nitrogen in natural form into ammonium salts in food.

There are three major methods developed on the basis of determination of gasified nitrogen or ammonium salts. Dumas method was developed in France in 1831. After that, Kjeldahl method was developed in Danish in 1883. The Ter Meulen method was developed in the Netherlands in 1924. Then, these methods are modified for several times.

Methods based on colorimetric principles

Colorimetric analyses can be applied to both macro and micro levels. By colorimetric methods, not only proteins in the dairy products, but also peptides and amino acids can be detected.

Protein determination by colorimetric method; it is the reaction of peptide bonds or amino acid residues with a suitable chemical chromophore group. The coloured proteins are measured by the spectrophotometer principle of light absorption.

Bi-urea method; in the strongly alkaline environment, the proteins in the food react with the copper compounds to form a red-violet or red-purple (purple) compounds. Since the intensity of the colour formed depends on the amount of protein in the environment, protein determination methods based on bi-urea reaction were developed.

FCL (Folin-Ciocalteu-Lowry) method; the Folin solution reacts with the proteins in the food and creates a blue colour in this method.

Formol titration method

This method is one of the fastest methods to determine the amount of milk proteins in dairy products. The amine group (-NH₂) is converted to the methylene amino group (-N = CH₂) by addition of formaldehyde to the amino acids in the proteins. The released carboxyl (COOH) group is titrated by the adjusted base and the result is calculated. In this method, the amount of base spent on titration is directly proportional to the amount of protein.

ELISA “Enzyme-linked Immunosorbent Assay” method

It is a biochemical-immunological method, allowing the antibody to bind with the antigen. It is very sensitive to antibody and antigen determination in the samples. Unbound or non-specific proteins are removed by washing. Enzyme-linked secondary antibodies are added and bound to the antigen-antibody construct (sandwich structure). The amount of protein is measured spectrophotometrically by adding the substrate to interact with the enzyme.

Electrophoresis method

Electrophoresis is the migration of solutes or particles loaded in a liquid medium under the influence of an electrical field. Since electrophoresis provides migration of all particulate species, the term “iontophoresis” refers to the migration of small ions in particular. The most common electrophoresis applications include whey proteins, hemoglobin and isoenzymes.

X-ray crystallography method

The method works on the basis of X-ray transmittance in the sample. α -strand and

β -layer motifs contained in proteins can be determined by this method. Information about chemical bonds in the protein can be obtained. By collecting all data, the three-dimensional structure of the protein can be understood. The fact that some proteins do not crystallize restricts the use of this method.

Nuclear Magnetic Resonance (NMR)

NMR is used to investigate the three-dimensional structure of proteins. For NMR, it is necessary to use high purity protein sample. This method is applicable for natural or recombinant proteins and suitable for structure analysis of small proteins (35 kDa).

Chromatographic methods

The proteins can be detected by analytical devices compatible with liquid chromatography due to their lack of thermal stability and their polar ligands. Peptides and proteins are separated based on differences in surface hydrophobicities or surface charges. These methods are thin layer chromatography, ion exchange chromatography (IEXC), affinity chromatography, hydrophobic interaction chromatography (HIC), gel filtration chromatography, reverse phase chromatography (RPC).

Thin layer chromatography is decomposition of proteins according to their dissolution ability. The sample impregnated on a solid surface (cellulose) is placed in the solvent surface. By dissolving the solvent solution on the surface, the proteins in the sample are separated according to their dissolution ability. Ion exchange chromatography is separation of proteins according to ionic

loads. Hydrophobic interaction chromatography is chromatographic separation technique commonly used for purification of macromolecules such as proteins and polynucleotides. Purification schemes are mostly developed by combining HIC with ion exchange, size exclusion and affinity chromatography. Affinity chromatography ensures separation of proteins to chemical groups. Gel filtration chromatography allows the separation of proteins according to their size.

Reverse phase high performance liquid chromatography (RP-HPLC) involves the separation of molecules on the basis of hydrophobicity. The separation depends on the hydrophobic binding of the dissolved molecule to the immobilized hydrophobic ligands, which are bound to the stationary phase from the mobile phase to the sorbent (Mant & Hodges, 1996). In reverse phase method, analyses are performed using C4, C8 and C18 filled non-polar columns. The C18 hydrophobic phase is suitable for separating peptides smaller than ~ 2000-3000 Da. The C4 hydrophobic phase is suitable for the separation of peptides and proteins larger than ~ 3000 Da (Aguilar & Hearn, 1996). Milk proteins can be detected by photo-diode array (PDA) and diode array (DAD) detectors with expanded UV and visible region properties in RP-HPLC technique. However, precise and accurate results are difficult to achieve with the combination of analytical devices that work only with light absorption. Due to interference elements and chromatographic separation difficulties, it is not possible to reach reliable analysis results. The exact solution of a correct analysis is possible by using mass selective detectors. The use of MS in

chromatography has several advantages. MS is a very sensitive detection technique. MS provides the separation of peptides/proteins by molecular weights. MS can detect proteins or peptides as specific mass (Premstaller, Oberacher & Walcher, 2001). Finger print of proteins can help identify of peptides/proteins milk origin (ZACHAR, 2011).

CONCLUSION

Chromatographic techniques have developed into powerful separation techniques, capable of separating large numbers of proteins and peptides. As a result, combining chromatography techniques has become a widespread method for protein analyses and separations in dairy products. Reversed-phase HPLC technique has become an essential technique in the separation and analysis of milk proteins and peptides in dairy products. It is widely used in the life science to characterize proteins and to analyse them for product identity and impurities. Reversedphase HPLC combination of mass spectrometry provides a powerful technique for milk protein analysis. Mass spectrometry interfaces with reversed-phase HPLC by means of the electro spray ion (ESI) source. The polar and ionized groups scattered on the surface of the protein particles determine the electrical charge and electrical properties of the protein molecule. Amino groups take protons and form cations ($\text{NH} + 4$). These groups are soluble in aqueous media and form ions. Carboxyl and phosphate groups gain anionic property by giving proton (H^+) to the environment. It is very difficult to ionize large molecules with ESI soft ionization technique. However, thanks to these ions, it is possible to analyze milk

Taner Erkaymaz, Firuze Ergin, Gizem Yildiz, Ahmet Kucukcetin
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Interfacial behavior of casein-inulin interactions at the oil and water interfaces

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casein, dietary fiber

Abstract. Mixtures of proteins and dietary fiber are frequently used in many technological applications in food industry. In many of these applications' protein-dietary fiber mixtures are used in the production of processed dispersions containing two or more immiscible phases such as aqueous, oil and/or gas phases in the forms of emulsions or foams. Due to their large interface areas, the dispersions are spontaneously unstable systems and prone to destabilization. The instability of these systems is achieved by a protective surface layer around the particles. The properties of this interface layer are controlled by the composition and structure of the adsorbed material. The aim of present study is to investigate the interfacial properties of protein- dietary fiber interactions at oil/water interfaces. For this reason, 1% solutions of casein, as a model compound, and mixed with the inulin, an important dietary fiber, have been prepared. The BiCone rotor has a diameter of 68 mm and a cone angle of 10° was used and the data were recorded at 25 °C. The rotational as well as oscillatory experiments were conducted and the interfacial shear stress (τ_i), interfacial viscosity (η_i) and interfacial modulus (G_i' , G_i'') values were recorded. Water and oil interfacial properties of samples were evaluated in terms of time, stress, strain and frequency sweep measurements. The G_i' values were higher than G_i'' ($G_i' > G_i''$) at studied frequency and the η_i was measured 1.616×10^{-3} Pas.m at the shear rate of 100.

INTRODUCTION

Proteins are commonly used amphiphilic molecules which widely find applications in food dispersions such as foams and emulsions. In contrast to small molecule surfactants, proteins not only reduce the interfacial tension during adsorption, they can also form a viscoelastic (multi) layer in the interface to protect oil droplets

against flocculation and coalescence (Wang et al., 2011).

Dietary fibers which is mostly provided by the cell wall of vegetables, fruits and cereals, include polysaccharides (pectin, cellulose and hemicellulose) and lignins. Both soluble and insoluble fibers may be present; however, higher amounts of

insoluble fibers are used for food fortifying purposes (Staffolo, Bertola, & Martino, 2004). The fiber may interact with other food components during processing. These interactions can lead to changes in the bioavailability of nutrients, texture or flavors of the product (Fernandez-Garcia & McGregor, 1997). Due to providing a desired structure to the foodstuffs, biopolymer mixtures are widely used in the food industry. Protein-polysaccharide complexes formed by electrostatic interactions have been reported to increase the stability of emulsions (Roudsari, Nakamura, Smith, & Corredig, 2006; Tran & Rousseau, 2013). Therefore, the knowledge of mechanisms occurring in casein-polysaccharide mixture systems is of great importance (Bourriot, Garnier, & Doublier, 1999a). Casein micelles have a relatively large and highly complex structure (diameter 20-600 nm). This molecular assembly is a supramolecular association of individual casein subunits of α s1-, α s2-, β - and κ -caseins. These fractions are organized in micelles according to hydrophobic and hydrophilic groups (Bourriot, Garnier, & Doublier, 1999b).

The interfacial rheology describes the functional interaction of the deformation of an interface, the forces exerted on it, and consequently the flows in the adjacent phases of the fluid. This can be determined by applying dilatation and shear forces. The shear rheology of the interfacial layers at the gas/liquid or liquid/liquid phase boundaries is related to a wide range of technical applications, especially in colloidal systems including large interfaces such as foams and emulsions. The interfacial flow behavior of such systems is controlled by the presence of particles present in the system such as

proteins, surfactants, lipids, which will be occurred due to the adsorption of interfacial active molecules and attachment of particles or by spreading or layer formation of the amphiphilic substances. The application of shear deformations to the interface layers provides indirect access to inter- and intramolecular interactions in the interfaces (Krägel & Derkatch, 2010). In the interface rheology, the interface area is kept constant and the information about the elastic or storage module (G') and the viscous or loss module (G'') depends on the frequency (Krägel & Derkatch, 2010; Oliveira, Santos, Vieira, Fraga, & Mansur, 2017). The BiCone geometry, magical rod and the du Noüy ring was used for the measurement of the surface shear rheology and various proteins such as β -lactoglobulin and hydrophobins have been studied using these attachment (Li et al., 2016). However, there are few studies on the interfacial rheology of proteins and dietary fibers and their interactions at oil/water interfaces. The interfacial rheology of casein which is the major fragment of the milk protein and the inulin as a dietary fiber was used to investigate the interfacial viscoelastic behavior adsorption layer at the water and oil interfaces using a rotational rheometer equipped with BiCone geometry.

MATERIAL AND METHODS

The inulin used in this study was kindly purchased from the Orafiti Food Ingredients (High performance inulin, HP, Belgium), the casein from bovine milk was from Sigma-Aldrich, USA. The sunflower oil was purchased from a local market.

The aqueous phase of samples was prepared with the equal amount of inulin and casein. The total of these two ingredients in the mixture was 1%. After weighing and preparation of aqueous phase, the samples were subjected to continuous stirring for 12 h on a magnetic stirrer.

Interfacial rheology for the determination of the effects on sunflower oil/water interface was studied with a peltier system rheometer (Haake Mars II, Karlsruhe, Germany) with BiCone probe (BC 68 / 5Ti). Before starting the analysis, the micro stress calibration, device and probe calibration was conducted carefully. The liquid form (water), which had a high density and which would be at the bottom, was filled up to the specified line spacing and the gap height was determined for the device. As a result of this measurement, data manager system was opened and curve fit of F_n against h values was plotted. The zero-crossing point x_0 was calculated and used as the measuring gap for the rheological measurements at interface layer. Dynamic shear interfacial rheology analyzes were performed with time sweep, frequency sweep and strain sweep tests. The time sweep test was performed with amplitude value of $\omega = 0.1$ %, angular frequency $\gamma = 1$ rads⁻¹ for 1 hour. The frequency sweep test was run at $\gamma = 0.1-10$ rads⁻¹ and $\omega = 0.1$ % linear region. The strain sweep test was conducted at $\omega = 0.01-100$ % and $\gamma = 1$ rads⁻¹ (Baldursdottir, Fullerton, Nielsen, & Jorgensen, 2010)

For both rotational as well as oscillatory test the measured raw data was modified in a such way that the contributions from the two bulk fluids are subtracted from the total results. The following equations was

used for the calculation of the G_i' and G_i'' of the sample.

$$G_i' = G'_{total}(\omega) - \frac{G'_A(\omega)}{2} - \frac{G'_B(\omega)}{2} \quad (1)$$

$$G_i'' = G''_{total}(\omega) - \frac{G''_A(\omega)}{2} - \frac{G''_B(\omega)}{2} \quad (2)$$

where

$G_i'(\omega)$ is interfacial storage modulus as a function of the applied angular frequency

$G_i''(\omega)$ is loss modulus as a function of the applied angular frequency

$G'_{total}(\omega)$ is the total storage modulus signal from the measurement with two liquids and interfacial layer as a function of the applied angular frequency

$G''_{total}(\omega)$ is the total loss modulus signal from the measurement with two liquids and interfacial layer as a function of the applied angular frequency

$G_A'(\omega)$ is total storage modulus from the bulk fluid A

$G_B'(\omega)$ is total storage modulus from the bulk fluid B

$G_A''(\omega)$ is total loss modulus from the bulk fluid A

$G_B''(\omega)$ is total loss modulus from the bulk fluid B

RESULT AND DISCUSSION

In order to investigate the effect of inulin and casein interaction at the oil/water interface, time sweep test were conducted and the elastic modulus (G_i'), loss modulus (G_i'') and interfacial complex viscosity (η_i^*) were measured at a frequency of 1 rads⁻¹ and a strain amplitude of 0.1 % as shown in Figure 1. Moreover, the time evaluation of G_i' and G_i'' of the sample was illustrated in Figure 1. The value along with the variation in the G_i'' was larger than that of G_i' . Also, the interfacial complex viscosity increased during the time sweep experiment. The structure and conformation of the casein-

inulin may support viscoelasticity and interfacial adsorption. In a previous study, it has been reported that protein-polysaccharide systems exhibit stronger dilatational viscoelastic properties than protein alone (Jourdain, Schmitt, Leser, Murray, & Dickinson, 2009).

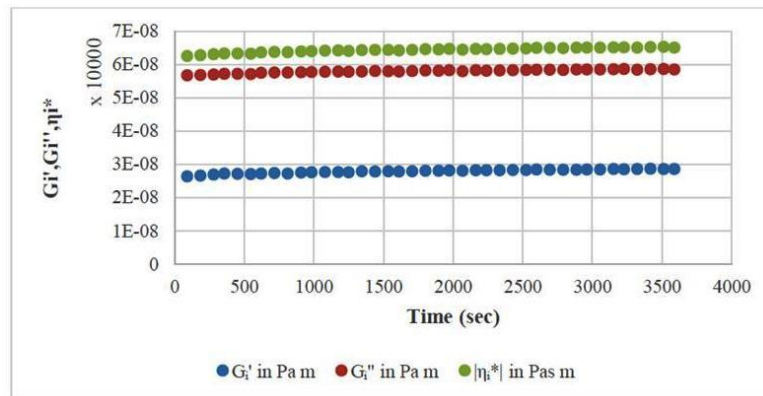


Figure 1
 Time evaluation of the interfacial elastic modulus (G_i'), viscous modulus (G_i'') and interfacial complex viscosity (η_i^*) of the sample at oil/water interface

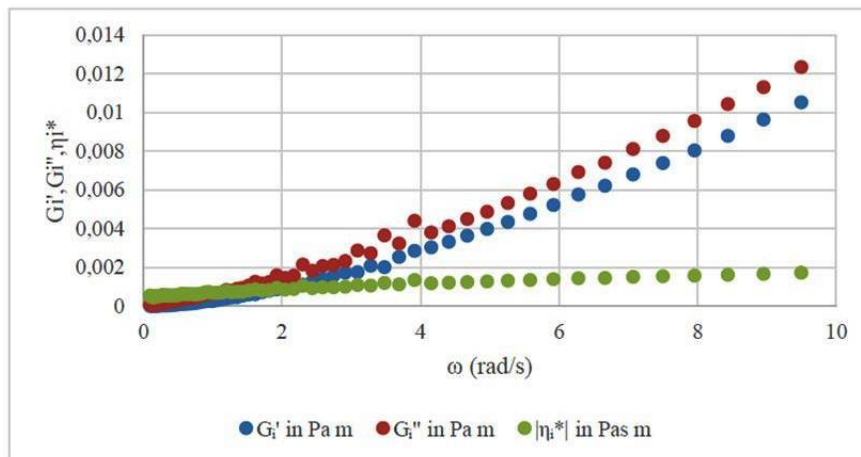


Figure 2
 Frequency sweep experimental results of samples at the oil/water interfaces

Frequency sweep test was performed at 25°C, $\gamma=0.1\%$ and the 0.1-10 rads⁻¹ frequency range and the results were illustrated at Figure 2. Both the elastic and viscous interfacial modulus of the casein-inulin at oil water interfaces was found to be dependent on the frequency, over the measured frequency range unlike the interfacial complex viscosity. While the η_i^* of the sample was independent of the frequency, the G_i' and G_i'' values of the sample was increased as the applied frequency was increased. The prepared sample was exhibited viscous behaviors with the $G_i'' > G_i'$ at studied frequencies. Strain sweep measurements were performed in order to trace the possible fracture mechanism of the samples. Figure 3 exhibited the strain dependence of the both interfacial elastic modulus and interfacial viscous modulus of the samples studied at the oil and water interface.

As can be easily seen from this figure, linear trend except some of the data was observed in the G_i' and G_i'' values of the casein and inulin samples at the measured frequency. G_i'' values were over the G_i' . The presence of dietary fiber may had an influence on the conformation of the casein molecules at the oil and water interface. In previous study, it is also reported that the presence of polysaccharides may hinder the conformational changes β -conglycinin at the oil/water interface, thus leading to a delay in reaching the adsorption rate (Li et al., 2018). The results of this study indicated that the dietary fiber and protein interaction may affected the interfacial rheological properties of the emulsions at oil and water interfaces. It should be considered that the film formation and emulsion stability of food products could be attributed to these results.

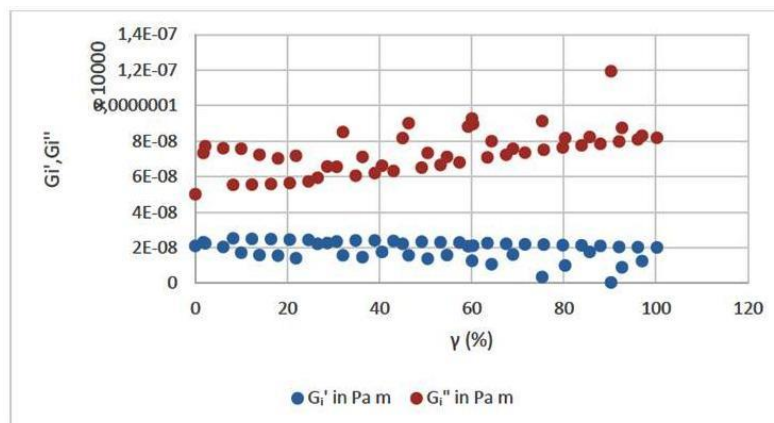


Figure 3
The strain dependency of elastic and viscous modulus of caseininulin at the oil/water interface

CONCLUSIONS

In this work, the interfacial rheological properties of casein and inulin at oil and water interface has been studied. Shear, time and frequency sweep measurements with the aid of BiCone geometry was done in order to characterize the samples. The results suggested that interfacial shear rheological properties may strongly affected by the dietary fiber and protein interaction. Besides, this study indicates that protein and dietary fiber may significantly improve the emulsifying and rheological properties of inulin-casein samples and provides useful information for the preparation of high emulsifying food products.

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Duygu Aslan, Mahmut Dogan, Meryem Goksel Sarac
Interfacial behavior of casein-inulin interactions at the oil and water interfaces

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Adem Kaya, Cavit Aktar, Osman Kadir Topuz
Effect of fluidized bed drying on the fatty acid content of giant red shrimp
(*Aristaeomorpha foliacea*) byproducts

Effect of fluidized bed drying on the fatty acid content of giant red shrimp (*Aristaeomorpha foliacea*) byproducts

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Keywords:

Red shrimp, fatty acids,
shrimp byproduct,
fluidized bed drying

Abstract. Giant red shrimp (*Aristaeomorpha foliacea*) is commercially valuable shrimp species found in Mediterranean Sea. During the shrimp processing, depending on the species, size, and shelling procedure, about 40-50% of the raw material weight is discarded as nonedible parts and named as byproduct. Byproduct of shrimp consist of meat, peels and other residues. These byproducts still contain valuable nutrients and functional compounds such as fatty acids, mineral salts, proteins, chitin, and pigments. The important human health benefits are associated with Omega-3 fatty acids particularly eicosapentaenoic (EPA, 20:5 n-3) and docosahexaenoic acid (DHA, 22:6 n:3). Recovering of bioactive compounds such as Omega-3 fatty acid rich oils has increased greatly during past few decades due to the its commercial value. The aim of this study was to recover of Omega-3 fatty acid rich shrimp oil from byproducts by applying of different biomass drying methods including fluidized bed drying method (FBD) and conventional oven drying method (ODS). The results showed that Omega-3 fatty acid content and health lipid indices (AI and TI) of shrimp byproducts were significantly ($P < 0.05$) affected by biomass drying methods. Omega-3 fatty acid content of fluidized bed dried shrimp byproduct was significantly higher than conventional oven dried byproduct.

INTRODUCTION

Depending on the species, size and shelling procedure, byproducts of shrimp comprise 40-50% of the whole shrimp weight. Although shrimp byproducts contains valuable nutrients such as proteins, free amino acids, Omega-3 rich oil, chitin, carotenoids, flavours, minerals and enzymes, it discarded as waste (da Silva et al., 2017; Prameela et al., 2017;

Sila et al., 2014). Polyunsaturated fatty acids (PUFA) are important fatty acids and contain more than one double bond in their carbon chain. PUFAs are categorized into two main sections; Omega-6 and Omega-3 depending on the position of the first double bond from the methyl end group of the fatty acid (Venegas-Calerón, Sayanova, & Napier, 2010). Eicosapentaenoic acid (EPA) and docosahexaenic acid (DHA) are most

Adem Kaya, Cavit Aktar, Osman Kadir Topuz
*Effect of fluidized bed drying on the fatty acid content of giant red shrimp
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valuable fatty acids found in aquatic origin biomaterials. EPA and DHA are reported to be in relation with prevention of cardiovascular diseases and have certain efficacy in preventing illnesses with an inflammatory component. It is postulated that they reduce hypertension, asthma, immune system disorders, susceptibility to mental illness, protection against heart disease, and improved brain and eye functions (Topuz, Yerlikaya, Yatmaz, Kaya, & Alp, 2017; Yerlikaya, Topuz, Buyukbenli, & Gokoglu, 2013). Unsaturated Omega-3 fatty acids, such as DHA and EPA are sensitive to oxygen, high temperature and ultraviolet light. During drying, chemical and physical reactions occur and therefore digestibility is increased owing to the protein hydrolyzation, but some thermolabile compound such as PUFA is often oxidized (Finot, 1997). Fluidized bed drying method and conventional oven drying methods are the common biomass drying method for the extraction of bioactive compounds from biomaterials. The aim of the study was to compare effect of fluidized bed drying and conventional oven drying on the fatty acid profile of shrimp byproduct.

MATERIAL AND METHODS

Giant red shrimps (*Aristaeomorpha foliacea*) were obtained from the seafood market in Antalya, Turkey. Shrimps are transported in cold chain and its byproducts, consisting heads, cephalothorax and shells were obtained manually. The shrimp byproducts were washed thoroughly with distilled water and spread over on filter paper for 10 min to remove excessive water on surface. Shrimp byproduct powder was divided

two groups prior to drying process and stored at -80°C in laboratory type deep freezer (Dairei Europe, ULTF 80).

Drying process: First group of shrimp byproducts was dried in fluidized bed dryer (Retsch, TG 200, Germany) at 60 °C with air speed of 150 m³/hour up to water activity of 0.35 (aw: 0.35) (approximately for 3-4 hours) and marked as 'FBD'. Second group was dried in conventional oven dryer at 60°C for 28 hours up to water activity of 0.35 (aw: 0.35) and marked as 'ODS'. All dried byproducts were ground to fine particles size with laboratory type grinder (Bosch mkm 6000, Turkey) and passed through a 1.5 mm mesh screen.

Oil extraction: Oil extraction from shrimp byproduct was performed according to method of (Blig & Dyer, 1959). 10 g byproduct powder was mixed with a mixture of 10 ml chloroform and 20 ml of methanol for 3 min. 10 ml additional chloroform was added to mixture and mixture was blended 30 sec. And then, 10 ml distilled water was added and blending continued for 30 sec. Mixture was filtered through Whatman no:1 filter paper and filtrate collected in graduated cylinder. After allowing the filtrate to separate two layers, the volume of the chloroform layer was passed to rotary evaporator to evaporate chloroform. After the chloroform was evaporated completely, dryness of oil was ensured using nitrogen stream.

Fatty acid composition analysis: Methyl esters were prepared by transmethylation method using 2 M KOH in methanol and n-hexane, according to the method of (Özogul, Özogul, & Alagoz, 2007). The

Adem Kaya, Cavit Aktar, Osman Kadir Topuz
*Effect of fluidized bed drying on the fatty acid content of giant red shrimp
(Aristaeomorpha foliacea) byproducts*

fatty acid composition was analysed by a gas chromatography device (Clarus 500 Perkin-Elmer, USA) equipped with a flame ionization detector and a fused silica capillary SGE column (30 m x 0.32 mm ID x 0.25 µm BP20 0.25 UM, USA). The fatty acid composition analyses were performed in triplicate and the results were given in chromatography area % as mean values.

Health lipid indices: Data of fatty acid profile was used to determine the atherogenicity (AI) and thrombogenicity index (TI). AI shows the inhibition of the aggregation of plaque and diminishing the levels of esterified fatty acids, cholesterol, and phospholipids, thereby preventing the appearance of micro-and macro-coronary diseases. TI shows the tendency to form clots in the blood vessels. AI and TI index

were calculated as follows (Ulbricht & Southgate, 199).

$$AI = [12:0 + (4 \times 14:0) + (16:0)] / (\Sigma MUFA + \Sigma PUFA n-6 + \Sigma PUFA n-3)$$

$$TI = (14:0 + 16:0 + 18:0) / [(0.5 \times \Sigma MUFA) + (0.5 \times \Sigma PUFA n-6) + (3 \times \Sigma PUFA n-3) + (n-3) / (n-6)]$$

Statistical analysis: All experiments were conducted in duplicate, and all analyses were done at least in duplicate. Statistical analysis was conducted according to the statistical analysis software of SAS institute (Statistical Analysis System, Cary, NC, USA). Differences among the mean value of samples were tested by Duncan's Multiple Range Test and significance was defined at P<0.05.

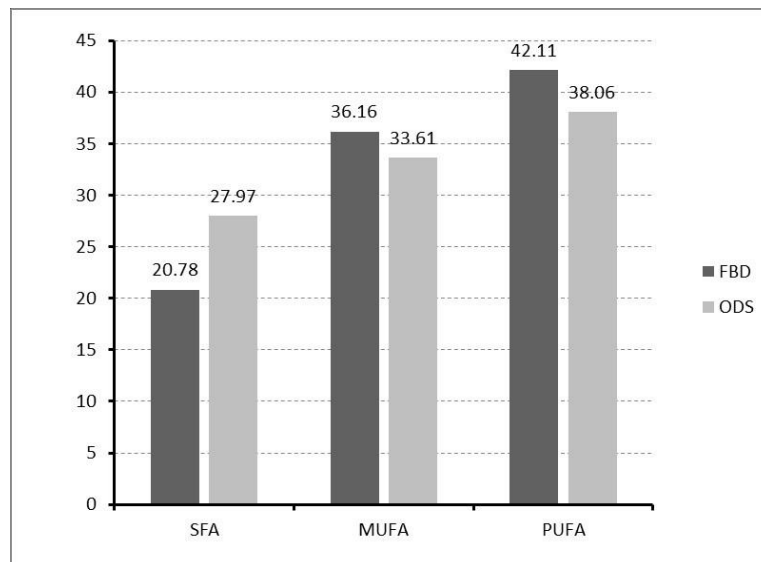


Figure 1
Fatty acid composition of oil extracted from shrimp byproducts.

RESULTS AND DISCUSSION

Fatty acid composition of red shrimp byproducts

Figure 1 shows the saturated (SF), monounsaturated (MUFA) and polyunsaturated (PUFA) fatty acid composition of oil extracted from red shrimp (*Aristaeomorpha foliacea*) byproducts. MUFA and PUFA contents of fluidized bed dried byproducts (FBD) were significantly ($P < 0.05$) higher than conventional oven dried byproducts (ODS) whereas SFA content was significantly ($P < 0.05$) lower than ODS. Fluidised bed drying has been recognised as a rapid, economic, gentle and uniform drying method with a high degree of efficiency compared with other drying techniques (Borgolte & Simon, 1981). PUFA content of FBD (42.11 g/100 g) was higher than that of red shrimp meat (38.88 g/100 g), whereas PUFA content of ODS was similar to PUFA content (38.06 g/100 g) of oil extracted from raw shrimp meat (Yerlikaya et al., 2013).

Figure 2 shows Omega-3 and Omega-6 fatty acid content of oil extracted from red shrimp (*Aristaeomorpha foliacea*) byproducts. Omega-3 fatty acid content of fluidized bed dried byproduct (FBD) was significantly ($P < 0.05$) higher than conventional oven dried byproducts (ODS) whereas Its Omega-6 fatty acid content was lower than ODS (Figure 2). Omega-3 fatty acid contents of both FBD (23.79 g/100 g) and ODS (17.76 g/100 g) were lower than that of raw meat of red shrimp (24.56 g/100 g) (Yerlikaya et al., 2013), whereas Omega-6 content of FBD (18.32 g/100 g) and ODS (20.3 g/100 g) was almost five fold higher than that of

Yerlikaya et al. (2013) (4.48 g/100 g). It could be stemmed from drying processes took place at high temperatures. Type and amount of consumed essential fatty acids and balanced intake of omega-3 and omega-6 are important for a healthy life. It is essential to decrease Omega-6 intake while increasing Omega-3 to prevent chronic disease (Simopoulos, 2002).

Figure 3 shows health lipid indices (atherogenicity (AI) and thrombogenicity (TI) indexes) of oil extracted from red shrimp (*Aristaeomorpha foliacea*) byproducts. As seen Figure 3. atherogenicity index (AI) and thrombogenicity index (TI) of oil extracted from fluidized bed dried byproducts (FBD) (0.269 and 0.184, respectively) was significantly ($P < 0.05$) lower than that of conventional oven dried biomass (ODS) (0.328 and 0.325, respectively). Seafood consumption are recommended by health authorities, not only for their high-quality protein and mineral content, but also for their healthful fatty acids. AI shows the inhibition of the aggregation of plaque and diminishing the levels of esterified fatty acids, cholesterol, and phospholipids, thereby preventing the appearance of micro-and macro-coronary diseases. TI shows the tendency to form clots in the blood vessels (Ulbricht & Southgate, 1991).

Atherogenicity indexes (AI) of both FBD (0.269 and ODS (0.328) were considerable higher than that of other species of red shrimp (*Aristeus antennatus*) (0.24) (Rosa & Nunes, 2004). Thrombogenicity index (TI) of FBD (0.184) was almost similar to lower TI (0.18) of *Aristeus antennatus*, whereas TI value of ODS (0.247) was considerably higher.

Adem Kaya, Cavit Aktar, Osman Kadir Topuz
Effect of fluidized bed drying on the fatty acid content of giant red shrimp
(*Aristaeomorpha foliacea*) byproducts

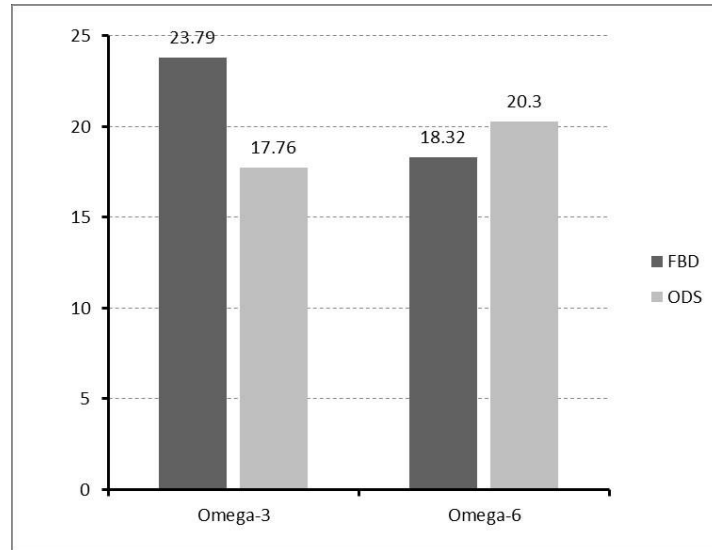


Figure 2
Omega-3 and Omega-6 fatty acid contents of shrimp by products.

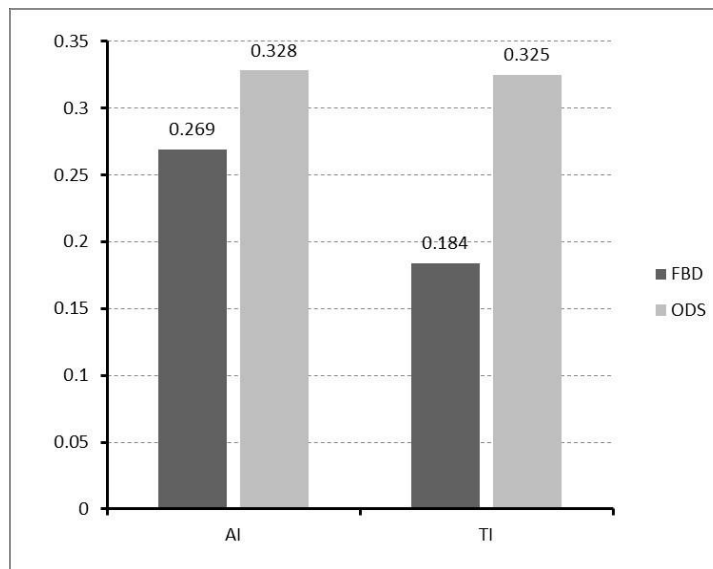


Figure 3
Atherogenicity (AI) and thrombogenicity index (TI) of shrimp by products.

Adem Kaya, Cavit Aktar, Osman Kadir Topuz
*Effect of fluidized bed drying on the fatty acid content of giant red shrimp
(Aristaeomorpha foliacea) byproducts*

CONCLUSIONS

The results of this study reveal the high nutritional quality of red shrimp byproducts oil. Fluidized bed drying of shrimp byproduct biomass contributed to its nutritional quality with preserving its omega-3 fatty acids. Conventional oven drying of shrimp byproduct biomass had lowering effect on the Omega-3 fatty acid content of oil extracted from red shrimp byproducts since oven drying of byproducts was taken place at high temperatures for long time. Its concluded that fluidized bed drying method could be used for drying of biomass containing valuable and sensitive bioactive compounds such as omega-3 fatty acids.

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Akdeniz University has 74,000 students and 2,700 members of the academic staff. The main campus of our university is located between Dumlupınar Boulevard and the Uncalı district. The main campus has an area of 3,483,589m², which includes 615,105m² of built up area. The campuses have a total indoor area 681,598m². Today, Akdeniz University continues its various activities in the fields of education, research and service to society in 24 Faculties, 7 Institutes, 1 School, 1 Conservatory, 12 Vocational Schools and 54 Research and Application Centres.



Drying Kinetics and Color Properties of Lemon Balm (*Melissa officinalis*) Leaves Dried by Convective Hot Air Drying
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Keywords:

Lemon balm,
hot air drying,
mathematical modelling,
color

Abstract. Lemon balm (*Melissa officinalis*) leaves with the moisture content of 3.18 g water/g dry base were dried by convective hot air drying at 50, 60 and 70°C until the moisture content fell down to 0.15 g water/g dry base. Drying experiments were completed between 17 and 50 min. depending on drying temperatures. For the selection of the most suitable thin layer drying model, five mathematical models (Page, Modified Page, Logarithmic, Lewis, Henderson and Pabis) were applied to the drying treatments. The higher correlation coefficient (R^2), and reduced root mean square error ($RMSE$), Chi square (χ^2) were used to identify the excellence of fit model for drying of lemon balm leaves. As a result of the statistical tests, Page and Modified Page were considered to be the best models for 60 and 70°C hot air drying experiments when compared to the other models. In addition, Logarithmic model resulted in preferable statistical values than other thin layer models at 50°C. The color values such as L^* , b^* , C^*_{ab} and h° decreased, while a^* value increased after drying. The effective moisture diffusivity ($Deff$) values of dried lemon balm leaves increased with the rise of drying temperatures and ranged between 2.03×10^{-8} to 7.13×10^{-8} m²/s. Total phenolic content and antioxidant capacity of dried lemon balm samples were both increased after drying. The total phenolic content and antioxidant capacity was obtained as the highest from 50°C treatment when compared with all cases.

INTRODUCTION

Lemon balm (*Melissa officinalis* L.), which is a member of *Lamiaceae* (formerly *Labiatae*) family, is grown as an ornamental plant in countries with a Mediterranean climate. It is native to

southern Europe and northern Africa, and east as far as the Caucasus and northern Iran. Geographically, it has spread to countries such as France, Bulgaria, Germany and Romania. On the other hand it is widely grown in Aegean and Mediterranean Regions of Turkey and also Istanbul and Bursa provinces. The

subspecies of *M. officinalis* are evaluated in domestic markets and they are also on the list of the exported medicinal and aromatic plants (Gasquet et al., 1993).

Lemon balm is a thin-leaved perennial herbaceous plant with yellow or whitish flowers at a height of 3-5 meters. Lemon balm, which has been known to have a calming effect since ancient times, is quite effective in the treatment of many diseases from stress to stomach disorders and it has a comforting feature due to its lemon like smell. It has also antispasmodic, antimicrobial and antimicrobial effects. Rosmarinic acid plays an important role in the chemicals obtained from melisa plant (Abad et al., 1997).

In a study, the essential oil of lemon balm was determined as 0.2%. In addition, the most important components were geranial (E-citral), neral (Z-citral), citronellal, Ecaryophyllenne and geraniol respectively (Dias et al., 2012). Carnat et al. (1998), also studied aroma components in lemon balm tea infusion, by GC-MS and determined geranial, neral and citronellal compounds respectively in the ratios of 43.53%, 30.15% and 16.81%.

Material and Method

Material and drying process

Fresh lemon balm leaves supplied from a local market in Bursa were stored in the refrigerator at a temperature of 4 ± 0.5 °C until drying process. After the samples were washed, the water was removed from the surface of leaves by paper towel. The

initial moisture content of samples was obtained by moisture analyzer (Sartorius MA150, Germany) and the average moisture content of lemon balm leaves was determined as 3.18 g water/g dry base.

Drying trials were performed in a hot air convective dryer which was produced by Yucebas Machine Analytical Equipment Industry (Y35, Izmir, Turkey) with the technical features of 220 V, 50-60 Hz, 200 W. 20 g lemon balm leaves were placed uniformly on an aluminum plate and dried at 50, 60 and 70°C with the constant 20% relative humidity. During drying samples were removed at intervals and weighed. The weight loss of samples was recorded by using a digital balance (Mettler Toledo, MS3002S, Greifensee, Switzerland) with the accuracy of 0.01 g. All weighing processes were completed in 10 s during drying process.

Mathematical modelling of drying data

Moisture ratio (MR) and drying rate of lemon balm leaves during drying were calculated by employing the following equations (Eq.1, Eq. 2).

where, *MR* is moisture ratio, *M* is the moisture content at a certain time (g water/g dry base), *M_i* is the primary moisture content (g water/g dry base), *M_e* is the equilibrium moisture content (g water/g dry base), *M_t* and *M_{t+dt}* are the moisture content at *t* and *t+dt* (g water/g dry base) respectively, and *t* is drying time (min) (Dadali et al., 2007).

$$MR = \frac{M - M_e}{M_i - M_e} \quad (1) \quad \text{Drying rate} = \frac{M_{t+dt} - M_t}{dt} \quad (2)$$

$$RMSE = \left[\frac{1}{N} \sum_{i=1}^N (MR_{exp,i} - MR_{pre,i})^2 \right]^{1/2} \quad (3)$$

$$\chi^2 = \frac{\sum_{i=1}^N (MR_{exp,i} - MR_{pre,i})^2}{N-n} \quad (4)$$

The equations in Table 1 were used to find most convenient model for explaining the drying curve of lemon balm leaves. Root mean square error (*RMSE*) gives deviation between the estimated and experimental values for the models. To identify the thin layer drying characteristics of lemon balm leaves, the model with the higher correlation coefficient (*R*²), and reduced *RMSE* and chi-squared (χ^2) was selected

as a best model (Ozbek and Dadalı, 2007). These parameters were calculated using the sequent equations (Eq 3, Eq 4): where, *MR_{exp,i}* is the empirically dimensionless moisture ratio for test *i*, *MR_{est,i}* is the estimated dimensionless moisture ratio for test *i*, *N* is the count of observation and *n* is the count of constants in the model (Avhad and Marchetti, 2016).

Table 1. Mathematical models applied to drying curves of lemon balm leaves

Model no	Model name	Model	References
1	Page	MR = exp(-kt ⁿ)	Wang et al. (2007)
2	Modified Page	MR = exp [(-kt) ⁿ]	Toğrul (2006)
3	Logarithmic	MR = a exp(-kt) + c	Darıcı and Şen (2015)
4	Lewis	MR = exp(-kt)	Doymaz (2006)
5	Henderson and Pabis	MR = a exp(-kt)	Evin (2011)

Calculation of effective moisture diffusivity

Fick's second diffusion law has been widely used to explain the drying process of food products during the falling rate period (Doymaz, 2008). The solution of Fick's second law for an infinite slab is showed in Equation (5), assuming dimensional moisture movement volume

change, constant temperature and diffusivity coefficients, and negligible shrinkage (Crank, 1975);

where, *Deff* is effective moisture diffusivity (m²/s), *L* is the half thickness of the slab in samples (m), and *n* is a positive integer. In practice, only the first term Equation (5) is written in a logarithmic form as follows:

$$MR = \frac{8}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{(2n-1)^2} \exp \left(-\frac{(2n-1)^2 \pi^2 D_{eff} t}{4L^2} \right) \quad (5)$$

$$MR = \frac{8}{\pi^2} \exp\left(-\frac{\pi^2 D_{eff} t}{4L^2}\right) \quad (6)$$

$$D_{eff} = -\frac{slope 4L^2}{\pi^2} \quad (7)$$

The effective moisture diffusivity were determined using the method of slopes by plotting experimental drying data in terms of $\ln MR$ versus drying time, using the following equation (7).

Color analysis

Color measurements of the samples were determined over the outer surface of the samples by using a chroma meter (Konica Minolta CR-5, Bench-top, Japan). L^* , a^* , b^* values were displayed as lightness/darkness, redness/greenness and yellowness/blueness respectively. Analyzed CIE-L, a and b values were used to calculate chroma and hue angle to characterize color changes during drying (Mujumdar, 2000; Demir, 2018). Chroma (C^*) changed from 0 (dull) to 60 (vivid) and was calculated with the first equation (1). Hue angle (h°) value, demonstrated in the second equation (2) is defined by the angles of 0, 90, 180 and 270°, representing the colors of red, yellow, green and blue, respectively (Karaaslan and Tuncer, 2008).

$$Chroma (C^*) = \sqrt{(a^*)^2 + (b^*)^2} \quad (8)$$

$$h^\circ = \arctan\left(\frac{b^*}{a^*}\right) \quad (9)$$

Extraction of samples for total phenolic content and antioxidant capacity

Extractions were carried out according to Capanoglu et al. (2008). Extracts were

prepared by adding 5 mL 75% aqueous methanol containing 0.1% formic acid in a cooled ultrasonic bath for 15 min and 10 min of centrifugation at 4°C and 2700 × g, after which the supernatants were collected. The extraction procedure was repeated three times, and all the extracts were stored at -20°C until analysis.

Determination of total phenolic content and antioxidant capacity

Folin-Ciocalteu spectrophotometric method was used to determine total phenolic content as described by Spanos and Wrolstad (1990). Gallic acid was used for the calibration of the standard curve ($R^2=0.9835$). The phenolic content was expressed as gallic acid equivalents of dry weight (mg of GAE/100g dw).

Antioxidant capacity of the fresh and dried lemon balm samples were measured with 2-diphenyl-1-picrylhydrazyl (DPPH), method (Katalinic et al., 2006). Trolox was used as the calibration of the standard curve ($R^2=0.9929$). The results were given as μmol Trolox equivalent (TE) per g dry weight ($\mu\text{mol TE/g dw}$).

Statistical analysis

The experiment was conducted in a completely randomized design with three replications. The results were statistically evaluated by one-way analysis of variance

(ANOVA) using the JMP software package version 6.0 (SAS Institute Inc. NC, 27513). When significant differences were found ($P < 0.05$), the Least Significant Difference (LSD) test was used to determine the differences among means.

RESULTS AND DISCUSSION

Drying characteristics of lemon balm leaves

The lemon balm leaves were dried in a hot air dryer using different temperature until

the moisture content reached 0.15 g water/g dry base. The changing of the moisture content versus drying time at various temperatures was given in Figure 1. The drying process took 50, 28 and 17 min at 50, 60 and 70°C, respectively. It was apparent that drying time decreased continuously with increasing temperature. This observation is in agreement with previous studies on drying of tomatoes (Doymaz, 2007), mint leaf (Therdthai and Zhou, 2009) and kiwifruit (Orikasa et al., 2008).

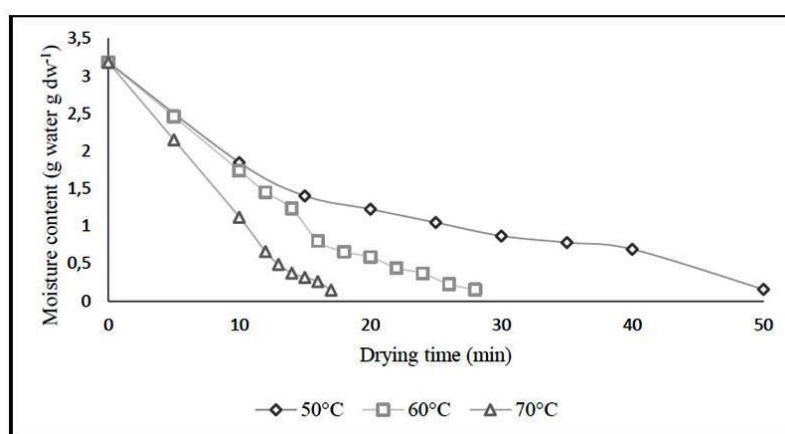


Figure 1

Moisture content of lemon balm leaves as a function of drying time at different drying temperatures

Results of drying rate during drying time, obtained in drying of lemon balm leaves carried out at three temperatures were presented in Figure 2. As can be seen from this figure, there is no constant rate period in drying curves, and all the drying processes occurred at a falling rate period. The results showed that moisture movement in the lemon balm leaves is governed by diffusion (Doymaz, 2005). Similar findings were reported on drying

of various food products (Akpınar et al., 2003; Senadeera et al., 2003; Wang et al., 2007).

Mathematical modelling of drying curves

Table 2 shows drying model coefficients and comparison criteria (R^2 , RMSE and χ^2) of the five thin layer drying model. The statistical parameter estimations showed in all cases that R^2 , RMSE and χ^2

values ranged from 0.8837 to 0.9918, 0.005350 to 0.109539 and 0.000361 to 0.121489, respectively. As a result of the statistical tests, Page and Modified Page models provided higher R^2 and lower RMSE and χ^2 values when compared to the other models for 60 and 70°C hot air drying experiments. For a temperature of 50°C, Logarithmic model gave better statistical values than the other models. Madamba et al. (1996) and Toğrul and Pehlivan (2003) for Logarithmic model and Dadali et al. (2007) and Çakmak et al. (2016) for Page and Modified Page models working with the garlic, apricot, okra and strawberry, respectively also recorded similar results.

Table 2. Statistical results obtained from the selected models

Model no	Drying processes	Model coefficient	RMSE	χ^2	R^2
1	50°C	n=0.8958 k=0.0662	0.011510	0.001533	0.9090
	60°C	n=1.4383 k=0.0234	0.005350	0.000361	0.9918
	70°C	n=1.6657 k=0.0255	0.005582	0.000412	0.9913
2	50°C	n=0.8958 k=0.0483	0.011510	0.001533	0.9090
	60°C	n=1.4383 k=0.0736	0.005350	0.000361	0.9918
	70°C	n=1.6657 k=0.1105	0.005582	0.000412	0.9913
3	50°C	k=0.0423 a=0.8299 c=0.0494	0.018581	0.004661	0.9786
	60°C	k=0.1323 a=1.3423 c=0.0484	0.038613	0.023855	0.9078
	70°C	k=0.2052 a=1.4235 c=0.0459	0.056000	0.042336	0.9209
4	50°C	k=0.8837	0.109539	0.121489	0.8837
	60°C	k=0.0915	0.022209	0.006457	0.9308
	70°C	k=0.1513	0.027955	0.007912	0.9157
4	50°C	k=0.0500 a=1.0672	0.017598	0.003584	0.8855
	60°C	k=0.108 a=1.3991	0.035285	0.017929	0.9589
	70°C	k=0.1757 a=1.3985	0.047437	0.026039	0.9376

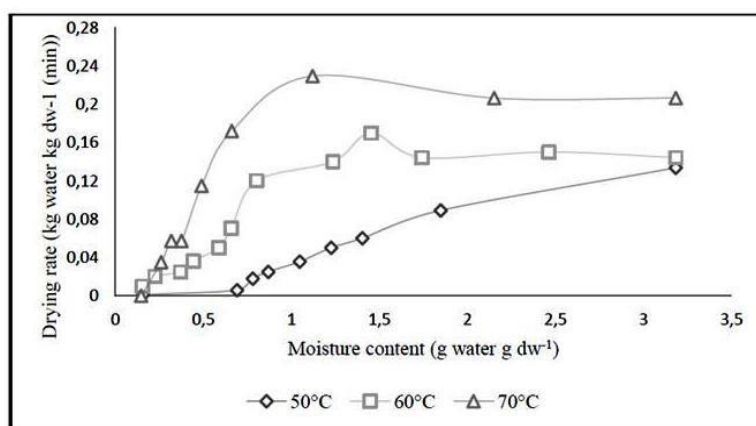


Figure 2

Drying rate of lemon balm leaves as a function of moisture content at different drying temperatures

Table 3: Values of effective moisture diffusivity obtained for lemon balm leaves at different temperatures

Drying processes	Deff (m ² /s)
50°C	2.03×10 ⁻⁸
60°C	4.38×10 ⁻⁸
70°C	7.13×10 ⁻⁸

Effective moisture diffusivity (Deff)

The effective moisture diffusivity (Deff) values for different drying temperatures, calculated from Equation 7, ranged from 2.03×10⁻⁸ to 7.13×10⁻⁸ m²/s (Table 3). It can be seen that the values of Deff increased greatly with increasing temperature. This result can be explained by the easier evaporation of the product in high temperature and the increase in drying rate (Mengeş and Ertekin, 2006). The values of Deff in our study were within the general range 10-12-10⁻⁸ for drying of food materials (Demiray and Tulek, 2017). Our findings are in line with the results informed by Doymaz (2006) who also acquired an increase in Deff values of mint leaves from 3.067×10⁻⁹ m²/s to 1.941×10⁻⁸ m²/s with the rise in drying temperature.

Color analysis

The results of color changes in fresh sample for all drying conditions were given in Table 4. The L* value were significantly affected by different drying treatments (*p* < 0.05) and resulted with a 2.05-26.62% decrease. The lowest L* value obtained from hot air 70°C dried samples which had darker color than other drying methods. Compared to the fresh sample, *a* (redness) values significantly increased (*p* < 0.05)

with all hot air drying treatments. The increase of *a** value could be a result of the Maillard reaction and degradation of pigments such as carotenoids (Maskan, 2001; Xiao et al., 2012). *b** values of dried lemon balm were decreased with respect to fresh samples between the ratios of 41.70-65.60%. This decrement was closely followed for Chroma (*C*_{ab}*) values, which were used to comprehend intensity of color. Hawlader et al. (2006) reported that, the reduction in *h*^o values is an expression of darker color. Hot air drying at 70°C caused a smaller reduction of *h*^o values. Additionally, pigment decompositions, non-enzymatic and enzymatic reactions are responsible for the formation of browning pigments (Albanese et al., 2013).

Total phenolic content and antioxidant capacity

The total phenolic content and antioxidant capacity of fresh and dried lemon balm samples were given in Table 5. The highest total phenolic content was attained by 50°C treatment with 2308.26±26.74 mg GAE/100 g dw (*p* < 0.05). Generally, total phenolic content was increased with drying treatments between the ratios of 5.85-255.07% when compared to fresh sample. Similar increment in total phenolic content was reported by Pricina and Karklina (2014) and Türkmen et al. (2005).

Table 4: Color values of fresh and dried lemon balm samples

Drying processes	L^*	a^*	b^*	C^*_{ab}	h°
Fresh sample	34,52±0,44 ^a	-8,89±0,84 ^c	19,71±2,62 ^a	21,62±2,74 ^a	114,38±0,91 ^a
50°C	33,81±1,52 ^a	-1,61±0,16 ^b	6,79±2,42 ^c	6,99±1,69 ^c	104,26±5,09 ^b
60°C	28,79±1,02 ^b	2,11±0,41 ^a	11,49±0,09 ^b	11,69±0,14 ^b	79,62±1,97 ^c
70°C	25,33±1,25 ^c	1,83±0,47 ^a	6,78±1,30 ^c	7,04±2,42 ^c	74,44±4,04 ^c

^{a-c} Different letters in the same column display significant difference ($P < 0.05$)

Table 5: Total phenolic content and antioxidant capacity of fresh and dried lemon balm samples Total phenolic content

Drying processes	Total phenolic content (mg GAE/100g dw)	Antioxidant capacity ($\mu\text{mol TE/g dw}$)
Fresh sample	650.09±51.49 ^b	1.00±0.34 ^d
50°C	2308.26±26.74 ^a	33.55±0.28 ^a
60°C	795.97±60.68 ^b	17.32±0.90 ^b
70°C	688.59±53.52 ^b	4.48±0.49 ^c

^{a-d} Different letters in the same column display significant difference ($P < 0.05$)

Antioxidant capacity of fresh lemon balm was significantly lower compared to dried samples ($p < 0.05$). The highest antioxidant capacity was obtained from hot air-50°C treatment with 33.55±0.28 $\mu\text{mol TE/g dw}$ while the lowest antioxidant capacity was determined in hot air drying at 70°C (4.48±0.49 $\mu\text{mol TE/g dw}$). Vega-Galvez et al. (2012) determined an increase in DPPH free radical scavenging activity of hot air dried peppers. Additionally, Priccina and Karklina (2014) reported an increment in antioxidant activity of some vegetables.

CONCLUSION

This study determined the effects of different hot air drying temperatures on drying characteristics, color, total phenolic content and antioxidant capacity of lemon

balm leaves. Our results showed that, the fastest and the shortest drying times were obtained from 70 and 50°C, respectively. Among all applied mathematical models, Page, Modified Page and Logarithmic models were found to be the best models to describe the drying characteristics of lemon balm leaves. Fick's model of moisture diffusion fitted all experimental data with acceptable correlation coefficients. In the evaluation of color parameters, L^* , b^* , C^* and h^* values decreased while a^* value increased after drying. Total phenolic content and antioxidant capacity of dried lemon balm samples were both increased after drying. Among all samples, the total phenolic content and antioxidant capacity was obtained as the highest from 50°C treatment. In consequence, between hot air drying treatments, "50°C" was found

applicable for lemon balm related with the enhanced bioactive properties.

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