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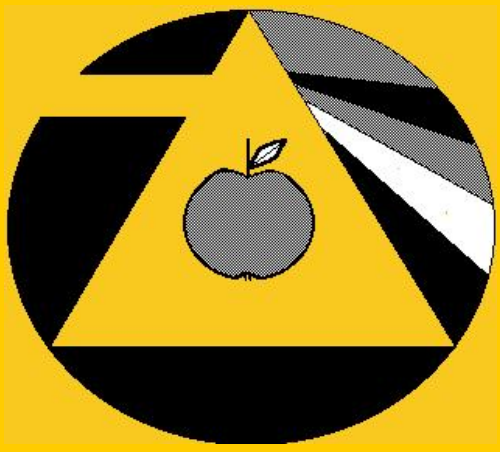

INTERNATIONAL SOCIETY OF FOOD PHYSICISTS

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HUNGARIAN BIOPHYSICAL SOCIETY

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EDITORIAL

This is the combined XXVIII/XXIX Volume of the Journal of Food Physics, and as You know the first issue was published in 1988, almost 3 decades ago. Many thanks for your kind help, cooperation, support and understanding the editors during this period of not easy existence.

We are sure, also this issue gives the opportunity for the readers to get interesting and useful information about some special questions of food physics. The topics of the scientific articles cover the following fields: differentiation and integration in natural science, drying kinetics, rheological parameters, food rheology, nutritional value, physics experiments in grammar shools, statical compressive properties, centrifugal freeze concentration, sensory characteristics, effect of the soaking pretreatment, starch gelatinization.

These papers were reported as lectures at the ISFP Debrecen Conference, 2016. 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. We will be really happy to have the possibility to continue the organisation of the ISFP conferences, the next conference will be organized in Turkey, 2018.

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

Andras S. Szabo
editor-in-chief

http://www.foodphysics.net

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XIIth International Conference of Food Physicists

http://mek.unideb.hu/index.php/hu/tudomany/icfp2016

The XIIth International Conference of Food Physicists was held in Debrecen, Hungary from the 6th to 8th, July 2016, organized by the International Society of Food Physicists and the University of Debrecen. The focus area of this biannual conference is all the physics related research fields, what contributes to the food sciences on any fields: food technology and unit operations, quality analysis and assurance, raw material production, health and environmental sciences. The conference provided an open channel for presenting and dissemination the new scientific results and applications, indicating creative discussions among the experienced researchers, students and the industry for finding the new opportunities and ways for further collaborations and common thinking.

23 oral lectures and 18 posters were presented during the first two days of the conference. The opening presentation showed the role of physics in the food science and raised awareness that this science is a part of technology, unit operations and quality analysis and its importance is as high as the importance of chemistry. The other presentations provided an overall overview of this fact; thermal kinetics during drying processing, low temperature cooking processing, use of ultraviolet light against mycotoxin contamination, rheological properties of milk products and cereals, modelling of physical parameters and changes and several other interesting presentations could be hear and seen during the conference.

Beside the lectures, scientific and social programs were made the conference more colourful: the pilot food processing plant of the University of Debrecen was visited and a tasting of traditional and local food products were organized during the first two days. On the third day of the conference was the poster session, where the best posters were awarded, and a visit to the Hungarian Academy of Sciences Institute for Nuclear

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Research and Department of Biophysics and Cell Biology of University of Debrecen closed the official program.

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 Turkey in 2018. We hope that all the participants and their colleagues will join to us and we will have a beautiful conference in the topics of our science, food physics.

Peter Sipos, Istvan Fekete, Andras S. Szabo

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DIFFERENTIATION AND INTEGRATION IN NATURAL SCIENCE, IMPORTANCE OF FOOD PHYSICS, AS A PART OF FOOD SCIENCE AND APPLIED PHYSICS

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Keywords:

applied physics,
food physics a bridge,
development of food physics,
interdisciplinary food physics,

Abstract. The development and modification of the science, forming and establishment of new fields is a normal process, carried out in general by 2 ways: differentiation and integration. This trend of development is typical for the segments of natural science, as well, including e.g. physics, chemistry, biology.
Analysing some special fields of natural science the paper tries to find true answers to the following questions:

- what are the dominant parts of food science and applied physics?
- 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 sub science?
- what are the development trends of food physics (Quo Vadis Cibus Physicorum)?

DIFFERENTIATION AND INTEGRATION

Figure 1. shows the process of differentiation and integration in physics.

Differentiation in classical physics:
- mechanics (statics, dynamics)
- optics
- acoustics
- electricity and magnetism
- hydrodynamics
- aerodynamics
- astrophysics
- nuclear physics
- statistical physics

Differentiation in modern physics:
- quantum mechanics
- wave mechanics
- particle physics (e.g. neutron physics)
- solid state physics
- high energy (speed) physics
- cosmic physics
- atomic (nuclear) physics
- reactor physics
- laser physics

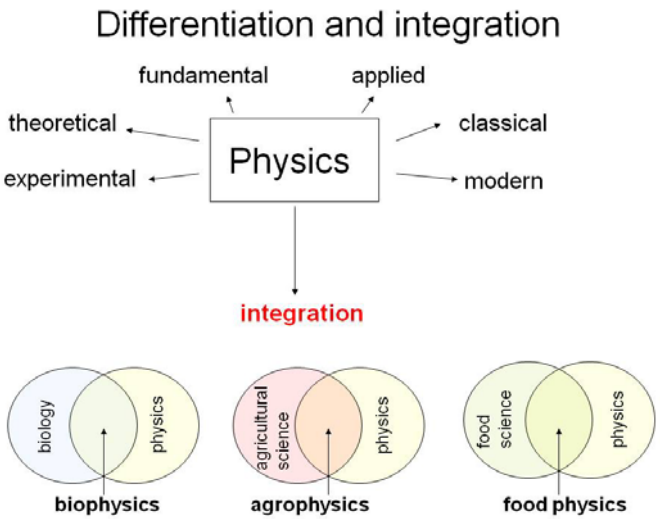

Figure 1
Process of differentiation and integration in physics.

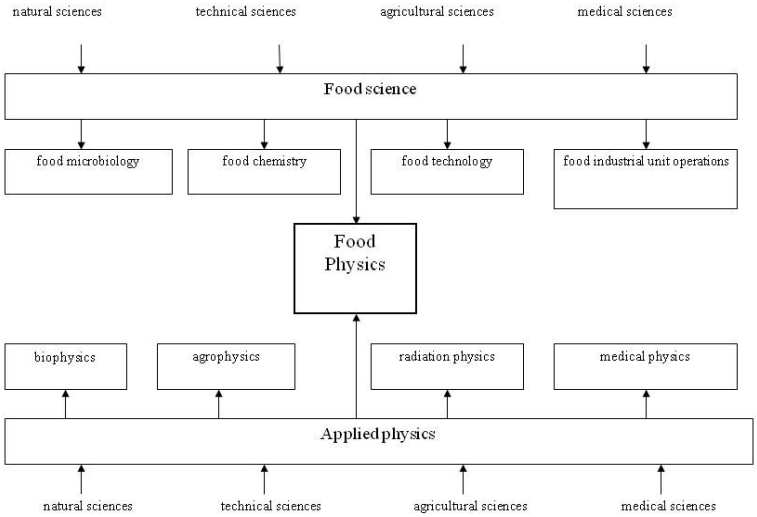

Figure 2
The food physics as a bridge between food science and applied physics

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FOOD PHYSICS AS A BRIDGE

Figure 2. shows the food physics as a bridge between food science and applied physics.
3 main topics of food physics:

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

INTERDISCIPLINARY SCIENCES AND SOME OTHER FIELDS OF APPLIED PHYSICS

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 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.

Some other fields of applied physics:

- soil physics
- geophysics
- atmosphere physics
- climate physics
- chemical physics
- polimer physics
- radiation physics
- medical physics
- biophysics

TRENDS IN THE DEVELOPEMENT OF FOOD PHYSICS (QUO VADIS CIBUS PHYSICORUM?)

Figure 3. shows the technology hill.
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

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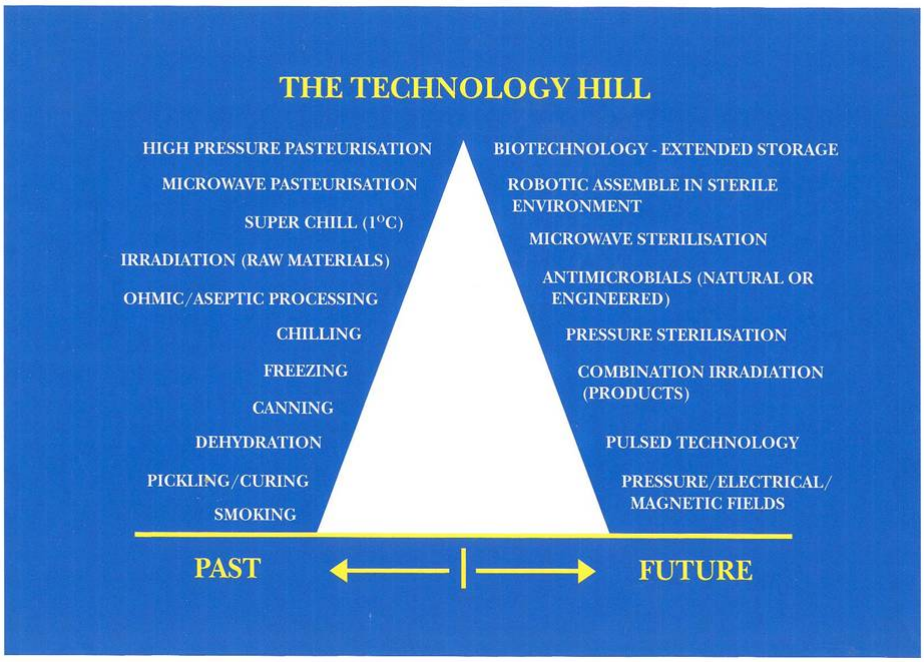

Figure 2
The technology hill

Nondesctructive measurement 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, plasto-metry, penetrometry, fructometry) for texture and consistence analysis

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Convective Drying Kinetics and Phenolic Contents of Olive Leaves

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Abstract. Olive leaves have considerable amount of "oleuropein" which is a kind of polyphenol. This compound could lower blood pressure, cancer risk and oxidative damage of cells. Because of the health benefits of this excellent antioxidant material, olive leaves are the subject of this study. Results of the research will contribute to information about drying behaviour of and drying impact on olive leaves.
Leaves were collected from *Olea europaea* trees from the garden located in Osmaniye Korkut Ata University, Turkey. After collection, they were washed, waited in room temperature to evaporate residual water and pulverized in a commercial blender. Leaf powders were dried in a conventional oven at 50, 60 and 70°C and the weight changes of samples were recorded manually in every 10 minutes for 50 and 60°C and 5 minutes for 70°C. Moisture ratio values were fitted into 13 different thin layer drying models and Parabolic and Cubic models were the best and had the highest R^2 value. On the other hand, total phenolic contents of dried powders were determined with Folin-Ciocalteu method and the highest phenolic amount (633.85 mg / kg gallic acid equivalent) belonged to the 60°C. Colour is accepted as one of the major quality characteristic of leaves and L^*, a^* and b^* values of leaf powders were determined. Temperature had an important effect on a* and b* values of samples ($P < 0.05$).

Keywords:
Olive leave, Drying, Mathematical modelling, Phenolich compound, Color

INTRODUCTION

The specific tree of Mediterrenean region and Asia is *Olea europaea* and known as its therapeutic effects dated from ancient times (Satish and Ansari 2013; Ben Salem et al. 2014). The products of this tree like extracts, teas, fruits and powder are attributed to biophenolic compounds such as secoiridoid oleuropein, tyrosol and hydroxytyrosol and some of these components inhibit microbial activity (Renis 1969; Hirschaman 1972). Several researches have indicated that especially olive leaf extract prevents muscle spasms, reduces blood pressure, stimulates blood flow (Bahloul et al. 2009) and demonstrates antidiabetic, hypolipidemic and anti-inflammatory activity (Ben Salem et al. 2014).

Infusion or decoction are main methods for preparation of olive leaves. Leaves are usually dried because of making easier of extraction procedure and lowering moisture content consequently. Drying is an oldest method for preservation of food stuffs and plays an important role in food processing (Ertekin

and Heybeli, 2013). Mathematical modelling of drying kinetics is also significant and required for process optimization and product quality (Karathanos and Belessiotis 1999).

The aims of this study are (i) to determine the temperature effect on drying kinetics of olive leaves, (ii) to fit experimental data to various semi-empirical mathematical models, (iii) to evaluate total phenolic contents of olive leaves.

MATERIAL AND METHOD

Material

Olive leaves which were used for conventional drying experiments were collected from Fakiusagi region (37° 2'44.47"N;36°13'25.40"E) in Osmaniye, Turkey and harvesting of olive leaves was conducted in May, 2016 at nearly temperature of 27 ºC.

Method

50, 60 and 70 ºC were selected temperatures for convective drying of leaves and drying was conducted at a constant air velocity (1.5 m/s) with a laboratory type dryer (Memmert, Germany). Firstly, olive leaves were milled using a commercial blender (Waring, Germany) into 4 mm diameter, then 6-7 grams of powdered olive leaf were put into a petri dish and dehydration was started. Weight of samples were collected in every 10 minutes for 50-60 ºC and every 5 minutes for 70 ºC manually using a 4-digit balance (Ohaus Pioneer, USA) until no weight changes were observed.

Mathematical modelling of drying curves

13 thin layer drying models were used for fitting experimental data and shown in Table 1. The calculation of moisture ratio (MR) was made with the help of Equation (1)

$$MR = \frac{M}{M_0} \qquad (1)$$

where M is the moisture content at time t (g water / g sample) and M_0 is the initial moisture content (g water / g sample). Equilibrium moisture content was negligible (Doymaz, 2004).

OriginPro 2016 (OriginLab, USA) software was used for regression analysis. Correlation coefficient (R^2), root mean square error (RMSE) (Equation 2) and reduced chi square (χ^2) (Equation 3) were important parameters to choose superior model. The highest R^2 and the lowest RMSE and χ^2 demonstrated the goodness of the fit.

$$RMSE = \left[\sum_{i=1}^{N} \left[\left(MR_{exp,i}\right) - MR_{pre,i}\right)_2\right]^{1/2} \qquad (2)$$

$$\chi^2 = \frac{\sum_{i=1}^{N}\left(MR_{exp,i} - MR_{pre,i}\right)^2}{N - y} \qquad (3)$$

where, N is the number of experimental data, y is the number of constant in model, $MR_{exp,i}$ is experimental moisture ratio at *i*, $MR_{pre,i}$ is the predicted moisture ratio at *i* (Doymaz, 2007).

Table 1: Semi-emphirical models for convective drying available in literature

Model Name	Equation	Reference
Lewis	MR = exp (-kt)	Bruce (1985)
Page	MR = exp ($-kt^n$)	Page (1949)
Modified Page	MR = exp $(-kt)^n$	White et al. (1981)
Henderson and Pabis	MR = a exp (-kt)	Henderson and Pabis (1961)
Logarithmic	MR = a exp (-kt) + c	Togrul and Pehlivan (2002)
Two-term	MR = a exp ($-k_0t$) + b exp ($-k_1t$)	Henderson (1974)
Midilli et al.	MR = a exp ($-kt^n$) + bt	Sharma and Prasad (2004)
Wang and Singh	MR = 1 + at + bt^2	Wang and Singh (1978)
Weibull	MR = exp $(-(t/b)^a)$	Corzo et al. (2008)
Parabolic	MR = a + bt + ct^2	Sharma and Prasad (2004)
Cubic	MR = a + bt + ct^2 + dt^3	Dalvand et al. (2012)
Sigmoid	MR = a + $\frac{b}{1 + e^{k(t-c)}}$	Figiel (2009)
Thompson	t = a ln(MR) + b [ln $(MR)^2$]	Thompson et al. (1968)

Effective moisture diffusivity and activation energy

Hashemi et al. (2009) and Bahloul et al. (2009) reported that the effective moisture diffusivity should be considered for optimization of dehydration process. Also, activation energy (Equation 4) could be defined as the amount of heating energy required for moving water from food (Aghbashlo et al. 2009) and stated an Arrhenius type equation.

$$D_{eff} = D_0 \exp(-E_a/RT) \quad (4)$$

where, D_{eff} is the effective moisture diffusivity (m^2/s), E_a is the activation energy (kJ/mol), R is the universal gas constant (8.3143 J/mol K), T is medium temperature (K) and D_0 is the exponential factor (m^2/s).

Total phenolic content (TPC)

Total phenolic contents (TPC) of dried leaves were confirmed according to the Folin-Ciocalteu (FC) method. 0.5 ml aliquot of the sample was put into a tube and 0.5 ml of FC reagent was added after adding 2 ml of Na_2CO_3 solution (200 g/L). The solution was mixed and the reaction proceeded for 15 min. at medium temperature. Then, 10 ml of distilled water was added and the formed precipitate was removed by centrifugation for 5 min at 4000 g. Finally, absorbance was measured at 725 nm (Huang and Prior, 2005).

Colour determination

In order to calculate total colour change (ΔE); L*, a*, b* values of leave powders were measured from top, center and bottom regions by Minolta Chroma meter CR 400 color meter and ΔE was calculated with the aid of equation (6). Unprocessed leave powders were accepted as reference.

$$\Delta E = \sqrt{\left[\left(L^* - L_{ref}^*\right)^2 + \left(a^* - a_{ref}^*\right)^2 + \left(b^* - b_{ref}^*\right)\right)^2} \quad (5)$$

Statistical analysis

The statistical differences of total phenolic content and colour were evaluated by Duncan test using SPSS software version 18.0 at 5 % confidence interval.

RESULTS AND DISCUSSION

Drying kinetics and mathematical modelling of drying curves

The initial and final moisture content of leaves were determined as 52.6 % and 6.0 % respectively according to method of AOAC, 1990. On dry basis, 1.109 kg water was found in leaves at the beginning. Drying temperature had a positive effect on drying kinetics. When temperature rose, drying rate increased, hence drying time reduced. Figure 1 demonstrates moisture ratio (MR) versus time during dehydration process. No constant rate period was observed in studied temperature range. Falling rate period was actively seen in whole process.

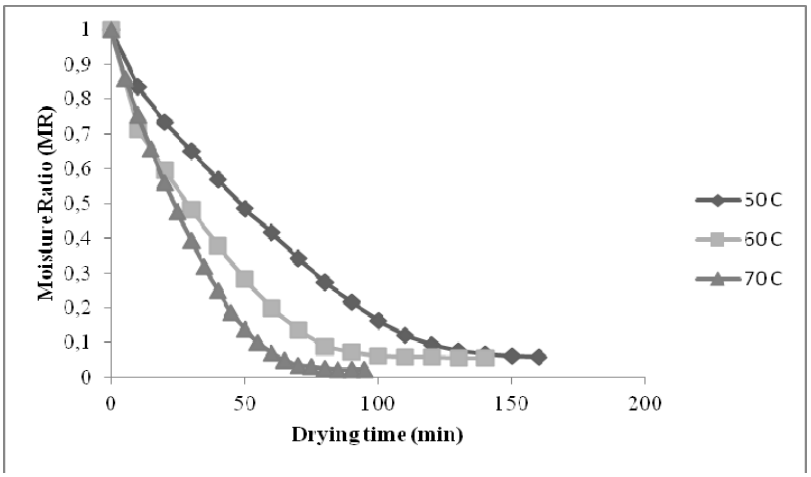

Figure 1
Moisture ratio values versus drying time at different temperatures

Experimental moisture ratio data provided a basis for mathematical modelling. Thirteen thin-layer drying models were analogised in terms of statistical results containing R^2, RMSE and reduced χ^2. In convective drying of olive leaves in temperature range of 50-70 ºC, R^2 was changed between 0.977 and 0.999; RMSE between 0.008 and 0.044; reduced χ^2 0.00007 and 0.00214 for applied all models. Parabolic and Cubic models were the best models describing dehydration of olive leaves and their statistical results and model constants were shown in Table 2.

The experimental moisture diffusivities of leave powders were found as directly proportional with temperature. 1.321×10^{-10}, 1.650×10^{-09} and 3.2×10^{-09} m^2/s were calculated moisture diffusivities for 50, 60, 70 ºC respectively. These results were

compatible with literature (Zang et al. 2010; Bahloul et al. 2009). Activation energy of samples was estimated as 147.736 kJ/mol and this value was higher than statements by Aghbashlo et al. (2008). They indicated that E_a values of agricultural products were in the intervals of 12 and 110 kJ/mol. This could be originated from powder form of leaves. Because powder forms have extra surface area when compared to other products.

Table 2: Parabolic and cubic model parameters of olive leaves

	Temperature (ºC)	***R^2***	***RMSE***	***χ^2***
Parabolic Model	50	0.998	0.01195	$1.734x10^{-4}$
	60	0.988	0.03102	0.0012
	70	0.999	0.00925	$1.006x10^{-4}$
Cubic Model	50	0.998	0.01185	$1.842x10^{-4}$
	60	0.995	0.01992	$5.410x10^{-4}$
	70	0.999	0.00758	$7.191x10^{-5}$

	Temperature (ºC)	***a***	***b***	***c***	***d***
Parabolic Model	50	0.975	0.012	$3.816x10^{-5}$	-
	60	0.921	0.016	$7.515x10^{-5}$	-
	70	0.987	0.024	$1.521x10^{-4}$	-
Cubic Model	50	0.972	0.012	$3.445x10^{-5}$	$1.545x10^{-8}$
	60	0.963	0.021	$1.560x10^{-4}$	$-3.848x10^{-7}$
	70	0.997	0.026	$1.927x10^{-4}$	$-2.849x10^{-7}$

Total phenolic content (TPC) and colour

2385.5 mg/kg gallic acid (GA) equivalent was the initial phenolic content of olive leaves. The maximum phenolic amount was observed in samples dried at 60 ºC (633.85 mg / kg GA equivalent). Phenolic levels were recorded as 303.61 mg / kg GA eqv. for 50 ºC and 447.05 mg / kg GA eqv. for 70 ºC. Duncan test results also showed that the temperature had an important effect on TPC ($P<0.05$). Similar results were found by Gamli et al. (2016) that the highest phenolics were found in olive leaves dried at 360 W rather than 90, 180, 600 and 900 W power levels.

L^*, a^*, b^* values of olive leaves were expressed in Table 3. Drying temperature had a significiant effect on only a^* and b^* levels ($P<0.05$). a^* and b^* levels were increased with increasing dehydration temperature. L value was not affected from temperature importantly ($P>0.05$). ΔE values were calculated as 7.989; 5.122; 10.014 for 50, 60, 70 ºC respectively.

Table 3: Colour parameters of dried olive leaves

Temperature (ºC)	***L****	***a****	***b****
50	48.35 ± 0.96^{a}	-4.40 ± 0.33^{a}	24.66 ± 0.56^{a}
60	46.31 ± 0.72^{a}	-3.70 ± 0.11^{b}	24.04 ± 0.51^{a}
70	48.16 ± 1.57^{a}	-2.07 ± 0.23^{c}	22.72 ± 0.20^{b}

Different superscript letters in the same column indicate significant difference at $P<0.05$.

CONCLUSION

In this research, convective drying kinetics of *Olea europaea* leaves were studied. Increasing of dehydration temperature had a shortened effect on drying time. Thirteen thin layer drying models were applied to drying data and parabolic and cubic models were chosen as the best. Diffusion coefficients were affected from temperature strongly and the highest moisture diffusivity pertained to 70 ºC. Activation energy of leaf powders was seemed to be higher according to researches available in literature. TPC values of samples showed the maximum level at 60 ºC. There were no significiant differences and correlation between L (brightness) of dried olive leaf powders ($P<0.05$).

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Rheological Parameters of Cereals

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Abstract. Rheology is a widely used tool in the qualification of cereals and cereal products. Maybe the most specific rheological analytical equipments were developed and used in the analysis of the dough made from flour and water. It is not a surprise as the dough is a very specific material. The moistened gluten proteins forms a three dimensional network in the dough what is a complex chemical system, and the chemical knowledge of this netwotk is hard to analyse and also hard to translate for the prediction of technological behaviour. On the other hand, the high starch content with the fiber content of the flour and the mechanical properties of the flour modify the usability of dough. The rheological analysis of this complex system is a rapid and practically useful way of qualification. The Farinograph, Alveograph, Extensigraph, Mixograph and Amylograph are specific complex equipments for the quality prediction and this paper will summarize the principles of these methods and their use in the qualification.

Keywords:

cereals,
rheology,
qualification,
wheat flour

MATERIALS AND METHODS

Rheologial methods in flour analysis

Specific rheological methods have a special emphasis in the cereal and especially in the wheat flour analysis. They can give special information about the technological suitability of flours, what depends on the starch and protein related functions of dough made from the flour. From the starch related parameters the activity of amylase enzymes and the gelatinization properties are the most important ones. Especially the high amylase activity, because it hinders the use in bakeries. The specific measurement method for the amylase activity is the Falling number. In this evaluation a flour-water suspension is stirred in hot water bath, then the rod falls down in the starch suspension. This falling slowed down due to the gelatinization of starch, but the amylases start to breakdown it. The higher amylase activity result faster starch degradation and faster decrease of viscosity, therefore the rod will be got down sooner.

The most important rheological tests of wheat flour is the ones which evaluates the behavior of dough made from water and flour. The most important tests for this are the Farinograph, Alveograph and Extensograph tests.

During the Farinograph test the dough in knead using two z-arm mixer arms. The machine continuously records the force what is required to maintain the constant deformation. As it can be seen, the resistance of dough against the kneading is

increases up to a specific point – this is the dough development time what is necessary to reach the optimal consistency for forming. The next phase is the stability when the strength of dough does not change. It can be determined by the length of the middle line of the curve measuring the time while it is horizontal, or measuring the time while the upper part of the curve is over the maximum consistency line. The next phase is the softening when the strength of dough decreases. In some countries the degree of softening after a predefined time is the last parameter. In Hungary the baking value is the most important parameter, what is calculated by the area between the maximum resistance line and the middle line of the curve. (D'Apollonia and Kunerth, 1990; Cornish et al., 2001).

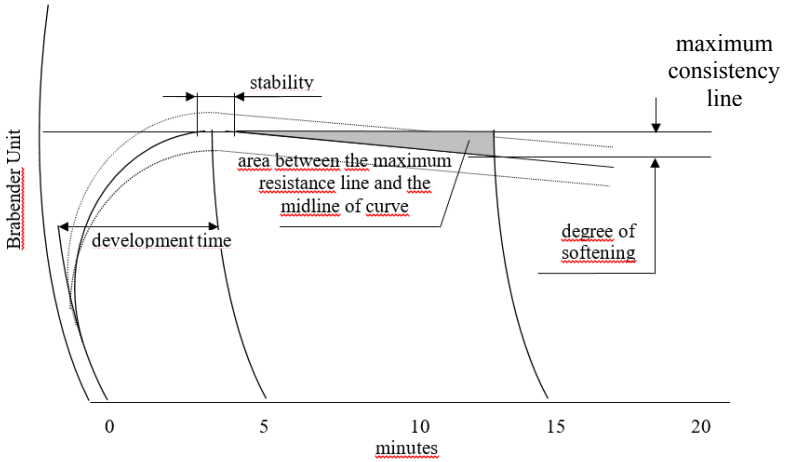

Figure 1
Representative Farinograph diagram

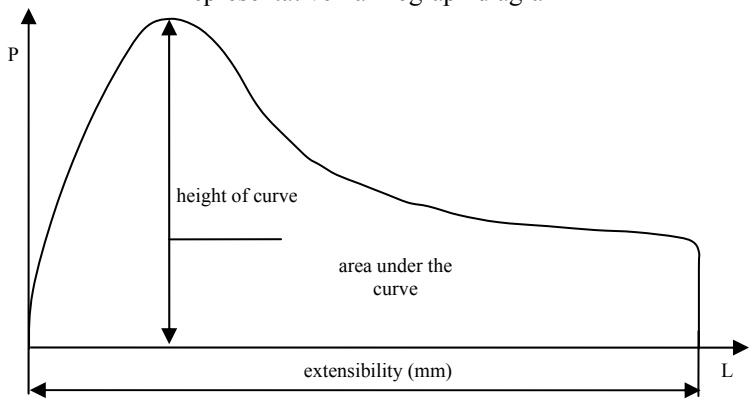

Figure 2
Representative Alveograph diagram

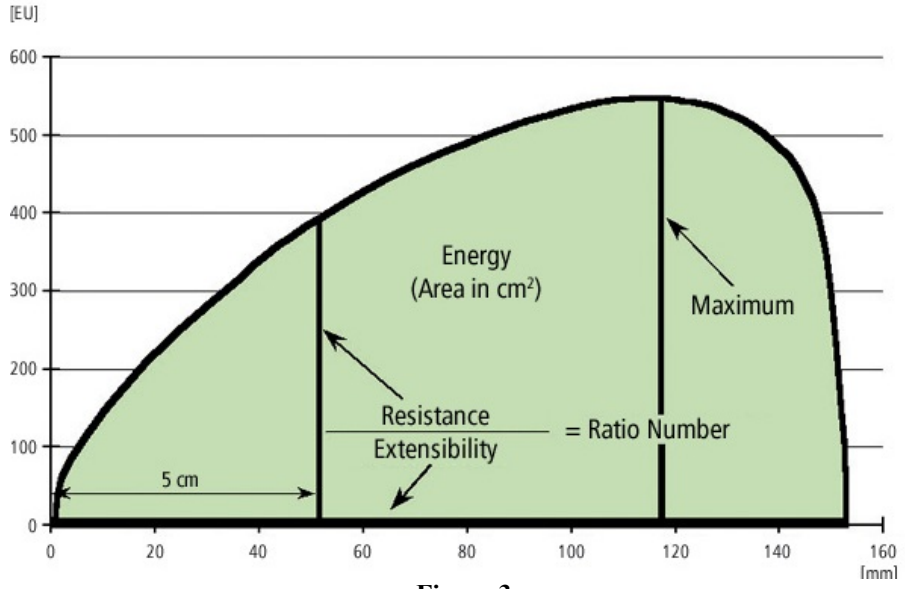

Figure 3
Representative Extensigraph diagram

The Alveograph test is the evaluation of dough made from salt solution and flour and formed into a disc. This disc is inflated until it gets torn and the equipment records the changes of pressure during the evaluation time (Figure 2). The higher pressure value (P value) is due to the stronger dough structure and the longer time to torn (L value) means more extensible dough. The quality parameter is the quotient of them (P/L value) and the deformation work (W value) what is the area under the curve from the beginning of inflation to the torn. (Rasper et al, 1986; Faridi and Rasper, 1987; Schöggl, 1998).

The Extensograph test is more or less similar to the Alveograph test; a dough formed into a rod and it is exposed to deformation until it stretches. The equipment records the resistance of dough against the pulling deformation and measure the resistance of dough (what is the maximum force measured during the pulling), the extensibility (what is the length of the curve) and the energy (the deformation work, determined by the measurement of the area under the curve) (Figure 3) (Müller and Hlynka, 1964).

Although these tests are different, there are some similarities in the properties. All of them evaluates the strength and the extensibility of the dough, therefore they characterize similar parameters. In this study we have evaluated the similarities and differences amongst them.

The evaluated samples

In the University of Debrecen the rheological parameters of winter wheat flours have been analysed for decades. In this study we have evaluated that how these parameters are changes under the same effects and is there any connection between the parameters of different methods. For this comparison we have

evaluated the effect of different flour additives (flour treatment agents and salts) on the Farinograph (MSZ ISO 5530-3:1995), Alveograph (AACC 54-30.02) and Extensograph (AACC 54-10.01) parameters and we made statistical analysis (correlation and regression analysis) to reveal the connections amongst the parameters and to try to estimate the different parameters from each others.

RESULTS AND DISCUSSION

The different flour additives significantly influenced the theological properties of the evaluated fine wheat flour (BL55 type), but their effects were different on the behaviour of the dough. The control flour had the highest Alveographic W value, relatively high Farinograph baking value and the second highest Extensographic energy value. However, when the flour was completed by 10% artichoke flour, the baking value showed a very slight decrease in the baking value (about 2%), while the Extensigraph energy value increased by almost 50% while the W value decreased to the third of the data of control flour. Other flour additives had similar effects on the different rheological parameters, for example one developed for biscuit making resulted almost the lowest baking value and W value and the dough was so soft that the Extensograph parameters were not evaluable. A classic Hungarian flour improver resulted the lowest baking value, but the Alveograph and Extensigraph readings proved that the dough became stronger by its use.

In the second experiment the effect of sodium chloride was evaluated on the rheological parameters. The sodium addition influences the water absorption capacity, increases the development time and stability while making the dough more strength by the slight rise of pH, which decreases the number of positive ions and helps the formation of cross-bindings (Preston, 1981; Danno and Hoseney, 1982). These references were proved in several cases as the addition of sodium chloride in increasing ratios increased the baking value, W value and Extensograph energy value of winter wheat flours, but it has no effect on the water absorption capacity and the Extensofraph extensibility. The increasing sodium chloride addition did not influenced the bread crumb hardness measured by texture analyser measured a day after baking, but its effect could be seen 3 days later when significantly lower hardness values were measured in the breads containing more salt.

When correlation analysis was performed on the results it was not a surprise that the connection between the baking value and the Extensograph energy value was moderate only (r=0,40), but it was much more stronger with the W value (r=0,71). Similarly there is only a moderate strong connection between the Extensograph energy and W value (r=0,43). It is much more interesting that the correlation coefficient is only 0,2 between the L value and the Extensograph extensibility and 2,21 between the P value and the Extensograph resistance value. Based on these connection it is also not a surprise that the stepwise regression analysis did not resulted good estimations for one rheological parameter based on the readings of the other quality data.

CONCLUSIONS

Rheological methods used in the cereal qualification are suitable for the prediction

of technological quality, but the different methods based on similar principle result different result. All the methods are suitable for the differentiation amongst the different samples, but their results are not comparable to each others. The prediction of rheological data using other quality parameters do not result reliable result.

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Application of Fractional Calculus in Food Rheology

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Abstract. In fractional calculus the order (β) of differentiation or integration is not an integer number, generally β is a fractional number between 0 and 1. The root of fractional calculus goes back to the 18th century, and this calculus is intensively developing nowadays, too. Application of fractional calculus can be found in rheology, in electrical impedance spectroscopy, in physiological description. It is interesting, that the description of viscoelastic properties of biological material is much more accurate with fractional calculus than with ordinary differentiation and integration. In this work the creeping and recovery curves of a simple sweet, - gum candy –and bread slice, were approached with ordinary and fractional calculus. The rheological parameters of gum candy were determined. The fractional calculus gave better fit on the measured creep recovery curve points, than classical rheology models containing discrete elastic and viscous elements.

Keywords:

fractional calculus, viscoelastic, food, creep, recovery

INTRODUCTION

In many cases the experimentally observed relaxation function exhibit a stretched (Kohlrausch) exponential decay $F(t) = F_0 \exp(-(t/\tau)^{\alpha})$, where F denotes the relaxing physical quantity (for example light intensity, stress relaxation in viscoelastic material, dielectric relaxation, etc.), t is the time, τ is a constant and $0 < \alpha < 1$ is a number (Schiessel et al., 1995). An appropriate tool to describe these relaxation processes is the fractional calculus (Süli, 2012).

The so called “Fractional Calculus” was born more than 300 years ago. In a letter dated September 30th, 1695 L'Hopital wrote to Leibniz asking him about a particular notation he had used in his publications for the nth-derivative of the linear function $f(x) = x$, $\frac{D^n x}{Dx^n}$ L'Hopital's posed the question to Leibniz, what would the result be if $n = 1/2$. Leibniz's response: "An apparent paradox, from which one day useful consequences will be drawn."

Within the 20th century especially numerous applications and physical manifestations of fractional calculus have been found (Mainardi and Spada, 2011; Schiessel et al., 1995). While the physical meaning is difficult to grasp, the definitions themselves are no more rigorous than those of their integer order counterparts.

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Schiessel et al., 1995). While the physical meaning is difficult to grasp, the definitions themselves are no more rigorous than those of their integer order counterparts.

α order fractional integral according to Riemann-Liouville for a *f(t)* real or complex function can be given by the formula

$$(J^{\alpha}f)(x) = \frac{1}{\Gamma(\alpha)}\int_0^x (x-t)^{\alpha-1} f(t)dt$$

where *t* is real variable, x>0, α>0 is a real number and $\Gamma(\alpha) = \int_0^{\infty} e^{-y} y^{\alpha-1} dy$ is the Gamma-function. There are several definitions for α order fractional derivative, practically as many as many mathematicians dealt with fractional calculus. A definition in which the Riemann-Liouville fractional integral is used can be given by the next expression:

$$(D^{\alpha})f(t) := \begin{cases} \dfrac{d^m}{dt^m}\left[\dfrac{1}{\Gamma(m-\alpha)}\displaystyle\int_0^t \frac{f(\tau)d\tau}{(t-\tau)^{\alpha+1-m}} d(\tau)\right] \\ \dfrac{d^m}{dt^m} f(t) \end{cases} \begin{matrix} , \; m-1 < \alpha < m \\ , \; \alpha = m \end{matrix}$$

where m-1< α <m and m is an integer number, α is a real number (Loverro, 2004).

In this work a stretched exponential function was fitted on creep and recovery curves of various food materials.

MATERIALS AND METHODS

The investigated materials: bread and gum candy were purchased in the local market. The creep-recovery test (CRT) curves were measured with a texture analyser TA-XT2 from Stable Micro System (Godalming, United Kingdom). The bread slices were pressed with a plexi cylinder of 36 mm diameter, and gum candies were pressed a metal cylinder of 75 mm diameter. The CRT test consists of four segments. The first segment is loading the sample with constant speed of measuring head until a pre-set force is reached. In the second segment the deformation is creeping under the constant force during a pre-set period. In the third segment (unloading) the probe is raised until the force on head becomes zero. In the fourth segment - in recovery - the relaxation of sample continues so, that the measuring head is raised when the relaxed sample reaches the probe. In our measurements the pre-set time was 60 s for both creeping and recovery period. The force in creeping period was 5 N and 2,5 N for gum candy and bread, respectively.

The creeping and recovery part of CRT are suitable for the determination of rheological parameters of sample material with model functions. The four-element Burgers model (Fig. 1) can describe both the creeping and recovery processes

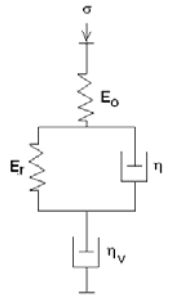

Figure 1.
The Burgers model. E_0 and E_r, represents the elastic modulus of the two spring elements and the η_r and η represents the viscosity of the two dashpots

The differential equation of four-element Burgers model (Sitkei, 1981) for σ stress and for ε strain in normal calculus

$$\frac{d^2\varepsilon}{dt^2}+\frac{1}{T_r}\frac{d\varepsilon}{dt}=\frac{1}{E_o}\left[\frac{d^2\sigma}{dt^2}+\left(\frac{E_o}{E_rT_r}+\frac{E_o}{\eta}+\frac{1}{T_r}\right)\frac{d\sigma}{dt}+\frac{E_o}{T_r\eta_v}\sigma\right]$$

and the solution of it is for creeping strain $\varepsilon(t)$ in time, t, when the stress is constant $\sigma=\sigma_0=\text{const}$:

$$\varepsilon(t)=\frac{\sigma_0}{E_0}+\frac{\sigma_0}{E_r}\left(1-e^{-\frac{t}{T_r}}\right)+\frac{\sigma_0}{\eta_v}t, \qquad (1)$$

where $T_r=\frac{\eta}{E_r}$, the retardation time and the solution of it for recovery strain $\varepsilon(t)$ in time, t, after t_1 time (time elapsed to beginning of recovery) when the stress becomes zero ($\sigma=0$):

$$\varepsilon(t)=\frac{\sigma_0}{E_r}\left(1-e^{-\frac{t_1}{T_r}}\right)e^{-\frac{t}{T_r}}+\frac{\sigma_0}{\eta_v}t_1 \qquad (2)$$

The differential equation of Burgers model in fractional calculus:

$$\frac{d^{\nu+1}\varepsilon}{dt^2}+\frac{1}{T_r}\frac{d^{\nu}\varepsilon}{dt}=\frac{1}{E_o}\left[\frac{d^{\nu+1}\sigma}{dt^2}+\left(\frac{E_o}{E_rT_r}+\frac{E_o}{\eta}+\frac{1}{T_r}\right)\frac{d^{\nu}\sigma}{dt}+\frac{E_o}{T_r\eta_v}\sigma\right]$$

and the solution is for creeping and recovery parts:

$$\varepsilon(t)=\frac{\sigma_0}{E_0}+\frac{\sigma_0}{E_r}\left(1-e^{-(\frac{t}{T_r})^{\beta}}\right)+\frac{\sigma_0}{\eta_v}t \qquad (3)$$

and

$$\varepsilon(t)=\frac{\sigma_0}{E_r}\left(1-e^{-(\frac{t_1}{T_r})^{\beta}}\right)e^{-(\frac{t}{T_r})^{\beta}}+\frac{\sigma_0}{\eta_v}t_1 \qquad (4)$$

respectively, where $0<\nu<1$ and $0<\beta<1$

The (1) and (3) expressions were fitted on creeping curves and the (2) and (4) expressions on the recovery curves. The elastic modulus, viscosities and β parameter were determined.

RESULTS AND DISCUSSION

Typical creep-recovery curve of bread can be seen on Fig. 2. Similar CRT curve was measured on gum candies, too. The relative quick load part is followed by much longer creeping part. The creeping part of all CRT curves was approached with Burgers model of normal and fractional calculus, with equation (1) and (3). After the creeping part there is a relative quick unload segment which is followed by recovery curve. The recovery part of CRT curves was fitted by Burgers model both with normal and fractional calculus, with expressions (2) and (4).

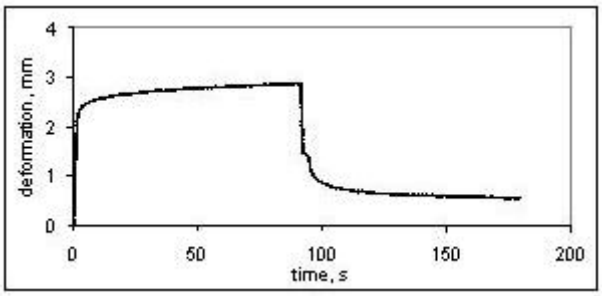

Figure 2.
A typical creep-recovery curve of a bread slice.

The result of curve fitting is demonstrated on Fig. 3. The stretched exponential function gave better approach of measured points especially in the beginning of both creeping and recovery part. The value of parameters from creeping period is very similar to the values from recovery period (Table 1.) for both investigated objects. Generally parameter values are lower from approaching the recovery part according to parameter values from creeping part.

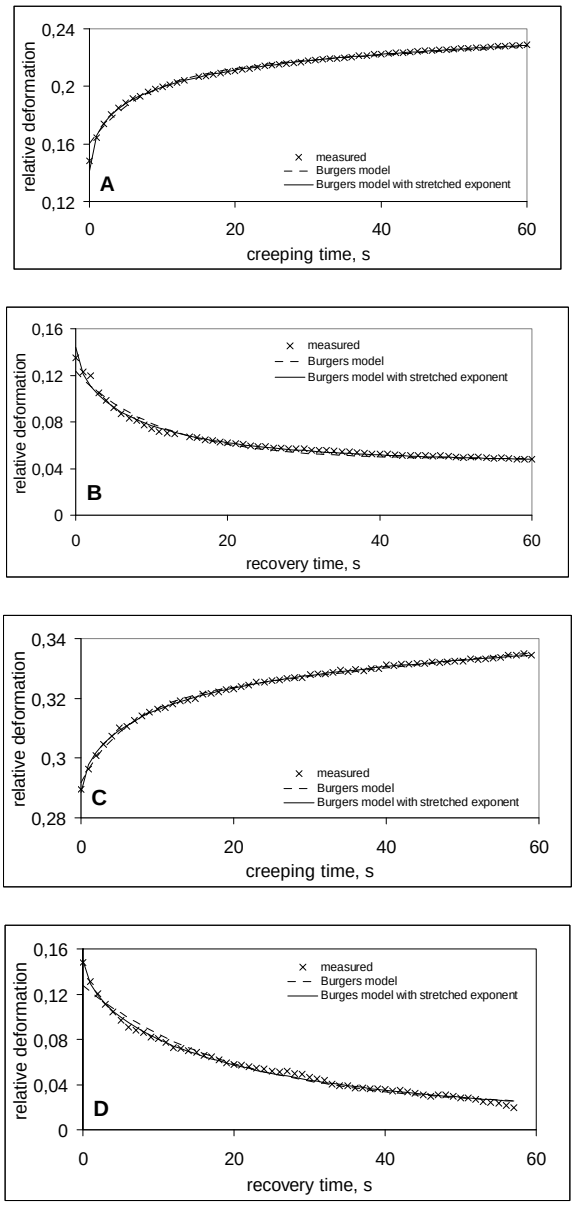

Figure 3
The fitting of creeping and recovery parts of CRT curve of bread (A,B)
and of gum candy (C,D)

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Table 1: The parameters of Burgers model from approaching the measured curves

Material	Calculus	part	E_0, kPa	E_r, kPa	η, MPas
bread	normal	creeping	16,2±0,8	52,2±3,2	0,39±0,02
		recovery		34,6±2,4	0,37±0,04
	fractional	creeping	18,4±1,0	31,8±3,5	0,25±0,06
		recovery		26,0±2,2	0,24±0,04
gum candy	normal	creeping	47,8±2,6	502,7±13,2	3,12±0,12
		recovery		127,2±8,2	2,57±0,19
	fractional	creeping	48,3±1,1	332,7±18,2	3,32±0,23
		recovery		88,2±7,5	1,72±0,10

Material	Calculus	part	η_V, MPas	β
bread	normal	creeping	8,73±0,72	
		recovery	5,11±0,64	
	fractional	creeping	16,39±0,98	0,521
		recovery	5,45±0,57	0,628
gum candy	normal	creeping	52,73±8,57	
		recovery	60,31±10,24	
	fractional	creeping	133,63±35,97	0,612
		recovery	258,73±29,76	0,625

It can be explained by the fact, that in the recovery period the stress is already zero, but in creeping period is about 2-3 kPa. Our earlier investigation showed, that both elastic moduli and viscosities of Burgers model for gum candy linearly increased, if the stress on the sample increased (Csima, 2015). This increase can be caused by structure changes under stress.

The lower parameter values for bread according to parameter values for gum candy can be explained with the lower elasticity and hardness of bread compared to gum candy.

CONCLUSIONS

The approach of creep and recovery part of CRT curves was proved more precise with fractional calculus than with normal calculus. It seems that the stretched exponential function better describes especially the quick processes of both creeping and recovery curves.

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INCREASING THE NUTRITIONAL VALUE OF SAHLEP

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Abstract. Innovations on technology and changing consumer expectations forced the development of dairy industry for producing novel products possessing different properties. In recent years, nutritionally enriched functional products have been prepared by the addition of highly valued nutritional compounds to milk. Sahlep, is a milk beverage, not only used as a drinking purpose to keep body the warm but also used for having the benefits of nutritional constituents and alleviating various diseases in Turkey for a long time. Whey proteins, contain, amino acids and various nutritionally high components. In addition, antioxidant, anticarcinogenic and hypocholesterotemic effects are the known some bioactivities. In this study, sahlep milk beverage was supplemented with whey protein concentrate (WPC 35) (1%, 2% and 3%). The pH, viscosity and sensory characteristics were analyzed. When sahlep milk beverage was supplemented with WPC 35 showed no effect on the pH value; however, viscosity was increased. According to sensory evaluations, supplemented with WPC 35 affected on colour, structure, taste and smell properties of sahlep milk beverage. The panelists gave the highest score to sahlep milk beverage supplemented with WPC 35 (3%), which is close to the control. It could be concluded that sahlep supplemented with WPC 35 (3%) could be prepared and suggested as a novel and nutritionally high product.

Keywords:

Sahlep,
milk beverage,
whey protein concentrate,
nutrition,
sensory,
viscosity

INTRODUCTION

To prepare the sahlep beverage, firstly, milk is mixed with sugar and sahlep powder, then it is boiled, and finally some cinnamon powder is strewed on top of it and it is drunk as hot; it is a traditional, thickened milk drink (Tamer et al., 2006).

Sahlep is the name given to the tubers of the genera of *Orchis, Ophyris, Serapias, Platanthera,* and *Dactylorhiza*. The plant has Western Asia origins, a tuberous and herbaceous structure, flat and long leaves, and white, pink, red, lilac or purple flowers. It has been found that *Orchidacea* family, which includes the sahlep plants, has approximately 24 genera and 90 types (Tamer et al., 2012).

The word sahlep has been taken from the Arabic language which means fox. Sahlep is a drug that has been recorded in the medical books since the time of Dioscorides. Dioscorides, in his work "Materia Medica", gave information on the colours, leaves and flowers of orchids.

Avicenna, in his work "Kanun", provided vast information on sahlep (Öğretmen et al. 2012).

Sahlep plant grows in the geographical mid-zone of the world, and it provides healing effects for the people. Sahlep grows in almost every place in our country and its tubers are gathered and sold in some regions. When the climate conditions are available in Turkey, 8-10 tons of sahlep is produced per year. In addition, 10 tons of sahlep comes to Turkey through border trade from Iran, Iraq, Azerbaijan, Georgia, Syria and Macedonia (Güzel, 2012).

When the tubers are unearthed, they are washed by rinsing with plenty of water. The cleaned tubers are boiled in water, ayran (drink made of yoghurt and water) and seldom in milk for 15-30 minutes. The reason for boiling is to stop the development of the tubers. Moreover, the process of boiling helps create the unique taste and scent of sahlep. The boiled tubers are dried directly under the sun. The dried tubers are hardened and grinded in mills to obtain the fine powder. The powdered sahlep is generally used in making the sahlep beverage and Kahramanmaraş ice-cream (Sezik, 2012).

The functional properties of sahlep depend on its type and chemical composition, particularly its glucomannan content (Yaşar, 2010). Glucomannan, which is found in sahlep, has very good stabilizer characteristics, and it gives the proper, hard, flexible homogenous structure and stability to ice-cream (Tekinşen, 2010). As the glucomannan amount in the sahlep powder increases, the stability and aroma of the prepared sahlep beverage increases as well.

Tekinşen and Güler (2010), stated that the composition of the sahlep powder, which is produced from ten *Orchidaceae* species growing in Turkey, varied between 9.35-12.40% for moisture, 3.11-4.95% for protein, 0.95-2.83% for ash, 17.7-54.6% for glucomannan, and 5.44-38.7 for starch. Farhoosh and Riazi (2007) informed that the composition of two Iran sahlep powders varied between 12.85-13.56% for moisture, 3.09-7.35% for protein, 2.10-3.84% for ash, 22.13-58.22% for glucomannan and 1.67-6.15 for starch.

It has been stated that sahlep keeps the body warm in cold days, increases resistance against common cold and prevents diarrhea. The most important component of sahlep is glucomannan (Tamer et al. 2012). Glucomannan is the name of the fibers that dissolve in neutral water and it is known to have many beneficial health effects (Yasar et al., 2009). Glucomannan has a polysaccharide structure, and it is a hemicellulose which consists of linear β-D-mannose and α-D-glucose monomers that are linked with β-1-4 links. Glucomannan is a stabilizer with a high water holding capacity. It can also be used for the treatment of chronic constipation (Tamer et al. 2012). Glucomannan contributes to the normalization of natural blood glucose, and lessening stress in pancreas, moreover, it has a preventive effect on blood sugar anomalies such as hypoglycemia. In addition, it plays a role in preventing the chronic diseases and controlling weight (Farhoosh and Riazi, 2007).

Whey is the name of the liquid remaining after milk has been curdled and casein is separated while producing cheese. Whey is an important by-product of the milk industry that makes cheese or casein. This by-product constitutes almost 85-90% of milk volume, and approximately 55% of milk nutrition remains in this by-product (Brandelli et al., 2015).

Whey can be turned into food additives through simple drying or by removing mineral elements, lipids and lactose. Whey products such as whey protein concentrates (WPC) and whey protein isolates (WPI) are developed by using different techniques such as ultrafiltration, microfiltration, reverse osmosis and ion exchange (Segat et al. 2014).

Whey protein products are considered as perfect food additives because of their unique functional properties. Whey proteins are added to foods not only because of their functional properties, but also because of their high nutritional value and GRAS status (El Salam et al., 2009).

Whey is a source of important proteins, and it is used as an agent increasing viscosity, forming gel, stabilizing emulsions or foams and hydrating in the production of many complex foods. In fact, since whey proteins include a high amount of β- Lactoglobulin and α-Lactalbumin and have other important minor compounds (protease peptone fraction), they are also responsible for hydration, gelation, emulsifying and foaming properties (Segat et al., 2014).

Whey contains only 5 basic proteins; β –Lactoglobulin, α-Lactalbumin, glycol-macropeptide, immunoglobulins and serum albumin (Krissansen, 2007).Whey includes many components that are beneficial for health, which are essential amino acids, bioactive peptides, antioxidants and immune-globulins. Among the important roles played by whey proteins are radical scavenging, anti-inflammatory, antitumor, immune-stimulatory, hypotensive, gut homeostasis, anti-obesity, anti-diabetic, muscle bio-synthesis, osteo-protective and radio-protective functions (Patel, 2015).

The purpose of this study is to study the impact of whey protein added to sahlep on the rheological and sensory properties of sahlep.

MATERIALS AND METHODS

Raw Material and Excipients

Milk: The milk obtained from morning milking in Osmaniye private milk enterprise was taken and then homogenized and used.
Whey protein concentrate (WPC 35): It was obtained from Enka Milk (Konya, Turkey) company.
Starch: Corn starch was used (Dr.Oetker, İzmir, Turkey).
Sahlep: Sahlep was supplied from a private company in Kahramanmaraş (Turkey).
Sugar: The sugar was supplied from Torku Company (Konya, Turkey).
Ginger: The ginger was bought from Bağdat Baharat (Kahramanmaraş, Turkey).
Cinnamon: The cinnamon was bought from Bağdat Baharat (Kahramanmaraş, Turkey).

Method

Sahlep Production

The controls of the raw milk were performed; its fat rate was adjusted to 3.5%, and it was homogenized. Whey protein concentrate (WPC 35)**,** sahlep, sugar, starch, cinnamon, and ginger were added in the amounts indicated in Table 1, and the mixture was mixed to become homogenous by using an homogenizer (Ultra Turrax, Janke & Kuntel KG, IKA, Werk, Germany). Double-walled steam boilers were used to heat the mixture at 85˚C for 10 minutes, and then it was cooled down to 25˚C. The manufactured

sahlep samples were stored at +5˚C during the analyses. Sahlep production is illustrated in Diagram 1.

Sahlep Analyses

pH Analysis: Orion Star™ A 211 pH Benchtop Meter digital pH meter (Thermo Scientific, Waltham, MA) was used for measuring this.

Viscosity Analysis: The viscosity of the samples was measured by using the Selecta STS-2011 Viscometer (Barcelona, Spain) device at 40ºC.

Sensory Analysis: In order to make a comparative assessment of the sahleps with the sensory method, a panelist group of 13 was formed with people from the Osmaniye Korkut Ata University,,Food Engineering Department, and the panelists were trained. Panelists gave Hedonic-type scores (1-9 points) independently from each other and in a comparative manner (Meilgaard et al., 1999).

Statistical Analysis: An analysis of variance was made by using the SPSS 18.0 package program for the physical, chemical and microbiologic data that were gathered. Duncan multiple comparison test was used to identify the differences. (Düzgüneş et al., 1987).

Table 1: The ratios of additives used in sahlep production

Samples	Milk (mL)	Whey protein concentrate (WPC 35) (g)	Sahlep (g)	Sugar (g)	Starch (g)	Ginger (g)	Cinnamon (g)
Control	1000	-	8	80	2	1	1
%1	1000	10	8	80	2	1	1
%2	1000	20	8	80	2	1	1
%3	1000	30	8	80	2	1	1

Table 2: pH and viscosity values of sahlep samples

	Control	Sample 1 (%1)	Sample 2 (%2)	Sample 3 (%3)
pH	$6.73^{a} \pm 1.01$	$6.75^{a} \pm 0.05$	$6.73^{a} \pm 0.17$	$6.70^{a} \pm 0.14$
Viscosity	$105.50^{c} \pm 9.19$	$123.50^{b} \pm 2.12$	$137.65^{b} \pm 3.53$	$165.72^{a} \pm 7.07$

The difference between the values indicated in the same line with different letters is statistically significant ($p<0.05$).

Milk
↓
Control (degree of acidity, pH, fat, antibiotic)
↓
Milk fat adjustment and homogenization
↓
Adding the additives (whey protein concentrate (WPC 35)**,** sahlep, sugar, starch, cinnamon, ginger)
↓
Mixing (until the additives become homogenous)
↓
Heat Treatment (at 85˚C for 10 minutes)
↓
Cooling (25˚C)
↓
Storage (+5˚C)

Diagram 1
Flow Chart for Sahlep Production

Table 3: Sensory analysis results of sahlep samples

Quality Attribute	Control	Sample 1 (1%)	Sample 2 (2%)	Sample 3 (3%)
Color	$8.38^{a} \pm 0.80$	$6.92^{b} \pm 1.35$	$7.35^{b} \pm 0.94$	$8.04^{a} \pm 0.72$
Structure	$7.88^{a} \pm 0.76$	$6.19^{c} \pm 0.89$	$6.73^{b} \pm 0.92$	$7.77^{a} \pm 0.95$
Scent	$7.92^{a} \pm 0.80$	$6.27^{c} \pm 1.04$	$6.85^{b} \pm 0.73$	$7.73^{a} \pm 1.0$
Taste	$8.15^{a} \pm 0.67$	$6.19^{b} \pm 0.90$	$6.65^{b} \pm 0.89$	$7.73^{a} \pm 0.92$
Overall	$8.19^{a} \pm 0.75$	$6.15^{c} \pm 1.12$	$6.85^{b} \pm 1.05$	$8.15^{a} \pm 0.83$

The difference between the values indicated in the same line with different letters is statistically significant ($p<0.05$).

RESULTS AND DISCUSSION

Table 2 presents the pH and viscosity results of sahlep samples. In terms of pH, there was no statistically significant difference among samples ($p>0.05$). However, as the whey amount increased, the viscosity values of samples increased as well, and this increase was found to be statistically significant ($p<0.05$).

Table 3 presents the sensory analysis results of sahlep samples. As the figure indicates, there was significant difference among samples in terms of colour ($p<0.05$). The control sample and the sample with 3% additive received the highest scores, and the difference between them was insignificant ($p>0.05$). The difference among samples in terms of structure was found to be significant ($p<0.05$). The control sample and the 3rd sample with 3% additive received the highest structure values, and the difference between them was statistically

insignificant ($p>0.05$). Likewise, the control sample and the 3rd sample with 3% additive received the highest score in terms of scent, and the difference between them was also insignificant ($p>0.05$). The lowest value belonged to the 1st sample with 1% additives. The difference among samples in terms of taste was found to be significant ($p<0.05$). The difference between the control sample and the 3rd sample with 3% additive was insignificant ($p>0.05$) and those samples received the highest scores in terms of taste. According to the overall evaluation criteria, the highest scores were received by the control sample and the 3rd sample with 3% additive, and the difference between these samples was insignificant ($p>0.05$). The lowest value belonged to the sample with 1% additive.

CONCLUSION

According to the result of the analysis, the viscosity level increased as the amount of whey proteins increased. According to the evaluation criteria of colour, scent, structure, taste and general acceptability, the control sample and the 3rd sample with 3% additives received the highest scores. The difference between the scores of the control sample and the 3rd sample was statistically insignificant ($p>0.05$), and the sample that was the closest to control sample was the 3rd sample. Adding 3% whey protein concentrate (WPC 35) can be recommended to increase the nutritional value of the sahlep drink and improve its biological property.

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TEACHING OF PHYSICS IN SCHOOLS WITH HELP OF EXPERIMENTS OF FOOD INVESTIGATION

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Keywords:

teaching of physics
investigated food materials,
boiling point
galvanic battery
fresh egg and boiled (cooked) egg

Abstract. Teaching of physics (and other branches of natural science, like chemistry, biology) in the schools the experiments are of primary importance (Sprott, 2015).The pupils are satisfied if the investigated materials are food samples, wellknown for them. The paper deals with 3 simple experiments of physical type. The following foodstuffs were investigated: sugar, salt, apple, potato, lemon, egg. For teaching the following experiments were prepared and carried out (Szabo et al., 2015), (Bozi et al., 2016), (Szabo, Izsak, 2016):

boiling point measurement in case of sugar and salt (NaCl) solutions for determination of the concentrations

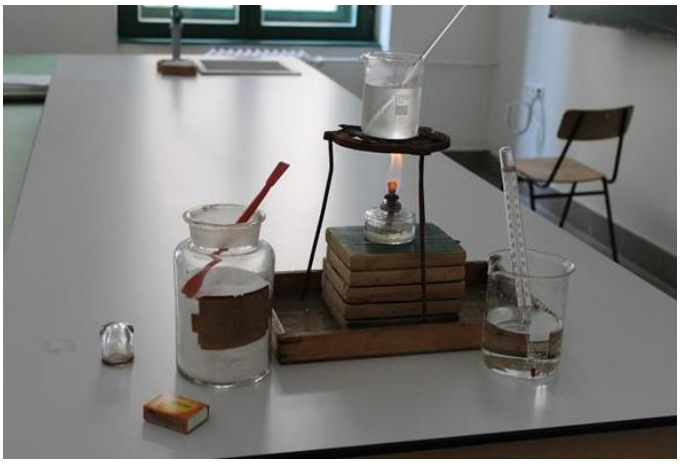
creation of galvanic battery using agricultural products, like lemon, potato or apple

differentiation between raw (crude) fresh egg and boiled (cooked) egg without cracking (breaking) the egg shell

BOILING POINT MEASUREMENT IN CASE OF SUGAR AND SALT SOLUTIONS

The practical aim of such type measurements can be the determination of the concentration. These experiments show not only the energy-demand (boiling is an endotherm process) of state changes, but the fact that the boiling point is a function of concentration of liquids, as well. If the concentration of salt (NaCl) and sugar ($C_{12}H_{22}O_{11}$) is high enough (over 10 %) , it is possible even with simple ordinary stick thermometer to show the increase of boiling point of solutions in the a volumetric flask in comparison with tap water used for the preparation of the solutions (Picture 1).

Table 1 shows the solubility of sucrose and NaCl as a function of temperature, and table 2 informs about some sugar-type carbohydrates.



Picture 1
Measuring set for boiling point determination

Table 1: Solubility of NaCl and sucrose

Substance	**Solubility g/100 g solvent**		
	15 °C	20 °C	25 °C
NaCl	35,9	36,0	36,2
sucrose	66,3	67,1	67,9

Table 2: Some sugar-type carbohydrates (mono- and disaccharides)

name	**composition**	**found in**
glucose	$C_6H_{12}O_6$	grape
fructose	$C_6H_{12}O_6$	fruits
sucrose	$C_{12}H_{22}1O_{11}$	sugar beet
lactose	$C_{12}H_{22}1O_{11}$	milk
maltose	$C_{12}H_{22}1O_{11}$	beer

(chemically cane sugar, beet sugar, palm sugar are identical)

The boiling point of the solvent above a solution will be greater than the boiling point of the pure solvent whether the solution contains a non-volatile solute or a volatile solute. However, for simplicity, only non-volatile solutes will be considered here.

Experimentally, we know that the change in boiling point of the solvent above a solution from that of the pure solvent is directly proportional to the molal concentration of the solute:

$$\Delta T = K_b m$$

where:

ΔT is the change in boiling point of the solvent,

K_b is the **molal boiling point elevation constant**, and

m is the molal concentration of the solute in the solution.

Note that the molal boiling point elevation constant, K_b, has a specific value depending on the identity of the solvent.

The graph (Fig. 1) shows the normal boiling point for water (solvent) as a function of molality in several solutions containing sucrose (a non-volatile solute). Note that the normal boiling point of water solution increases as the concentration of sucrose increases.

It is appropriate to point out the following:

1. from point of view of concentration we should differentiate unsaturated, saturated and oversaturated solutions
2. the rate of dissolution of different materials is different, therefore there is a difference between the concentration of the saturated solutions of salt and sugar substances
3. the temperature of boiling depends on the air pressure, so when the experiment is carried out on altitudes, significantly higher (e.g. the top of the Matra, appr. 1000 m above sea level) the measured boiling point would be definitely lower.
4. the concentrations and the boiling points are well correlated, so using calibration curve (or table data) based on the boiling point measurements the degree of concentration can be calculated
5. the concentration of the solution affects not only the boiling point, but affects the freezing point, as well (the sea water does not freeze at 0 ° C), however the freezing point is not a function of air pressure
6. for determination of concentrations not only the boiling point increase, but also the freezing point decrease is suitable, so using this technique you can detect for example the milk adulteration (application of precision thermometer is necessary)

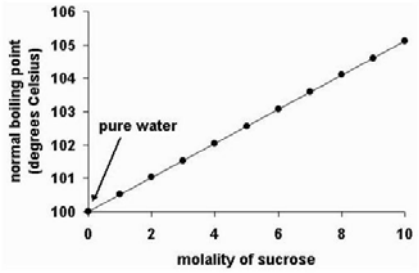
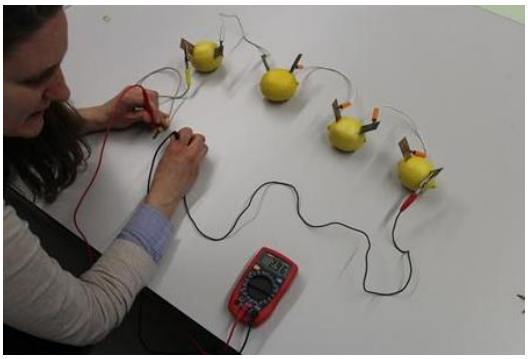


Figure 1
Boiling point as a function of molality



Picture 2
Galvanic battery with lemon

Table 3: Electrode potentials (V)

metals	**potentials in Volt**
Mg/Mg^{2+}	- 2.38
Al/Al^{3+}	- 1.70
Zn/Zn^{2+}	- 0.76
Fe/Fe^{2+}	- 0.44
Pb/Pb^{2+}	- 0.12
Cu/Cu^{2+}	+0.34
Ag/Ag^{+}	+0.80
Hg/Hg^{2+}	+0.85
Au/Au^{+}	+1.50

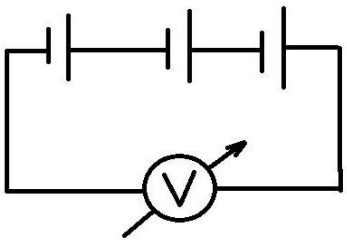


Figure 2
Cascading several galvanic units

CREATION OF GALVANIC BATTERY USING AGRICULTURAL PRODUCTS

Thanks to italian natural scientists Luigi Galvani (1737-1798) and Alessandro Volta (1745-1827) the so-called galvanic battery is known already more than 200 years, which has become known as an electric power source. The essence of the principle was the recognition that if two different metals are put in alkaline or acidic or salty solution, containing ions, then a voltage difference is created between them. How large is the voltage difference this can be calculated, based on the electrode potentials, shown as the Volta's voltage line. Voltage is about the order of 1 V. The electrode potentials You can see in table 3.

The creation of the test equipment - that is, the galvanic battery - is very simple, we insert into a lemon (or apple or potato) on the opposite sides a copper and zinc plate (the 2 electrodes) and close the circuit! The set is on the picture 2. The produced potential can be measured by a voltmeter, the electricity by an amperometer or to put into the circuit a small flashlight bulb. So even we can enjoy the recognition of enthusiastic pupils that a natural light source has been created. Fig. 2 shows the cascading several galvanic units.

We should call the attention to the following:

- lemons, potatoes, apples represent in this case the electrolyte, that is a solution in which ions (cations and anions) are. Ask the pupils what kind of ions can be expected in foodstuffs! (The clever pupils surely will mention e.g. potassium and calcium cations and chloride, citrate and phosphate anions)
- The water content of these raw food electrolytes are approx. as follows: 76% potato, 90% apple, 89 % lemon. Mineral content (what is called practically as ash content) is the following: potato – 1.1 %, apple - 0.4%, lemon - 0.6%.
- Ask the question, is the value of the produced voltage affected in what product (food) the electrodes were inserted?, That is to say, does it change the voltage e.g. in the case if instead of apple or lemon pears or oranges are used?
- If we have time to carry out the experiment also with other metals (e.g. iron, silver, aluminum), we should point out the difference between the electrode potentials, hence the differences between the developed voltages.
- Be sure to mention that cascading several galvanic units (batteries connected in line) the voltage can be increased

DIFFERENTIATION BETWEEN RAW (CRUDE) FRESH EGG AND BOILED (COOKED) EGG WITHOUT CRACKING (BREAKING) THE EGGSHELL

If for some reasons you accidentally mixed the raw and the cooked eggs, it is rather easy to distinguish between them with a simple physical method. Try to spin the eggs (with about the same weight) appr. with the same power on the table or other flat surface. The spin is performed holding the eggs between the

index finger and thumb bringing the eggs successively into motion. Picture 3 shows the spinning of eggs, using for measurement a chronometer.

Picture 3
Spinning of fresh and boiled eggs

It can be measured well, that cooked eggs will spin for a longer time, however in case of raw eggs the spin time is significantly shorter. Eg. 20 seconds in the first case and only 12 seconds in the second one. Let's try to interpret the cause of the difference!

The difference is due to the situation, that boiled eggs - physically - behave as a rigid body, where the positions of each mass points are fixed. The time of rotation depends on the spin strength, egg weight and the friction between egg and table, however the drag (air resistance) is negligible. On the other side the raw egg considered as a system in which the white and the yolk (as viscous fluids) do not rotate together with the eggshell, and strong enough friction occurs between the fluid and the solid shell. This friction slows down the rotation, that is, outside of the friction between the table and egg shelll acts further a braking force of friction between the liquid part of the egg and the egg shell.

Finally let us mention that the energy of rotation is a function of the geometric parameters of the objects, with mass m and angulat velocity omega. The energy of rotation:

$$\mathbf{E_r = ½\ J\ \omega^2}$$

where:
J – inertia momentum

$$J=\sum_{i=1}^{n} m_i * r_i^2$$

Data for the J inertia momentum as a function of geometry of the objects are shown in table 4.

Table 4: Inertia momentum as a function of geometry

geometry	**inertia momentum**
globe	$m\ r^2$
cylinder	$m\ R^2$
pipe	$m\ (m\ R+^2r^2)$

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Compressive properties of Golden Delicious fruits

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Keywords:

apple, compression,
modulus of elasticity,
rupture point

Abstract. The study is aimed on the experimental evaluation of the apple cultivar Golden Delicious (*Malus domestica L.*) fruits at statical compressive loading in lateral direction. Mechanical properties such as rupture force and deformation as well as modulus of elasticity can be used to evaluate the behaviour of the fruits mechanically under the static loading. Apparent moduli of elasticity were determined on the base of elastic Hook theory and Hertz theory (ASAE, 2004). A testing machine Andilog Stentor 1000 (Andilog Technologies, Vitrolles, France) was employed for compression tests. The behaviour of the hemisphere of fruit was studied between two parallel plates and the indentation with the flat – faced cylindrical intender of diameter 8 mm and cone intender of diameter 6.4 mm and angle 60 degree was also studied.

INTRODUCTION

Mechanical properties of the apple fruits are required for the best management of the production process. Processing of apple tissue is in the many cases realized by the compress methods. Afterward material properties of the apple tissue represent very important feature of the material knowledge. The texture of apple flesh is important in assessing the eating qualities of the fruit (Khan and Vicent, 1993). Flesh firmness is a key quality parameter, since it is directly related to fruit ripeness, and is often a good indicator of shelf – life potential (De Ketelaere et al., 2006). Vozary and Meszaros (2007) interested in Idared apple cylinder of 20 mm diameter and of 15 mm length, cut out from whole apple in radial direction. The real part of impedance decreased as the deformation, or the stress increased, and the imaginary part of impedance increased under increasing stress or deformation. Extensive test have shown that if the initial part of force-deformation curves of soft biological tissues are taken into consideration, the initial part of the curves are usually concaved towards the force axis. This is exactly opposite the force-deformation curves for polymeric materials which is usually convex towards the force axis (Mohsenin, 1986).

MATERIALS AND METHODS

Samples

The apple fruits of cultivar Golden Delicious (*Malus domestica L.*) were tested. The fruits were obtained in the conventional shop and stored one day at the temperature 4°C and the air humidity (40 – 60) % in the refrigerator. Twenty samples of the fruits were collected and used for the testing. Each fruit was cut into two hemispheres. Each test was realized on the one hemisphere sample.

Methods

Statical compressive loading in lateral direction was used for the fruit testing. A testing machine Andilog Stentor 1000 (Andilog Technologies, Vitrolles, France) was employed for compression tests. The experiments were carried out at loading speed 10 mm.min^{-1}. The force F (N) and the compression D_c (m) were measured by the acquisition software RSIC ver. 4.06.
The loading curves of dependency of the force on the deformation or of the stress on the strain were realized.

Four methods were applied for testing of fruits. Ten tests were applied for each of four methods. The compression of fruit hemisphere between two parallel plates was realized. First method was based on the elastic theory and the Hook's law was used. Values of module of elasticity were calculated as the slope of the linear part of the stress – strain curves on the base of regression method. The second applied method was the determination of apparent module of elasticity realized on the base of the Hertz equations for contact stresses used in solid mechanics also at the compression of fruit hemisphere between two parallel plates. Apparent module of elasticity were determined from the equation (ASAE, 2004):

$$E_a = \frac{0.338\,K^{\frac{3}{2}}F\left(1-\mu^2\right)}{D_c^{\frac{3}{2}}}\left[\left(\frac{1}{R_U}+\frac{1}{R'_U}\right)^{\frac{1}{2}}\right] \quad (1)$$

where: E_a is the apparent modulus of elasticity, Pa,
D_c is the compression, m,
μ represents Poisson's ratio, - ,
F is the force, N,
R_U, R'_U are the minimum and the maximum radii of curvature of the convex surface of the sample at the point of contact with the upper plate, m,
K is the constant determined on the base of contact angle.

Third method was based on the penetration of fruit hemisphere by flat – ended cylindrical indenter of diameter 8 mm. Determination of the apparent module of elasticity on the base of the Hertz equations were realized from the equation (ASAE, 2004):

$$E_a = \frac{0.338\,K^{\frac{3}{2}}F\left(1-\mu^2\right)}{D_c^{\frac{3}{2}}}\left[\left(\frac{1}{R_U}+\frac{1}{R'_U}+\frac{4}{d}\right)^{\frac{1}{2}}\right] \quad (2)$$

where: all quantities are the same as in the equation (1),
d is the indenter diameter curvature, m.

Fourth method was based on the penetration of fruit hemisphere by cone indenter of diameter 6.4 mm and angle 60 degree. Determination of the apparent module of elasticity was realized on the base of the elastic theory by the equation (McKee et al., 2011):

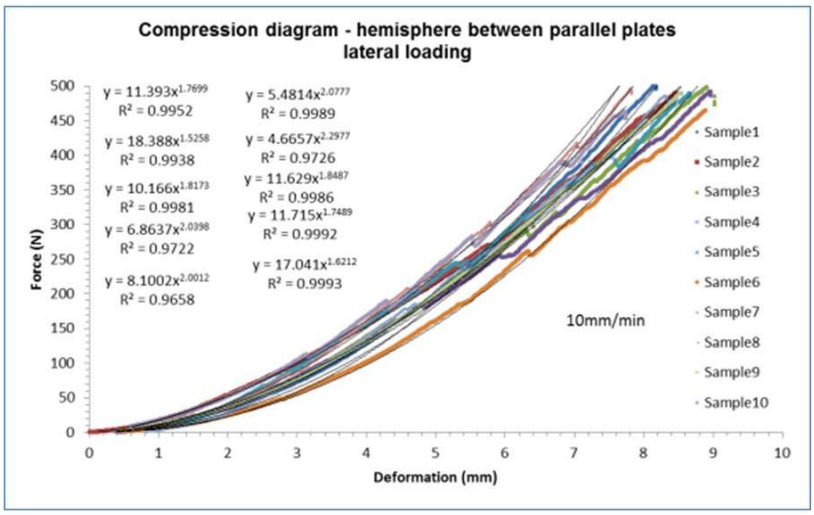
$$F = \frac{2}{\pi}\tan\alpha\,\frac{E}{(1-\mu^2)}\,D^2 \quad (3)$$

where:
E is the modulus of elasticity, Pa,
F is force, N,
μ is Poisson' ratio, - ,
α is the half angle opening of the cone, ° ,
D is the depth of penetration deformation, mm

RESULTS AND DISCUSSION

The compression diagrams of the hemisphere fruits samples compressed between two parallel plates are presented on the Figure 1. Compression force – deformation curves (Figure 1.a.) and stress – strain curves (Figure 1.b.) of the apple fruit Golden Delicious in the lateral loading represent the nonlinear viscoelastic behaviour because the average power coefficient of the power functions applied on the force – deformation curves (Figure 1.a.) reached the value 1.875 and on the stress – strain curves (Figure 1.b.) reached the value 1.866. The elastic theory assumed the value of the power coefficient 1 and Hertz theory 1.5

a.



b.

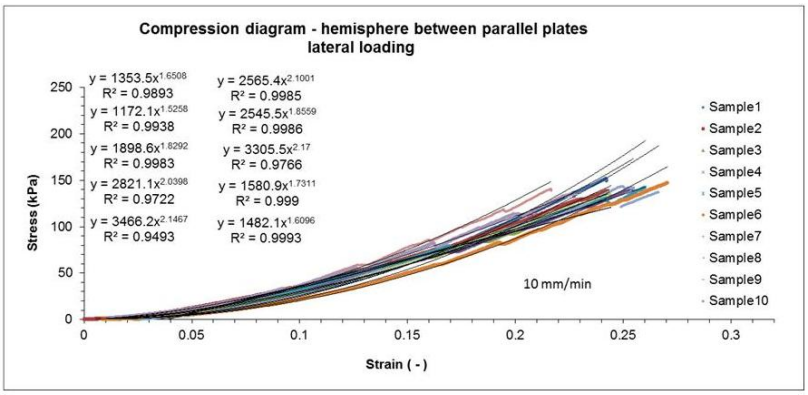


Figure 1
Compression force – deformation curves (a) and the stress – strain curves (b) of apple fruits Golden Delicious in the lateral loading – nonlinear viscoelastic characteristics

Mechanical behaviour in nonlinear curves we can describe, when we will determine the differential modulus of elasticity in the each point of the dependence. Average value of the differential modulus of elasticity obtained from the linear parts of the stress – strain curves in the Figure 1.b. by regression method (strains ranged from 0.1 to 0.3) was E = 785.536±73.597 kPa. Average value of the differential modulus in the initial region of the stress – strain curves (strains ranged from 0.04 to 0.12) reached value E = 485.845±67.591 kPa.

Apparent module of elasticity were determined from the Eq. 1. Poisson's ratio μ was assumed 0.22 (ASAE, 2004). K was the constant determined on the base of contact angle, K = 1.349. Apparent moduli of elasticity depended on value of the deformation D_c (mm). Dependencies are presented on the Figure 2. In the level of deformation from 1 mm to 9 mm, realized by the compression between two parallel plates, the experimental values of the apparent module of elasticity ranged from 500 kPa to the 2800 kPa.

Third realized method was the compress deformation of the fruit hemisphere by flat – faced cylindrical indenter of diameter 8 mm. This method enabled the measurement of mechanical properties of apple's skin with mesocarp. Compression force – deformation curves and the stress – strain curves are presented on the Figure 3.a. and Figure 3.b.

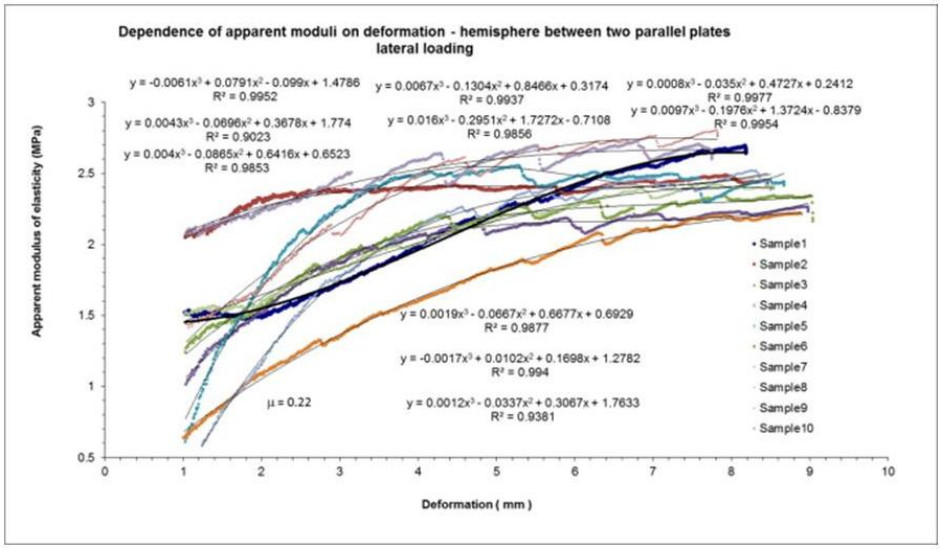
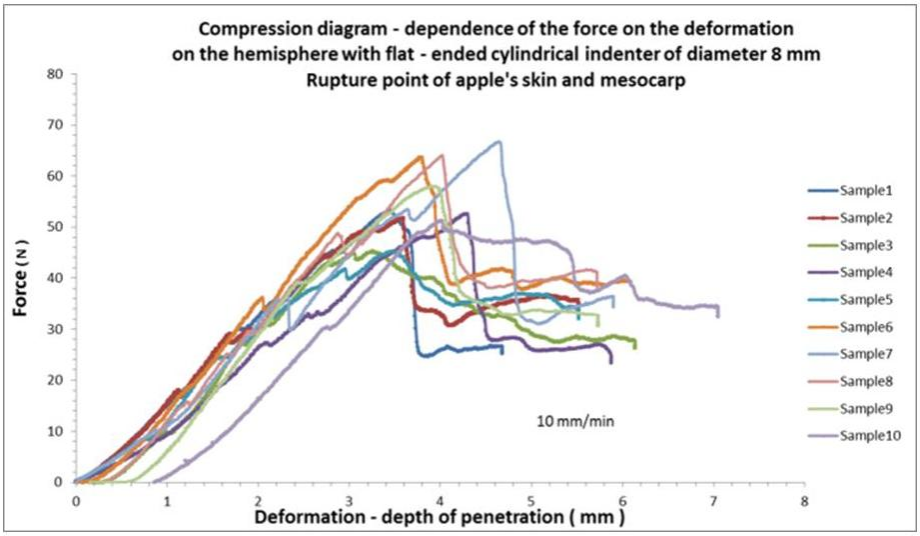


Figure 2
Dependences of the apparent module of elasticity on the deformation of apple hemisphere fruits Golden Delicious
in the lateral loading – second method – Hertz theory

a.



b.

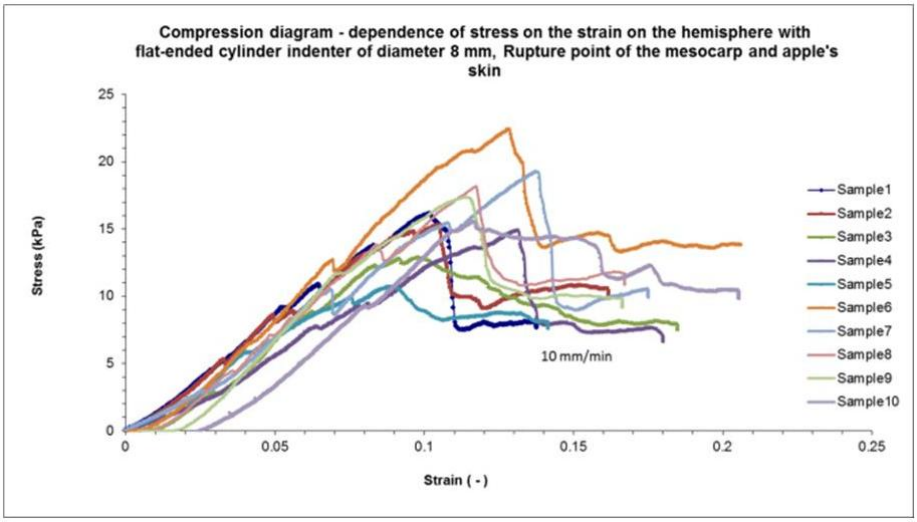


Figure 3
Compression force – deformation curves of the apple's skin fruit and mesocarp (a) and the stress – strain curves (b) of Golden Delicious fruit hemispheres in the lateral loading and the rupture points of the mesocarp and the apple fruit skin – third method

The rupture points of apple‘s skin and mesocarp (maximum of the force where apple’s skin with mesocarp is punctured by the indenter) were determined. Average value of the maximal force in the rupture point F_R=55.251±8.152 N and the maximal deformation in the rupture point D_R=3.942±0.475 mm were determined. Average value of the maximal stress in the rupture point was σ_R=16.177±3.297 kPa and the maximal strain in the rupture point was ε_R =0.113±0.016. Module of elasticity were determined on the base of Hook’s theory of elasticity from the linear part of the compression curves (deformation ranged from 0 to 2.4 mm) Average values of the module of elasticity in this region, where the strains ranged from 0.01 to 0.06, reached value E=170.168±24.273kPa. Apparent module of elasticity were determined from the Eq. 2. Poisson’s ratio μ was assumed 0.22 (ASAE, 2004). K was the constant determined on the base of contact angle, K = 1.349. Apparent modulus of elasticity depended also on value of the deformation D_c (mm). Dependencies are presented on the Figure 4. In the level of deformation from 1 mm to 7 mm the experimental values of the apparent module of elasticity obtained by the deformation of hemisphere fruits by flat – faced cylindrical indenter of diameter 8 mm, ranged from 200 kPa to the 2000 kPa.

Fourth applied method consisted in the deformation of the fruit hemispheres by the cone indenter of diameter 6.4 mm and angle 60°. The length of the cone of diameter 6.4 mm was 6 mm and then the diameter enlarged and continued as the cylinder of the diameter of 8 mm on the length 3.6 mm. Then diameter of the cylinder again continued with the diameter of 6.4 mm. The method also enabled the measurement of mechanical properties of apple‘s skin with mesocarp. Compression force – deformation curves are presented on the Figure 5. The rupture points of apple‘s skin and mesocarp were also determined. We identified two rupture points on the deformation curves. The first rupture point corresponds of the diameter 6.4 mm and the depth of penetration of 6 mm of the cone and the second point corresponds of the enlarging of the diameter of the cone on the 8 mm and the depth of the penetration from 7 mm to 9.6 mm.

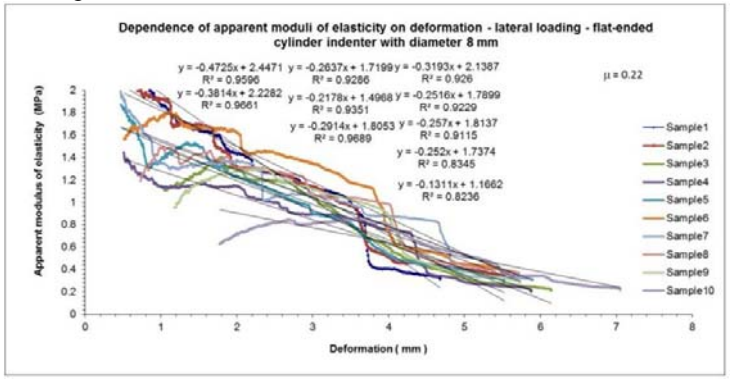


Figure 4
Dependences of apparent module of elasticity on the deformation of apple hemisphere fruits Golden Delicious in the lateral loading – third method – Hertz theory

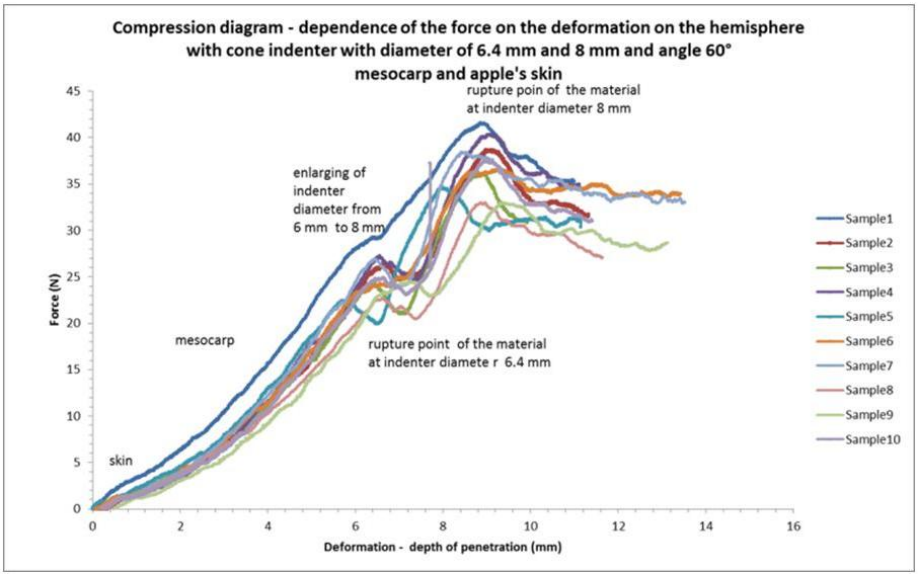


Figure 5
Compression force – deformation curves of the apple's skin fruit and mesocarp of Golden Delicious fruit hemispheres in the lateral loading and two rupture points of the mesocarp and the apple fruit skin – fourth method

Average value of the maximal force in the first rupture point F_R=24.824±0.774 N and the maximal deformation in the first rupture point D_R=6.409±0.154 mm were determined. Average value of the maximal force in the second rupture point F_R=36.826±1.016 N and the maximal deformation in the second rupture point D_R=8.838±0.142 mm were also determined. When we realized regression method by the power function model on the experimental data of the force – deformation dependencies on the Figure 5 in the range of the deformations from 1 mm to 6 mm (mesocarp of apple), we obtained very good correspondence with the Hertz theory (Figure 6). The experimental equation obtained as the average results from the ten dependencies was:

$$F = 1.443\, D^{1.540} \quad (4)$$

The coefficient 1.540 confirmed good correspondence with the Hertz theory. Apparent module of elasticity were determined from the Eq. 4. Poisson's ratio μ was assumed 0.22 (ASAE, 2004). The half angle opening of the cone was 30 °.

Apparent modulus of elasticity depended also on value of the deformation D (mm). Dependencies are presented on the Figure 7. In the level of the deformations from 2 mm to 6 mm the experimental values of the apparent module of elasticity obtained by the deformation of hemisphere fruits by the cone indenter of diameter 6.4 mm and angle 60°, ranged from 1500 kPa to the 4500 kPa.

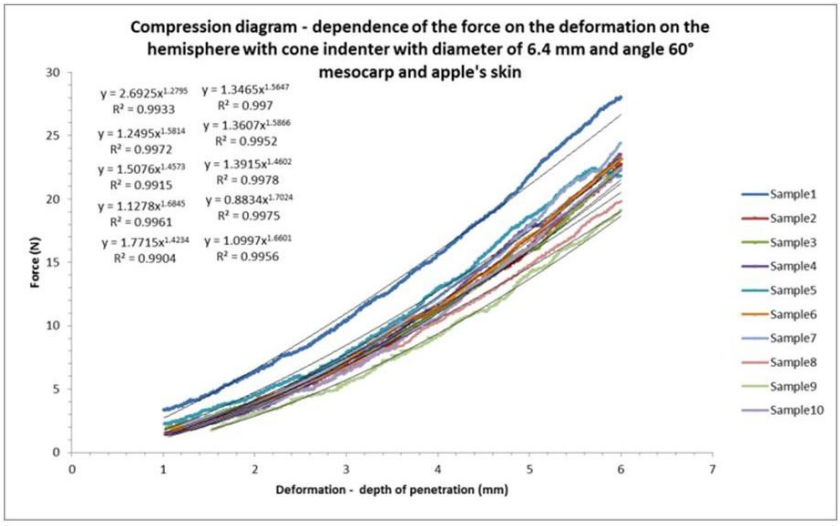


Figure 6
Compression force – deformation curves of the apple's skin fruit and the mesocarp of Golden Delicious in the lateral loading – depth of penetration from 1 to 6 mm fourth method – Hertz model

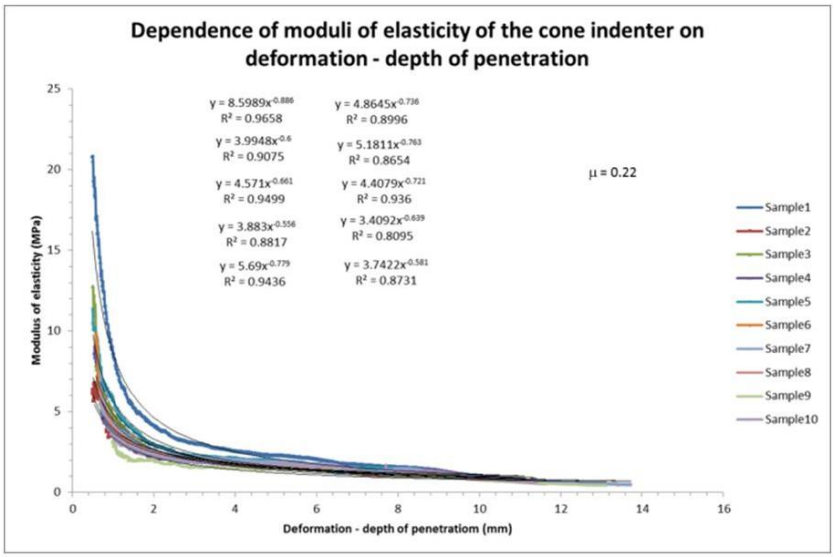


Figure 7
Dependences of apparent module of elasticity of the Golden Delicious apple fruit hemispheres on the deformation – fourth method

A lot of the authors interested in the study of the compressive properties of the apples. Cen et al. (2013) interested in the measurements of the stress – strain curves of Golden Delicious apple, which measured for each of the five storage times and reached values of the stress from 0.2 MPa to 0.45 MPa at the strain from 10% to 15%. Arnold and Mohsenin (1971) interested in the study of Yellow Delicious apple cultivars. They obtained values of the module of elasticity E = 3030 kPa and E = 4200 kPa at the compressive speed v = 2.54 mm/min. Costa et al. (2011) measured 86 apple cultivars at the compressive speed v = 100 mm/min and 300 mm/min and obtained the values of the module of elasticity from 1000 kPa to 2 000 kPa. Shirvani et al. (2014) studied the cultivars of Golden Delicious apples and on the base of the Boussinesq's theory determined the modulus of elasticity E =1530 kPa, on the base of the Hertz's theory also obtained the modulus of elasticity E =1530 kPa and on the base of the Hooke's theory determined the modulus of elasticity E =2680 kPa. Winisdorffer et al. (2015) studied 5 cultivars of the apples and reached the moduli of elasticity from 1000 kPa to 4500 kPa. Alamar et al. (2008) studied apple cultivars Braeburn and Jonagored and reached from the first part of stress-strain curve the moduli of elasticity in the range from 350 kPa to 420 kPa. They obtained the moduli of elasticity in the range from 1720 to 2010 kPa at the 80% of the stress.

CONCLUSIONS

The values of the module of elasticity of the apple fruit hemispheres which were determined on the base of the first method (Hooke's law) were not consistent with Hertz's theory. The values obtained on the base of the first method (Hooke's law) were consistent with the results of Alamar et al. (2008). The apparent module of elasticity obtained on the base of Hertz's theory for the lateral loading of the hemispheres between two parallel plates were consistent with the module determined by the cylinder flat – end indenter and the cone indenter. The values of the apparent module of elasticity depended on the deformation at which were calculated. This is indication that several of the assumptions on which the Hertz's equations are based were not completely satisfied. The apple fruit is not an ideal elastic solid body.

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A Survey on the Centrifugal Freeze Concentration Method For Mulberry Molasses

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Abstract. Centrifugal freeze concentration is a new technique to obtain the concentrated solution from liquid foods in food industry, which is a method for recovering a food solute from a solution based on the separation of pure ice crystals from a freeze concentrated aqueous phase. The aim of this study was to investigate the concentration of mulberry molasses by the centrifugation freeze concentration method. For this purpose, the mulberry molasses (73.2 ^{o}Bx) was diluted to different total soluble dry matter contents (20, 25, 30, 35, 40, and 45 ^{o}Bx) and the method of freeze concentration assisted by centrifugation was applied. The concentrated solution which was obtained after the removal of formed ice by filtration was frozen again at same conditions and same processes were repeated (cycle). The percentage of concentrate, efficiency of concentration and recovered solute values were calculated for each cycle. According to the results of the successive three cycles, the amount of remaining solutions after the removal of ice content decreased and in contrary to increase in total soluble solid content, the concentration efficiency decreased. The percentage of concentrate changed between 79.4% and 94.8% and the recovered solute values reached to 0.80 and 0.93 kg/kg.

Keywords:

Freezing,
Freeze Concentration,
Centrifuge,
Mulberry Molasses

INTRODUCTION

Molasses (Pekmez), a kind of fruit juice concentrate, is a traditional Turkish food made of different fruits such as grape, mulberry, carob, watermelon, apricot, prune, fig, apple and sugar beet (Tosun and Ustun, 2003; Batu, 2005; Karababa and Işıklı, 2005; Liyana-Pathirana et al., 2006). Pekmez is a naturally nutritious food with total soluble solid content of 50–80% (Arıcı et al., 2004), high amounts of sugar, minerals and organic acids (Sengul et al., 2005; Yoğurtçu and Kamışlı, 2006).

Freeze concentration is a method for concentrating a food solute in a solution based on the separation of pure ice crystals to from a freeze-concentrated solution (Petzold et al., 2012). In this method water is removed at low temperatures preserving the quality of the original materials (Miyawaki et al., 2005). These foods can be kept without spoiling for a long time or it's ready for subsequent processing steps such as drying (Dinçer and Topuz, 2009).

One of the main unit operations in the food industry is the concentration of

aqueous solutions. Generally, three techniques are used for concentration of food. These are evaporation, reverse osmosis and freeze concentration. As compared to evaporation and membrane technology: freeze concentration has some significant potential advantages for producing a concentrate with high quality because the process occurs at low temperatures where no vapor/liquid interface exists resulting in no loss of volatiles, the flavor and quality of freeze-concentrated products are exceptionally high, especially relative to their evaporated counterparts. These benefits make freeze concentration particularly suitable for the concentration of some products, such as fruit juices, coffee and tea extracts, and aroma extracts (Morison and Hartel, 2007).

Freezing is an important stage of this method and three techniques are used for the growth of ice crystals. These are suspension freeze concentration, film freeze concentration (progressive or falling film freeze concentration) and block freeze concentration (Aider et al., 2009; Sanchez et al., 2011). A typical installation for suspension freeze concentration consists of scraped surface heat exchangers to generate ice nuclei, recrystallisers, to increase ice growth and a system for separation of the ice from the liquid, usually by wash columns, operated at elevated pressures. (Van Weelden, 1994; Lee and Lee, 1998). Film freeze concentration is based on film crystallisation, which consists of the formation of a single crystal that grows layer by layer from the solution to be concentrated. In this process the fluid to be concentrated flows down over a chilled surface, which causes the crystallization of ice and the further growth of the ice crystals on the surface (Sanchez et al., 2009). The separation of ice and concentrated solution occurs because the ice adheres to the surface, while the concentrated liquid flows down along the surface. In block freeze concentration, also known as freeze–thaw concentration, the solution to be concentrated is completely frozen and then partially thawed to recover a fraction of liquid with a higher concentration (Aider et al., 2009; Nakagawa et al., 2010). Block freeze concentration consists of three stages: freezing, thawing and separation of the concentrated liquid fraction (Moreno et al., 2013). Additionally, the process can be repeated in successive cycles to increase the concentration index (Aider and Ounis, 2012). Assisted techniques improve the efficiency of processing for block freeze concentration. These techniques are the application of ultrasound, ice nucleation agents, vacuum, and centrifugation (Petzold and Aguilera, 2013). In a study of Petzold et al. (2012), the suction was generated by connecting a vacuum pump to the bottom of the frozen sample at ambient temperature and under vacuum in the freeze concentration of sucrose solutions. Watanabe and Arai (1994) have studied the application ice nucleation activity to freeze concentration. Centrifugation is a type of separation where the force of gravity is largely replaced by a higher driving force, through the application of centrifugal force (Toledo, 2007). An alternative for separating the concentrated solution from the ice fraction is the use of centrifugation. Centrifugation has been proposed by Petzold et al. (2015) in frozen blueberry and pineapple juices. In this study, frozen samples transferred to a centrifuge operated at 20˚C for 10 min at 4600 rpm to

force the separation of solutes from the frozen samples. This technique has high values of total soluble solid content, recovered solute and percentage of concentrate after the third cycles for both juices, values close to 0.74 kg solute per 1 kg initial solute, and reaching approximately 60% of the percentage of concentrate. In the study of Petzold and Aguilera (2012), sucrose solutions were transferred to a centrifuge operated at 20˚C for 15 min different centrifugation speeds (800, 1600 and 2400 RCF). Sucrose solutions reached approximately 0.73 kg of sucrose obtained per 1 kg of initial sucrose at 1600 RCF of centrifugation speed, independent of initial concentration of sucrose (5 to 20 wt.%) and freezing procedure (radial or undirectional freezing).The aim of this study is to investigate the concentration of mulberry molasses by the block freeze concentration method applied by centrifugal separation.

MATERIALS AND METHODS

Materials

Mulberry molasses (73.2 ˚Brix, SEREL Inc.) were obtained from a local market in Izmir, Turkey. Diluted samples with the total soluble solid contents of 20, 25, 30, 35, 40, and 45 ˚Brix (˚Bx) were prepared for the experiments by mixing with water.

Methods

Freezing and centrifugation

Diluted mulberry molasses (25g) were placed into plastic centrifugal tubes (internal diameter D=3cm) were frozen in a static freezer at -20˚C for 12 hours. Then, the frozen samples were removed from the freezer (Vestel SVC-145, Turkey) and rapidly transferred to a centrifuge (Nüve NF800, Turkey) operated at 4100 rpm for 15 min at room condition. After centrifugation, the concentrated solution (solute) was collected, and the remaining frozen core (ice fraction) was thawed so that the total soluble solids content was determined in both fractions. The total soluble solids content of separated ice fraction (C_i) and total soluble solid content of concentrated sample (C_c) were analyzed at ambient temperature with a refractometer (HANNA HI 96801 Digital Refractometer, USA). The concentrated solution which was obtained the after removal of formed ice by filtration was frozen again at same conditions and same processes were repeated three times (cycle).

Modelling of Rheological Behaviour

Rheological properties of diluted and concentrated of mulberry molasses samples were measured by Brookfield Viscometer (Model LVDV-II pro, Brookfield Engineering Laboratories, USA) at 25 ˚C . Shear stress- shear rate data were obtained from experimental measurements. Data was fitted to 4 different rheological models (Newtonian, Bingham, Power Law, Herschel-Bulkley) (Rao, 2014) by using IBM SPSS Statistics Package (vers. 20).

Newtonian Model: $\sigma = \eta \dot{\gamma}$ (5)

Power Law Model: $\sigma = k(\dot{\gamma})^n$ (6)

Bingham Model: $\sigma - \sigma_0 = k \dot{\gamma}$ (7)

Herschel-Bulkley Model:

$$\sigma - \sigma_0 = k(\dot{\gamma})^n \quad (8)$$

where σ is shear stress (Pa), $\dot{\gamma}$ is shear rate (1/sec), σ_0 is yield stress (Pa), n is flow behaviour index, k is consistency coefficient (Pa.s ^{n}).

Calculations

The calculated values of thawing fraction (f) (Nakagawa et al., 2010, Miyawaki et al., 2012), percentage of concentrate (PC,%), efficiency (E, %), and recovered solute (Y, kg solute/kg initial solute) were calculated as defined in the references (Nakagawa et al., 2010, Petzold et al., 2013).

Freeze concentration is a method for concentrating a food solute in a solution based on the separation of ice crystals to from a freeze-concentrated solution. Block freeze concentration consists of freezing, thawing and separation stages. The concentrated phase is melting more quickly than ice phase, therefore, concentrated phase is separated from the ice phase. Freeze concentration occurs at low temperatures where no vapor/liquid interface exists resulting in no loss of volatiles, therefore, the flavor and quality is higher than evaporation.

Fig. 1 shows the total soluble solid content (^{o}Bx) values of concentrated molasses at each cycle. A general increase in total soluble solid content (^{o}Bx) was observed by increasing the number of cycles. The highest increase was seen in the initial total soluble solid content of 20 ^{o}Bx sample and reached to 41.4 ^{o}Bx after the third cycle. On the contrary, the increase in initial total soluble solid content (^{o}Bx) leads to a decrease in total soluble solid content of the later cycles since the water in the sample decreases and by increasing the number of cycles, water in the frozen sample also decreases. Generally by increasing the number of cycles, total soluble solid content of ice fraction (^{o}Bx) increased. According to Petzold et al. (2015), by increasing the number of cycles, total soluble solid content (^{o}Bx) in the concentrated fraction and the separated ice fraction increased. Fig. 2 shows the calculated thawing fraction values at each cycle. The thawing fraction was used to follow the development of process with increasing the number of cycles, thawing fraction also increased. The highest thawing fraction was observed at the total soluble solid content of 40°Bx.

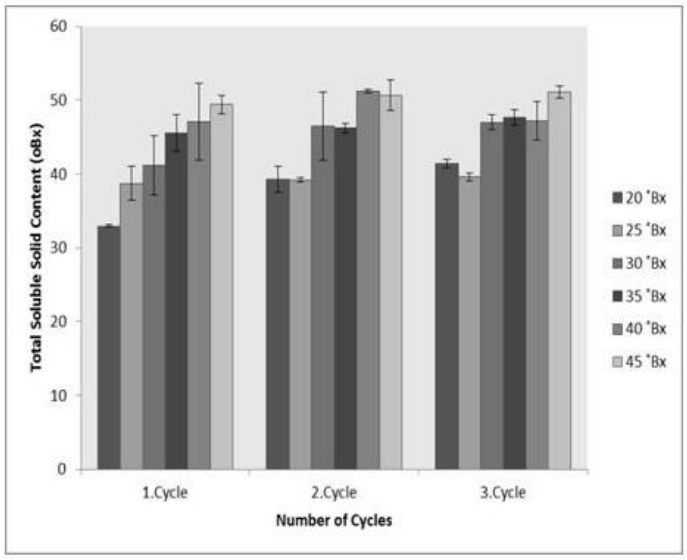

Figure 1
The total soluble solid content of concentrated molasses

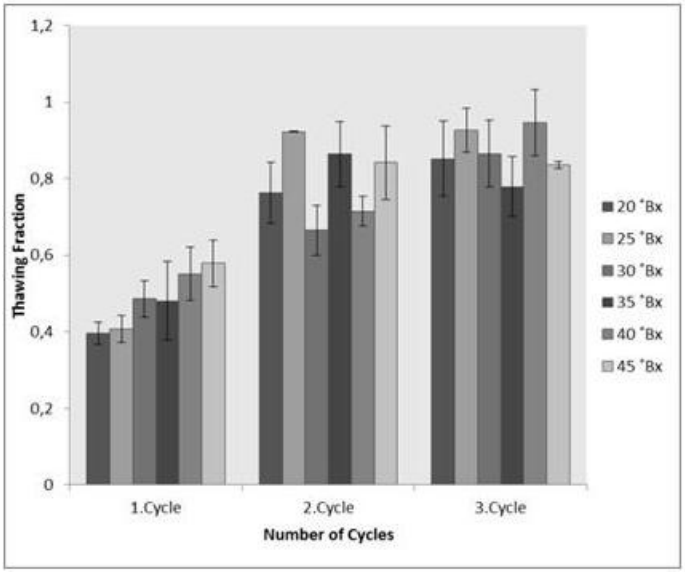

Figure 2
The thawing fraction for each cycle

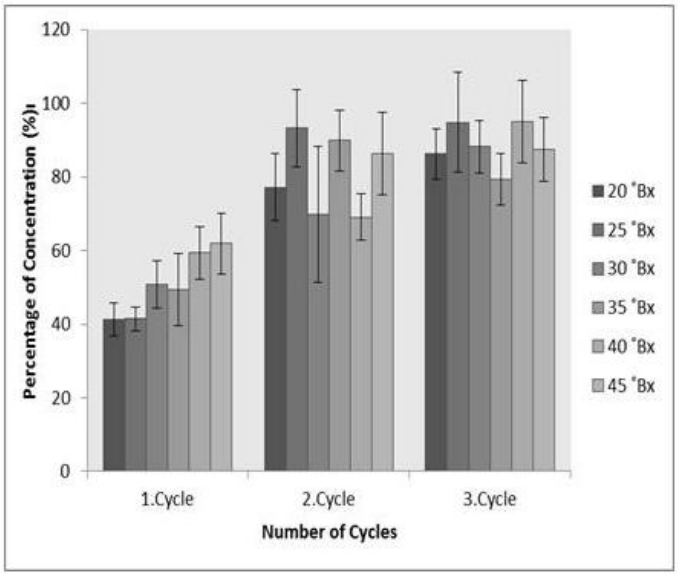

Figure 3
The percentage of concentrate for each cycle

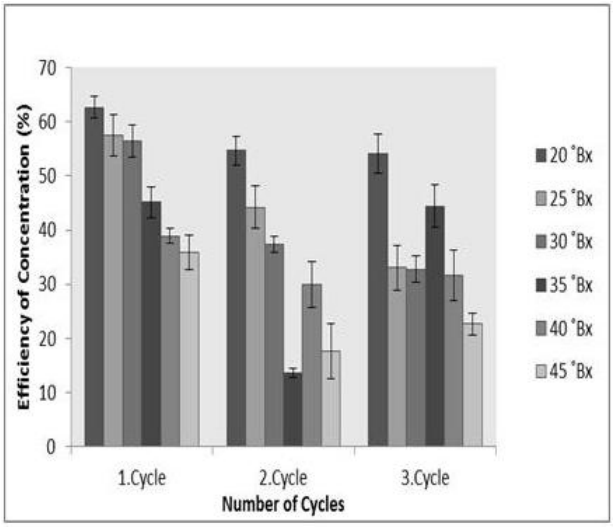

Figure 4
The efficiency of concentration for each cycle

Fig. 3 shows the percentage of concentrate at each cycle. As the number of cycle increases, percentage of concentrate increased. At end of the third cycle, the percentage of concentrate changed between 79.4% and 94.8%. At the research of Petzold et al. (2015), the percentage of concentrate increased for blueberry and pineapple juices at each cycle and reached to 60 % for the blueberry juice and 61% for the pineapple juice. Fig. 4 shows the efficiency of concentration at each cycle. As the number of cycles increased, the efficiency of concentration decreased. The efficiency of concentration was found to be at the highest at 20 ^{o}Bx.

Fig. 5 shows the recovered solute (expressed as kg solute/kg initial solute) at each cycle. As a general trend, the amount of recovered solute increased after each cycle. At the end of the third cycle, the recovered solute values ranged between 0.80 and 0.93 kg solute/kg initial solute. The recovered solute values of 0.67 to 0.74 kg/kg for blueberry and 0.48 to 0.73 kg/kg for pineapple juice was reported by Petzold et al., 2015.

Knowledge about the flow behavior of concentrated samples is pertinent to quality control, sensory evaluation and food processing and handling operations (transport, mixing, homogenization, sterilization, concentration) (Rao et al., 1984). The rheological properties of diluted (25, 35 and 45 $^{\circ}$Bx) and freeze concentrated of mulberry molasses samples were measured (0-100 rpm, 18 spindle number) and R^2 and RMSE (Root-Mean-Square Error) values were calculated to determine the model describing the reological changes.

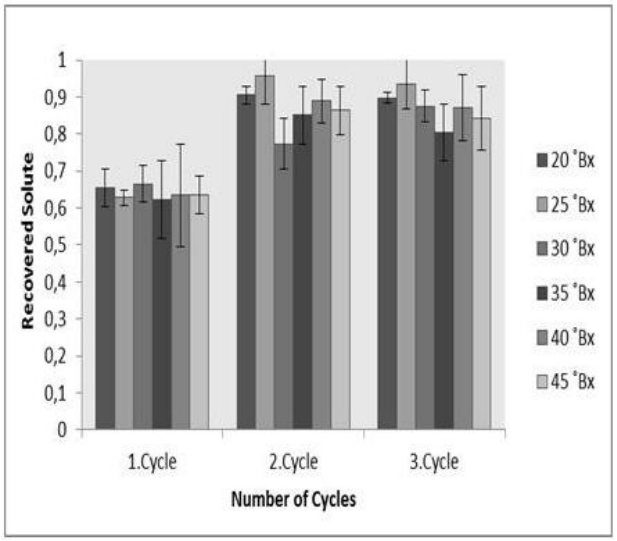

Figure 5
The recovered solute for each cycle

Table 1: (a., b., c., d., e., f.,): Statistical evaluation of the rheological model best fitting the experimental data of samples

a.

Models	Parameters	Inıtıal Concentration (˚Bx)	Initial	1.Cycle
Newtonian	η	25 ˚Bx	0.018±0.001	0.035±0.002
		35 ˚Bx	0.038±0.001	0.075±0.001
		45 ˚Bx	0.084±0.001	0.117±0.001
	R^2	25 ˚Bx	0.936	0.872
		35 ˚Bx	0.970	0.999
		45 ˚Bx	0.994	0.999
	RMSE	25 ˚Bx	0.217	0.453
		35 ˚Bx	0.281	0.278
		45 ˚Bx	0.273	0.278
Power Law	k	25 ˚Bx	0.002±0.001	0.198±0.054
		35 ˚Bx	0.020±0.008	0.093±0.019
		45 ˚Bx	0.057±0.009	0.098±0.007
	n	25 ˚Bx	1.457±0.099	0.623±0.061
		35 ˚Bx	1.141±0.090	0.953±0.044
		45 ˚Bx	1.084±0.033	1.038±0.016
	R^2	25 ˚Bx	0.985	0.967
		35 ˚Bx	0.977	0.992
		45 ˚Bx	0.997	0.999
	RMSE	25 ˚Bx	0.151	0.221
		35 ˚Bx	0.245	0.263
		45 ˚Bx	0.206	0.263

b.

Models	Parameters	Inıtıal Concentration (˚Bx)	2.Cycle	3.Cycle
Newtonian	η	25 ˚Bx	0.049±0.001	0.048±0.002
		35 ˚Bx	0.069±0.001	0.069±0.001
		45 ˚Bx	0.106±0.001	0.090±0.001
	R^2	25 ˚Bx	0.963	0.942
		35 ˚Bx	0.997	0.994
		45 ˚Bx	0.997	0.999
	RMSE	25 ˚Bx	0.404	0.344
		35 ˚Bx	0.161	0.217
		45 ˚Bx	0.240	0.145
Power Law	k	25 ˚Bx	0.079±0.028	0.103±0.039
		35 ˚Bx	0.048±0.004	0.048±0.007
		45 ˚Bx	0.110±0.015	0.078±0.007
	n	25 ˚Bx	0.895±0.078	0.832±0.083
		35 ˚Bx	1.079±0.017	1.080±0.033
		45 ˚Bx	0.992±0.029	1.032±0.018
	R^2	25 ˚Bx	0.969	0.958
		35 ˚Bx	0.999	0.997
		45 ˚Bx	0.997	0.999
	RMSE	25 ˚Bx	0.345	0.318
		35 ˚Bx	0.087	5.086
		45 ˚Bx	0.239	0.132

c.

Models	Parameters	Inıtıal Concentration (˚Bx)	Initial	1.Cycle
Bingham	σ_0	25 ˚Bx	-0.209±0.020	0.632±0.171
		35 ˚Bx	0.008±0.005	0.388±0.155
		45 ˚Bx	-0.073±0.018	0.006±0.114
	η	25 ˚Bx	0.020±0.001	0.028±0.002
		35 ˚Bx	0.038±0.002	0.071±0.001
		45 ˚Bx	0.085±0.002	0.116±0.001
	R^2	25 ˚Bx	0.953	0.949
		35 ˚Bx	0.970	0.996
		45 ˚Bx	0.994	0.999
	RMSE	25 ˚Bx	0.186	0.276
		35 ˚Bx	0.280	0.184
		45 ˚Bx	0.270	0.184

d.

Models	Parameters	Inıtıal Concentration (˚Bx)	2.Cycle	3.Cycle
Bingham	σ_0	25 ˚Bx	0.547±0.112	0.701±0.040
		35 ˚Bx	-0.149±0.087	-0.020±0.009
		45 ˚Bx	0.303±0.109	-0.022±0.009
	η	25 ˚Bx	0.043±0.001	0.040±0.001
		35 ˚Bx	0.071±0.001	0.069±0.002
		45 ˚Bx	0.103±0.001	0.090±0.001
	R^2	25 ˚Bx	0.990	0.992
		35 ˚Bx	0.998	0.994
		45 ˚Bx	0.998	0.999
	RMSE	25 ˚Bx	0.181	0.151
		35 ˚Bx	0.141	0.216
		45 ˚Bx	0.175	0.144

e.

Models	Parameters	Inıtıal Concentration (˚Bx)	Initial	1.Cycle
Herschel-Bulkley	σ_0	25 ˚Bx	0.060±0.007	0.060±0.012
		35 ˚Bx	0.360±0.096	0.658±0.138
		45 ˚Bx	0.381±0.078	0.380±0.052
	k	25 ˚Bx	0.012±0.001	0.148±0.076
		35 ˚Bx	0.008±0.002	0.041±0.009
		45 ˚Bx	0.037±0.008	0.073±0.004
	n	25 ˚Bx	1.533±0.164	0.675±0.101
		35 ˚Bx	1.321±0.146	1.112±0.047
		45 ˚Bx	1.165±0.044	1.094±0.010
	R^2	25 ˚Bx	0.986	0.969
		35 ˚Bx	0.985	0.998
		45 ˚Bx	0.998	1.000
	RMSE	25 ˚Bx	0.445	0.500
		35 ˚Bx	0.208	0.148
		45 ˚Bx	0.533	0.148

f.

Models	Parameters	Inıtıal Concentration (˚Bx)	2.Cycle	3.Cycle
Herschel-Bulkley	σ_0	25 ˚Bx	0.862±0.090	0.992±0.056
		35 ˚Bx	0.104±0.077	0.395±0.082
		45 ˚Bx	0.649±0.072	0.197±0.065
	k	25 ˚Bx	0.013±0.004	0.013±0.002
		35 ˚Bx	0.042±0.005	0.027±0.004
		45 ˚Bx	0.063±0.005	0.064±0.008
	n	25 ˚Bx	1.240±0.060	1.235±0.039
		35 ˚Bx	1.105±0.026	1.189±0.032
		45 ˚Bx	1.098±0.017	1.070±0.026
	R^2	25 ˚Bx	0.997	0.999
		35 ˚Bx	0.999	0.999
		45 ˚Bx	1.000	0.999
	RMSE	25 ˚Bx	0.096	0.123
		35 ˚Bx	0.079	0.087
		45 ˚Bx	0.081	0.116

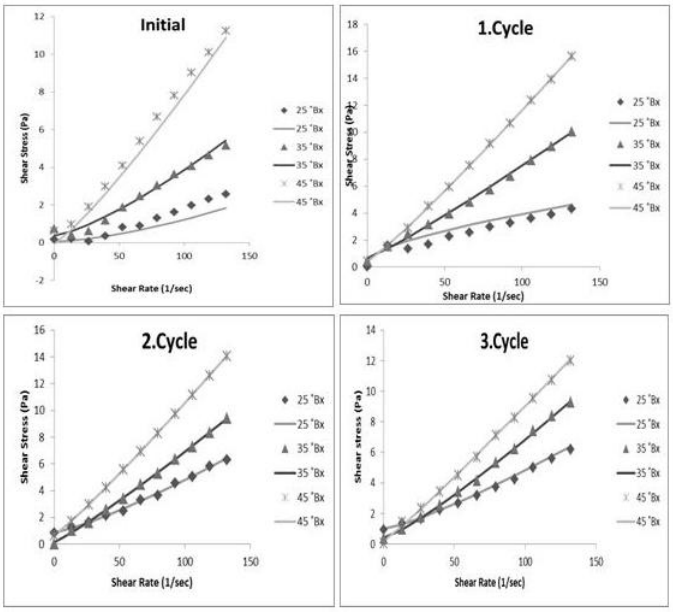

Figure 6
The experimental data and Herschel-Bulkley model predictions and experimental data for shear stress-shear rate relationship for original samples (a) and after each cycle (b, c, d) of freeze concentration (Symbols show the experimental data, lines show the model prediction)

Table 1 shows the statistical evaluation of the reological models by fitting the experimental data of samples. The best fit for all samples were observed with the Herschel-Bulkley Model. In Herschel-Bulkley Model, consistency coefficient (k) of the samples increased according to beginning. At the end of the third cycle, maximum increase of consistency coefficient was 0.064 $Pa.s^n$. Fig. 6 shows the model predictions and experimental data for shear stress-shear rate relationship for original samples (a) and after each cycle (b, c, d) of freeze concentration 25, 35 and 45 ˚Bx.

CONCLUSION

The aim of this study was to investigate of concentration of mulberry molasses by freeze concentration method assisted by centrifugation. Centrifugation technique is used improve the efficiency of processing for freeze concentration. By the increasing the number of cycles, the values of concentration, thawing fraction, percentage of concentrate, efficiency of concentration, recovered solute and viscosity increased. The rheological behaviour of molasses samples were best modeled with Herschel-Bulkley Model for both the original samples and after each cycle of freeze concentration.

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Sensory Characteristics and Textural Changes during Storage of Sponge Cake with Functional Ingredients

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Keywords:
sponge cake,
functional ingredients,
textural characteristics,
storage,
sensory analysis

Abstract. Sensory and textural characteristics of sponge cake prepared with different functional ingredients (einkorn wholemeal flour, Jerusalem artichoke powder, cocoa husk powder) in replacement of the part of the wheat flour were investigated during 6 days storage (on the 1st, the 3rd and the 6th day). Descriptive sensory analysis was used to compare the new and the control sponge cakes. The functional ingredients did not produce significant changes in the sensory characteristics of the sponge cakes, except of crumb's texture. The texture changes are characterised by elastic modulus, hardness and cutting force. These parameters of sponge cakes changed differently during the storage period. During the 6 days storage period the samples with functional ingredients preserved in better way the elastic modulus in comparison with the control sample, because they are more reach in fibre. The sponge cake, containing 20% Jerusalem artichoke powder, keeps their freshness longer time.

INTRODUCTION

The texture is one of the main characteristics of the sponge cakes that can be affected by the addition of functional ingredients. It can be determined by instrumental or sensory methods. Instrumental methods offer some advantages over sensory analysis because they are rapid and objective. Sahi & Alava (2003) studied the crumb structure of sponge cakes to evaluate the effect of different emulsifiers. Texture profile analysis of cake crumb was performed by Singh, Rosell, Sharma, & Singh, (2003) to study the effect of sodium lauryl sulphate. Kamel & Rasper (1988) investigated the effect on cake crumb firmness of preparing reduced-calorie cakes with sorbitol or polydextrose to replace sugar.

The storage stability or the shelf life of the baked products could be defined as maintenance the sensory and physical characteristics associated with the freshness such as crumb tenderness, compressibility and moistness by preventing alteration associated with staling during the storage (Baixauli, R., Salvador, A., & Fiszman, S. M., 2008). However, sensory methods are the only ones that make it possible to assess consumer acceptance. Consumers expect a product with a soft, spongy, tender crumb, but also a certain degree of resistance, not crumbling easily; these characteristics worsen during the storage and, in general, consumer rejection of the product occurs before any microbiological spoilage makes it unsuitable for human consumption

(Hough, G., Langohr, K., Gomez, G., & Curia, A., 2003). Different methods maybe used to determine the sensory characteristics of a food product using consumer data.

Different flour types have been investigated for developing cakes of lower cost and better quality in terms of consumer acceptance (Karaoglu, M.M. & Kotancilar, H.G., 2009). The baking time and temperature have an impact on the morphology and the texture of bread and on its quality (specific volume, crust colour, crust/crumb ratio, crumb firmness and moisture content). This morphology affects the kinetics of moisture transfer during the aging and, consequently, the mechanical properties. Among the different physical properties, which can be considered as characterizing the cakes, the porosity is important not only for the mechanical properties of the crumb but also for moisture transfer within the product. B1aszczak, Sadowska, Fornal, and Rosell (2004) found that during the staling, the porosity decreased and the crumb pores became smaller and rounder. A static compression mode, such as texture profile analysis (Carson & Sun, 2001) (determined using a texture analyzer), firmness (based on force-deformation), stress relaxation, penetration, and compression tests have provided data about bread crumb mechanical changes associated with the staling process (Angioloni & Collar, 2009). Other aspects related to staling may also be considered such as the loss of resilience. Goasaert, Slade, Levine, and Delcour (2009) measured the firmness and resilience of bakery products after 6 days of storage using a texturometer and concluded that an increase in crumb firmness led to a decrease in crumb resilience due to a less flexible gluten network. Instrumental and sensory evaluations on sponge cake textures have been widely performed, but it is important to establish which tests and probes are more appropriate for describing the sensory attributes of texture, thereby ascertaining which objective test correlates in best way with the sensory perception of the texture.

The objectives of this study are to compare the influence of replacing 20%, 35% and 50% of wheat flour with different functional ingredients on the textural characteristics of the sponge cake, freshly baked and stored for 6 days, and to assess the sensory characteristics of the fresh sponge cakes.

MATERIAL AND METHODS

Cake batters preparation

Standard raw materials such as wheat flour of type 500 – ash 0.5% (GoodMills, Bulgaria EAD), granulated sugar (Zaharni zavodi AD), eggs (local market) used in the current study are authorized by the Ministry of Health as manufactured in Bulgaria. The control cake was prepared, following a traditional technology and formulation (Angelov et al., 1974).

The batter formulation of the control cake was as follows (based on flour weight): egg yolk 43.23%, egg white 96.77%, refined granulated sugar 83.87%, and wheat flour 100%. In particular, a double mixing procedure was applied by partitioning whipping of whites and egg yolks. Jerusalem artichoke powder (JAP), cocoa husk powder (CHP) and einkorn wholemeal flour (EWF) were added into sponge cake flour at different levels 20, 35 and 50%, by replacing wheat flour, respectively (Table 1, Figure 1).

Each sponge cakes batter of 95 g was poured out into metallic forms and baked

in an electric oven (Rahovetz - 02, Bulgaria) at 180°C for 30 min. The sponge cakes were stored at standard conditions (at temperature of 18°C and 75 % relative humidity) up to the sixth day from production date according to standard requirements (BSS, 1982). The humidity and the temperature were kept constant by means of a desiccator supplied with psychrometer, and put in a thermostat with accuracy of ± 0.5°C.

Table 1: Sponge cake batters formulations

Ingredients	Amount based on:			
	flour weight, [%]	flour mix /wheat flour and functional ingredient / weight, [%]		
	control sample	with 20 % Jerusalem artichoke powder	with 35 % cocoa husks powder	with 50 % einkorn wholemeal flour
Yolk of egg	43.23	43.23	43.23	43.23
White of egg	96.77	96.77	96.77	96.77
Refined granulated sugar	83.87	83.87	83.87	83.87
Wheat flour type 500	100.00	80.00	65.00	50.00
Functional ingredient	-	20.00	35.00	50.00

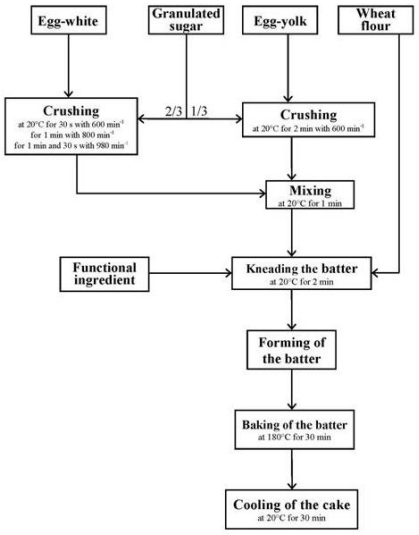

Figure 1
Technology flowsheet of the sponge cake preparing with functional ingredient

Sensory characteristics

The descriptive test for a quantitative sensory profiling was used to establish the sensory characteristics (shape, colour, cell size and uniformity, odour, sweetness, aftertaste, crumb tenderness) of the sponge cakes, 6 h after baking, following the ISO 8586:2014 and ISO 13299:2011 methods. The sponge cakes samples were ready 1 h before the evaluation. Samples of different cakes were kept in coded plates covered with aluminium foil. Twelve trained panelists were selected to guarantee the evaluation accuracy. The intensity of each sensory characteristic was recorded on a ten-point linear scale after 1 h orientation sessions of the panelists, where they specified terminology and anchor points on the scale. The coded samples were shown simultaneously and evaluated in random order among the panelists. The sensory characteristics are shown in Table 2.

Table 2: Sensory characteristics and their definitions

Sensory characteristics	Definition	Evaluation method	Scale
Shape	Visual assessment of surface and preserved form of sponge cake.	Presence of large cracks on the surface	0-3
		Numerous smaller cracks	3-6
		Without cracks, smooth upper surface	6-9
Colour	Visual assessment of the colour of the crust and crumb of sponge cake (to control crust the colour of the crust is golden brown and the crumb has a light yellow colour)	A significant discolouration	0-3
		Unevenly colour of surface and in the crumb of the cakes	3-6
		Characteristic colouring of crumb and crust	6-9
Cell size and uniformity	Visual assessment of the size and even distribution of pores in crumb crust sponge cake	Visible differences in the size and distribution of pores	0-3
		A small difference in the size and distribution of pores in the crumb	3-6
		Evenly distributed small and uniform in size pores	6-9
Odour	Rating odour during consumption of sponge cake	Presence of strong, unusual odour – pushy	0-3
		Slight uncharacteristic odour	3-6
		Characteristic pleasant odour	6-9
Sweetness	Evaluation of the sweet taste of sponge cake	Insufficient pronounced sweet taste	0-3
		Moderate sweetness	3-6
		Strong sweet taste	6-9
Aftertaste	Evaluation of the aftertaste after consumption of sponge cake	Missing aftertaste or unpleasant taste sensation (palatability)	0-3
		Presence of slightly discernible side aftertaste	3-6
		Strong, unpleasant after taste	6-9
Crumb tenderness	Evaluation of the applied compressive force required for deformation of the crumb	The need to apply more force to deformation	0-3
		Applying moderate compression force to deformation	3-6
		Use less force to deformation	6-9

Textural characteristics

The textural parameters of the sponge cakes were investigated by texture analyzer Stable Micro Systems XT2Ai equipped with loading cell 50 kg and specialized software „Texture Exponent". The software allows force calibration and sample height calibration before the measurements. The instrument works in the deformation range 0 - 500 mm (with resolution 0.001 mm) and the force range 0 - 500 N (resolution: 0.001 N, minimal measured force: 1 N).

The sample preparation includes measurement of the sample sizes (length, width and height) by digital calliper, measurement of the sample weight by laboratory scales.

1. Investigation of the sponge cake elasticity: The test was done at low deformation rate 1mm/s till 20% strain was achieved using a cylinder probe with diameter 50 mm. The elastic modulus (ε, kPa), defined as the slope of the linear part in the stress/strain curve was determined from this measurement.
2. Determination of the sponge cake hardness by rupture test (imitation of the biting process). The test was done at high deformation rate 5 mm/s using a thin cylinder probe with diameter 5 mm till 40 mm deformation. The stress (σ, kPa) of the rupture point was determined from this test.
3. Cutting of the sponge cakes. The test was done with a "knife" probe, which is wider and higher than the sizes of the cake. The cutting was done till 100% of the sponge cake height with cutting rate 2 mm/s. The maximum cutting force (F, N) was determined.

Physico-chemical characteristics

For the determination of the sponge cake structure, optical photographs were taken of the top surface and of the cross sections of the half-cut cake**.**

Determination of dietary fibre

The total, soluble and insoluble dietary fibre content was determined by the enzymatic-gravimetric method AOAC 985.29, using the total dietary fibre assay kit TDF 100A (Sigma-Aldrich) and the instructions provided by the manufacturer.

Determination of total fat

The total fat content was determined by the Randall extraction method (ISO 11085:2008).

Data analysis

Depending on the type of the studied characteristic from 3 to 12 repetitions of each measurement were done. For the assessment of the measured results accuracy a statistical method with level of significance $p \leq 0.05$ was used. The data were analyzed and presented as average values ± standard deviation.

RESULTS AND DISCUSSION

Sensory analysis

The results of the sensory analysis suggested that the addition of functional ingredients in the cake formula interfere positively on the product acceptability, in fact the samples with the 50% einkorn wholemeal flour showed the highest value of "overall acceptability". It was observed that the higher crumb tenderness scores for control resulted an increase in the overall liking values. The cakes had similar shape with an exception for the cake with 35% cocoa husks powder (Figure 2). This cake had the smallest height, and its surface had visible cracks (Figure 3).

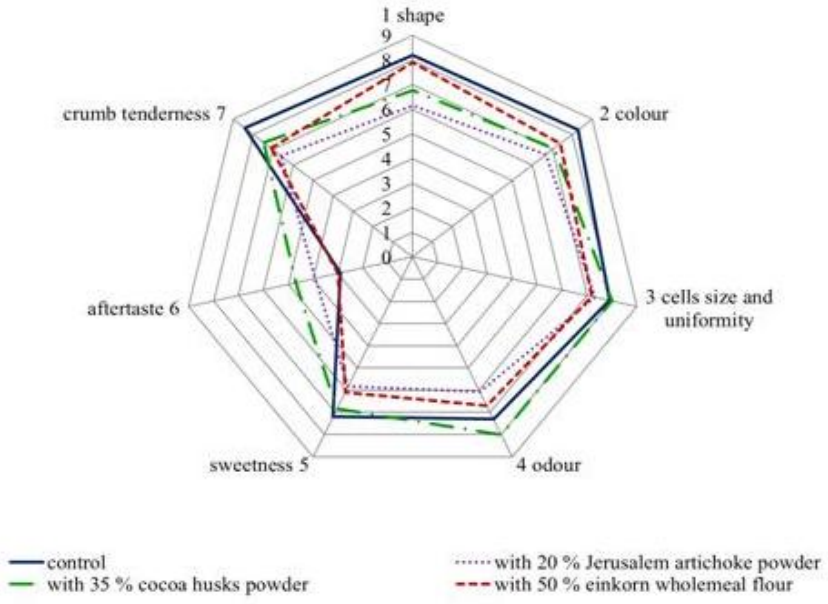

Figure 2
Sensory profiles of sucrose-sweetened sponge cakes*
*A scale from 0 to 9 was used to evaluate sensory characteristics. Nine is ideal for the third sensory characteristic when the cells are small and equal in size.

a) Without an additive control cake-sample	**b)** With an additive of 20 % Jerusalem artichoke powder;	**c)** With an additive of 35 % of cacao husks powder;	**d)** With an additive of 50 % einkorn wholemeal flour.

Figure 3
Photographs of top surface and of cross sections of the fresh

sucrose-sweetened sponge cakes

The crumb pore cells of the cakes with 35% cocoa husks powder had thicker walls, and they were larger and equal in size (Figure 2 and Figure 3). The cells of the sponge cake-sample were smaller and almost uniformly distributed in the crumb, with thinner walls. The cake-samples had a crust and crumb with more pronounced light-yellow colour due to the presence of the color components in the yolks of the egg (carotenoids). The colour of the crust and crumb of the cakes with 35% cocoa husks powder was brown, of cakes with 50% einkorn wholemeal flour is light-yellow - light-brown, cakes with 20% Jerusalem artichoke powder – gray brown (Figure 3). The odour of the cakes with 20% Jerusalem artichoke powder was more strongly expressed and more specific towards the control sample odour, and was not perceived by the sensory panelists as unpleasant. The intensity of the sweetness for all investigated sponge cakes is close, but when the adding of the functional ingredients is greater and aftertaste was read.

Instrumental texture

The elastic modulus (Figure 4) for all the investigated cakes was very similar on the first day and increased during the storage. The increasing was the smallest for the control cake and the highest for the sample with 50% einkorn wholemeal flour.

The hardness (puncture stress) of the cakes is shown on Figure 5. The hardness of the control cake had a very small decreasing. For sponge cakes containing functional ingredients, the hardness increased during the sixth day storage. The increasing was the highest for the sponge cakes with 20% Jerusalem artichoke powder and 50% einkorn wholemeal flour and the smallest for cake with 35% cocoa husks powder.

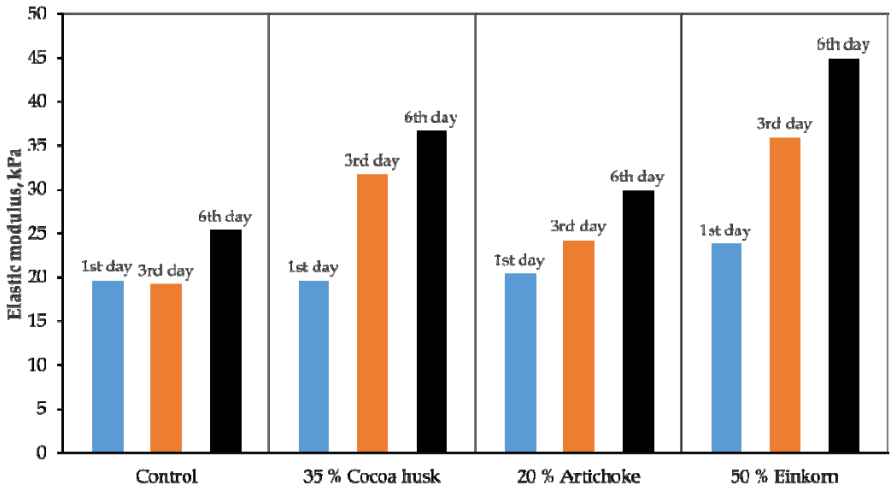

Figure 4
Elastic modulus of sponge cakes during the storage

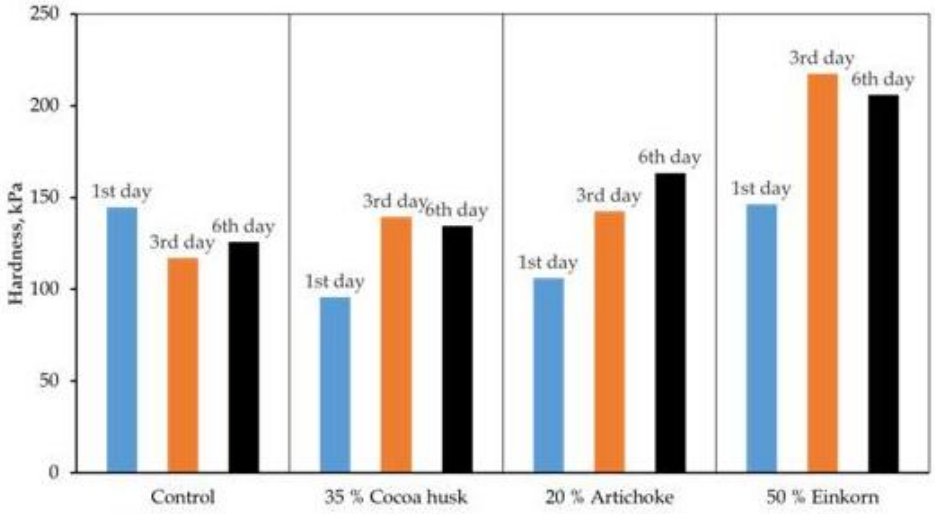

Figure 5
Puncture test (hardness) of sponge cakes during the storage

The changes of the cutting force is shown on Figure 6. The cutting force of the control sample showed an increasing during the storage. The samples with 35% cocoa husk powder and 20% Jerusalem artichoke powder showed the highest cutting force on the 3rd day, and after that smaller like on the first day, because they became to crackly texture.

The observed differences in the textural properties of sponge cakes with functional ingredients are due to their different composition.

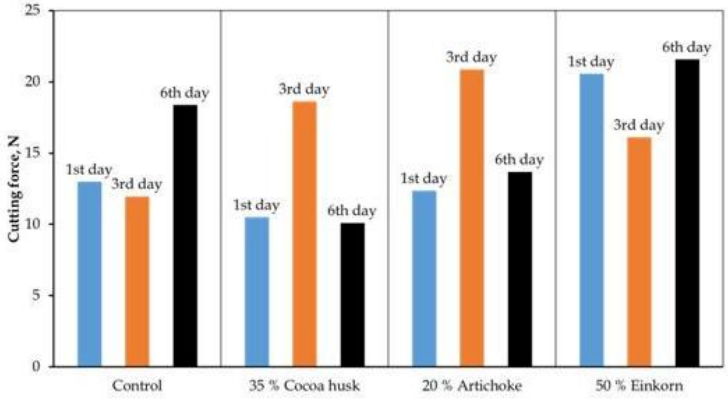

Figure 6
Cutting force pf sponge cakes during the storage

The sponge cake with 50% einkorn wholemeal flour contains large amounts of fiber, most of which are insoluble (Table 3). As a result, the cake is characterized by the highest elasticity and hardness. During the storage the elasticity and the hardness grow fastest.

The sponge cake with 35% cocoa husk powder contains the greatest amount of insoluble fiber and fat (Table 3). Perhaps the larger amounts of fat in the cake with 35% cocoa husk powder causes less elasticity and hardness of the cake crumb in comparison to the sponge cake with 50% einkorn wholemeal flour.

The sponge cake with 20% Jerusalem artichoke powder contains large amounts of soluble fibre (Table 3). As a result, the elasticity of the cake is retained to the greatest extent during the storage (i.e., the changes are minimal).

Table 3: Average values of the dietary fiber and of the total fat of the sponge sucrose-sweetened cake samples without and with functional ingredient (per 100 g product)

Type of sponge sucrose-sweetened cake	Dietary fiber, [%]			Total fat, [%]
	insoluble	soluble	total	
Control	1.56±0.07	0.45±0.51	2.01±0.58	6.32±0.03
With 20 % Jerusalem artichoke powder	2.24±0.05	1.43±0.58	3.67±0.63	4.06±0.27
With 35 % cacao husks powder	3.97±0.03	0.34±0.39	4.31±0.42	7.03±0.12
With 50 % einkorn wholemeal flour	2.73±0.06	0.48±0.27	3.21±0.33	6.79±0.11

CONCLUSIONS

The texture is the most important parameter in the product design because the texture and food matrix are linked to the micro- and macrostructural composition of foods, which determines the sensory perception. Any change in the formulation or processing directly influence the structural composition of the foods. The biggest challenge in the food manufacturing is to produce food products with a consistent high quality. The influence of the ingredients on slowing down starch retrogradation was initially tested by instrumental methods and later correlated to the results of a sensory panel.
On the first day of storage the greatest cutting force is applied to the sponge cake with 50% einkorn wholemeal flour, i.e. it has the strongest internal resistance, and the less power is used for cutting the cake with 35% cocoa husks powder. During the storage, the smallest change was recorded in the cakes containing more dietary fiber – with 35% cocoa husks powder and with 50% einkorn wholemeal flour.

The addition of 50% einkorn wholemeal flour in sponge cake produced a harder texture: the samples were harder than the others and showed higher elastic modulus. During the 6 days storage period the samples with functional ingredients remained stable elastic modulus not like the control sample.

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THE DETERMINATION OF THE EFFECTS OF SOAKING AS A PRETREATMENT FOR SOYBEANS

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Keywords:

Pre-treatment, soaking, soybeans, weight gain, solid loss

Abstract. In this study, it was intended to determine the soaking behaviour of soybeans. Soybeans are legumes, which, as a family are rich in proteins, carbohydrates, dietary fiber, vitamins, and minerals. In order to obtain the edible form of legumes, they are processed through pre-treatments such as soaking, grinding and cooking. The soaking process, which is one of the most important pre-treatments, results in gaining water, easiness of the cooking, and reduction of the cooking time. In this study, the effects of different soybean:water ratios (1:2.5, 1:5, 1:10, and 1:20 (weight:weight) (w:w)) and the salt concentrations (1, 2, and 5%) and temperatures of the soaking medium (4, 25, and 80°C) on the total soluble solid content of soaking medium and colour values of the soybeans were examined with their effects on the weight gain of soybeans. According to the results of the analyses, a higher rate of weight gain values was observed for the experiments performed at 80°C. In general, the total soluble solid contents of the soaking medium, which indicates the loss of solids, were found to be inversely proportional to the amount of water both for the experiments in water and brine.

INTRODUCTION

Soybeans (*Glycine max*) being a high protein legume, can be processed into many food products which have advantages of cost and utilization, when compared to most foods and proteins of animal origin (Beleia et al., 2006). It is an important crop which is used in human and animal nourishment with % 18-20 oil and % 40-45 protein content (Unakıtan & Aydın, 2012).

The soaking process which is one of the most important pre-treatments, results in gaining water, easiness of the cooking, and reduction of the cooking time. Main components of the solid lost into the soaking and cooking water are known to be carbohydrates and proteins (Wang et al., 1979). On the other hand, the solid loss has the advantage of losing some antinutritional factor such as phytates, enzyme inhibitors (trypsin, chymotrypsin and a-amylase) and hemaglutinins, which are primary reasons for the discomfort associated with consuming legumes (Abd El-Hady & Habiba, 2003). However, high solid loss during soaking and cooking of legumes decreases the nutritional and economic value of the final product (Unakıtan & Aydın, 2012). The loss was

reported to be between 2 and 19% (wet basis, wb) of the total solid, depending on the water temperature, type of seeds and physiochemical defects on seeds (Agustin et al., 1989; Kon, 1979; Seena &Sridhar, 2005).

Temperature increases the rehydration rate but usually does not play a significant effect on rehydration capacity. The effect of temperature on rehydration rate is due both to the decreased viscosity of the immersion medium and to the effects of temperature on the food material structure. Temperature effects are usually described by an Arrhenius-type relationship (Ilincanu et al., 1997; Thakor et al., 1995).

In this work, the aim was to determine the soaking behaviour of soybeans. According to the experimental data the moisture content, rehydration rate, colour changes and the total soluble solid content changes of the soaking medium were determined depending on the changes of soybean:water ratio, salt concentration and temperature.

MATERIAL AND METHOD

Material

The soybeans in dried, commercial form were obtained from a local market in Izmir, Turkey.

Methods

The moisture content of the soybeans and rehydrated soybeans was determined according to AOAC, 2000. The colour values of the soybeans (L*, a*, and b* values) were measured with the Minolta CR-400 Colorimeter, Japan and the results were expressed in accordance with the CIE Lab. System.

Rehydration experiments were carried out in distilled water at refrigeration temperature (4±1°C), room temperature (25±1°C) and in hot water (80±1°C). The samples (10±0.5g) were weighed in a string bag and placed inside a beaker. For the experiments, the soybean:water ratios were adjusted to 1:2.5, 1:5, 1:10, and 1:20 (w:w). To observe the effects of different salt concentrations, brines of 0%, 1%, 2%, and 5% salt concentrations (w:w) were prepared and at every soybean:water ratio the experiments were repeated.

The samples were removed from water, drained, and their weight was measured for every 15 min for the first hour of the rehydration, then repeated every 30 min until the end of the 6th hour . After this, samples were weighed every hour until the 10th hour of the rehydration. The rehydration ratio of the samples were determined by using Eq. (1) (Filli & Nkama, 2007) and the weight gain calculations were done by using Eq.(2) (Svihus et al., 1997).

The total soluble solid content (TSSC) of the rehydration medium was determined by digital refractometer (RFM 330, UK).

The data was analyzed using the statistical software SPSS 21.0 (SPSS Inc., USA). The data was subjected to an analysis of variance (ANOVA) which was used to determine the difference between means. The drying and rehydration experiments were replicated twice and all the analyses were triplicated

$$Rehydration\ Ratio - \frac{Weight\ of\ the\ rehydrated\ material\ (kg)}{\textbf{Weight of the initial material (kg)}} \quad (1)$$

$$Weight\ Gain(\%) = \frac{\left[\left(Weight\ of\ the\ rehydrated\ material\ at\ a\ time(kg)\right) - \left(\textbf{Weight of the initial material}\ (kg)\right)\right]}{\textbf{Weight of the initial material (kg)}} x100 \quad (2)$$

RESULTS AND DISCUSSION

The legumes are sold in dry form and they have to be processed prior to consumption (Agustin et al., 1989; Vidal-Valverde et al., 2002). Soaking is an integral part of a number of treatments, such as cooking, canning, germination, and fermentation. It consists of hydration of the seeds in water, usually until they reach maximum weight, with or without discarding of the soaking medium, and the results obtained depend of factors such as legume genus, species and variety, process duration, temperature, pH, salinity of the soaking media, and also the storage conditions undergone before processing (Prodanov et al., 2002). The initial moisture content of the soybeans was found to be 39.95% (wet basis, wb). The results of the moisture content of analysis for rehydrated soybeans are given in Table 1.

The results showed that the moisture content values of the rehydrated soybeans, generally increases with the increasing water to soybean ratios and temperature of the soaking medium. The increase of water to soybean ratio beyond 5:1 (w:w) significantly affects the moisture content values compared to the ratio of 2.5:1 (w:w) ($P<0.05$), however, further increase does not have any significant effect ($P>0.05$). Similarly, the effects of temperature and salt concentration of the soaking medium were found to be significantly affecting the moisture content ($P<0.05$). Considering the same soybean to water ratio values, increase of temperature leads to an increase of the moisture content values due to the increased rate of molecular movement and possible structural deformations at high temperatures. Depending on the results of the moisture content determination, the salt concentration of the soaking medium and the moisture content are inversely proportional whereas, the ratio of water:soybean and the moisture content values are directly proportional.

The initial color values (L*, a*, and b*, CIE Lab System) of the soybeans were found to be 71.19 ± 2, 2.13 ±0.06, and 9.7 ±0.14 respectively. As shown in Fig. 1, the a* values of the rehdyrated soybeans decreased, however, the b* value fluctuated during soaking pretreatment. The brightness values of the rehydrated soybeans were not significantly affected the soybean:water ratios ($P<0.05$). The results of the colour measurements indicated that the brightness (L*) values of the soybeans are increasing depending on the salt concentrations for 25°C and 80°C, however, the values at 4°C shows a decrease with salt concentrations. On the contrary, considering a* values it was observed that the L* values were increasing with increasing salt concentration for the experiments carried on for 4 and 25°C, however, a decreasing trend in general was observed for 80°C. The measured b* values were changing quite much so that no tendency was estimated (The data were not given).

The rehydration ratio values based on the increase of weight of the soybeans are shown in Table 2. The rehydration ratio values of the soybeans were found to be in range between 2.34 and 2.89 for the experiments performed in destilled water.

Table 1: The moisture content (wet basis) values of the rehydrated soybeans

Moisture Content			
Soybean:Water Ratio / Temperature (°C)	**Salt Concentration (%)**	**1:2.5**	**1:5**
4	**0**	54.86 ± 1.19^{axk}	60.76 ± 1.62^{byk}
	1	54.96 ± 1.00^{axk}	58.61 ± 0.36^{byk}
	2	$51.49\pm0.26^{a(xy)k}$	58.70 ± 2.87^{byk}
	5	47.62 ± 1.83^{azk}	51.77 ± 0.26^{bxk}
25	**0**	56.25 ± 0.43^{atk}	61.17 ± 0.76^{bzk}
	1	55.12 ± 0.97^{azk}	59.17 ± 0.42^{bxk}
	2	53.36 ± 0.32^{ayk}	$59.67\pm0.45^{b(xy)k}$
	5	$49.32\pm0.27^{ax(kl)}$	60.16 ± 0.23^{byk}
80	**0**	62.33 ± 0.11^{ayl}	62.76 ± 1.92^{axk}
	1	55.26 ± 2.24^{ayk}	61.50 ± 0.60^{bxl}
	2	56.07 ± 1.79^{ayk}	61.46 ± 0.57^{bxk}
	5	52.94 ± 0.55^{axl}	60.16 ± 0.41^{bxk}

Moisture Content			
Soybean:Water Ratio / Temperature (°C)	**Salt Concentration (%)**	**1:10**	**1:20**
4	**0**	60.41 ± 0.07^{bzk}	60.18 ± 0.72^{byk}
	1	59.61 ± 0.02^{czk}	60.18 ± 0.57^{cyk}
	2	57.34 ± 0.58^{byk}	57.97 ± 0.75^{byk}
	5	51.16 ± 0.46^{bxk}	50.06 ± 1.04^{bxk}
25	**0**	61.36 ± 0.05^{byk}	61.88 ± 0.56^{bxk}
	1	60.14 ± 0.47^{bxk}	61.29 ± 0.27^{cxk}
	2	$61.00\pm0.39^{c(xy)l}$	61.32 ± 0.17^{cyl}
	5	62.45 ± 0.14^{czk}	62.89 ± 0.10^{dzl}
80	**0**	63.09 ± 0.75^{axk}	63.85 ± 0.28^{azl}
	1	63.84 ± 0.38^{cxl}	63.28 ± 0.25^{czl}
	2	63.28 ± 0.34^{cxm}	$62.17\pm0.41^{(bc)ym}$
	5	60.11 ± 0.40^{bxk}	61.34 ± 0.29^{cxl}

$^{a–d}$ shows significant difference in the samples according to soybean:water ratio ($P<0.05$).
$^{x-t}$ shows significant difference in the samples according to salt concentration ($P<0.05$).
$^{k-m}$ shows significant difference in the samples according to temperature ($P<0.05$).

Table 2: The calculated values of rehydration ratios for soybeans

Rehydration Ratio			
Soybean:Water Ratio / Temperature (°C)	**Salt Concentration (%)**	**1:2.5**	**1:5**
4	**0**	2.34 ± 0.07^{ayk}	2.88 ± 0.13^{byk}
	1	2.31 ± 0.06^{axk}	2.75 ± 0.11^{cyl}
	2	2.27 ± 0.08^{axk}	2.60 ± 0.03^{cyl}
	5	2.34 ± 0.10^{ayk}	2.41 ± 0.06^{bxk}
25	**0**	2.35 ± 0.16^{ayk}	$2.60\pm0.17^{(ab)yk}$
	1	2.58 ± 0.12^{ayk}	2.73 ± 0.02^{azl}
	2	2.18 ± 0.15^{axk}	2.36 ± 0.09^{bxk}
	5	2.42 ± 0.12^{byk}	2.43 ± 0.14^{ayk}
80	**0**	2.42 ± 0.07^{axk}	$2.50\pm0.10^{(ab)xk}$
	1	2.50 ± 0.12^{axk}	2.48 ± 0.16^{axk}
	2	2.33 ± 0.05^{axk}	2.40 ± 0.07^{bxk}
	5	2.38 ± 0.04^{axk}	2.52 ± 0.03^{bxk}

Rehydration Ratio			
Soybean:Water Ratio / Temperature (°C)	**Salt Concentration (%)**	**1:10**	**1:20**
4	**0**	2.89 ± 0.13^{btm}	2.79 ± 0.12^{byk}
	1	2.67 ± 0.03^{bzl}	2.65 ± 0.07^{byl}
	2	2.58 ± 0.05^{byk}	$2.61\pm0.12^{c(xy)k}$
	5	2.49 ± 0.05^{cxl}	2.49 ± 0.10^{cxk}
25	**0**	$2.64\pm0.06^{(ab)zl}$	2.77 ± 0.11^{czk}
	1	2.50 ± 0.02^{ayk}	2.38 ± 0.09^{axk}
	2	2.53 ± 0.07^{byk}	2.54 ± 0.15^{byk}
	5	2.20 ± 0.09^{axk}	2.48 ± 0.11^{cxk}
80	**0**	2.50 ± 0.06^{axk}	2.66 ± 0.06^{bxk}
	1	2.50 ± 0.11^{axk}	2.69 ± 0.18^{byl}
	2	2.56 ± 0.15^{dyk}	2.53 ± 0.11^{cxk}
	5	2.86 ± 0.03^{dzm}	2.72 ± 0.17^{cyl}

$^{a-d}$ shows significant difference in the samples according to soybean water ratio ($P<0.05$)
$^{x-t}$ shows significant difference in the samples according to salt concentration ($P<0.05$)
$^{k-m}$ shows significant difference in the samples according to temperature ($P<0.05$)

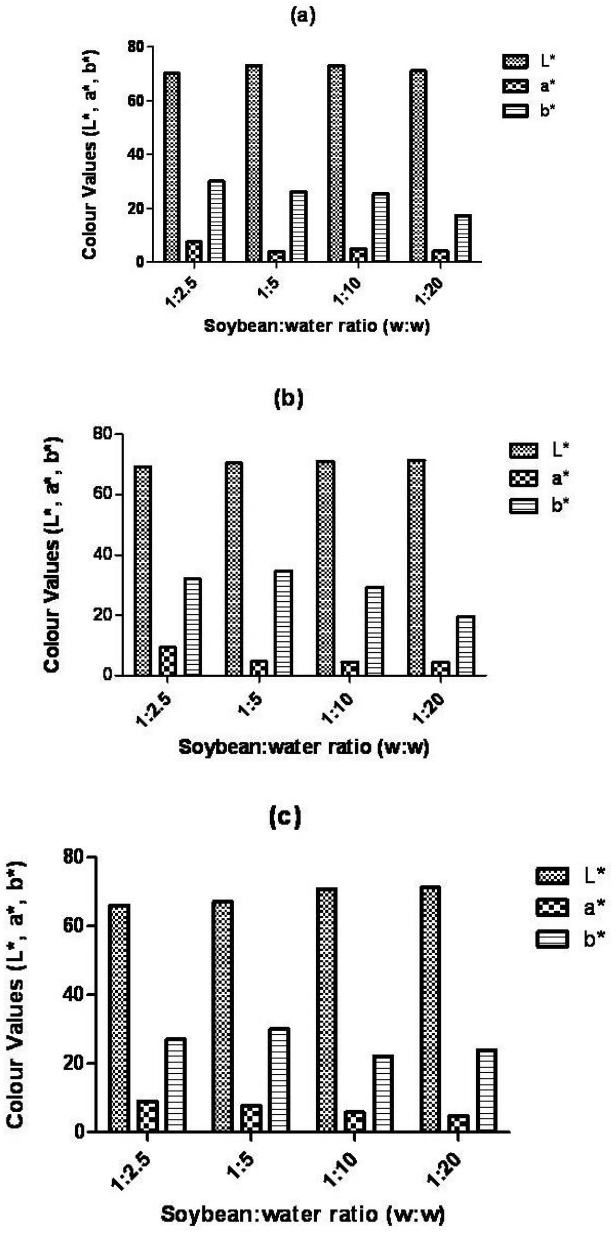

Figure 1
The colour values of rehydrated soybeans at 4°C (a), 25°C (b) and 80°C (c)

The results showed that, the rehydration ratio values of the soybeans were significantly affected by the soybean:water ratio ($P<0.05$) and higher rehydration ratio values were evaluated for 4°C soaking medium temperature. The rehydration ratio of the soybeans ranged between 2.18 and 2.86 for the experiments performed in different salt concentrations brines. The highest rehydration ratio was observed in 80°C soaking medium temperature, 5% salt concentrated, 1:10 soybean:water ratio. The results showed that, the rehydration ratio values of the soybeans were significantly affected by the salt concentrations, temperature of soaking medium and soybean:water ratio values ($P<0.05$).

The weight gain values (%) calculated based on the water absorption of the rehydrated soybeans are shown in Fig. 2. The results indicate that the weight gain occurred rapidly at the initial stage of rehydration and after a while, it started to be stationary due to filling of capillaries on the surface of the samples. After filling of the free capillaries and intercellular spaces of sample with water, water uptake started to decrease and became constant. According to Fig. 2, it was observed that the samples processed at all temperatures reached an equilibrium. The change of weight gain (%) values at 4°C and 25°C are similar in their trends, however, higher values were observed for the rehydration at 4°C. On the other hand, increasing the rehydration medium temperature to 80°C increased the rate of rehydration at the initial period and the equilibrium values attained at a very short time periods. The results showed that the weight gain values (%) observed for the soybeans soaked in brine were the same with the ones in water for the experiments at 80°C, however, significantly lower values were observed for 4 and 25°C at the end of the rehydration experiments ($P<0.05$) (The data were not given).

The results of the total soluble solid content measurements are shown in Table 3. During the soaking pretreatment of soybeans, a colour change was observed in the process water indicating soluble matter loss. While soaking pretreatment, some valuable compounds of legumes are leached out to the soaking medium and this decreasing the overall quality and desirability of the final product. The lowest total soluble solid content of rehydration medium was found in 25°C soaking medium temperature and 1:20 soybean:water ratio for the experiments at distilled water. Compared to other rehydration temperatures, higher values of TSSC of rehdyration medium was observed in 80°C soaking medium temperature for destilled water, most probably due to the structural deformation in interior structure. In general, higher values of TSSC were observed for the rehydration experiments in brine solutions. The analysis of the statistical evaluation showed that the soybean:water ratio, salt concentration of the brine, and the temperature of the soaking medium were significantly affecting the TSSC values of the samples ($P<0.05$).

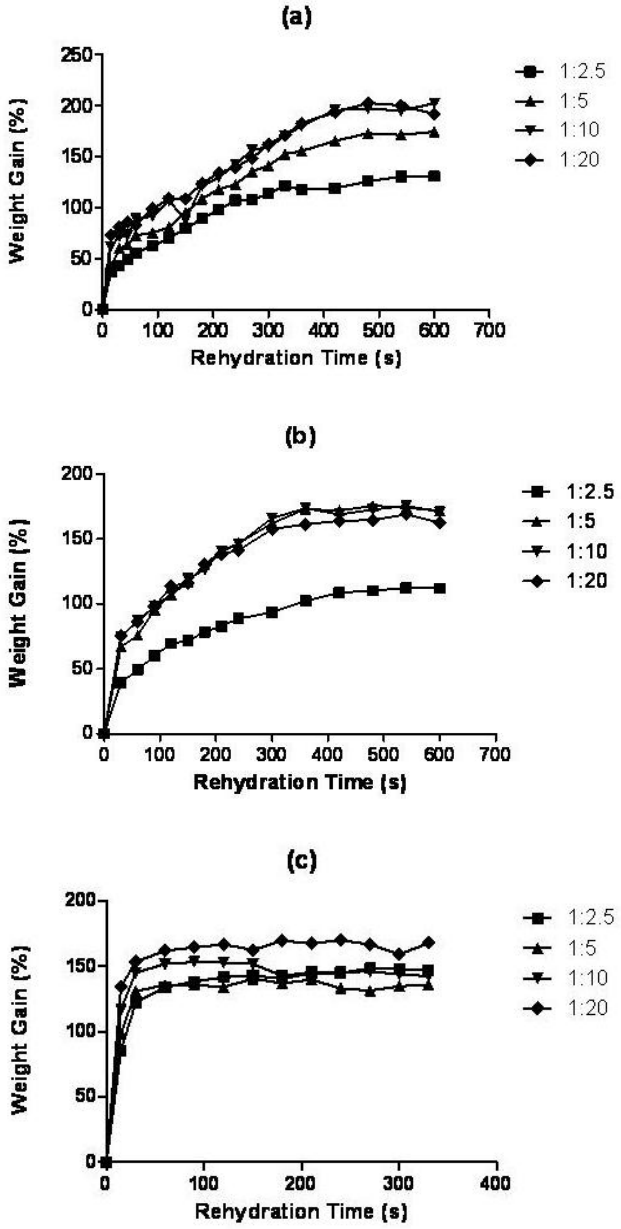

Figure 2
Weight gain (%) of rehydrated soybeans at 4°C (a), 25°C (b) and 80°C (c)

Table 3: The Total Soluble Solid Content (TSSC) (°Brix) values for rehydration medium

	Total Soluble Solid Content		
Soybean:Water Ratio / Temperature (°C)	**Salt Concentration (%)**	**1:2.5**	**1:5**
4	**0**	3.23 ± 0.26^{cxk}	2.10 ± 0.24^{bxk}
	1	4.20 ± 0.24^{cyl}	2.60 ± 0.08^{byk}
	2	5.40 ± 0.18^{czl}	3.90 ± 0.19^{bzk}
	5	8.00 ± 0.22^{ctl}	6.30 ± 0.26^{vtk}
25	**0**	3.13 ± 0.19^{cyk}	1.73 ± 0.12^{bxk}
	1	2.30 ± 0.13^{bxk}	2.60 ± 0.16^{cyk}
	2	4.60 ± 0.25^{dzk}	3.70 ± 0.06^{czk}
	5	7.70 ± 0.15^{dtk}	6.50 ± 0.13^{ctk}
80	**0**	4.83 ± 0.19^{bxl}	$3.27\pm0.63^{(ab)xl}$
	1	8.80 ± 0.26^{dym}	5.40 ± 0.12^{cyl}
	2	10.5 ± 0.17^{dzm}	6.50 ± 0.17^{czl}
	5	13.5 ± 0.14^{dtm}	9.40 ± 0.21^{ctl}

	Total Soluble Solid Content		
Soybean:Water Ratio / Temperature (°C)	**Salt Concentration (%)**	**1:10**	**1:20**
4	**0**	1.00 ± 0.16^{axk}	0.60 ± 0.14^{axl}
	1	2.00 ± 0.15^{ayk}	1.60 ± 0.14^{ayk}
	2	3.00 ± 0.16^{azl}	2.90 ± 0.11^{azk}
	5	6.00 ± 0.12^{btl}	5.80 ± 0.03^{atk}
25	**0**	1.73 ± 0.31^{bxl}	0.33 ± 0.05^{axk}
	1	2.20 ± 0.03^{byk}	1.50 ± 0.10^{ayk}
	2	3.10 ± 0.12^{bzl}	2.70 ± 0.17^{azk}
	5	6.10 ± 0.10^{btl}	5.90 ± 0.14^{atk}
80	**0**	$2.87\pm1.45^{(ab)xm}$	0.97 ± 0.17^{axl}
	1	3.30 ± 0.28^{bxl}	2.10 ± 0.11^{ayl}
	2	2.00 ± 0.21^{axk}	3.20 ± 0.13^{bzl}
	5	5.30 ± 0.25^{ayk}	6.60 ± 011^{btl}

$^{a-d}$shows significant difference in the samples according to soybean water ratio ($P<0.05$).
$^{x-t}$ shows significant difference in the samples according to salt concentration ($P<0.05$)
$^{k-m}$ shows significant difference in the samples according to temperature ($P<0.05$).

The total soluble solid content of the soaking medium was found to be proportional to salt concentration of soaking medium. In a study by Sayar et al. (2011), it was reported that the TSSC was proportional to salt concentration of soaking medium and solid loss of the chickpea samples as 2.5% at 20°C for 900 min soaking and 10.2% at 100°C for 60 min cooking where solid loss was determined by drying the whole seeds before and after the soaking treatment. In the same study it was reported that the main compounds lost to the soaking or the cooking medium are known to be carbohydrates and proteins. Therefore, the data for solid loss can be used as an indicator of the quality for soaked and/or cooked legumes.

CONCLUSION

In this study, the moisture content, colour change, rehydration behaviour of the soybeans and total soluble solid content increase in the rehydration medium were determined both in water and brine as the soaking medium. The highest moisture content value was observed for 1:20 soybean:water ratio at 80°C for water and similar values were observed at all salt concentrations for 1:10 and 1:20 soybean:water ratio values at rehydration medium temperature of 80°C. The rehydration ratio generally increased with the increase of rehydration water amount (w:w) and the highest rehydration ratio was observed for 1:10 soybean:water ratio at 4 °C. The TSSC values (°Bx) of the soaking medium were inversely proportional to amount of water and generally proportional to the soaking medium temperature. The results in general showed that 1:10 soybean:water ratio is more appropriate for the soaking pre-treatment due to efficient use of water, requirement of lower temperatures leading to energy efficiency and obtained compearable results at lower salt concentrations.

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EFFECT OF EMULSIFIERS OF THE STARCH GELATINIZATION IN SPONGE CAKE BATTER

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Abstract. The present research investigated the effect of three types of emulsifiers on the starch gelatinization in sponge cake batter. The method of differential scanning calorimetry (DSC) was applied to determine the changes in the gelatinization temperature and the endothermic transition enthalpy during the baking of sponge cake batters. The following 4 samples were examined – control sponge cake, prepared by traditional recipe, sponge cakes with added 1% of one of the following emulsifiers-polyglycerol monostearate ester (E475), sucrose stearate ester (E473) and modified inulin palmitate ester (HP-25). It was found that the presence of emulsifier affected both the gelatinization temperature and the phase transition enthalpy. The starch gelatinization in the control was carried out in the lowest temperature range and was characterized with the lowest transition energy (enthalpy of the endothermic transition) - 1.78 J/g. The addition of emulsifiers let to narrowing the temperature range where the gelatinization is observed and to increasing the gelatinization temperature and the transition energy. The most pronounced effect was observed for the samples, which were with the sucrose stearate ester. The transition energy in this case was 2.68 J/g. The results obtained confirmed the emulsifiers retard the starch gelatinization in sponge cake batter.

Keywords:

sponge cake batter,
emulsifiers,
starch gelatinization,
DSC thermograms

INTRODUCTION

The degree of water absorption in starch determines the change of the foamed mixture into a stable porous structure during baking in the production of sponge cake (Baeva, Terzieva, & Panchev, 2003; Guadarrama-Lezama, Carrillo-Navas, Pérez-Alonso, Vernon-Carter, & Alvarez-Ramirez, 2016; Handleman, Conn, & Lyons, 1961; Muzukoshi, 1985). The incorporation of emulsifiers in the batter composition affects the degree of starch gelatinization (Chevallier, Colonna, Della Valle, & Lourdin, 2000; Gomez, et al., 2004; Kohajdová, Karovičová, & Schmidt, 2009). The emulsifiers control this process by shifting the starch gelatinization temperature to higher values (Buck & Walker, 1988; Derby, Miller, Miller, & Trimbo, 1975; Handlema et al., 1961; Kim & Walker, 1992; Richardson, Langton, Bark, & Hermansson, 2003). Some studies (Buck, & Walker, 1988; Eliasson, 1985;

Richardson et al., 2003) report the application of emulsifiers such as monoglycerides, diglycerides and polyglycerol esters, which affect the initial temperature of gelatinization.

The amount of added emulsifiers from in the concentration range of 1 to 3% from the weight of the flour increases the gelatinization temperature from 67 °C to 86 °C (Buck, & Walker, 1988; Eliasson, 1985). It has been shown (Eliasson, 1985; Kim & Walker, 1992; Marcotte, Sablani, Kasapis, Baik, & Fustier, 2004; Nunes, Moore, Ryan, & Arendt, 2009; Richardson, Kidman, Langton, & Hermansson, 2004; Sahi & Alava, 2003) that this effect is due to the emulsifier's ability to absorb water and to bind to the starch macromolecular chains in a way, similar to the sucrose (Baeva, Terzieva, & Markov, 1997; Chevallier, Colonna, Della Valle, & Lourdin, 2000; Perry & Donald, 2002; Spies & Hoseney, 1982; White & Lauer, 1990; Wootton & Bamunuarachchi, 1980).

The complex between the emulsifier and the amylose is described as an amylose helix formation around the hydrophobic part of the emulsifier (Richardson et al., 2004). The use of emulsifiers with a higher degree of polymerization significantly increased the retarding effect on the starch gelatinization (Sahi & Alava, 2003). The retarded starch gelatinization increases the extent of air bubbles expansion, which determines the formation of the porous structure in the baked product (Baeva et al., 2003; Guadarrama-Lezama et al., 2016; Handleman et al., 1961; Muzukoshi, 1985). Thus, the addition of emulsifiers to the bakery products leads to specific product features - extending the shelf life of bread and bakery products, reducing the stickiness of the batter and controlling the rheological properties of batter (Adheeb Usaid, Premkumar, & Ranganathan, 2014; Fessas & Schiraldi, 2000; Gomez, et al., 2004; Kohajdová et al., 2009; Münzing, 1991; Nunes et al., 2009).

The aim of the present research is to investigate the effect of different kinds of emulsifiers on the starch gelatinization in sponge cake system using the method of the differential scanning calorimetry.

MATERIALS AND METHODS

Sponge cake preparation

Standard raw materials such as wheat flour of "Type 500" – ash 0.5% (GoodMills, Bulgaria EAD), granulated sugar (Zaharni zavodi AD), eggs (local market), glycerol, water and emulsifiers used in the current study are authorized by the Ministry of Health as manufactured in Bulgaria. Three different emulsifiers (food surfactants or surface active agents) are considered as optional additives and are used as batter and cake improvers: polyglycerol monostearate E475 (Radiamuls Poly 2248K) (Oleon Group, Belgium), sucrose stearate E473 (Sisterna SP70-C) (SISTERNA, Netherlands) and modified inulin palmitate (HP-25), produced by University of Food Technologies, Department of Organic Chemistry and Microbiology, Bulgaria. Emulsifiers are added to the formulation at 1% (amount based on the batter).

The batter formulations of the studied sponge cakes are given in Table 1. The mixture for the control sponge cake (batter-control) was prepared following a traditional technology and formulation (Angelov, Bekirov, Genadieva, & Atanasov, 1974), according a double-bowl mixing procedure. The recipe for making batter included preliminary separation of egg whites and yolks. The batter with the

addition of surfactant as an emulsifier gel is prepared by a single-bowl mixing process of the components.

The sponge cakes were baked in a metallic pan containing 75 g of batter and placed in an electric oven (Rahovetz -02, Bulgaria) for 30 min at 180 °C.

Physical characteristics of the batters and cakes

The thermal properties of the sponge batter were characterised by means of a differential scanning calorimeter DSC 204F1 Phoenix (Netzsch Geräebau GmbH, Germany). The calorimeter was calibrated by indium standard. Sponge cake batter samples (10 - 15 mg) were closed hermetically in aluminium pans and heated in the calorimeter from 20 to 150 ^{o}C at a rate of 5^{o}C/min. These conditions are similar to those of the center of the batter during the baking. The temperatures and the enthalpy of the thermal transitions were determined with the use of instrument's software Proteus Analysis (Netzsch, Germany). The enthalpy values were expressed as J/g.

The moisture content in the batter and the crumb cake (2 h after baking) was determined using the AACC method 44–11 (AACC, 2000) after drying out in an oven at 105 °C to constant weight. The measurements were done in triplicate and the mean values were presented.

The water activity of the batter and the crumb samples was determined using a Novasina AG CH-8853 water activity meter (Zurich, Switzerland) at 20 °C. The samples were put in sample cups and hermetically covered before analysis to avoid moisture loss or gain. The a_w-metre was calibrated with a saturated sodium chloride (NaCl) salt solution. The water activity was measured in triplicate on cake batters and crumb cakes.

For the determination of the sponge cake crumb structure, optical photographs were taken of the cross sections of the half-cut cake.

Data analysis

All analyses were conducted in triplicate and the average values are reported. For the assessment of the measured results accuracy a statistical method with level of significance $p \leq 0.05$ was used. The data were analyzed and presented as mean values ± standard deviation.

RESULTS AND DISCUSSION

The progress of the starch gelatinization process is related to the absorption of energy required for destroying the native structure of the starch grains, accompanied by water adsorption (swelling). The observed changes in the endothermic peaks in the DSC curves are due to the different degree of the starch swelling in the sponge batters during the heating. Therefore the change in the energy value of the sponge system is caused to some extent by the change in the forms of the bound water. As a result the water activity of the sponge system correlates to the forms of the bound water. The water activity characterizes the reaction ability (chemical potential) of the water in the system. If the water activity decreases, the chemical potential and decreases too, and then the reactions involving the water in the colloidal system will require more energy in comparison with the free water. The factors affecting the water activity: type and concentration of the ingredients and the temperature of the environment, determine the forms of the bound water, which is involved in the process of the starch gelatinization.

Table 1: Sponge cake batters formulations

Ingredients	Amount based on wheat flour, [%]			
	control sample	with 1% polyglycerol monostearate ester (E475)	with 1% sucrose stearate ester (E473)	with 1 % modified inulin palmitate ester (HP-25)
Yolk of egg	43.22	43.22	43.22	43.22
White of egg	96.77	96.77	96.77	96.77
Refined granulated sugar	83.87	83.77	83.87	83.87
Wheat flour type 500	100.00	100.00	100.00	100.00
Emulsifier (Surfactants)	-	6.45	12.90	19.36
Water	-	3.87	6.45	12.90
Glycerol	-	-	3.87	3.61

This fact enables to recognize the relationship between the sponge batter ingredients and to analyse their impact on the quality of the final crust. The amount of the total water in the system depends on the type of the components in the batter formulation (Table 1), moisture content and water activity of the batter and the crumb of the baked cake (Table 3).

The surfactants (emulsifiers) used in the present research play an important role in the retarding of the starch gelatinization during the baking process and in this way the immobilized in the batter air bubbles can sufficiently increase their volume before the solidification of the sponge system. The sponge batter, which is in the state of liquid aerated mixture (foam) transforms to porous stable structure. As a result of these processes a crumb with specific porous structure and high volume is formed. The energy, transmitted during the heating to the sponge system with added surfactants, is involved in the destruction of the native starch structure in three ways: absorption of water and starch swelling during the gelatinization process, destruction of the bonds between the emulsifier and the amylose in the starch, destruction of the bonds between the sucrose and the starch molecules (Baeva, Terzieva, & Markov, 1997; Chevallier, Colonna, Della Valle, & Lourdin, 2000; Perry & Donald, 2002; Spies & Hoseney, 1982; White & Lauer, 1990; Wootton & Bamunuarachchi, 1980).

Therefore the change in the energy value of the sponge system is caused to some extent by the change in the water state. Depending on the balance between the three processes an endothermic phase transition of the starch gelatinization in the sponge mass with added surfactants forms.

An increase in the initial starch gelatinization temperature in sponge batters with added emulsifiers in comparison to the control batter is clearly detected. The lowest value of the initial starch gelatinization temperature (70 °C) is detected for the control batter. The starch gelatinization for all the samples containing emulsifier gels starts at 72 °C. Therefore the binding of water in the starch gelatinization in its initial degree of swelling starts for the control batter at lower temperatures and with lower energy

consumption compared to other samples – Fig. 1-4 and Table 2. This fact is due to the relatively high water activity of the control batter in comparison to the other batters – Table 3.

Table 2: Values of the thermal transitions

Type of sponge cake batter	Gelatinization temperature ranges (ΔT), [°C]	Peak gelatinization temperature, (T_p), [°C]	Gelatinization enthalpies (ΔH), [J/g]
Sponge cake batter control	(70 - 92)	Peak 1 – 80.5 Peak 2 – 88.1	1.78±0.08
Batter with 1% polyglycerol monostearate ester (E475) /PGE/	(72 – 100)	Peak 1 – 87.3 Peak 2 – 90.0	2.66±0.51
Batter with 1% sucrose stearate ester (E473) /SE/	(72 – 100)	Peak 1 – 85.5 Peak 2 – 89.3	2.68±0.33
Batter with 1 % modified inulin palmitate ester (HP 25)	(72 – 100)	Peak 1 – 86.5	2.46±0.48

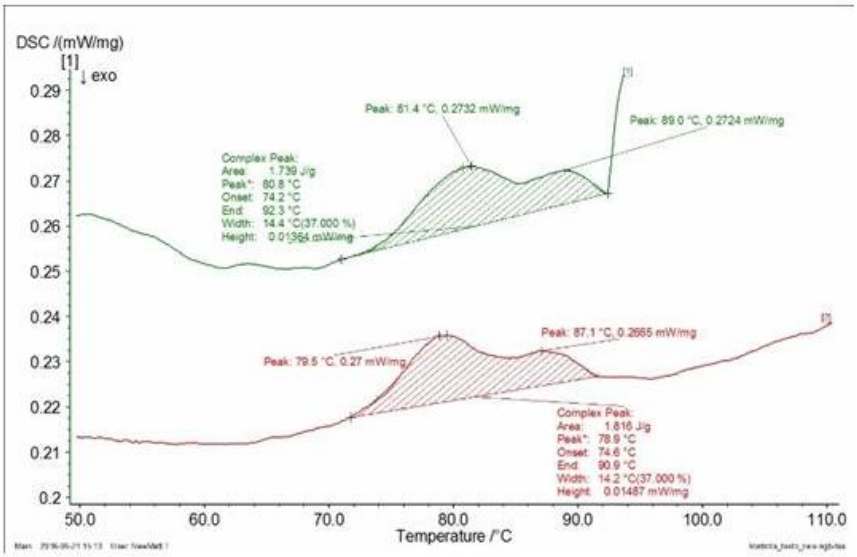

Figure 1
Thermograms for the starch gelatinization in batter sponge cake control

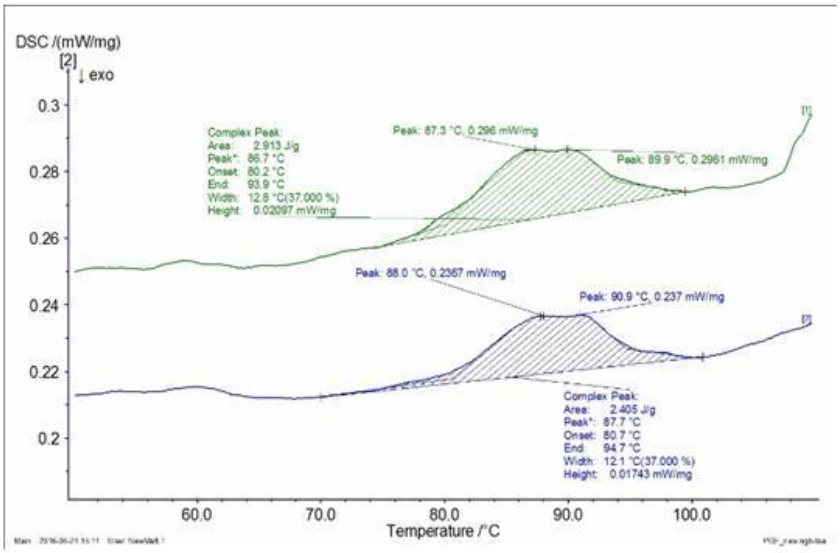

Figure 2
Thermograms for the starch gelatinization in batter sponge cake the addition of polyglycerol monostearate ester (E475)

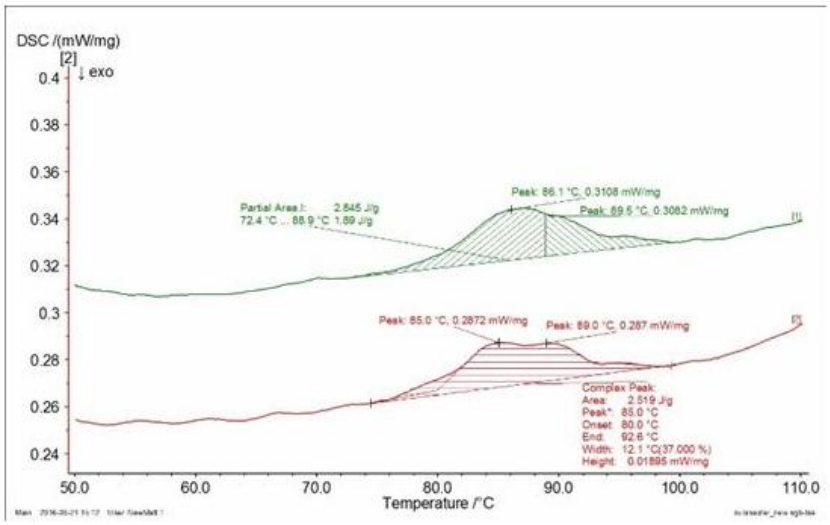

Figure 3
Thermograms for the starch gelatinization in batter sponge cake the addition of sucrose stearate ester (E473)

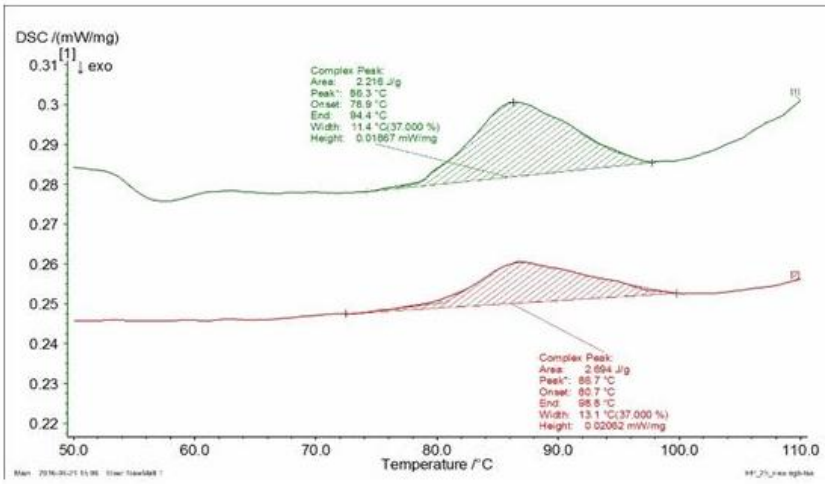

Figure 4
Thermograms for the starch gelatinization in batter sponge cake the addition of modified inulin palmitate ester (HP-25)

Table 3: Mean values[1] of the moisture content and the water activity of the batter and crumb cake

Sample	Physical characteristics[2] of the batters and cakes			
	a_w, cake batter	moisture cake batter, [%]	a_w, crumb cake	crumb moisture, [%]
Control	0.977 ± 0.001	36.44 ± 1,71	0.808 ± 0.001	29.88 ± 0.43
With addition of polyglycerol monostearate ester (E475)	0.916 ± 0.001	39.28 ± 0.34	0.908 ± 0.001	28.80 ± 0.20
With addition of sucrose stearate ester (E473)	0.904 ± 0.001	38.48 ± 0.53	0.898 ± 0.001	28.66 ± 0.69
With the addition of inulin palmitate ester (HP-25)	0.898 ± 0.001	43.89 ± 1.33	0.899 ± 0.001	35.35 ± 1.36

[1] The values are mean ± SD ($p \leq 0.05$).
[2] The temperature of the batter and crumb is on the average 20.0 ± 0.5 °C.

a) **b)** **c)** **d)**

Figure 5

Photographs of cross sections of sucrose-sweetened sponge cakes:
a) Without an emulsifier (control cake-sample);
b) With addition of 1% polyglycerol monostearate ester (E475);
c) With addition of 1% sucrose stearate ester (E473);
d) With addition of 1% inulin palmitate ester (HP-25).

The higher water activity of the control batter determines the higher degree of starch gelatinization, which affects the formation of porous structure in the final product (Figure 5).

The thermal transitions of starch gelatinization in the samples with added emulsifiers are characterized with higher enthalpies than the control batter. This fact could be explained with the relatively lower water activity for the batters with added emulsifiers in comparison with the control batter (0.977 ± 0.001) (Table 3). As shown in Table 2 the starch gelatinization process in the control batter finishes at lower temperature of 92°C. Higher energy values are required for starch gelatinization when emulsifiers are added to the batters and therefore the temperatures of the endothermic phase transitions are shifted to higher values (Table 2 and Fig. 1-4). Similarly the enthalpy of starch gelatinization for the control sample is the lowest (1.78 J/g) (Table 2), and it is higher for the batters with added sucrose stearate ester and polyglycerol monostearate ester (2.68 – 2.66 J/g).

The temperatures of the endothermic phase transitions in the sponge batters with added emulsifiers are shifted to higher values corresponding to higher energy levels (enthalpies). Therefore, the energy for starch gelatinization in the sponge batter with emulsifier gels is about 1.5 times bigger than the energy in the control batter (Table 2).

The most pronounced retarding effect on the process of starch gelatinization has the addition of 1% inulin-palmitate ester. The water activity of the sponge batter is this case is the lowest (0.898 ± 0.001). The DSC curve of heating the batter with inulin palmitate ester is characterized by sharp single endothermic peak (Fig. 4).

The role of the surfactants in the retarding of the starch gelatinization is confirmed by the measured values for the moisture content of the cake batters and the crumb cake. A relatively low water content (36.44 ± 1.71) but the highest water activity in comparison to the samples with added emulsifiers is observed for the control batter. This may be due to the different degree of dilution of the surfactant emulsifiers in the preparation of the gel in the composition of the sponge batter, and to the binding effect of the emulsifier to water in the system. This tendency of binding of the

emulsifiers to the water is retained in the process of baking, and it is most pronounced for sponge system with inulin palmitate ester whose water activity is statistically indistinguishable in the batter and in the cake crumb.

The cake with addition of polyglycerol monostearate ester is characterized by the highest water activity (0.908 ± 0.001), and the water activity of the cake with inulin palmitate ester is comparable to that of the cake with addition of sucrose stearate ester (0.898 - 0.899). The change in the water activity during the thermal treatment is the greatest for the sponge system without addition of emulsifier. It is the highest at the initial state (control batter) and the lowest in the baked cake (0.808 ± 0.001). The water activity of the control cake was significantly lower than those with added surfactants, confirming a high degree of starch gelatinization in the control sample (Figure 1).

Therefore, effected by the addition of emulsifiers in the sponge batter, the starch gelatinization proceeds in a wider temperature range at higher temperatures and with higher energy consumption. Based on these facts, one could consider that the emulsifiers exhibit retarding effect on the starch gelatinization in sponge batter. The retarding effect of the emulsifiers on the starch gelatinization delays the solidification of the dough and extends the period of gas expansion in it, and hence affects the structure of the sponge cake (Figure 5).

CONCLUSIONS

1. Partial crystallization is observed for all investigated sponge cake batters. The process proceeds to varying degrees and finishes at different temperatures intervals covering the range of 70-100 ^{o}C. The incomplete starch gelatinization contributes to solidification of the batter and turns it into colloidal-porous structure.

2. The starch gelatinization in the presence of the emulsifier polyglycerol monostearate ester (E475), sucrose stearate ester (E473) and modified inulin palmitate ester proceeds in wider temperature range and at higher energy consumption. The highest enthalpy of gelatinization is calculated for batters with addition of sucrose stearate ester and polyglycerol monostearate ester (2.68 – 2.66 J/g).

3. It has been shown that the emulsifiers exhibit retarding effect on the starch gelatinization in sponge cake batters. The most pronounced retarding effect possess the inulin palmitate ester. The changes in the thermal transitions during the starch gelatinization are related to the emulsifier binding capability to both water and starch, which determines the lower degree of gelatinization. The shifting of the endothermic transitions to higher temperatures determines the strongest binding role of emulsifiers on the ability of starch to bind the water in the system. It is assumed that the concurrent binding of the emulsifier with the water and the amylose determines a low degree of starch gelatinization in the sponge system.

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