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Effect of storage temperatures on Kashkaval texture

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Abstract

Introduction. The aim of the present study was to investigate the effect of storage temperature on the texture parameters of cow milk Kashkaval cheese.

Materials and methods. Kashkaval cheese samples were prepared according to a classic technology and stored at different temperatures (4.0±1.0 °C; 1.0±1.0 °C; 7.5±0.5 °C and -18.0±1.0 °C). Texture analysis was performed by StableMicroSystems TA-XT2i analyser equipped with a loading cell 50 kg.

Results and discussion. The obtained results showed a significant difference in the values of the hardness index between the cheese samples stored in a refrigerated state and those stored in a superchilled and frozen state. With increasing storage temperature of cheese, there was a tendency to decrease ($p<0.05$) the values of the cohesiveness indicator. The results obtained in the present study showed that the storage temperature had a decisive influence on the changes in the springiness of the cheese. Higher storage temperatures (4.0±1.0 °C) were accompanied by a significant decrease in the springiness of the cheese. The storage of the Kashkaval cheese in a refrigerated state was accompanied by a significant increase ($p<0.05$) in its adhesiveness. This trend intensified with the increase of the storage temperature. With the increase of the storage temperature of cheese, more significant decrease ($p<0.05$) of the values of the gumminess index was observed. In a comparative analysis of the changes in the hardness and gumminess of the experimental Kashkaval cheese samples, it was found that within the same temperature regime the decrease in the values of gumminess was greater than that of the hardness. As all three indicators declined in the process of refrigerated storage, it reflected significantly on the gumminess.

Conclusion. More intense changes in the texture of the studied Kashkaval cheese samples were observed with the increase of the storage temperature.

Introduction

The texture of foods, in particular cheeses, is a subject of mandatory research over the last decade (Bolhuis and Forde, 2020). The structural and mechanical characteristics of cheeses are essential and determine their sensory perception by the consumer (Foegeding and Drake, 2007).

Modern technologies, such as curd or cheese freezing, are applied during storage in order to achieve a longer cheese shelf life (Alinovi and Mucchetti, 2020; Marcuzzo et al., 2012). During the storage of the cheeses, changes in their texture occur. Many authors prove that the texture of the cheese is influenced by a number of factors, such as milk composition, water content, salt content, pH and degree of proteolysis during ripening (Hill and Kethireddipalli, 2013). The texture profile of the cheeses also changes during their storage, as the temperature and the storage time are determining factors for the degree of changes. Some authors suggest that the decrease in hardness and increase in the softness of fresh cheeses is directly related to the proteolytic activity of lactic acid bacteria, which breaks down proteins into peptides and subsequently into low molecular weight peptides and amino acids (Fox et al., 2017). This disrupts the integrity of the protein network in the cheeses during storage and gives them softness (Fox et al., 2017).

Some researchers suggest that cheeses with high protein and low fat content have higher values of hardness and elasticity (Borges et al., 2020). The same trend is observed for the stability of the gumminess indicator, which in turn is associated with the hardness and springiness of the cheese by other author teams (Diamantino et al., 2014). It is found that the decrease in pH resistance leads to a loss of colloidal maintenance of calcium phosphate by the casein submicel, as result of which small aggregates are formed. This in turn leads to softening of the cheese (Buriti et al., 2005).

According to Diefes et al. (1993), the dehydration of proteins and the formation of ice crystals in the cheese during freezing and its storage in the frozen state can cause the destruction of the structure of the proteins. This in turn allows the small oil balls to come into contact with each other and thus form granules. Kuo and Gunasekaran (2003) reported that prolonged storage of cheese in a frozen state could cause greater damage to its structure as a result of recrystallization of ice crystals.

After thawing, proteins are not able to completely bind water. As a result, water remains free in the protein network, leading to the formation of a porous protein matrix of the cheeses, which are stored frozen for a longer time (Degner et al., 2014). Experimental studies with an electron microscope have shown that freezing and thawing cause the formation of larger cavities between the protein fibres in cheese samples due to the growth of ice crystals (Graiver et al., 2004).

Kuo and Gunasekaran (2009) investigated how freezing and frozen storage affect the microstructure of mozzarella cheese and pizza cheese. The authors observed visible differences in the microstructures of the two types of cheese. Freezing has a different effect on siren matrices. It causes the mozzarella protein matrix to break, while pizza cheese cracks and accumulates bacteria. The authors found that the protein matrix of pizza cheese was more susceptible to freezing due to freezing than that of mozzarella cheese, with tempering of the samples mitigating the negative effect of freezing. Disturbances in the structure of the cheese caused by freezing and storage in a frozen state can be minor with proper storage and tempering.

Although there are published data on the possibilities of using frozen curd in the production of Kashkaval cheese, no data examining the influence of the storage regime on its texture were found.

The *aim* of the present study was to investigate the effect of storage temperature on the texture parameters of cow milk Kashkaval cheese.

Materials and methods

Materials

The used raw cow milk corresponded to the national and European regulations (Regulation 853/2004). It was used in order to produce Kashkaval cheese samples stored at different temperatures with a known composition (Ivanov and Markova, 2020) and sensory profile (Ivanov et al., 2020).

Starter culture containing *Strepococcus thermophiles*, *Lactobacillus helveticus* and *Lactobacillus delbrueckii* ssp. *bulgaricus* was supplied by Lactina Ltd. Calcium dichloride solution (50%) as well as the rennet enzyme were purchased from Biokom Trendafilov Ltd.

Methods

Kashkaval preparation. Kashkaval samples were produced in a local dairy plant according to a classic applied technology (Kozhev, 2006). Cow milk was standardized in order to achieve casein to fat ratio equal to 0.69. It was further heat-treated at 65±1 °C for 15 s and cooled to temperature of incubation 33±1 °C. The prepared cow milk was inoculated with a thermophilic starter culture (*Streptococcus thermophilus* and *Lactobacillus delbrueckii* subsp. *bulgaricus*) and calcium chloride 50% (diluted 1:10 with water, in amount 30 cm^3/100 L milk) and commercial animal rennet enzyme was also added (diluted 1:10 with water, in such amount that the first coagulation started 9-10 min after enzyme addition). When a firm coagulum was formed (after 30 min), the curd was cut and held in rest for 5 min. A stirring process of the curd started for 20 min. It was further heated at 38-40 °C for 40 min with continuous agitation. The formed whey was separated from the curd and it was collected in the form of a compact mass in order to assure the cheddaring process which took place at pH 5.3 (about 2 h). The curd submitted to cheddaring was cut, milled, salted and stretched in a concentrated salt solution (13%) at 72 °C. The fresh Kashkaval was moulded into 1 kg parallelepiped forms. It was dried for 15 h and was further vacuum-packaged in polyethylene bags. The fresh Kashkaval was subjected to ripening at 9.0±1.0 °C for 60 days. Storage of ripened Kashkaval was conducted at different temperatures (refrigerated at 4.0±1.0 °C; refrigerated at 1.0±1.0 °C; frozen at 7.5±0.5 °C and deep frozen at -18.0±1.0 °C) for 12 months. Different stages (1^{st}, 3^{rd}, 6^{th}, 9^{th} and 12^{th} month) of the storage period were investigated.

Texture Profile Analysis. Cheese cubes with dimensions 25·25·25 mm were prepared. A measurement of the sample sizes (length, width and height) by digital caliper and measurement of the sample weight by laboratory scales was done.

A texture analyzer StableMicroSystems TA-XT2i (Stable Micro System, Ltd, UK) equipped with loading cell 50 kg and specialized software „Texture Exponent 6.1 was used in order to determine their textural parameters. The applied software allowed calibrating the force and the sample height before the measurements. The deformation range for the instrument work was 0-500 mm (with resolution 0.001 mm) and the force range was 0-500 N (resolution: 0.001 N, trigger force was 0.05N, minimal measured firmness force was 1 N).

The texture profile analysis was made by a flat plate probe (P 50-Stainless steel cylinder probe, Ø=50 mm) with test speed 1 $mm.s^{-1}$, a strain of 50% and two bite time intervals of 5 s. The test was repeated on 3 cubes.

The textural characteristics of the cheese were determined as follow: Firmness force (F) was determined as the maximum force coordinate of 1^{st} peek of the curve; Adhesiveness was defined as the stress, necessary to pull up the measure probe after the 50% compression

(negative integrated area, Nmm); Springiness was the distance of the detected height of the product on the second compression divided by the original compression distance where (Length 2 /Length 1); Cohesiveness was defined as the ratio between the 2nd and 1st loading area (A_2/A_1); Gumminess was computed like cohesiveness multiplied by the firmness; Chewiness was computed like cohesiveness multiplied by the firmness and the springiness (Zheng et al., 2016). The test was repeated on 3 cubes.

Statistical analysis. All statistical procedures were computed using the Microsoft Excel and Sigma Plot 2001 software. Statistical analysis was conducted by one-way ANOVA analysis (Donchev et al., 2002). Results were presented as mean of n=4 (2 batches with 2 repetitions) ± standard deviation (SD) and were considered as statistically different when $p<0.05$ (Petrova, 2002; Dilcheva and Kinova, 2008).

Results and discussion

The obtained results for the change of the hardness of the experimental samples of Kashkaval cheese in the process of refrigerated storage are presented in Figure 1.

There was a decrease in the values of the hardness index in all tested samples. The smallest decrease in hardness was found in Kaskaval cheese stored superchilled (-7.5±0.5 °C) and frozen (-18.0±1.0 °C). At the end of storage, the hardness values in these cheese samples were reduced to 61.4±1.5 N. In the present study, no statistically significant ($p<0.05$) differences in the hardness of Kashkaval cheese stored in superchilled and frozen state were found. This was probably due to similar changes in the microstructure of these samples due to the absence of intense proteolytic processes in the cheese matrix.

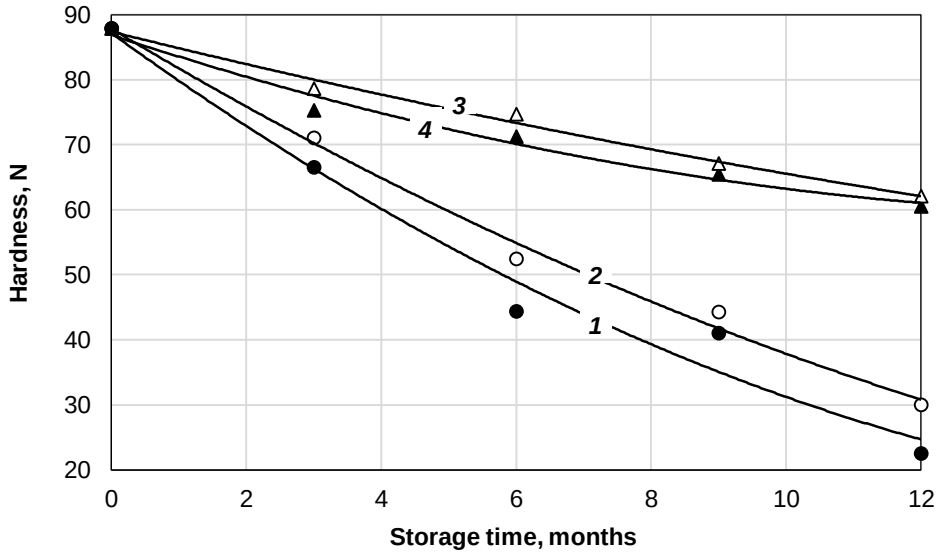

Figure 1. Changes in the hardness index of the analysed Kashkaval cheese samples from cow's milk stored in a refrigerated, superchilled and frozen state:

1 – Refrigerated Kashkaval (4±1 °C);
2 – Refrigerated Kashkaval (1±1 °C);
3 – Superchilled Kashkaval (-7±1 °C);
4 – Frozen Kashkaval (-18±1 °C).

With increasing storage temperature of Kashkaval cheese, more significant decrease ($p>0.05$) in the values of the hardness index was observed. During the 12-month storage period of the Kashkaval cheese samples at temperatures of 1.0±1.0 °C, their hardness decreased from 87.8±4.2 N to 29.9±3.3 N. For the cheese stored at the temperature regime of 4.0±1.0 °C, the decrease in the values of the hardness index was from 87.8±4.2 N to 22.5±2.8 N. The obtained results (Figure 1) indicated the direct dependence of the changes in the hardness of the cheese with the storage temperature. Alvarenga et al. (2011) investigated the effect of freezing on the rheology of sheep cheese samples. The authors found that the hardness index in frozen cheese samples was significantly higher than in chilled cheese samples. Diefes et al. (1993) suggested that this was due to the partial dehydration of the proteins in the cheese matrix of frozen cheeses. As a result, the cheeses acquire a firmer (harder) and elastic structure.

The obtained results showed that even the minimum temperature difference of 3 °C in the two storage modes of the refrigerated Kashkaval cheese samples (1.0±1.0 °C and 4.0±1.0 °C) led to significant differences in their hardness. The significant difference in the values of the hardness index between the cheese samples stored in a refrigerated state and those stored in a superchilled and frozen state was significant ($p<0.05$). As it was found during the storage of Kashkaval cheese in superchilled and frozen state, the proteolytic processes were almost completely inhibited (Ivanov and Markova, 2020). This favoured the preservation of its structure, which explained the higher hardness of these samples. Probably the phase transition of water in the process of superchilling and freezing accompanied by the formation of ice crystals destroyed to a lesser extent the structure of Kashkaval cheese compared to proteolysis. On the other hand, the cheese stored in a refrigerated state lacked a phase transition of water and the formation of ice crystals, but the ongoing proteolytic processes significantly weakened the paracasein matrix, which was accompanied by a decrease in its hardness.

The obtained results for the change of the indicator during storage are presented in Figure 2. In the present study, no statistically significant change in the values of this indicator was found in cheese stored in superchilled (-7.5±0.5 °C) and frozen (-18.0±1.0 °C) state. This indicator characterized the strength of the bonds in the paracasein matrix of Kashkaval cheese (Gunasekaran and Ak, 2004).

After 12 months of storage, the values of the cohesiveness index in the analysed cheese samples were maintained at the levels of 0.47±0.02. This showed that these two refrigeration regimes provided minimal changes in the structure of the cheese, which corresponded to the observed tendency for the change in the hardness of the analysed samples. The absence of intensive proteolytic processes in the cheese samples stored in superchilled and frozen state contributed to the preservation of their structure and their greater hardness compared to cheese stored in a refrigerated state.

With increasing storage temperature of cheese, there was a tendency to decrease ($p<0.05$) the values of the cohesiveness indicator. During the 12-month storage period of the cheese samples at temperatures of 1.0±1.0 °C, their cohesiveness decreased from 0.49±0.02 N to 0.43±0.01 N. These changes in the cohesivenes indicator were minimal, but statistically significant ($p<0.05$). A more significant decrease in the cohesiveness values was observed during the storage process at 4.0±1.0 °C. For the whole period of refrigerated storage (12 months) the decrease in the values of the cohesiveness indicator in these samples was from 0.49±0.02 to 0.24±0.02.

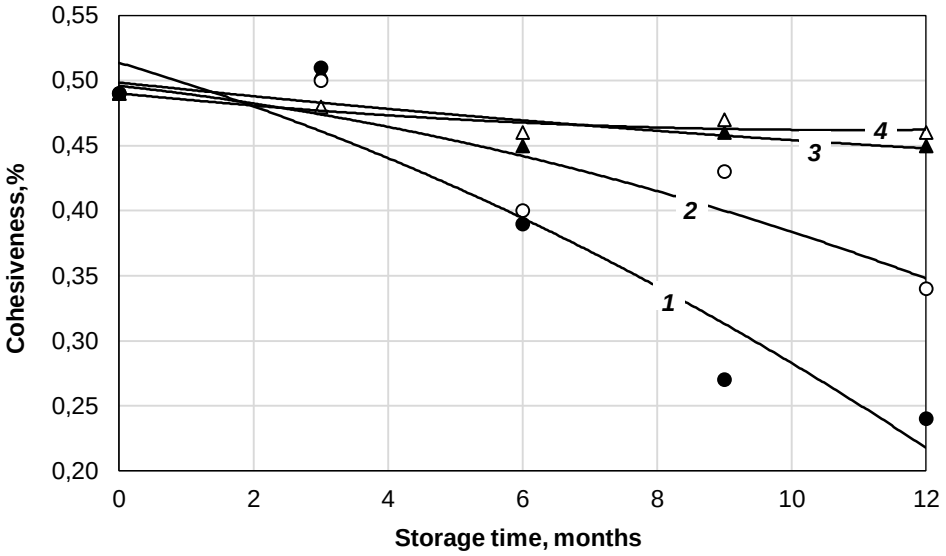

Figure 2. Changes in the cohesiveness index of the analysed Kashkaval cheese samples from cow's milk stored in a refrigerated, superchilled and frozen state:
1 – Refrigerated Kashkaval (4±1 °C);
2 – Refrigerated Kashkaval (1±1 °C);
3 – Superchilled Kashkaval (-7±1 °C);
4 – Frozen Kashkaval (-18±1 °C).

Everar et al. (2006) investigated the structural and mechanical properties of cheddar cheese left to mature for 9 months under refrigerated conditions. The indicators hardness, springiness, cohesiveness, adhesiveness and gumminess were studied. After the 9th month of storage, there was a decrease in the values of springiness, cohesiveness, adhesiveness and gumminess in the tested cheese samples whist was in correspondence of our results. Aday et al. (2010) examined white semi-hard cheese placed to mature at 2–4 °C for 12 months. After 9 months of cheese samples storage, the authors found that the cohesiveness decreased.

The obtained results in the present study (Figure 2) indicated a statistically significant ($p<0.05$) influence of the temperature regime of Kashkaval storage on the changes in the cohesiveness index. This effect was most noticeable when the cheese was stored in a refrigerated state, in which even the minimum temperature difference of 3 °C between the two experimental modes (1.0±1.0 °C and 4.0±1.0 °C) led to significant differences in the values of the cohesiveness indicator.

The data obtained in the present study on the change of the springiness index of the experimental samples of Kashkaval cheese in the process of refrigerated storage are presented in Figure 3. This indicator reflected the speed and extent to which a deformed object regained its original shape and size (Gwartney et al., 2004).

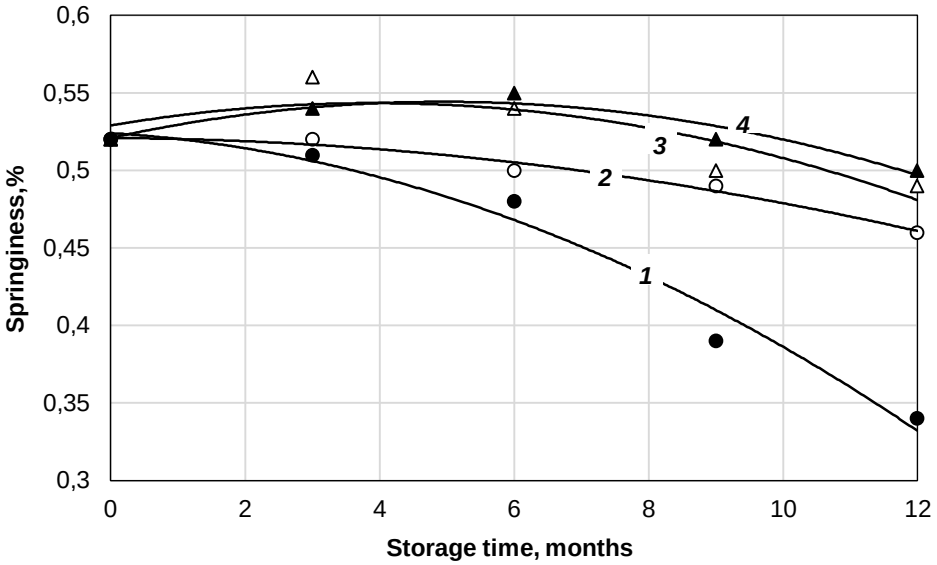

Figure 3. Changes in the springiness index of the analysed Kashkaval cheese samples from cow's milk stored in a refrigerated, superchilled and frozen state:
1 – Refrigerated Kashkaval (4±1 °C);
2 – Refrigerated Kashkaval (1±1 °C);
3 – Superchilled Kashkaval (-7±1 °C);
4 – Frozen Kashkaval (-18±1 °C).

In the present study, no statistically significant change in the springiness of the Kashkaval cheese samples stored in the superchilled (-7.5±0.5 °C) and frozen state (-18.0±1.0 °C) was found. At the end of storage, during these two refrigeration regimes, the elasticity of the cheese was preserved at about 0.49±0.02%. This trend corresponded to the lack of statistically significant changes in the indicators of hardness and cohesiveness of yellow cheese samples stored in superchilled and frozen conditions.

The storage of the cheese in a refrigerated state was accompanied by a decrease ($p<0.05$) in its springiness. This trend intensified with the increase of the storage temperature. During the 12-month storage period of the cheese samples at temperatures of 1.0±1.0 °C, their springiness decreased from 0.52±0.01% to 0.46±0.02%. Similar to the changes in the homogeneity of these samples, the observed decrease in the values of the elasticity index was minimal but statistically significant ($p<0.05$). A more significant decrease in the elasticity of the cheese was observed during storage at 4.0±1.0 °C. For the whole period of refrigerated storage (12 months) the decrease in the values of the elasticity index in these samples was from 0.52±0.02% to 0.34±0.02%. Zheng et al. (2016) examined the texture of cheddar cheese. The authors found that with increasing storage temperature, the springiness of the studied cheese samples decreased. The correlation between storage temperature and springiness index was negative.

The results obtained in the present study (Figure 3) showed that the storage temperature had a decisive influence on the changes in the springiness of the cheese. Higher storage temperatures (4.0±1.0 °C) were accompanied by a significant decrease in the springiness of the cheese, which was probably due to the ongoing proteolytic processes leading to weakening of the bonds in the paracasein matrix.

The data on the change of the adhesiveness index of the yellow cheese in the process of refrigerated storage are presented in Figure 4.

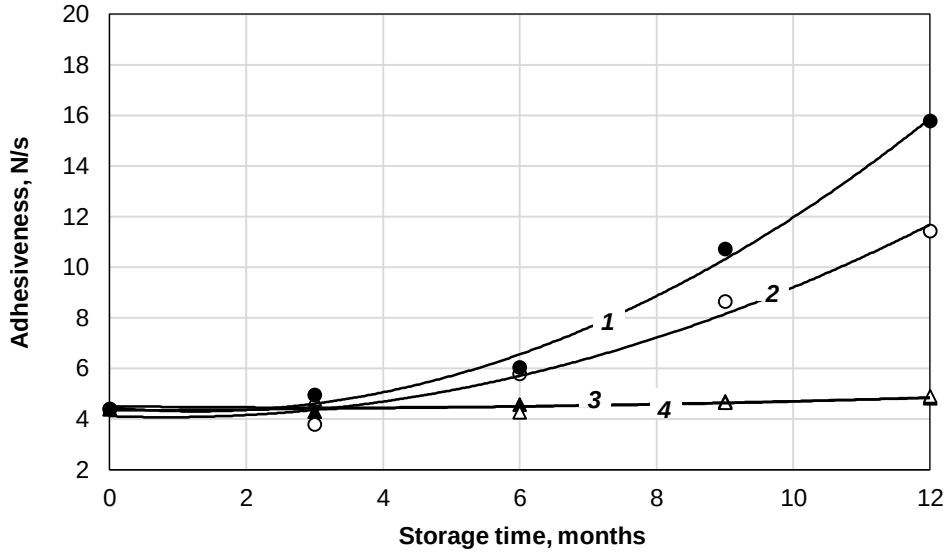

Figure 4. Changes in the adhesiveness index of the analysed Kashkaval cheese samples from cow's milk stored in a refrigerated, superchilled and frozen state:
1 – Refrigerated Kashkaval (4±1 °C);
2 – Refrigerated Kashkaval (1±1 °C);
3 – Superchilled Kashkaval (-7±1 °C);
4 – Frozen Kashkaval (-18±1 °C).

This indicator reflected the effort required to overcome the adhesion forces of the sample to the working tool of the apparatus (Tunick, 2000). Sensory, this was the degree of adhesion of the sample to the teeth of the consumer in the process of its chewing (Gwartney et al., 2004). In the present study there was a minimal but statistically significant increase in the values of the adhesiveness index in the process of storing cheese in superchilled (-7.5±0.5 °C) and frozen state (-18.0±1.0 °C). At the end of storage under these two refrigeration regimes, the adhesiveness of the yellow cheese reached values of 4.81±0.08 N.s. The observed increase in the values of this indicator was most likely due to the partial rupture of the bonds in the paracasein matrix of the cheese as a result of the formation of ice crystals during the refrigeration, superchilling and freezing treatments. However, the lack of intensive proteolytic processes in the cheese stored at these two temperatures did not allow a significant increase in its stickiness (Ivanov and Markova, 2020).

The storage of the Kashkaval cheese in a refrigerated state was accompanied by a significant increase ($p<0.05$) in its adhesiveness. This trend intensified with the increase of the storage temperature. During the 12-month storage period of cheese samples at

temperatures of 1.0±1.0 °C, their adhesiveness increased from 4.39±0.07 N.s to 11.43±0.09 N.s. A more significant increase in the stickiness of the cheese was observed during storage at 4.0±1.0 °C. For the whole period of refrigerated storage (12 months) the increase in the values of the adhesiveness index in these samples was from 4.39±0.07 N.s to 15.77±0.08 N.s.

The results obtained in the present study (Figure 4) corresponded to the data on proteolysis in Kashkaval cheese stored at the same conditions (Ivanov and Markova, 2020). Higher storage temperatures (1.0±1.0 °C and 4.0±1.0 °C) were accompanied by a higher degree of hydrolysis of casein to low molecular weight compounds, the accumulation of which in Kashkaval led to an increase in its adhesiveness. Similar trends were found by other authors (Chevanan et al., 2006; El-Bakry et al., 2011). Zheng et al. (2016) investigated the influence of storage temperature on the physicochemical composition and structural and mechanical parameters of mozzarella cheese. The authors found that with increasing storage temperature of the studied cheese samples and the adhesiveness index increased. According to the authors of the study, the low protein and high fat content softened the cheese matrix, which in turn increased its stickiness.

The results obtained for the change of the gumminess of the experimental samples of Kashkaval in the process of refrigerated storage are presented in Figure 5.

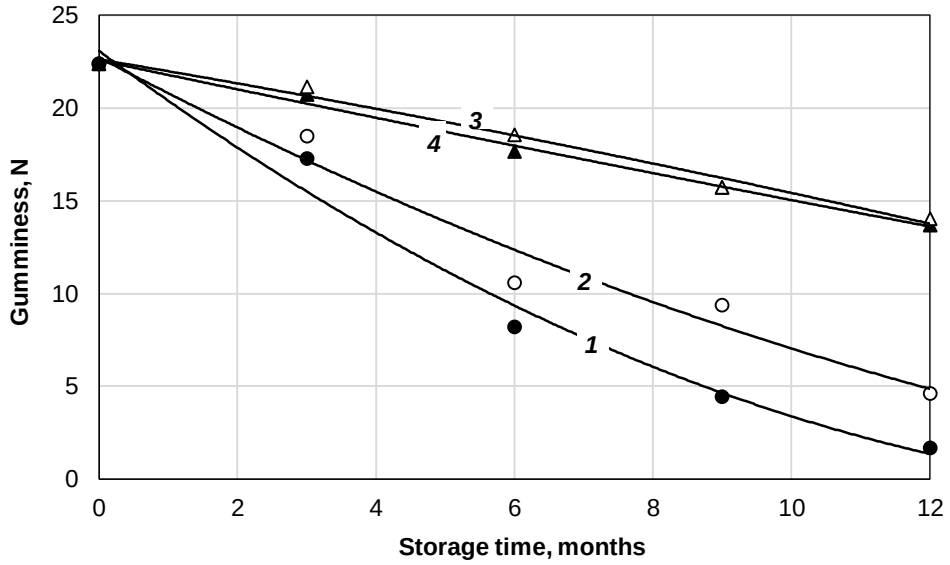

Figure 5. Changes in the gumminess index of the analysed Kashkaval cheese samples from cow's milk stored in a refrigerated, superchilled and frozen state:
1 – Refrigerated Kashkaval (4±1 °C);
2 – Refrigerated Kashkaval (1±1 °C);
3 – Superchilled Kashkaval (-7±1 °C);
4 – Frozen Kashkaval (-18±1 °C).

This indicator reflected the energy required to destroy the structure of the cheese to the extent which was absorbed by the consumer (Chevanan et al., 2011). As shown in the presented data (Figure 5), the gumminess of Kashkaval cheese decreased during storage for

all four experimental refrigeration modes. Due to the direct relationship existed between the gumminess and hardness of cheese, the trends in the changes of these two indicators were similar.

In the present study, the smallest decrease in gumminess was found in cheese stored in superchilled (-7.5±0.5 °C) and frozen (-18.0±1.0 °C) state. At the end of storage, the values of the gumminess index in these samples of Kashkaval cheese decreased to 13.8±1.1 N. In the present study, no statistically significant ($p<0.05$) differences in the gumminess of cheese stored in the refrigerated and frozen state were found. This was probably due to the similar changes in the microstructure of these samples due to the ongoing crystal formation processes during their refrigeration processing and storage, as well as to the absence of intensive proteolytic processes in the cheese matrix.

With the increase of the storage temperature of cheese, more significant decrease ($p<0.05$) of the values of the gumminess index was observed. During the 12-month storage period of the cheese samples at temperatures of 1.0±1.0 °C, their gumminess decreased from 22.4±1.1 N to 4.7±0.8 N. For the cheese stored at 4±1 °C, the decrease in the values of the gumminess index was from 22.4±1.1 N to 1.8±0.5 N. Mushtaq et al. (2015) reported a decrease in the values of the gumminess index in buffalo cheese samples. The data from the conducted research showed that with the increase of the storage temperature the gumminess indicator decreased. The authors explained this with a change in pH values. The reduction of the active acidity in the cheese led to demineralization and destabilization of casein micelles. This in turn led to the formation of low molecular weight compounds and softening of the texture of the cheese (Buriti et al., 2005).

In a comparative analysis of the changes in the hardness and gumminess of the experimental Kashkaval cheese samples, it was found that within the same temperature regime the decrease in the values of gumminess was greater than that of the hardness. The reason for this was the complex nature of the gumminess indicator, taking into account the combined perception of hardness, cohesiveness and springiness of Kashkaval cheese. As all three indicators declined in the process of refrigerated storage, it reflected significantly on the gumminess.

Conclusions

The changes in the structural and mechanical parameters of Kashkaval cheese in the process of its refrigerated storage were directly dependent on the applied temperature regime. During Kashkaval storage in refrigerated, superchilled and frozen state, a decrease in the values of the indicators hardness, springiness, cohesiveness, adhesiveness and gumminess was established. The most insignificant changes were found in the texture of Kashkaval samples stored in superchilled and frozen state. More intense changes in the texture of the studied Kashkaval cheese samples were observed with the increase of the storage temperature.

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Nutritional value of protein in wheat-rye bread manufactured with addition of flour from low-alkaloid cultivars of lupin

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Abstract

Introduction. The aim of this study was to assess the nutritional value of protein in wheat-rye bread manufactured with an addition of flour from low-alkaloid cultivars of lupin.

Material and methods. Flours from yellow lupin cultivars: Juno, Polo, Legat and Markiz, were used in wheat-rye bread baking. The following were determined in breads: protein content, *in vitro* protein digestibility, amino acid composition, effective protein content (EP), chemical score (CS), essential amino acid index (EAAI), true digestibility of protein (TD), protein digestibility corrected amino acid score (PDCAAS) and protein efficiency ratio (PER).

Results and discussion. The addition of lupin flour resulted in an increase in the total protein and digestible protein in breads. The greatest increase in the EP, CS, PDCAAS and EAAI was observed when the bread was enriched with flour from Polo cultivar. No significant changes were noted in PER. Protein in the products with lupin flour contained more leucine, lysine, asparagine and arginine, than the cotrol sample. A tendency has been recently observed to enrich cereal products, mainly those made from wheat and wheat-rye mixes, with flours obtained from other plants, such as lupin, green peas, beans, hemp and buckwheat. These flours are highly valued because of their functional properties, which include solubility, emulsifying capabilities, foaming and gelling properties, and water retaining capability. Lupin flour and protein isolates from lupin seeds does not affect the taste of a final product. An addition of lupin flour to wheat bread improves considerably the quality and amount of protein and dietary fibre in a final product. The high values of indices PDCAAS and PER make it possible to compare lupin proteins and proteins of leguminous plants to animal proteins.

Conclusions. Protein of low-alkaloid cultivars of lupin can be a valuable component which increases the nutritional value of bread protein and therefore it should be recommended for use in baking.

Introduction

Cereal products, including bread, are at the base of many present food pyramids. They provide the human body with energy, protein of plant origin, vitamins B as well as macronutrients (magnesium, potassium, phosphorus) and micronutrients (iron, copper). Protein performs a number of functions in the body, with serving as building blocks being the most important of them. However, cereal proteins are regarded as having lower biological value than those of animal origin. This is because they contain, depending on the cereal species, smaller amounts of some essential amino acids – lysine and tryptophan (wheat and maize), methionine (rye) and threonine (rice) (Hryniewiecki, 2007).

Leguminous plants, particularly soybean, are used as a raw material in the production of food which provides the body with full-value protein. Lupin is an alternative source of protein, which can replace soybean imported from the warm climate zone. The use and consumption of products from seeds of this plant have been steadily growing in recent years (De Cortes-Sanchez *et al.,* 2005). Lupin is a good source not only of protein and essential amino acids, but also of fat, dietary fibre, minerals and vitamins (Martinez-Villaluenga *et al*., 2006, 2009; Zielińska *et al*., 2008; Kohajdoova *et al.,* 2011; Schumacher *et al*., 2011). Due to a high content of lysine in lupin protein, particularly in "sweet", low-alkaloid cultivars, it can be an excellent supplement of cereal proteins, in which the content of this amino acid is low.

In the current available literature, no data on the usefulness of the protein of bread baked from wheat-rye flour enriched with lupine flour have been found. It was decided that the nutritional value of the protein of the analyzed bread should, apart from traditional chemical quality indicators, be extended with the newest, recommended indicators taking into account the biological availability of this nutrient.

The aim of this study was to assess the nutritional value of protein in wheat-rye bread manufactured with an addition of flour from low-alkaloid cultivars of lupin.

Materials and methods

Sample preparation

Bread made from the mixture of wheat-rye (80 : 20) flours (K – control) and breads from wheat-rye flour with a 5% addition of flour from four low-alkaloid "sweet" cultivars of yellow lupin (75 : 20 : 5) were used as the study material. The experimental breads were marked after cultivars as: J – Juno, P – Polo, L – Legat, and M – Markiz. Lupin seeds were obtained from the Agricultural Experiment Unit of the University of Warmia and Mazury in Olsztyn, Poland. The bread was baked in a private bakery. The baking process was carried out as described in Skibniewska *et al.* (2003).

Analyses

The total protein (N x 6.25) was determined according to AOAC (1995a). The *in vitro* protein digestibility, i.e. its susceptibility to the action of digestive enzymes, was determined after incubation with a solution of pepsin, pancreatin and a bile extract at the enzyme-protein ratio of 1 : 20 (Ikeda, 1990). The *in vitro* protein digestibility was calculated from the following formula: ($D_{in\ vitro}$ = [a – b/a] x 100), where a – starting content of protein in the

bread under study as g/100 g of bread, b – content of protein in the bread under study following *in vitro* digestion, as g/100 g of bread.

Amino acid content (g/16 g N, equivalent of g/100 g of protein) was determined with an amino acid analyser Biochrom 20 Plus (Biochrom Ltd., Cambridge, UK). The Amino Acid Standard Solution by Sigma was used as the standard. The protein hydrolysate was prepared by acidic hydrolysis (6 M HCl, at 105°C for 24 h). The content of sulphur amino acids was determined separately in 6 M HCl following oxidative hydrolysis (formic acid + hydrogen peroxide at 9 : 1 ratio, 4°C, 16 h). Tryptophan was determined according to AOAC (1995b).

The content of essential amino acids in the breads under study was used as the basis for calculation of the chemical protein quality indices: the chemical score (CS) and the integrated essential amino acid index (EAAI). The amino acid composition of two standard proteins – FAO/WHO 1991 and DRI/USA 2005 (Jarosz *et. al.*, 2017) was used in the calculations. The CS index was calculated from the formula: $X_i = a_i / a_{is}$ x 100, where a_i – the content of a given essential amino acid in the protein under test, a_{is} – the content of a given essential amino acid in the standard protein, i = 1, 2…8 (Hryniewiecki, 2007). The calculations were conducted using the content of isoleucine (ILEU), leucine (LEU), lysine (LYS), sum of methionine and cysteine (MET + CYS), phenylalanine and tyrosine (PHE + TYR), threonine (THR), tryptophan (TRY) and valine (VAL). The CS is taken as the lowest ($X_{min.}$) of the eight ratios expressed as percent ($X_{min.}$ = CS), and the amino acid for which the value is the lowest is the one limiting the nutritional value of the protein.

The EAAI, introduced by Oser (1959), was calculated from the formula:

$$EAAI = 10^{lg\,EAA}.$$

In order to calculate lg EAA, the following formula, provided by Rakowska *et al.* (1978), was used:

$$lg\,EAA = 1/8\,(lg\,X_1 + lg\,X_2 + \ldots + lg\,X_8).$$

The content of effective protein (EP) was calculated from the respective CS (%) and the total protein content (TP, %) in the bread under analysis, from the following formula:

$$EP\,(\%) = TP \times CS/100$$ (Gawęcki & Jeszka, 1986).

The true digestibility (TD, %) of investigated bread samples was calculated as the weighted average of the true digestibility indices of individual components of breads and their percentage share. The TD values of individual components were taken after Boye *et al.* (2012): wheat flour – 89.4%, rye flour (barley) – 75.3%, lupin flour (soybean) – 80.0%.

The protein digestibility corrected amino acid score (PDCAAS) was calculated from the formula:

$$PDCAAS\,(\%) = CS\,(\%) \times TD\,(\%)/100$$ (Schaafsma, 2012, Ruhterfurd 2015).

The protein efficiency ratio (PER) was calculated from the content (g/16 g N) of leucine (LEU) and tyrosine (TYR) in the bread from the following formula:

$$PER = -0.468 + 0.454\ LEU - 0.105\ TYR$$ (Sujak *et al.*, 2006).

The chemical analyses were performed in triplicates. The results in Table 1 and 2 are presented as mean values with standard deviations. The statistical significance of differences between the mean values was tested with the Student's t-test at the significance level of $P \leq 0.05$, using the Statistica PL 10.0 software (StatSoft, Kraków, Poland).

Results and discussion

Protein content and *in vitro* protein digestibility in the breads under study

Table 1 shows the protein content and its *in vitro* digestibility in the tested samples. All breads manufactured with the addition of lupin flour contained more protein (10.1–11.0%) than the control bread (9.0%), with the bread enriched with the Markiz cultivar flour showing the highest percentage of protein. No significant differences were observed between the breads made with an addition of lupin flours. The content of protein following *in vitro* digestion was the highest in bread prepared with an addition of flour from the Juno (5.6%) cultivar, but again, no statistical differences between the enriched breads were observed. All values of this parameter were higher in the experimental samples than in the control. The protein in the bread enriched with the Juno cultivar was the most susceptible to the action of digestive enzymes (55.4%). The other digestibility indices were not statistically different and were 50.5%; 49.1% and 49.1% for breads with flours of the Polo, Legat and Markiz cultivars, respectively.

Table 1

Protein content and *in vitro* protein digestibility in the breads under study (mean ± SD)

Specification	Type of bread				
	Control K	**Juno J**	**Polo P**	**Legat L**	**Markiz M**
Total protein content, g/100 g	9.0±0.2^a	10.1±0.2^b	10.5±0.2^b	10.8±0.2^b	11.0±0.3^b
Protein content following *in vitro* digestion, g/100 g	4.4±0.1^a	5.6±0.1^b	5.3±0.3^b	5.3±0.1^b	5.3±0.3^b
In vitro protein digestibility, %	48.9±1.0^a	55.4±1.1^b	50.5±1.0^a	49.1±0.8^a	49.1±0.6^a

Different letters in a row denote the presence of differences statistically significant at $P \leq 0.05$

Amino acid composition of wheat-rye bread made with an addition of selected lupin flours

Table 2 presents the content of amino acids in protein of the investigated bread samples. Modification of the dough composition significantly increased the content of leucine level from 7.4 g/16 g N in the control sample to 7.7 g/16 g N in the bread with

an addition of flour from the Legat cultivar. Nevertheless, the content of this amino acid in all investigated breads exceeds its content in the standard protein FAO/WHO 1991 (6.6 g/16 g N) and DRI/USA 2005 (5.5 g/16 g N).

Table 2

Amino acid composition of wheat-rye bread made with an addition of selected lupin flours (mean ± SD)

Specification	Type of bread				
	Control K	Juno J	Polo P	Legat L	Markiz M
Essential amino acids, g/16 g N					
Thr	2.9±0.1^{a}	3.0±0.1^{a}	2.8±0.1^{a}	2.9±0.1^{a}	2.9±0.2^{a}
Met	2.1±0.1^{a}	1.7±0.1^{b}	1.8±0.1^{ab}	1.9±0.1^{a}	1.7±0.2^{ab}
Met + Cys	3.5±0.1^{a}	3.2±0.1^{b}	3.3±0.1^{ab}	3.1±0.1^{b}	3.1±0.3^{ab}
Val	5.4±0.1^{a}	5.2±0.1^{a}	5.2±0.1^{a}	5.3±0.1^{a}	5.1±0.3^{a}
Ileu	4.2±0.1^{a}	4.3±0.1^{a}	4.1±0.1^{a}	4.3±0.1^{a}	4.2±0.1^{a}
Leu	7.4±0.1^{a}	7.5±0.1^{a}	7.4±0.1^{a}	7.7±0.1^{b}	7.6±0.3^{ab}
Phe	5.3±0.1^{a}	5.0±0.1^{b}	4.9±0.1^{b}	4.9±0.1^{b}	5.2±0.1^{a}
Phe + Tyr	7.3±0.2^{a}	7.0±0.1^{a}	6.9±0.1^{a}	7.3±0.1^{a}	7.2±0.3^{a}
Lys	2.5±0.1^{a}	3.2±0.1^{b}	3.7±0.1^{b}	2.9±0.1^{b}	3.0±0.3^{b}
Try	0.8±0.1^{a}	0.8±0.1^{a}	0.8±0.1^{a}	0.8±0.1^{a}	0.8±0.1^{a}
Non-essential amino acids, g/16 g N					
Asp	4.4±0.1^{a}	5.3±0.2^{b}	5.3±0.2^{b}	5.2±0.1^{b}	5.3±0.2^{b}
Ser	4.6±0.1^{a}	4.5±0.1^{b}	4.5±0.1^{b}	4.6±0.1^{a}	4.4±0.3^{ab}
Glu	33.0±0.1^{a}	32.1±0.1^{b}	31.8±0.1^{b}	31.5±0.5^{b}	32.0±0.2^{ab}
Pro	11.3±0.1^{a}	9.7±0.1^{b}	9.5±0.1^{b}	9.7±0.1^{b}	9.9±0.4^{b}
Ala	3.4±0.1^{a}	3.4±0.1^{a}	3.6±0.1^{b}	3.5±0.2^{a}	3.4±0.2^{a}
Gli	3.6±0.1^{a}	3.6±0.2^{a}	3.6±0.2^{a}	3.6±0.1^{a}	3.7±0.1^{a}
His	2.0±0.1^{a}	2.0±0.1^{a}	2.2±0.1^{b}	2.1±0.1^{a}	2.2±0.3^{a}
Arg	3.5±0.1^{a}	5.0±0.5^{b}	5.0±0.1^{b}	4.7±0.1^{b}	4.8±0.2^{b}

Different letters in a row denote the presence of differences statistically significant at $P \leq 0.05$

Particular improvement of the protein quality in breads with the addition of lupin flour was observed in the case of lysine. Bread with the flour from the Juno cultivar contained 3.2 g/16 g N of lysine, that with an addition of the Polo cultivar – 3.7 g/16 g N; Legat – 2.9 g/16 g N, and Markiz – 3.0 g/16 g N. The content of lysine in protein of the control sample was

2.5 g/16 g N. Regarding the non-essential amino acids, a considerable increase in the amount of asparagine was recorded in all breads enriched with lupin flour: Juno – 5.3 g/16 g N, Polo – 5.3 g/16 g N, Legat -5.2 g/16 g N and Markiz – 5.3 g/16 g N, compared to the control sample (4.4 g/16 g N). An addition of flour of the Polo cultivar increased the content of alanine in the protein from 3.4 g/16 g/N (control bread) to 3.6 g/16 g N. The content of arginine increased considerably in each option of the bread enrichment with lupin flour. It was 5.0; 5.0; 4.7; 4.8 g/16 g N for the breads with the addition of flour from the Juno, Polo, Legat and Markiz cultivars, respectively, compared to the control sample – 3.5 g/16 g N. Conversely, all breads enriched with lupin flour contained considerably less methionine + cystine, while valine and phenylalanine + tyrosine remained at the comparable level to protein in the bread made from wheat and rye flour (Table 2).

Chemical and dietary indices of the protein nutritional value of wheat-rye bread made with selected lupin flours

The chemical and dietary indices of the nutritional value of protein were calculated using two amino acid standards – FAO/WHO 1991 and DRI/USA 2005 (Table 3). The content of effective protein (EP) increased after the addition of lupin flour from each cultivar under study, and it was the highest when the bread was enriched with flour from the Polo cultivar. The values obtained for this sample with the use of both of the standards were 6.7% and 7.6%, respectively. The addition of lupin flour increased the chemical score and the essential amino acid index of all investigated samples. Lysine was the limiting amino acid in each treatment. The highest CS was calculated when flour from the Polo cultivar was added (63.8%; 72.5%), likewise an increase in the EAAI calculated on the base of the FAO/WHO 1991 standard (104.7%). The highest EAAI was observed for the bread prepared with flour from the Markiz cultivar, when calculations were based on the DRI/USA 2005 standard (128.8%). The true digestibility of protein was slightly lower in all lupin enriched samples (86.2%) than in the control (86.6%). The PDCAAS increased considerably in the samples under analysis compared to the values calculated for the bread without the addition of lupin flour (37.3%; 42.4%). The largest increase was observed when flour from the Polo cultivar was added (55.0%; 62.5%). Similar values of the PER were calculated for the bread with the addition of flour from the Juno (2.73), Legat (2.75) and Markiz (2.77) cultivars, whereas the value for the bread with the addition of flour from the Polo cultivar was the same as for the bread from wheat-rye flour – 2.68 (Table 3).

Starvation, malnutrition and insufficient supply of nutrients are the problems that the contemporary world struggles with. There is a significant demand for food products of high nutritional value, obtained from stable and sustainable sources (Raikos *et al.*, 2014).

Staple foods in the majority of countries are breads of different kinds and other products made from flour. In general, one can claim that bread is the basic food, although its forms can be different in different cultures. Bakery products are made mainly from wheat flour. This material is highly valued because of its versatile physical and chemical properties, and the possibility of use in manufacturing many food products.

Table 3

Chemical and dietary indices of the protein nutritional value of wheat-rye bread made with selected lupin flours

Specification	Type of bread				
	Control K	Juno J	Polo P	Legat L	Markiz M
EP[1], %	3.9	5.6	6.7	5.4	5.7
EP[2], %	4.4	6.3	7.6	6.1	6.5
CS[1], %	43.1	55.2	63.8	50.0	51.7
Limiting amino acid[1]	Lys	Lys	Lys	Lys	Lys
CS[2], %	49.0	62.7	72.5	56.7	58.8
Limiting amino acid[2]	Lys	Lys	Lys	Lys	Lys
EAAI[1], %	100.0	102.3	104.7	102.3	102.3
EAAI[2], %	123.0	120.2	125.9	123.0	128.8
TD, %	86.6	86.2	86.2	86.2	86.2
PDCAAS[1], %	37.3	47.6	55.0	43.1	44.6
PDCAAS[2], %	42.4	54.0	62.5	48.9	50.7
PER	2.68	2.73	2.68	2.75	2.77

[1] Chemical indices of nutritional value of protein calculated on the basis of the amino acid standard FAO/WHO 1991.

[2] Chemical indices of nutritional value of protein calculated on the basis of the amino acid standard DRI/USA 2005

EP – effective protein; CS – chemical score; EAAI – integrated essential amino acid index; TD – true digestibility; PDCAAS – protein digestibility corrected amino acid score; PER – protein efficiency ratio

Discussion

A tendency has been recently observed to enrich cereal products, mainly those made from wheat and wheat-rye mixes, with flours obtained from other plants, such as lupin, green peas, beans, hemp and buckwheat. These flours are highly valued because of their functional properties, which include solubility, emulsifying capabilities, foaming and gelling properties, and water retaining capability (Raikos *et al.,* 2014). Economic, environmental and health-related factors play an important role in seeking new solutions and combinations in bakery. They include prices increase on the food market, the need to ensure supplies from stable and so far unused sources and preventing protein undernourishment. The last factor is a common

problem in developing and economically backward countries due to restricted access to animal protein (Bhat & Karim, 2009). With this issue in mind, the present study focuses on the usability of lupin in a form of flour in bread baking as an opportunity to improve the nutritional value of protein contained in this food product. Since bread, in its various forms, is highly valued around the world owing to its sensory properties, readiness to be consumed and, importantly, because of its price, one can claim that it is eaten by all social groups (Correia *et al.*, 2015). Therefore, improving the quality of protein consumed with this product can contribute to the considerable improvement of nourishment of many people.

The present study has demonstrated an alteration of the quality of protein in the wheat-rye bread prepared with an addition of flour from different lupin cultivars. An addition of lupin flour itself contributed to an increase in the total protein content in the product (Table 1). Lupin seeds have been consumed since antiquity but, apart from being used as food, they have many other applications (EFSA 2005). Interestingly, a 5% addition of lupin flour from sweet lupin cultivars has a positive effect on the organoleptic properties of bread and it delays the process of bread staling (Skibniewska *et al.* 2003). An addition of 10% lupin flour to bread generates a product with slightly different physical and chemical properties. These include rheological properties of dough, its density, colour and texture. It is encouraging that the consumer sensory assessment showed that the differences originating from the enrichment with lupin flour are practically imperceptible, which makes it possible to improve the nutritional quality without changing the product features, which mainly affect the consumers' choices (Correia *et al.*, 2015). Lupin flour and protein isolates from lupin seeds are widely used in creating new food products because their addition does not affect the taste of a final product (Gresta *et al.*, 2017). Studies have shown that an addition of lupin flour to wheat bread improves considerably the quality and amount of protein and dietary fibre in a final product. The growing interest in the use of lupin seeds in the global food production is also caused by the fact that it is not a genetically modified plant and it contains less phytoestrogens than soybean, and also that lupin flour provides less calories than refined wheat flour (Hall & Johnson, 2004; Villarino *et al.*, 2015a). Lupin seeds are rich in carotenoids, phenols and have high antioxidant potential (Siger *et al*., 2012). In Arab countries, flour from seeds of leguminous plants is used to improve the nutritional value of pita bread (traditionally made from wheat flour), because of a low level of lysine and a relatively high level of sulphur amino acids in wheat protein (Mubarak, 2001; Abu–Ghoush *et al.*, 2008). Moreover, enriching Arab bread with lupin flour has contributed to an increase in the content of total ash, dietary fibre, total fat and a decrease in carbohydrates content (Al Omari *et al.*, 2015). Lysine was the limiting amino acid in the protein of the wheat-rye bread under study. The chemical score increased after the addition of lupin flour, by even 25% in the case of the Polo cultivar (Table 3). Lupin protein is described as complementary and it perfectly supplements the diet with essential amino acids (Lampart–Szczapa *et al.*, 2003; Erbas *et al.*, 2005). Their content is estimated to be higher than in soybean and regarded as a perfect substitute of animal protein (Süssmann *et al.*, 2007). However, although European consumers express positive opinions on the consumption of plant protein, they are largely unaware that lupin seeds are not inferior to soybean in this regard (Lucas *et al.*, 2015). Lupin is an alternative to soybean in the production of fermented foods, such as Indonesian tempeh, Japanese miso and natto, and fermented spices (Rybiński *et al*., 2018). Inclusion of lupin in the human diet also brings other benefits than just the quality of protein. Studies have confirmed the positive effect of the consumption of lupin flours in diabetes because of the presence of dietary fibre, and the properties which reduce the risk of coronary disease owing to the presence of agents which decrease the blood lipid and sugar levels (Duranti, 2006; Rumiyati *et al.*, 2012; Rumiyati *et al.*, 2015; Tadele, 2015). Currently, lupin seeds, flour and

bran are used in Europe to enrich breads, pasta, cakes, muffins as well as extruded snacks and beverages. Lupin is also widely used in production of gluten-free food. Lupin germs are eaten in Australia. White lupin grown in Ethiopia under the local name of "Gibto" is baked and then used as raw material in production of alcoholic beverage called "Arekie". It is also used as a snack or a condiment called "Shiro" (Tizazu & Emire, 2010; Yeheis *et al.,* 2010). Efforts have also been made to replace animal and soy protein with lupin seeds in feeding pigs and broilers, which would have a beneficial effect on the fatty acid profile of pork and poultry (Zraly *et al.,* 2007; Mierlita 2015).

The dietetic role of lupin has been confirmed in studies on the possibility of effective regulation of appetite owing to an addition of lupin flour to wheat bread. Forty percent of wheat flour used to make dough was replaced with lupin flour. Analyses have revealed more than two-fold increase in the total protein content in bread with an addition of lupin compared to wheat bread, with an unchanged amount of total fat. Moreover, the amount of fibre increased almost four-fold. Bread with an addition of lupin flour was a rich source of protein and fibre, which partly replaced carbohydrates from wheat flour. The amount of carbohydrates was found to decrease by 30%. Consumption of bread enriched with lupin flour resulted in an increased feeling of satiety and lower (by approx. 20%) intake of energy in next meals. It affected secretion of ghrelin, glucose and insulin after a meal. This experiment has confirmed that the consumption of high protein diet rich in fibre reduces energy intake in subsequent meals more than consumption of high-carbohydrate diets (Lee *et al.,* 2006). Food enriching with lupin can be useful in treatment and prevention of obesity as well as some diseases caused by this state. As has been noted earlier in the present study, an addition of lupin flour to wheat-rye bread resulted in a considerable increase in the amount of arginine in the protein, regardless of which cultivar of lupin was used. A high content of arginine is typical of all lupin cultivars. This amino acid plays a key role in regulating lipid content in blood. A study conducted by Bähr *et al.* (2015) showed that consumption of products enriched with lupin protein (bread, sausages, vegetarian pâtés) has a beneficial effect on the blood lipid profile, in the same way as the consumption of products enriched with milk protein with an elevated concentration of arginine. Food enriched with lupin is said to have a nutraceutical potential, so it has a positive effect on human health in the treatment and prevention of diseases. Apart from these benefits, lupin proteins are known to decrease the sugar level in blood. It has been demonstrated that lupin protein extract is 10 times more potent than standard anti-diabetic drugs (Agrawal *et al.,* 2015). The main interest is focused on gamma-conglutin, which accounts for approx. 5% of all lupin seed proteins and which plays a role in the plants' defence against pathogens (Agizzio *et al.,* 2003). This bioactive peptide has not been found in bread made exclusively from refined wheat flour. In addition, such benefits like the facilitation of defecation and blood pressure decrease have been mentioned. Arginine is a precursor of the synthesis of nitrogen oxide. The mechanism of blood pressure decrease may involve relaxation of the blood vessel walls by nitrogen oxide, which is a potent endothelium relaxing factor (Sedlakova *et al.,* 2016). Other examples of bioactive lupin proteins include serine protease of the Bowman-Birk type, whose beneficial effect has been demonstrated in cancers, atrophy of skeletal muscles, angiogenesis, rheumatoid arthritis, neurodegenerative diseases and coronary disease treatments (Scarafoni *et al.,* 2008).

In the present study, the addition of lupin flour to wheat-rye bread considerably improved its *in vitro* digestibility (Table 1) and increased the PDCAAS (Table 3). A positive effect of the addition of flour from leguminous plants to wheat sourdough breads and gluten-free cakes on *in vitro* digestibility of proteins in the final product has been observed (Anyango *et al.,* 2011; Gularte *et al.,* 2012). Studies have shown that 5% addition of lupin flour

increases the PDCAAS by 15 to approx. 50%, depending on the lupin cultivar used. The findings of Villarino *et al.* (2015b) indicate that a 20% addition of lupin flour from various cultivars of narrow leaf lupin (*Lupinus angustifolius*, ASL) increases the PDCAAS by approx. 50%, regardless of the cultivar used. Such high values of these indices make it possible to compare lupin proteins and proteins of leguminous plants to animal proteins (Erbersdobler *et al.*, 2017a,b).

The effect of enriching bread with lupin flour on the protein efficiency ratio proved insignificant for all the lupin cultivars under study. The findings of animal studies presented by Monteiro *et al.* (2014) were similar and were indicative of a much greater effect of supplementation with casein on the PER.

Conclusions

The addition of lupin flour to wheat-rye bread resulted in an increase in total and digestible protein in the final product, regardless of the lupin cultivar used. Enrichment of wheat-rye flour with lupin flour from the Juno cultivar significantly increased the *in vitro* digestibility of protein in the bread. The greatest increase in the effective protein (EP), chemical score (CS), protein digestibility–corrected amino acid score (PDCAAS) and essential amino acid index (EAAI) was achieved when the bread was enriched with flour from Polo cultivar. Wheat-rye flour enriching with lupin flour from the Juno, Polo, Legat and Markiz cultivars, intended for bread making, did not have a significant effect on the protein efficiency ratio (PER). The addition of lupin flour in the process of bread making resulted in enrichment of the final products in essential amino acids lysine and leucine, and non-essential amino acids asparagine and arginine. Protein of low-alkaloid cultivars of lupin Juno, Polo, Legat and Markiz can be a valuable component which increases the nutritional value of bread protein and for this reason it should be recommended for the use in baking.

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Substantiation of a rational method of purification of sugar sorghum juice in the technology of food syrup production

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Abstract

Introduction. The purpose of research is to establish patterns for removal of macromolecular and color compounds, using different methods of purification sugar sorghum juice to obtain food syrup.

Materials and methods. Sugar sorghum hybrid 'Mamont' was used as a feedstock for food syrup. Native sorghum juice was subjected to enzymatic treatment to hydrolyze starch. For extraction of sugar-soluble non-sugars from sorghum juice, including macromolecular substances (MMS) and dyes, the cationic flocculant polyhexamethylene guanidine hydrochloride (PGMG GC) and natural mineral sorbent zeolite were used. Membrane filtration methods and ion exchange purification were used to intensify the purification process.

Results and discussion. Due to the use of zeolite at an optimal cost of 0.8–1.0% by weight of the juice, the discoloration effect is achieved at the level of 41–46%, and the effect of removal of the MMS by 20–22%.

The use of zeolite for purification of sorghum juice in combination with membrane filtration methods, such as mechanical filtration and ultrafiltration leads to improved technological performance of sorghum juice. Under these purification conditions, sugar sorghum juice with a purity of 90.72% and a colour of 245.8 ICUMSA units was obtained, and the efficiency of purification, removal MMS and proteins substances was 46.1, 82.3 and 69.5%, respectively.

Under the conditions of supplementing the above-method with ion exchange purification, we obtained an increase in the efficiency of purification, removal of the MMS and proteins in accordance with the values of 51.9, 98.5 and 89.2%.

Proposed methods of purification of sugar sorghum juice are effective in removing MMS, proteins and colored compounds and provide food syrups in which the optimal ratio of carbohydrates sucrose and glucose and fructose (65:35) % by weight of total sugar.

Conclusions. The best quality indicators were syrups obtained by purification methods, which included the use of adsorption purification with zeolite, membrane filtration and ion exchange purification.

Introduction

The priority of sugar sorghum over other sugar-bearing crops is that the juice of sugar sorghum stalks contains from 14 to 20% of carbohydrates, with sucrose amounting to 60−80% and reducing compounds 20−40%. In addition, the juice is a source of biologically active substances; it contains amino and organic acids, polyphenols, proteins, vitamins, and minerals (Eggleston G. et al., 2013; Umakanth A.V. et al., 2013). Most of the components present in the juice prevent the crystallisation of sucrose. Therefore, the juice is concentrated into a syrup that is transparent and has a mild taste, while the fructose present in it gives it a barely noticeable honey tone (Zhetkizgenkyzy G. et al., 2016). Basing on the analysis of the chemical composition of sugar sorghum syrup, that it can be compared (Willis O.O. et al., 2013) with sugar cane syrup; however, due to the high content of phenols and flavonoids, the syrup has excellent antioxidant properties, which allows its use in the food industry as a product with functional properties. Thus, obtaining food syrup from sugar sorghum is a good basis for the development and production of biologically valuable foods (Kovtunova N.A. et al., 2019; Karputina M. et al., 2014).

It was developed (Vukov K., 1987) a technology to a syrup, which included shredding stalks, juice settling, centrifugation, filtration and thickening into syrup. These studies are not technologically complete and require additional research to ensure that the syrup is of proper quality.

It is offered (Costa G. H. G. et al., 2015; Eggleston G. et al., 2016; Albuquerque F., 2011) to carry out clarification of sugar sorghum juices by application of various chemical reagents. Thus, it is suggested to clarifysugar sorghum juices using coagulants of calcium hydroxide and magnesium oxide (Costa G. H. G. et al., 2015) that are used for the removal of non-sugars from juice in the production of sugar from sugar cane (Albuquerque F., 2011). Therefore, using magnesium oxide coagulant by adding it to sorghum juice to pH 6.0 and 7.0 reduced the amount of sludge, phenolic compounds, and ash in the clarified juice, as well as improved the dynamics of the decantation process and the quality of the juice. When using calcium hydroxide, the starch content in the juice decreased. At the same time, during the decantation of non-sugars with calcium hydroxide at pH 7.0, the volume of the sludge increased.

For the production of an industrial batch of syrup, a technology was tested (Eggleston G. et al., 2016), which involved the decantation of deaerated juice in decantation tanks, hotpurification of juice at 80°C, defecation to pH 6.5 with lime milk at an application rate 360 g/l and by adding 5 ppm of polyanionic flocculant. In general, the proposed technology allowed to obtain an industrial batch of syrup of proper quality, as well as ensured uninterrupted supply of syrup to the consumer. At the same time, there is no information on the technological quality of the obtained product.

However, in the case of the use of chemical reagents in the technology of obtaining food syrup for further use as a ready food product, there is an urgent need for additional control of the source product for residual concentrations of chemical reagent in the end product.

It is proposed to use filtration and decantation of juice without the use of chemicals to obtain high-quality syrup (Nimbkar N. et al., 2006).

It is considered (Csefalvay E. et al., 2019) the possibility of using various existing technologies for the production of syrup based on the production of sugar from sugar beet and sugar cane. The efficiency of four-stage technology without using chemical reagents is shown, which provides centrifugal separation of insoluble starch from sorghum juice, application of ultrafiltration to remove proteins and microorganisms, nanofiltration to

concentrate carbohydrates to a dry matter content of 25%, and vacuum concentration to syrup. Under such conditions, it is almost possible to preserve the carbohydrate component, close to its initial ratio in the source juice. However, little information on indicators of technological quality of the product.

It was (Ospankulova G. et al., 2020) proposed a multi-stage scheme of juice clarification, which included mechanical filtration of juice, hydrolysis of starch by amylolytic enzymes, centrifugation, decolourisation of juice with active charcoal, chitosan treatment, sucrose inversion by enzyme invertase at a low-temperature evaporation mode (+58 °C). Under such conditions, good quality syrup with a slight accumulation of 5-hydroxymethylfurfural during storage and no reaction of crystallization of sucrose was obtained. The proposed technology is overloaded with technological processes, which is not always an economically sound solution.

Meanwhile, the effectiveness of natural sorbents, in particular zeolite, for clearing sugar sorghum juice, has not been studied. At the same time, the researchers (Husiatynska N. et al., 2018) proposed to use natural zeolite to improve the production quality of diffusion juice in the production of sugar from sugar beets.

Therefore, an important issue in the technology of obtaining a high-quality sugar-containing product is the maximum preservation of the carbohydrate component and useful biologically active compounds of sorghum juice.

The *aim* of the research was to establish the regularities of removal of macromolecular and colouring compounds when using different methods of sugar sorghum juice purification to obtain food syrup. To achieve this goal, the following tasks were identified:

- To carry out comparative studies of different methods of sugar sorghum juice purification in order to choose the optimal conditions of the process without the involvement of chemical reagents;
- To establish the efficiency of sugar sorghum juice clarification with zeolite sorbent and to determine effective methods of zeolite application in combination with membrane and ion exchange filtration methods at the stage of juice purification in processing natural sorghum juice;
- To establish the optimal application rate of zeolite sorbent for the process of juicepurification in order to improve production quality of the juice;
- To investigate the degree of removal of macromolecular compounds, proteins and colouring compounds from sorghum juice.

Materials and methods

Materials used in the experiment

Natural zeolite with fractions <0.3 (powder), 0.2–0.5 mm, and 1–3 mm (Transcarpathian Zeolite Plant, Sokyrnytsia, Khust district, Zakarpattia region, Ukraine.

Chemical reagent, cationic flocculant, is developed on the basis of polyhexamethylene guanidine hydrochloride (PHMG-HC) (20.0±1.5% mass), which belongs to low-hazardous substances (hazard class 4) and has approval for drinking water purification and solutions (Gembickij P.A. et al., 1998).

Membrane purification methods (Membrany, 2005), which involved the use of mechanical filtration, on a polypropylene cartridge filter Ecosoft 2.5x10" with a filtration degree of 5–10 μm and ultrafiltration on a membrane Aqua filter TLCHF-2T with a filtration degree of 0.02–0.1 μm.

Ion exchange regenerating mixture of resins DOWEXMB-50 of mixed action in the ratio of cations in H^-form and anions in OH^-form of 1.2:1.0, with a granule size of 0.35–1.2 mm and a total exchange capacity (g-equivalent/l) 1.8 H^+ and 1.0 OH^- (Dow Chemical LLC).

Sorghum stalks of sugar hybrid 'Mamont' (the breeder: Odesa Breeding and Genetic Institute − National Centre of NAAS) were used as a feedstock. They were harvested in the stage of milky-wax ripeness in the experimental field of the Institute of Bioenergy Crops and Sugar Beet NAAS (Ksaverivka 2, Vasylkiv district, Kyiv region). Cell juice was obtained from stems, which were previously cleaned of leaves and squeezed on a roller press. The obtained sorghum juice was filtered. Its physicochemical characteristics were determined in the average juice sample (Table 1).

Table 1

Technological indicators of sugar sorghum juice

Index	Average value
H^+ activity	5.0±0.22
Dry matter (DM), %	15.8±0.26
Macromolecular compounds (MMS), % DM	12.1±0.38
Proteins, %	0.87±0.1
Starch, %	3.1±0.3
Total sugars, %	12.9±0.1
Reducing sugars, %	4.5±0.05
Sucrose, %	8.4±0.05

Pre-clarification of sugar sorghum juice

Extraction of sugars from sorghum stalks was performed by pressing (Grigorenko, N.O. et al., 2017). The resulting juice, due to the content of natural starch, was subjected to enzymatic treatment (Hryhorenko N. et al., 2007). Hydrolysis of starch reduces the viscosity of juices during purification and increases the sugar content in the juice. The natural sorghum juice was heated to a temperature of 95−100 °C and kept at this temperature for 8±3 min for thermal coagulation of the MMS and proteins (Hryhorenko N. et al., 2007). Then the juice was cooled to the optimal for enzymes temperature and two-stage enzymatic hydrolysis of starch was carried out as follows: (1) dextrinization and simultaneous dilution of starch were performed with the addition of the enzyme product (α-amylase) in the amount of 2.5−3.0 units per 1g DM of starch for 30±10 min; (2) saccharification of dextrins to glucose was performed with the addition of glucoamylase in the amount of 3.0−4.0 units per 1 g of DM starch for 30±10 minutes. The resulting juice was used for research to purify and thicken to a syrup.

Technological characteristics of sorghum juice after enzymatic hydrolysis of starch are shown in Table 2.

Sorghum juice purification

Chemical reagents, natural adsorption mineral materials, membrane filtration methods, and ion exchange purification were used to cleansugar sorghum juice from macromolecular and colouring compounds. The proposed methods are shown in Table 3.

Table 2

Technological characteristics of sorghum juice after fermentation

Index	Value
H^+ activity	5.22
Dry matter (DM), %	16.3
Proteins, %	0.68
Macromolecular compounds (MMS), % DM	10.90
Total sugars, %	13.70
Reducing sugars, %	4.93
Sucrose, %	8.77
Purity, %	84.05

Table 3

Methods of sugar sorghum juice purification

Processes	Treatment1 (control)	Treatment 2	Treatment 3	Treatment 4
Pre-purification	+	+	+	+
Chemical clarification	+			
Adsorption purification		+	+	+
Mechanical filtering	+	+	+	+
Ultrafiltration	+		+	+
Ion exchange purification				+
Concentration	+	+	+	+

In order to effectively extract soluble non-sugars from sorghum juice, including MMS and colouring compounds, a chemical reagent, namelycationic flocculant polyhexamethylene guanidine hydrochloride (PHMG HC), and natural mineral sorbent, zeolite-clinoptilolite, were used in the research.

Treatment 1 − control. It is based on our previously developed technology (Hryhorenko N. et al., 2020), which provided removal of MMS and colouring substances after pre-purificationusing PHMG HC at an application rate of 0.004−0.005% a.i. and a temperature of 60 °C for 30 minutes, followed by separation of the coagulated sludge by filtration with a perlite filler on a vacuum filter and carrying out additional ultrafiltration of sorghum juice on a polyethylene terephthalate membrane with a pore diameter of 0.08−0.15 μm.

All purification methods stipulated concentration of purified juice to syrup with a dry matter content of 70−75%. The concentration process was performed using a laboratory rotary vacuum evaporator at a temperature of 60−80 °C.

Treatment 2. After pre-purification of the juice, to remove coagulated non-sugars and mechanical impurities, adsorption purification with sorbent zeolite-clinoptilolite in the amount of 1.0% masswas carried out at a temperature of 40 °C for 8−10 min followed by membrane filtration, i.e. mechanical filtration on the polypropylene cartridge filter Ecosoft 2,5x10”with a filtration degree of 5−10 μm.

Treatments 3 and 4. The juice purification was performed using zeolite-clinoptilolite sorbent in the amount of 1.0% to the weight of the juice at a temperature of 40 °C for 8−10 min, followed by mechanical filtration on a polypropylene cartridge filter Ecosoft 2.5x10"with a filtration degree of 5−10 μm, ultrafiltration using Aquafilter TLCHF-2T membrane with a filtration degree of 0.02−0.1 μm at an operating pressure of 0.1−0.15 MPa (*treatment 3*), and ion exchange sorption of sorghum juice using ion exchange regenerating resins of mixed action DOWEXM-50 in the cation exchange resin in H^- form to anion exchange resin in OH^- form ratio of 1.2:1.0 (*treatment 4*).

Determining the main technological quality indicators of sugar sorghum juice and food syrup

The research was conducted using conventional methods of control and analysisexisting in the sugar industries and accepted in international practice (Kupchyk M. et al., 2007).

The **content of total sugars**, **reducing substances**, and **sucrose** was determined by the iodometric method (Shtangeyeva N.I. et al., 2000), which is based on the reduction of alkaline copper solution with a solution of reducing substances and quantification of reduced copper oxide (I).

The purity index was calculated as the ratio of the total sugars content in the product to the dry matter content multiplied by 100% (Kupchyk M. et al., 2007).

The starch content was determined by the Morell Do Voil method (Panova T. et al., 2010**)**, which is based on measuring the optical density of the solution, acidified with acetic acid and treated with potassium iodide, using SF-46 spectrophotometer at a wavelength of 570 nm.

The **starch concentration** was determined according to the calibration curve constructed on standard solutions.

The **content of the macromolecular substances (MMS) and colloids** was determined by the method of Dumanskyy and Harin, modified by Korolkov and Silina, which is based on the properties of hydrophilic colloids to coagulate in solution after the addition of ethanol, followed by their quantitative determination by the weight method (Kupchyk M. et al., 2007).

The **protein content** was determined by the method of Reva and Simakhina (with biuret reagent) using spectrophotometer SF-46 at a wavelength of 600 nm (Kupchyk M. et al., 2007).

Dry matter content (DM, % to the weight of the product) was determined without sample preparation, at a temperature of 20 °C by the refractometric method (Kupchyk M. et al., 2007) using refractometer RPL-3.

The activity of H^+ ions was determined by the potentiometric method (Kupchyk M. et al., 2007) using universal ionometer EV-74.

The colour of the solutions was determined by photometric method, i.e. measuring the optical density of the solutions using photoelectrocolourimeter KFK-3, in a 10-mm cuvette, at a wavelength of 560 nm (Shtangeyeva N.I. et al., 2000).

Order of research

The application rate of zeolite for sorghum juice was 0−2.0% to the weight of juice. After stirring for 8−10 min, the precipitate was separated by filtration. Colour **(**Shtangeyeva N.I. et al., 2000**)** and MMS content **(**Kupchyk M. et al., 2007**)** were determined, and the

efficiency of decolourisation **(**Herasymenko O.A et al., 1992**)** and MMS removal **(**Herasymenko O.A et al., 1992**)** was calculated.

At the next technological stage, for the most complete extraction of MMS and colour compounds from sorghum juice, after treatment with zeolite in the amount of 1.0% tothe weight of juice at a temperature of 40 °C for 8−10 min, it was envisaged to carry out additional purification of the juice by membrane filtration methods, namely mechanical filtration (*treatment 2*), mechanical and ultrafiltration (*treatment 3*), with supplementing previous method by ion exchange purification (*treatment* 4). Juice purification using PHMGHC (*treatment*1) was used as a control.

At the next stage of research, to obtain an organic syrup from sugar sorghum juice, which would have improved technological quality and functional health properties, there is a need to replace the technology of purification using synthetic flocculant PHMG HC with a natural absorbent. Therefore, *treatments 3* and*4* involved the use of the natural mineral sorbent zeolite-clinoptilolite in sorghum juice purification.

Statistical analysis

Data were expressed as mean±standard deviation for three replications. Mathematical processing of the experimental data was performed using Mathcad Professional 2000 and Microsoft Excel 2007. The differences were considered significant at the LSD $\alpha = 0.95$.

Results and discussion

Study of methods of sugar sorghum juice purification and the quality of the obtained food syrups

The adsorption properties of zeolite-clinoptilolite are determined by its structure, which consists of silicon tetrahedra and alumina octahedral (Taran N.G.,1983). The high ion exchange activity of clinoptilolite is associated with the content of Al^{3+} ions (Milovych S. et al., 2010). In the case of natural sorbent, physicochemical processes of the interaction between anions of macromolecular and colouring compounds of sorghum with Al^{3+} cations of the sorbent occur due to specific adsorption forces because of their high polarization ability (Wanga Sh. et al., 2010).

In order to establish a rational application rate of zeolite-clinoptilolite and the duration of the process of sorghum juice purification, we carried out studies. Their results are given in Table 4.

The high efficiency of purification of sorghum juice is achieved by treating it with zeolite-clinoptilolite at an application rate from 0.3 to 1.5% to the weight of juice and the duration of 4–8 min. At such an application rate, a decrease in the content of the MMS was noted, which led to a decrease in the colour of sorghum juice. The optimal application rate can be considered0.8–1.0% to the weight of juice, which ensures discolouration efficiency from 41 to 46%, and the efficiency of MMS removal from 20 to 22%. The obtained results are consistent with the conclusions of other studies (Husiatynska N. et al., 2018).

Table 4

Technological characteristics of the purified sorghum juice as affected by the application rate of zeolite-clinoptilolite and the duration of the process

Index	**Application rate of reagent, % to juice weight**					
	0.1	**0.3**	**0.5**	**1.0**	**1.5**	**2.0**
Dry matter (DM), %	16.0	16.0	16.1	16.1	16.1	16.2
H^+ activity	5.13	5.13	5.13	5.13	5.13	5.13
Colour, ICUMSA units	905.6	736.6	697.8	507.3	508.2	516.7
Decolouration efficiency, %	3.68	21.65	25.78	46.04	45.95	45.04
MMS, % to DM	8.43	7.34	6.95	6.75	6.71	6.68
Efficiency of MMS removal, %	2.54	15.14	19.65	21.96	22.43	22.77

The high efficiency of purification of sorghum juice is achieved by treating it with zeolite-clinoptilolite at an application rate from 0.3 to 1.5% to the weight of juice and the duration of 4–8 min. At such an application rate, a decrease in the content of the MMS was noted, which led to a decrease in the colour of sorghum juice. The optimal application rate can be considered0.8–1.0% to the weight of juice, which ensures discolouration efficiency from 41 to 46%, and the efficiency of MMS removal from 20 to 22%. The obtained results are consistent with the conclusions of other studies (Husiatynska N. et al., 2018).

Study of the efficiency of purification of sugar sorghum juice using membrane technology

Technological characteristics of sorghum juice obtained in different treatments are presented in Table 5.

Table 5

Technological characteristics of purified sorghum juice

Index	**Treatment 1** (control)	**Treatment 2**	**Treatment 3**	**Treatment 4**
H^+ activity	5.22	5.22	5.22	4.99
Dry matter (DM), %	17.2	16.8	16.6	16.5
Macromolecular compounds (MMS), % DM	5.60	6.40	3.32	1.18
Proteins, %	0.30	0.37	0.12	0.01
Colour, ICUMSA units	254.0	469.7	245.8	128.9
Total sugars, %	15.62	14.70	15.06	15.12
Reducing sugars: % to product weight % DM	 5.82 33.84	 5.35 31.85	 5.23 31.50	 5.09 30.85
Sucrose: % to product weight % DM	 9.80 56.97	 9.35 55.65	 9.83 59.22	 10.03 60.79
Purity, %	90.81	87.50	90.72	91.64

Analysis of technological characteristics of purified juice (Table 5) confirmed that the use of cationic flocculant PHMG HC, mechanical and ultrafiltration in *treatment 1* (control) improves the quality of purified juice by precipitating MMS and colour compounds, which reduces the colour to 254.0 ICUMSA units and increase juice purity to 90.81%. This is consistent with the findings of previous studies (Hryhorenko N. et al., 2020).

It should be noted that in today's conditions (Grygorenko, N. et al., 2019) when there is a trend of excessive consumption of simple carbohydrates and synthetic sweeteners by the population, the need for organic and environmentally friendly products is becoming increasingly important. Therefore, we came to a reasonable decision, that is to withdraw chemical reagent PHMG HC from the technological process of sorghum juice purification (Hrushetskyi R.I. et al., 2016).

However, *treatment 2*, that is the use of only membrane filtration methods after pre- and adsorption purification of the juice, did not ensure its proper purification from macromolecular and colour compounds. Thus, the MMS content in purified sorghum juice remained quite high (6.40%), which significantly affected its colour, the value of which was quite high and amounted to 469.7 ICUMSA units. Accordingly, under such conditions, the purification of the juice did not significantly increase its purity (87.50%).

Efficiency sorghum sugar juice purification using zeolite

Comparative data on the technological quality of purified juices in *treatments3* and *4* (Table 5) confirmed the correct choice of purification method, which provides the most complete removal of MMS and colour compounds and improves its quality, including purity.

Thus, under the conditions of using zeolite-clinoptilolite, mechanical filtration, and ultrafiltration, there is a significant improvement in the technological characteristics of sorghum juice (*treatment3*). Under such conditions, sorghum juice was obtained with a purity of 90.72% and a colour of 245.8 ICUMSA units, which indicates the high productivity of the ultrafiltration process in combination with absorption purification of the juice with zeolite to remove suspensions substances, MMS and colouring compounds.

It should be noted that in *treatment3*, supplemented with ion exchange purification, we obtained purified juice with the lowest indicators in terms of MMS content (1.18%) and protein content (0.01%). Thus, *treatment4* provided the highest efficiency in the extraction of MMS and colouring compounds. The colour of the juice decreased almost twice compared to *treatment3* and amounted to 128.9 ICUMSA units. The purity of the purified juice was also the highest (91.64%) and comparable to all proposed methods of purification.

Confirmation of these research results is the efficiency of purification, MMS, and protein substances removal from sugar sorghum juice by the studied methods (Figure 1).

Thus, studies have shown that the best efficiency of sorghum juices purification was observed in *treatments 3* and *4*, specifically, absorption purification using zeolite-clinoptilolite, mechanical and ultrafiltration (*treatment3*), which ensured purification efficiency, removal of MMS and proteins of 46.1%, 82.3%, and 69.5%, respectively. Under the conditions of absorption purification by zeolite-clinoptilolite, mechanical filtration, ultrafiltration, and ion exchange purification (*treatment4*), purification efficiency, removal of MMS and proteins amounted to 51.9%, 98.5%, and 89.2%, respectively.

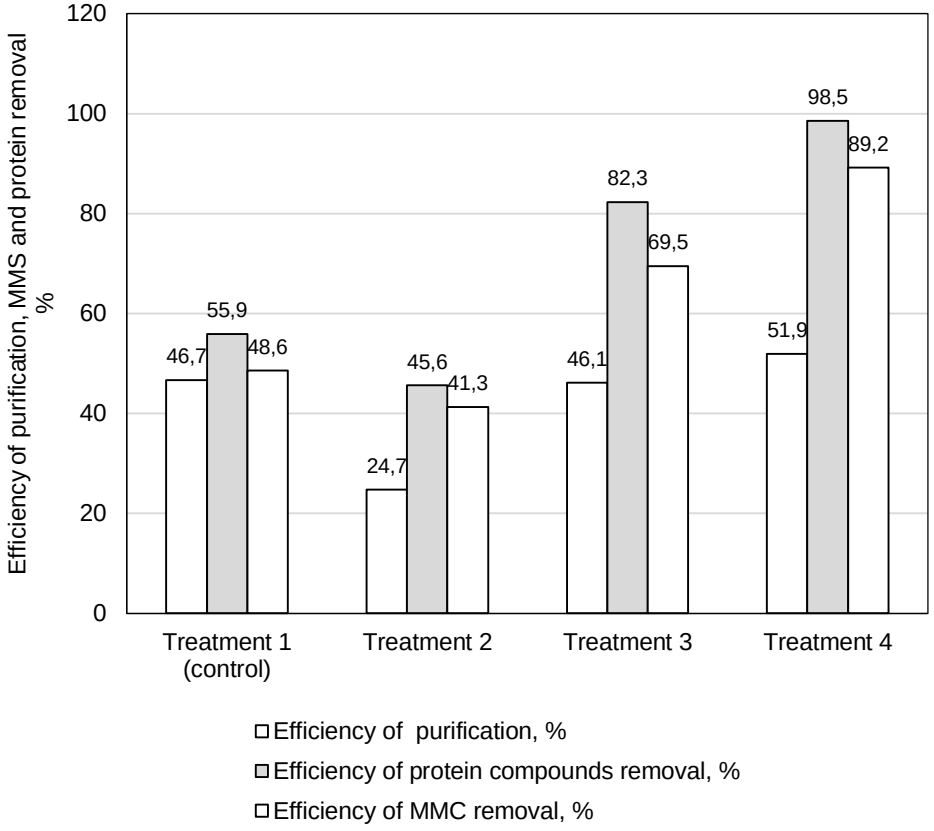

Figure 1. Efficiency of purification, MMS and protein compounds removal from sugar sorghum juice by different purification methods

Study of methods of purification of sugar sorghum juice on the quality of the obtained food syrups

The results of the studies on sensory and physicochemical characteristics of syrups obtained in accordance with the proposed methods of juice purification are presented in Table 6.

Analysis of the experimental data (Table 6) shows that the purification methods 1 and 2 did not fully ensure the removal of MMS and colouring compounds. A high colour index of the product due to the high content of MMS, proteins and colouring compounds was observed in the juices obtained by the relevant purification treatments. In addition, in the process of juice concentration to syrup at high temperatures, sugars enter into complex reactions of dehydration, melanoid formation (Mayar reaction), etc. (Eggleston G. et al., 2000; Vercellotti J. et al., 2010), which leads to intensive formation of colouring substances that affect food characteristics of the syrup in terms of colour and taste.

Table 6

Comparative sensory and physicochemical characteristics of the syrups obtained by different methods of sorghum juice purification

Characteristics	**Obtained sugar sorghum syrups**			
	Treatment 1 (control)	**Treatment 2**	**Treatment 3**	**Treatment 4**
	Sensory characteristics			
Look	Curdy consistency, transparent liquid	Curdy consistency, untransparent liquid	Curdy consistency, transparent liquid	Curdy consistency, transparent liquid
Colour	Light-brown	Dark-brown	Yellow	Light-yellow
Physical and chemical characteristics				
H^+ activity	5.25	5.24	5.22	4.87
Dry matter (DM), %	74.5	73.5	73.4	74.2
Colour, ICUMSA units	540.2	612.9	99.8	86.7
Total sugars, %	67.52	64.20	66.48	68.05
Reducing sugars: % to product weight % DM	 25.72 34.52	 24.72 33.63	 23.68 32.26	 23.20 31.27
Sucrose: % to product weight % DM	 41.80 56.11	 39.48 53.72	 42.80 58.31	 44.85 60.44
Purity, %	90.63	87.35	9.57	91.71

Confirmation of these assumptions is a more rapid increase in reducing sugars in the process of technological purification of juice and thickening to syrup in *treatments* 1 and 2 (Table 6), which led to a more intense increase in colour due to the interaction of monosaccharides with amino acids at a high temperature **(**Bugaenko I.F. et al., 2002**).**

At the same time, *treatment 3* provided quite good results in terms of syrup quality (99.8 ICUMSA units) and purity (90.57%). The best results of the quality of the syrup were obtained in *treatment 4*, with a colour of 86.7 ICUMSA units and a purity of 91.71%. Also, in these methods, there is no significant rearrangement in sugars, which indicates resource-saving conditions of technological processes of juice purification and thickening to syrup. In addition, it should be noted that these methods of purification are better from a sanitary point of view **(**Glebov A.B. et al., 2013**)** because they do not involve soluble chemical reagents **(**Patent 147898 Ukraine). To sum up the experimental data, it can be stated that the methods of sorghum juice purification used in *treatments 3* and *4* are efficient in removing MMS, proteins and colour compounds and ensure obtaining of transparent yellow and slightly amber syrup with a pleasant odour and balanced the optimal ratio of carbohydrates sucrose and glucose, fructose (65:35% mass). This fact makes it possible to recommend syrup for consumption as a natural sweetener. In addition, the developed technologies ensure the preservation of natural amino acids. We identified 19 amino acids in the products, of which seven are essential (threonine, valine, methionine, isoleucine, leucine, phenylalanine, and lysine). Of the minerals that are essential for human health, we found seven (iron, nickel,

zinc, copper, cobalt, manganese, chromium) vital trace elements that are necessary for metabolic processes, as a part of the molecules, enzymes, hormones, and vitamins. The obtained products contain micronutrients in the amount of about 2% to DM. The presence of amino acids and minerals necessary for the human body indicates their significant nutritional and biological value.

Accordingly, the syrup obtained by purification methods used in *treatments 3* and *4*, namely adsorption purification, membrane filtration, and ion exchange purification, can be used in organic food production and also be a ready-to-use sugar-containing product.

Conclusion

1. In the conditions of deteriorating food security and food quality, due to the use of synthetic sugar substitutes in food products instead of crystal sugar, the production of organic sugar-containing products from alternative feedstock, in particular, sugar sorghum, have big prospects.
2. It was found that juice purification methods that use zeolite adsorbent, mechanical filtration in combination with ultrafiltration, and ion exchange purification ensure obtaining an organic sugar-containing product with a well-balanced natural carbohydrate composition which can be widely used in the food industry as a sugar component.
3. The study shows the effectiveness of the natural adsorbent zeolite in improving the quality of sorghum juice, reducing its colour, and removing MMS and protein substances. At an application rate of zeolite of 0.8−1.0% to the weight of juice, the efficiency of discolouration is 41−46%, and the efficiency of MMS removal is 20−22%.
4. It is experimentally determined that the use of zeolite for the purification of sorghum juice in combination with membrane filtration methods, such as mechanical filtration and ultrafiltration (*treatment 3*) provides the efficiency of purification, MMS and proteins removal and at the level of 46.1%, 82.3%, and 69.5%, respectively. Under the conditions of supplementing the above-mentioned treatment with ion exchange purification (*treatment 4*) we obtained an increase in the efficiency of purification, MMS and proteins amounted to 51.9%, 98.5%, and 89.2%, respectively.

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Enzymatic hydrolysis of lactose in concentrates of reconstituted demineralized whey, intended for ice cream production

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Abstract

Introduction. The feasibility of using fermented concentrates of reconstitutd demineralized sweet whey as a source of whey proteins and monosaccharides in ice cream was proved.

Materials and methods. Physicochemical parameters of reconstituted fermented and unfermented concentrates of demineralized sweet whey with dry matter content from 10 to 40% were studied. The lactose content was determined by the accelerated colorimetric method, water activity – on the water activity analyzer.

Results and discussion. Rational regimes of enzymatic hydrolysis of lactose in concentrates of reconstituted demineralized sweet whey with dry matter content from 10 to 40% are temperature of 40–43 °C and a pH of 6.1–6.6, the recommended dose of liquid enzyme preparation GODO-YNL2, obtained from the yeast *Kluyveromyces lactis*, for concentrates with a lactose content of 7.5–30.0% ranges from 0.1 to 0.4%. The duration of enzymatic hydrolysis under these conditions for 4±2 h ensures the degree of lactose hydrolysis not less than 70%. In order to increase the degree of lactose hydrolysis simultaneously with the enzyme preparation in certain quantities single-strain lyophilized probiotic culture *«L. acidophilus LYO 50 DCU-S»* was used.

During the first 4 hours of incubation, the active acidity of the whey concentrates samples reaches values not lower than pH=5.7–5.9. At the specified acidity, the enzyme preparation GODO-YNL2 shows sufficient activity. The presence of lactose hydrolysis products to some extent stimulates the development of *L. acidophilus*. Due to the joint hydrolyzing action of enzyme and starter culture preparations for 6–8 hours, the degree of lactose hydrolysis can be achieved at the level of 80–85%.

The prospect of further research is the development of scientifically sound ice cream formulations based on hydrolyzed concentrates of reconstituted fermented whey. Ice cream enriched with whey proteins and probiotic culture of *L. acidophilus* will also have a lower content of disaccharides – sucrose and lactose.

Conclusions. The possibility of increasing the efficiency of lactose enzymolysis in concentrates of reconstituted demineralized whey to 80–85% by combining the specific action of the enzyme preparation GODO-YNL2 and starter based on *L. acidophilus* has been proved. The product of enzymolysis is of technological interest as a multifunctional ingredient in ice cream.

Introduction

Ice cream with a higher content of milk proteins is usually enriched with casein and caseinates, whey protein concentrates, as well as dry dairy products (Nadtochij et al., 2016; Polishchuk et al., 2020). It was proved the possibility of increasing the protein content by 30-90% in ice cream with a mass fraction of 10.5% fat by adding concentrates of whey proteins and milk proteins (Patel, 2006), but it significantly increases the cost of the finished product at its rather high-fat content. At the same time, reducing the caloric content of ice cream by limiting its fat content to not more than 5% is one of the urgent tasks in the field of its production. Decreased fat content or its absence in ice cream leads to defects of consistency (Goff and Hartel, 2013). Usually, problems with the formation of organoleptic quality indicators of low-fat ice cream are solved by using polysaccharides and products of their chemical modification or destruction (Azari-Anpar et al., 2017). These compounds only thicken the aqueous phase and do not affect the nutritional value of the product. Instead, whey proteins have a unique ability to mask the absence or low-fat content in ice cream (El-Zeini et al., 2016), which allows increasing the biological value of the product and ensure its high quality.

The cheapest source of biologically complete whey proteins in ice cream is condensed and dry whey. However, whey contains up to 70–75% of lactose from the total dry matter content, the excess of which in ice cream causes its excessive crystallization (Livney et al., 2007). Reducing the lactose content or its exclusion from the composition of ice cream by enzymatic hydrolysis prevents defects of consistency during storage of the hardened product (Özdemir et al., 2018; Chauhan et al. 2010). The products of lactose hydrolysis are also characterized by an increased degree of sweetness, which makes it possible to reduce the sugar content in ice cream composition. Partial hydrolysis of lactose can be achieved by incubating ice cream mixtures with lactic acid bacteria (Borovik et al., 2014). However, the combination of both methods of lactose enzymolysis in whey concentrates with the simultaneous use of enzyme and starter culture preparations has not been studied, which confirms the need for further study of this issue.

It is known that reconstituted whey is not inferior to milk as a nutrient medium for lactobacilli, and the rate of enzymatic reaction depends only on the initial lactose content in the dairy system (Lisak et al., 2011; Drgalic et al., 2005; Stehlik-Tomas et al., 2001). This gives rise to choose reconstituted whey not only as a source of protein but also lactose for its microbial fermentation.

Quite a high content of salts in the whey (up to 0.6–0.8% – in fresh, up to 8.5% – in dry) can also affect the quality of ice cream – its taste and texture (Goff and Hartel, 2013). The addition of 10 to 25% of dry whey to the ice cream reduces the cryoscopic temperature of the mixtures by more than 0.55 °C, which significantly impairs the shape stability of the ice cream (Robert et al., 2003). Therefore, it is advisable to use for the production of whey ice cream reconstituted concentrates of demineralized whey with a mass fraction of dry matter, which corresponds to their content in the finished product (20–40%).

At the same time, it is known that the most efficient whey hydrolysis occurs at a degree of demineralization of 70%. Exceeding this value reduces the content of magnesium and manganese ions in the whey, which activates the enzymatic activity of β-galactosidase (Sokolovskaja et al., 2017). Thus, hydrolyzed whey concentrates with a degree of demineralization of 70% are promising in the composition of ice cream, as they will reduce the need for sugar, prevent consistency defects and increase the nutritional value of low-lactose ice cream (Barukčić and Božanić, 2008).

The purpose of the research is to study the patterns of the process of lactose hydrolysis in reconstituted concentrates of sweet demineralized whey, intended for the production of ice cream with higher content of whey proteins and low lactose content.

So it is necessary to solve the following *tasks:*

- Choose a technologically feasible way to enrich ice cream with milk proteins;
- To determine the rational modes of enzymatic hydrolysis of lactose in concentrates of demineralized sweet whey;
- To check the possibility of lactose enzymolysis with simultaneous use of enzyme and starter culture preparations;
- To determine the possibility of partial replacement of sugar by hydrolyzed whey concentrates in ice cream composition.

Materials and methods

Materials

Demineralized whey powder was selected for the study, which contains in terms of dry matter: ash – not more than 2.5%, lactose – not less than 79%, protein – not less than 10.7%. The solubility index of dry demineralized whey is 0.5 cm ³ of the raw precipitate.

As an enzyme preparation, a liquid preparation of β-D-galactosidase hydrolase (lactase) with the trade name GODO-YNL2 (Danisko, Denmark) was used, which is a producer of selection strains of *Kluyveromyces lactis*. Under standard conditions of milk hydrolysis for 24 h at a temperature of 4.4-7.2 °C, the recommended amount of the preparation GODO-YNL2 (containing 10% β-galactosidase) is 100 g per 100 liters of milk.

For incubation of fermented samples with residual lactose content was used starter preparation *«L. acidophilus LYO 50 DCU-S»* (Danisko, Denmark), which is a single-strain lyophilized probiotic culture with the recommended dose of 5 g per 100 liters of milk.

Samples and their preparation

Dry whey was reconstituted in drinking water at a temperature of 40–45 °C, the obtained concentrates with a mass fraction of dry matter from 10 to 40% were filtered, pasteurized at a temperature of 85–88 °C for 3–5 min, cooled to a temperature of 40–43 °C (temperature range acceptable for enzymolysis with various preparations) and fermented with the preparation GODO-YNL2 and starter based on the starter preparation *«L. acidophilus LYO 50 DCU-S»* for different combinations, according to the schemes below.

The degree of lactose hydrolysis was determined by variable modes of fermentation in concentrates of reduced demineralized sweet whey with a mass fraction of dry matter from 10 to 40%. The mass fraction of the enzyme preparation was varied in the range from 0.1 to 0.4%, the duration of the fermentation process – from 1 to 10 hours.

The following schemes of fermentation process were adopted:

- **Scheme 1**. Fermentation of whey concentrates with the enzyme preparation GODO-YNL2;
- **Scheme 2**. Fermentation of whey concentrates with enzyme preparation GODO-YNL2 and starter preparation *«l. Acidophilus lyo 50 dcu-s».*

With the simultaneous application of the enzyme GODO-YNL2 and the starter preparation, it is assumed that during the lag phase of *L. acidophilus* development (2-4 h) the enzyme should have time to detect hydrolytic activity at active acidity pH ≥5.7.

Methods

Method for determining lactose content

The lactose content was determined by the accelerated colorimetric method (Teles et al., 1978) by changing the color of whey samples, which occurs due to the interaction between phenol, sodium hydroxide, picric acid, sodium bisulfide with lactose.

Methods for determining active and titrated acidity

The active acidity index was measured on a pH-meter «pH-150 MA» with a combined glass electrode «ESC 10601/4» (Tomovska et al., 2016).

Measurement of titratable acidity was carried out in accordance with a generally known method (Tomovska et al., 2016).

Method for determining water activity

Water activity was determined in whey concentrates before and after fermentation on a water activity analyzer "HygroLab 2" (Rotronic, Switzerland) at a temperature of 20 °C in the measurement range 0–1 Aw (0–100% rh) (Kuzmyk et al., 2021).

The degree of hydrolysis was expressed as a percentage, according to the lactose content of the fermented samples relative to its initial content (Livney Y. et al., 2007).

Statistical analysis

Data were expressed as means ± standard deviations for triplicate determination. Statistical analysis was performed using Microsoft Excel 2007. Differences were considered to be significant at a validity of $\alpha = 0.95$.

Results and discussion

Activity of the enzyme preparation for the lactose hydrolysis in reconstituted whey concentrates

According to scheme 1 of the fermentation process, the above, tested the effectiveness of the enzyme preparation GODO-YNL2 for the lactose hydrolysis in whey concentrates with a mass fraction of dry matter from 10 to 40%. The lactose content in the samples, respectively, ranged from 7.7 to 30.8%. The active acidity of the whey concentrates ranged from 6.6 ± 0.1 (10% dry matter) to 6.1 ± 0.1 (40% dry matter).

In table 1 shows the values of the degree of lactose hydrolysis with variable content and duration of the process. The degree of lactose hydrolysis, not less than 70% (the corresponding values are highlighted in the table with a light gray background), and the degree of lactose hydrolysis above 75% (moderately gray background) were considered high.

According to the results of the study (Table 1), a significant effect on the degree of lactose hydrolysis in variable parameters of this process (the content of the enzyme preparation and the duration of fermentation) was confirmed (Rosolen et al., 2015).

Table 1

Degree of lactose hydrolysis (%) in concentrates of reconstituted demineralized sweet whey of different dry matter content with the introduction of the enzyme preparation (P≥0.95; n = 3)*

Content of the enzyme preparation, %	Duration of fermentation, h					
	1	2	4	6	8	10
Mass fraction of dry matter 10% (mass fraction of lactose 7.7%)						
0,1	34,5±1,1	55,1±1,8	71,0±1,8	73,1±2,0	74,2±2,0	75,0±2,0
0,2	45,3±1,3	71,3±2,0	72,5±1,9	74,0±2,1	75,1±2,5	76,2±2,4
0,3	57,0±2,0	73,0±2,2	74,5±2,1	75,2±2,5	76,2±2,1	77,0±2,3
0,4	70,0±2,2	75,2±2,2	76,2±2,2	76,8±2,4	77,1±2,2	77,4±2,4
Mass fraction of dry matter 20% (mass fraction of lactose 15.4%)						
0,1	30,2±1,1	50,8±1,9	62,0±1,8	69,1±2,2	71,3±2,3	73,5±2,0
0,2	42,0±1,5	64,6±2,0	71,1±2,1	72,3±2,4	73,0±2,0	74,3±2,4
0,3	52,8±1,9	71,0±2,0	73,8±2,5	74,2±2,0	73,5±2,4	75,0±2,7
0,4	63,8±2,0	71,4±2,3	75,0±2,0	74,7±2,4	74,9±2,0	75,5±2,5
Mass fraction of dry matter 30% (mass fraction of lactose 23.1%)						
0,1	26,9±1,0	47,5±1,5	55,3±1,7	60,3±2,5	67,3±1,9	72,1±2,4
0,2	29,8±1,2	53,0±2,0	61,7±2,0	69,4±2,5	71,5±2,2	73,4±2,6
0,3	44,7±1,5	68,1±2,1	72,3±1,9	72,5±2,4	73,0±2,5	74,5±2,2
0,4	58,3±1,9	70,1±2,2	73±2,1	73,7±2,6	74,3±2,2	75,1±3,0
Mass fraction of dry matter 40% (mass fraction of lactose 30.8%)						
0,1	25,2±1,1	42,8±1,5	53,8±2,1	58,4±2,0	65,8±1,8	70,2±2,0
0,2	28,0±0,9	51,6±1,0	59,8±2,1	66,2±2,0	68,9±2,2	71,3±2,1
0,3	40,3±1,9	63,7±2,0	68,2±2,2	71,6±2,6	72,0±2,2	72,6±2,0
0,4	50,3±1,9	68,1±2,3	71,3±2,0	72,4±2,3	73,0±2,4	73,3±2,2

With the increasing duration of the process, the degree of hydrolysis increases. Moreover, in the first hours of fermentation, there is the greatest activity of the enzyme, which gradually decreases. This effect is probably due to a decrease in the lactose content in the samples and the accumulation of the content of the enzymolysis product – β-galactose, which is correlated with the known information (Mjalo et al., 2005; Hnitsevych et al., 2017). At the same time, the dynamic of the hydrolysis process of lactose is significantly influenced by the physicochemical characteristics of the samples of reconstituted demineralized whey, in particular the dry matter content. The degree of lactose hydrolysis with an increase in dry matter content from 20 to 40% is slightly reduced. This can be explained by the increase in viscosity of reconstituted whey concentrates (Sokolovskaja et al., 2017), which complicates the distribution of the enzyme in the volume of the substrate and, consequently, reduces the efficiency of the enzymatic process.

It should also be noted that with increasing lactose content in concentrates, the need for the enzyme preparation increases with a corresponding increase in the rational duration of the enzymolysis process. Thus, to obtain the maximum hydrolytic effect for a concentrate with a mass fraction of lactose of 7.7% (10% dry matter), the fermentation process is the shortest (1-2 hours) with a minimum need for the enzyme preparation – at 0.2%. To save the enzyme by reducing its content to the recommended 0.1%, the duration of hydrolysis should be extended to 4 hours. With a further gradual increase in the lactose content in concentrates to 30.8%, the degree of lactose hydrolysis is not less than 70% in the shortest time for this system (4-6 hours) is possible only with an increased content of enzyme preparation – 03-0.4%.

Therefore, increasing the content of the substrate requires the use of more enzyme preparation. At the same time, there are recommendations to reduce the need for enzyme preparation by increasing the dry matter content in the whey (Sokolovskaja et al., 2017), which requires further refinement for systems based on concentrated whey obtained in different ways.

The following pattern also attracts attention: a high degree of lactose hydrolysis (more than 75%) in the samples of reconstituted demineralized whey can be achieved only with a dry matter content of not more than 30%. Moreover, with increasing dry matter content in whey samples from 10 to 30%, the ranges of variable parameters of the hydrolysis process to achieve the maximum degree of enzymolysis are significantly narrowed, as illustrated in Table 1.

Fermentation of demineralized whey concentrates with the help of various preparations

In order to intensify the fermentation process of lactose, the possibility of combining the hydrolyzing action of the enzyme GODO-YNL2 and the lyophilized starter «*L. acidophilus LYO 50 DCU-S»* was investigated.

For this purpose, the following regularities of the fermentation process of whey concentrates with a mass fraction of dry matter from 10 to 40% by starter on the basis of the lyophilized preparation «*L. acidophilus LYO 50 DCU-S»* were established:

- Phase 1: within 3-4 hours the titrated acidity of fermented concentrates increased slightly – from 22–25 °T (pH = 6.1–6.6) to 38-42 °T (pH = 5.7–5.9), which characteristic of the lag phase of bacterial development;
- Phase 2: during the next 2–3 hours there was an active increase in acidity to values of 90-100 °T (pH = 4.2–4.36);
- Phase 3: the subsequent period was characterized by a decrease in the activity of acidophilic starter with increasing titrated acidity to 110–120 °T, which is a well-known pattern and is due to the preservative action of lactic acid as a product of lactic acid fermentation on microorganisms included in compositions of starters (Sharma et al., 2017).

Since the values of active acidity below 5.5–5.6 will inhibit the enzymatic activity of the preparation GODO-YNL2, it was decided to simultaneously add to the whey concentrates enzyme preparation GODO-YNL2 and activated starter «*L. acidophilus LYO 50 DCU-S».* The duration of the lag phase for 3-4 hours, which is observed during the incubation of whey concentrates, is sufficient for the enzyme preparation GODO-YNL2 to show its maximum activity.

To ensure the maximum possible degree of lactose hydrolysis by the enzyme preparation within 4 hours (according to Table 1) for samples of whey concentrates was selected such it amount:

- For whey concentrate with mass fraction of dry matter 10% selected 0.1% of the preparation GODO-YNL2;
- For 20% of concentrate – 0.2%;
- For 30% of concentrate – 0.3%;
- For 40% of the concentrate – 0.4%.

The degree of lactose hydrolysis in concentrates of reconstituted demineralized sweet whey with different dry matter content with simultaneous application of enzyme and fermentation preparations are given in table 2. Light gray background highlighted the values of the degree of lactose hydrolysis higher than 70%, gray – higher than 75%, dark gray – higher than 80%.

Table 2

Degree of lactose hydrolysis (%) in concentrates of reconstituted demineralized sweet whey of different dry matter content with the simultaneous introduction of enzyme and starter preparations (P≥0.95; n = 3)*

Mass fraction of dry matter in concentrate, %	Duration of fermentation, h					
	1	2	4	6	8	10
10	34,7±1,1	56,4±1,8	76,2±2,2	83,5±2,5	86,5±2,0	87,1±2,8
20	42,5±1,5	65,1±2,3	75,4±2,5	81,5±1,8	86,0±2,4	86,9±2,5
30	45,0±1,8	68,2±2,1	73,8±2,7	79,8±2,0	85,9±2,3	86,3±2,2
40	50,8±1,5	68,5±2,3	73,3±2,2	79,0±2,0	79,8±2,2	84,0±2,3

The combination of hydrolyzing action of the enzyme and starter preparations allows to increase the degree of lactose hydrolysis in the composition of lactose concentrates to 80% and above. To do this, it is necessary to ensure the duration of the total hydrolytic process in concentrates with a dry matter content of 10-30% for 6-8 hours. Prolongation of the hydrolysis process to 10 hours does not lead to a significant result in terms of changes in the composition of fermented samples of whey concentrates, except for the sample with the maximum dry matter content. It should also be noted that the presence of lactose hydrolysis products to some extent stimulates the development of *L. acidophilus* bacteria, the activity of which decreases slightly over time. This is due to the increase in osmotic pressure due to the accumulation of low molecular weight products of lactose hydrolysis in concentrates of reconstituted demineralized sweet whey from 10 to 40% (Sleator and Hill, 2002).

In contrast with recent studies (Schmidt et al., 2016; Sangwan, et al., 2015), based on the use or production of enzyme monopreparations of narrow direction, revealed a symbiotic effect of the joint interaction of the enzyme and the starter of *L. acidophilus*. The revealed symbiosis of the applied preparations increases the efficiency of lactose hydrolysis by 10–15%, compared with the use of only one enzyme GODO-YNL2.

Simultaneous use of acidophilic starter and enzyme not only increases the efficiency of lactose hydrolysis, but also has the following advantages:

- The possibility of enriching whey ice cream with probiotic culture;

– Providing ice cream with original organoleptic properties;
– Formation of a thick consistency and increasing of resistance to melting of ice cream in the presence of viscous exopolysaccharides produced by *l. Acidophilus*.

Water activity in fermented and unfermented whey concentrates with different dry matter content

The obtained products of lactose hydrolysis, in particular monosaccharides, will undoubtedly affect the osmotic pressure of the aqueous phase of ice cream and, accordingly, a slight decrease in the cryoscopic temperature of the mixtures. Shifting the cryoscopic temperature to values lower than – 4 °C is not desirable for ice cream, which is subjected to further low-temperature processing and long-term storage. Too low cryoscopic temperature reduces the melting resistance of ice cream (Polischuk et al., 2019). To check the effect of lactose hydrolysis products on the osmotic pressure of the aqueous phase of ice cream, the water activity (Aw) of fermented and unfermented concentrates of reconstituted whey with different dry matter content was studied (see Figure 1).

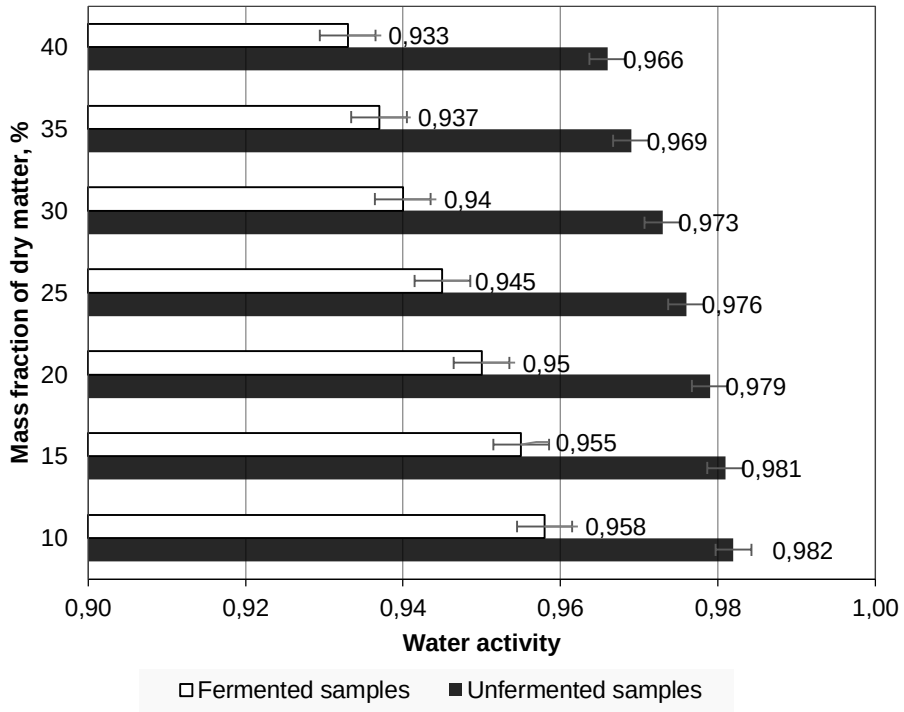

Figure 1. Water activity of unfermented and fermented samples of reconstituted demineralized sweet whey with different dry matter content

According to figure 1, the predictable and rather insignificant influence of monosaccharides on the state of water in the studied samples becomes clear (Polischuk et al., 2019). A slight decrease in Aw in fermented whey concentrates due to lactose hydrolysis will to some extent affect the processes of formation of ice cream physicochemical parameters as

a polydisperse food system. Therefore, in further work, it will be necessary to investigate the cryoscopic temperature of ice cream samples based on hydrolyzed concentrates of demineralized whey.

Degree of substitution of sugar for lactose hydrolysis products

From a physiological point of view, the feeling of sweetness is a general feeling of appetite that occurs when sweeteners stimulate specialized receptor proteins. Sweet foods, including ice cream, are energetically rich not only because of the presence of sugar itself but also because it is usually combined with fat. Even small amounts of sugar and fat together can provide a disproportionate amount of energy (Trumbo et al., 2021). Therefore, low-fat ice cream with lower sugar content will have increased demand from consumers.

It has previously been suggested that the need for sugar in ice cream may be reduced due to the greater degree of sweetness of hydrolyzed whey concentrates compared to non-hydrolyzed concentrates. Therefore, the sweetness degree of concentrates with different dry matter content was recalculated depending on the degree of lactose hydrolysis (Table 3) (Trumbo et al., 2021). The values of relative sweetness for lactose were taken as 0.16, and the values of the relative sweetness of glucose and galactose were 0.73 and 0.32, respectively.

Table 3

Relative sweetness of concentrates from demineralized whey at different dry matter content and degree of lactose hydrolysis

Mass fraction of dry matter in whey, %	The degree of lactose hydrolysis, %										
	0	10	20	30	40	50	60	70	80	90	100
10	0,012	0,015	0,017	0,020	0,023	0,025	0,028	0,031	0,033	0,036	0,039
20	0,024	0,029	0,034	0,040	0,045	0,051	0,056	0,061	0,067	0,072	0,078
30	0,036	0,044	0,052	0,060	0,068	0,076	0,084	0,092	0,100	0,108	0,117
40	0,047	0,058	0,069	0,080	0,091	0,101	0,112	0,123	0,134	0,145	0,155

According to the results of the calculation, the following is established. In the composition of milk-based ice cream, the sugar content is about 15%, and the estimated content of hydrolyzed whey concentrates can reach 50-80%. Therefore, on the example of the use of 30% fermented whey concentrate, to maintain the conventional degree of sweetness in ice cream it is possible to reduce the sugar content to 70.5-83.5%.

Thus, another, and very significant advantage of using fermented concentrates of demineralized whey in ice cream has been proved. This advantage opens up new possibilities for obtaining low-calorie ice cream, low in sugar and fat, enriched with whey proteins and probiotics, with high resistance to melting and a creamy consistency.

The prospect of further research is the development of scientifically sound formulations for low-calorie ice cream with high nutritional value based on hydrolyzed concentrates of reconstituted fermented whey. After establishing the lactose content in such ice cream, it can be attributed either to the lactose-free product (lactose < 0.1%) or to the product with reduced lactose content (lactose < 1%). With the increased content of whey proteins, the biological

value of the new product and its organoleptic and physicochemical parameters will be studied.

Conclusions

1. To enrich the ice cream with whey proteins, fermented concentrates of reconstituted demineralized sweet whey were selected, which can be not only a source of biologically complete proteins, but also perform the function of sweeteners and contain probiotics, improve the quality of the finished product.
2. Rational regimes of lactose enzymolysis in concentrates of demineralized whey with a mass fraction of dry matter from 10 to 40% were determined using the enzyme preparation GODO-YNL2 in the amount of 0.1–0.4% for the duration of the process 4±2 h at a temperature of 40–43 °C. The degree of lactose hydrolysis under these conditions reaches 70–75%.
3. The possibility of increasing the efficiency of the lactose enzymolysis process in concentrates of demineralized whey to 80–85% by combining the specific action of the enzyme preparation GODO-YNL2 and starter based on *L. acidophilus* has been proved.
4. According to the results of calculating the relative sweetness of hydrolyzed whey concentrates at different degrees of lactose hydrolysis, the possibility of replacing them with up to 70.5–83.5% of sugar in ice cream was previously established.

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Antimicrobial properties and application of fig seed oil as an additive for chitosan-based films

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Abstract

Introduction. In this study, we aimed to test whether the antimicrobial capacity of chitosan polymer could be increased synergistically when fig seed oil, plum kernel oil, apricot kernel oil were applied in different conditions.

Materials and methods. Antimicrobial effects of these oils against to certain bacterial species were evaluated with/without chitosan by agar disk/well diffusion and spectrophotometric measurement. Thin chitosan films enriched by seed oils were produced to test both antifungal and antibacterial effects *in vivo* and also in application.

Results and discussion. Although we could not obtain significant effect in culture conditions, fig seed oil singly or in combination with apricot and plum kernel oil was able to improve anti-spoilage properties of chitosan film. While fresh lemon and banana slices wrapped only ordinary cling film showed a complete deterioration, chitosan-film could substantially prohibit microbial spoilage. Moreover, almost a complete protection against microbial spoilage has been determined for these food particularly packed by chitosan-film enriched with fig seed oil. Besides, MD simulation was performed in order to evaluate putative interactions between oil compounds and chitosan. We have proposed that the most potential compound which is common in all theree oil extracts, is benzaldehyde. Herein, interaction through H-bonding was determined between between functional groups of the chitosan and benzaldehyde molecule by computational analysis, suggesting this might be one of possible factors for the observed contribution of fig seed oil and also other oil extracts to anti-spoilage effects of chitosan-based packaging film.

Conclusions. Addition of fig seed oil alone or in combination with various extracts into food packages has an application potential in order to extend the shelf life of food by alleviating deterioration for several days.

Introduction

Food deterioration or spoilage as a result of various chemical, biological, and/or physical changes, is a serious problem which is encountered during processes such as production, storage and distribution (Kong and Singh, 2011). Changes in mentioned features cause food to become unsuitable for consumption due to loss in tissue integrity, nutritional values, chemical/biological activity and benefit. Spoilage does not only render a food harmful and risky for human and public health but also causes economic losses (Rawat, 2015). The factors that cause food spoilage are classified in three main groups as chemical, physical and microbial (Odeyemi et al., 2020). Microorganisms such as bacteria and fungi (mold, yeast) are the leading biological agents which give rise to food spoilage. Some compounds produced by microorganisms on the food ruin the physical and biochemical properties such as colour, texture, nutrient content of the food and consequently poison the food (Rawat, 2015). Spoilage microorganisms include mold species such as *Penicillium*, *Aspergillus*, yeast species such as *Saccharomyces*, *Candida* and bacterial species such as *Escherichia*, *Bacillus*, *Staphylococcus* (Sperber, 2009). A variety of traditional and modern approaches such as drying, freezing, heat treatment and antimicrobial packaging, respectively, are usually applied to avoid or retard food spoilage (Amit et al., 2017).

Antimicrobial packaging, a relatively current approach, is based on the principle of constitution of a protective layer against to saprophyte microorganisms outside the food by incorporating antimicrobial agents in the packaging film (Malhotra et al., 2015). Certain natural essential oils from plants, as well as polymers such as polyvinylchloride (PVC), polypropylene (PP), high density polyethylene (HDPE), low density polyethylene (LDPE), polyethylene terephthalate (PET), polylactic acid (PLA) and chitosan are frequently prefered substances in antimicrobial films (Huang et al., 2019). The best example of antimicrobial polymers is chitosan with chemical formula [(C6H11NO4)n (C8H13NO4)m] (Marpu and Benton, 2018; Mahira et al., 2019). Chitosan has the opportunity to be used in many areas, especially food industry, and has superior properties compared to many other biopolymers in terms of its chemical and physical properties since it is prevalent and abundant in nature, less or non-toxic in human beings, biodegradable, biocompatible and antimicrobial (Marpu and Benton, 2018; Tan et al., 2015). Studies have shown that chitosan has inhibitory effects on the growth and reproduction of many microorganisms such as *Escherichia coli*, *Staphylococcus sp.*, *Bacillus sp.*, *Salmonella sp.*, *Listeria sp.*, *Micrococcus sp.*, *Vibrio sp.*, etc. (Goy et al., 2009). However, it has been determined that coating the food with chitosan film reduces the partial oxygen pressure in the package, contributes to keeping the temperature in balance by transfer of moisture between the food and its environment, prevents loss of water and delays enzymatic browning in fruits (Kahve and Duran, 2016).

On the other hand, essential and fixed oils, obtained from different parts of plants, such as flowers, buds, leaves, fruits, shells, seeds, have quite different biological activities and are another most widely used additives in the food industry (Huang et al., 2019). Some of the oils also show antimicrobial activity by altering membrane permeability of the microbial cells, causing cytotoxic effect or disrupting the cellular energy system (Chouhan et al., 2017). Due to such antimicrobial effects, certain oils are repurposed or tested in antimicrobial films/packages. For instance, grapefruit seed extract has been shown to exhibit a broad inhibition on microbial growth against both gram-positive and gram-negative bacteria (Heggers et al., 2004). In addition, chitosan-based composite films containing grapefruit seed extract as an antimicrobial and antifungal agent have been reported to have the potential to delay the incidence of fungal growth (Tan et al., 2015). In another study, various oils extracted from the seeds of jackfruit (*Artocarpus heterophyllus*), papaya (*Carica papaya*),

banana peels (*Musa acuminata*), tamarind (*Tamarindus indica*), pineapple (*Ananas comosus*), plum (*Prunus domestica*), musambi (*Citrus limetta*), strawberry (*Fragaria ananassa*), orange (*Citrus sinensis*) and guava (*Psidium guajava*) were screened for their potential antimicrobial activities (Debnath et al., 2011). Although extracts of banana, pineapple and musambi did not show any significant microbial activity contrary to bacterial and fungal strains, plum seed oil has been showed to have the strongest broad spectrum antibacterial activity and antifungal activity (Debnath et al., 2011; Savic et al., 2016). It has also reported that essential oil from apricot kernel had antimicrobial activity against various bacteria and yeasts (Lee et al 2014) and combining chitosan and apricot kernel oil in the packaging film has been shown to increase the antimicrobial effect and contributed to food preservation (Priyadarshi et al., 2018).

Despite the fact that presence of certain studies on antimicrobial properties of apricot kernel oil and plum kernel oil and their cooperation in chitosan films, the number of researches dealing with fig seed oil is almost negligible. The only existing study which published by Duman et al., revealed the chemical content of fig seed oil and demonstrated that fig seed oil had a great antifungal effect against *Candida albicans* and *Aspergillus flavus* strains and strong antibacterial effect on *Staphylococcus aureus*, *Pseudomonas aeruginosa*, *Escherichia coli*, *Enterococcus faecalis* but also weak effect on *P. aeruginosa* and *Klebsiella pneumoniae in vitro* (Duman et al., 2018). Nevertheless, application of fig seed oil as an additive in chitosan-based antimicrobial films has not been reported yet.

As it was reviewed above, the use of antimicrobial polymers such as chitosan in food packages are crucial to prevent food spoilage in order to extend shelf life. However, there are several ongoing studies about investigation of additives that are easily accessible, biocompatible, biodegradable, non-hazardous for the health and have low cost but high antimicrobial potential for antimicrobial films.

From the view of this point, we *aimed* to evaluate antimicrobial effects of fig seed oil singly and in combination with apricot kernel oil and plum kernel oil synergistically both in vitro and in vivo.

For this purpose, we have performed antimicrobial tests such as agar disk diffusion, agar well diffusion and spectrophotometric measurement against to *Escherichia coli* and *Bacillus subtilis* in the culture. Along with this, thin chitosan-based films including each oil individually and simultaneously together, were produced and tested against both saprophyte bacteria and fungi on various food by observing microorganismal colonization and by tracking the changes in physical conditions such as texture, browning, odour, rigidity, etc. Herein we demonstrated that fig seed oil alone and combination of fig seed oil, apricot kernel oil and plum kernel oil in chitosan-film conserved the food for a longer time rather than the film including chitosan only and hence we concluded that fig seed oil and the others ultimately exhibited a synergistic anti-spoilage effect in practice. Thus it has been the first study that examined the usage potential of these oils synergistically proposing a promising usage potential of fig seed oil and also in cooperation with tested oils for the antimicrobial packaging applications in near future.

Material and methods

Chitosan and seed oil extracts

Chitosan (medium molecular weight) was obtained from Sigma Aldrich (#448877-50G). The marketed seed oil extracts were supplied from reliable manufacturers in Turkey. Apricot

seed oil (*Prunus armeniaca*) and fig seed oil (*Ficus carica*) were purchased from Arifoğlu Co. (İstanbul, Turkey). Plum seed oil (*Prunus salicina*) was obtained from Neva Co. (İstanbul, Turkey). LB Broth and LB Agar (Caisson Labs, UT, USA) were prepared according to manufacturer's instructions.

Film preparation

2% (w/v) of chitosan polymer was dissolved in 1% (v/v) aqueous acetic acid (Merck, NJ, USA) solution. Solution was stirred at 70°C for 48 hours to obtain a completely homogeneous gel-like structure in liquid form. Tween 80 (0.2%, v/v) (Merck, NJ, USA) as an emulsifier and each oil (1.5%, v/v) according to experimental design (Table 1) were added into solution. First experimental group was prepared as 100 ml chitosan solution only. Each mixture was spread on the plates covered with cling film and waited for solidification at room temperature for 24 hours. By the way, one of the surface of a piece of ordinary cling film has covered with chitosan-based film as well.

Antimicrobial activity

Antimicrobial tests were carried out by using LB broth medium and LB agar-related methods. For agar well diffusion method *Escherichia coli* and *Bacillus subtilis* were streaked through the LB agar as widespread and then 1-mm diameter wells were punctured in agar to load 20 µl of solution. Concentration for oils and chitosan were used equivalent to amount in the film. In disk diffusion method, round pieces with 1.5-mm diameter were cut from the prepared thin films and placed on LB agar spreaded with bacteria. Petri dishes were incubated at 37°C overnight to measure inhibition zone around the wells or disks. Along with this, antimicrobial test were also performed by cultivation in LB broth liquid media. Chitosan and each oil were added into LB broth in a final concentration of 1.5% (w/v) and 1.5% (v/v), respectively. Upon incubation at 37°C for overnight, optic density (OD) was measured at 600 nm by UV spectrophotometer (Schimadzu, Japan) to compare relative bacterial growth between experimental groups.

Calculation of % Inhibition Rate

Escherichia coli and *Bacillus subtilis* were cultured in LB broth media containing chitosan only, seed oil extracts only and both in combination (Table 1). Bacterial growth in such conditions was measured at 600 nm wavelength to evaluate inhibitory effect relative to control by calculating % inhibition rate of substances alone or in combination. The following equation was used to calculate the percentage of inhibition on bacterial growth relative to control which does not contain either chitosan and oil in LB by using absorbance values (A) (Rolim et al., 2015).

$$\%\, inhibition\ = \frac{(Acontrol\ - Asample)}{Acontrol} \times 100$$

Absorbance obtained for each sample (Asample) was subtracted from the absorbance of control (Acontrol). Lastly, % inhibition rate was calculated by division of subtraction by the Acontrol and multiplication by %. Hence inhibition in control was accounted as 0% and inhibition rate in experimental groups have been calculated relatively compared to control. Experiment was performed in triplicate for *Escherichia coli* and average results were exploited for further analysis while it's attempted for only once for *Bacillus subtilis*.

MD Simulation

Molecular dynamics (MD) simulation was employed to explore the interaction between benzaldehyde and chitosan polymer chain. Then number of repeating unit for the polymer was considered to be 19. The ratio of the polymer/benzaldehyde molecules was considered to be 1/10. Canonical ensemble, NVT (constant number of molecules, constant volume, and constant temperature), at 300K were used to implement the molecular dynamics for a total duration of 5.0 ns using a 1.0 fs time step. AMBER force field (Bayly et al., 1995) was also used in the MD. All the simulations were performed using the LAMMPS software package (Plimpton, 1995).

Results and discussion

Antimicrobial test of chitosan, seed oil extracts and chitosan-seed oil combinations on agar culture

It was determined that the inhibition zones around the wells were quite small and there was no significant difference between the control and other groups. Observed inhibition zones were less than expected and these zones were not sufficient to measure and compare accurately since they were about/under only 1 mm on the agar *(data not shown)*. The results were similar for both types of bacteria. Because we have thought that oil extracts which were directly loaded in wells may not have been able to form an effective inhibition zone due to their inability to diffuse in water-based agar. We have attempted disk diffusion method to overcome this trouble. However, there was no notable and comparable inhibition zones around the films on agar plates seeded by *Escherichia coli* and *Bacillus subtilis* (data not shown). There were only restricted inhibition zones around contact areas of the well and film containing chitosan alone (data not shown).
Chitosan, apricot kernel oil, plum kernel oil, fig kernel oil, and chitosan-seed oil solutions were tested on *Bacillus subtilis* and *Escherichia coli* bacteria using both agar well diffusion and agar disk diffusion methods in obedience to Table 1.

Table 1

Design of control and experimental groups

Film Type	Chitosan	Apricot Kernel Oil	Plum Kernel Oil	Fig Seed Oil
Control	–	–	–	–
Ch	+	–	–	–
Ch+AKO	+	+	–	–
Ch+PKO	+	–	+	–
Ch+FSO	+	–	–	+
Ch+Triple	+	+	+	+

Antimicrobial test of chitosan, seed oil extracts and chitosan-seed oil combinations in broth medium

Results showed that chitosan alone was the most effective reducing *E. coli* growth by 48%, and unexpectedly the oil extracts could not contribute to antibacterial effect of chitosan in culture conditions (Table 2 and Figure 1).

Table 2

Average absorbance values and calculated % inhibition rates for *Escherichia coli* (average of 3 independent experiments)

Film Type	Mean Absorbance	Calculated Inhibition Rate (%)
Control	1.271	0
Ch+AKO	1.105	13
Ch+PKO	1.077	15
Ch+FSO	0.937	26
Ch+Triple	1.011	20
Ch	0.650	48

These unexpected results may have caused by the concentration of oils and density of bacterial culture. Similar with *E. coli*, chitosan was the most effective agent alone reducing bacterial growth by 92% *(data not shown)*. While Ch+AKO reduced growth by 53%, Ch+PKO, Ch+Triple and Ch+FSO had inhibition on bacterial growth with 46%, 45% and 25%, respectively obtained from only single experimental attempt *(data not shown)*. We preliminarily showed each oil with chitosan has relatively more bactericidal activity on *B. subtilis*, a gram-positive species. It was complied with a study reported gram-positive bacteria was more sensitive against certain essential oils rather than gram-negative bacteria (Erkmen & Özcan 2004). That was the reason why preliminary inhibition rates was high in *Bacillus subtilis*.

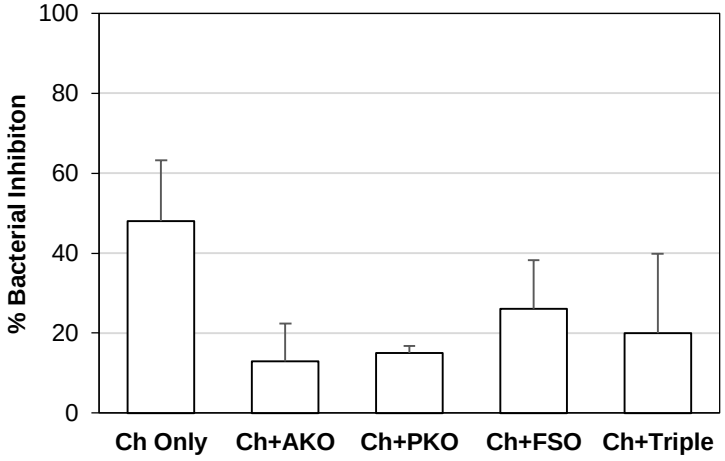

Figure 1. Relative inhibition rates on *E. coli* growth (Error bars represent SEM. #ns = statictically non-significant)

Contribution of Seed Oil Extracts to Protective Effects of Antimicrobial Chitosan Films

Chitosan-included films are already used in antimicrobial packaging. Even though we were not able to consistent results in culture conditions, we tested the usage and antimicrobial potential of apricot kernel oil, plum kernel oil and fig seed oil in the chitosan film-related application. For this purpose, we incorporated each oil extract alone or all of them synergistically into thin chitosan films and various fresh and well-conditioned foods (such as lemon, banana, bread and cheese) were packed. Slices with the similar size obtained from only the same food were preferred for packaging in order to eliminate variability and inequality such as genetic, morphological, biochemical and/or productional discrepancy. As a control, some pieces were wrapped by only cling film to isolate food from aerial environment. All of experimental groups and controls were kept in the same place and conditions (dry, dark, room temperature) and visible changes were regularly observed for 9 days.

We could acquire the most appreciable and dramatic results for lemon slices and afterward banana. At the end of the 9th day, control slice was completely decayed by observable microorganismal propagation and physical changes such as browning, dehydratation and so on (Figure 2 and Table 3). The slice wrapped by only chitosan film has stayed more durable than the control as expected even there was a trace of degradation. However, chitosan films with AKO, PKO and FSO, singly and in triplet could keep slices in more unspoilt condition relative to chitosan film alone. Along with this, same results were achieved for also banana slices. While control evolved into a bad manner, chitosan film could protect the fruit and the slice clinged with Ch+Triple film was the piece in the best condition (Table 3). However, we could not detect significant difference in the condition of bread and cheese among Ch film and Ch films including oil extracts (data not shown).

Table 3

Food spoilage rates detected according to sensory parameters such as color, odor, hardness, water loss, texture deterioration, and etc.

Deterioration level is relatively gradated between "+++++" (= most deterioration) and "+" (= least deterioration)

Film Type	Degradation Rate of Lemon Slice	Degradation Rate of Banana Slice
Control	+++++	++++
Ch	+++	+++
Ch+AKO	+	++
Ch+PKO	++	++
Ch+FSO	+	+++
Ch+Triple	+	+

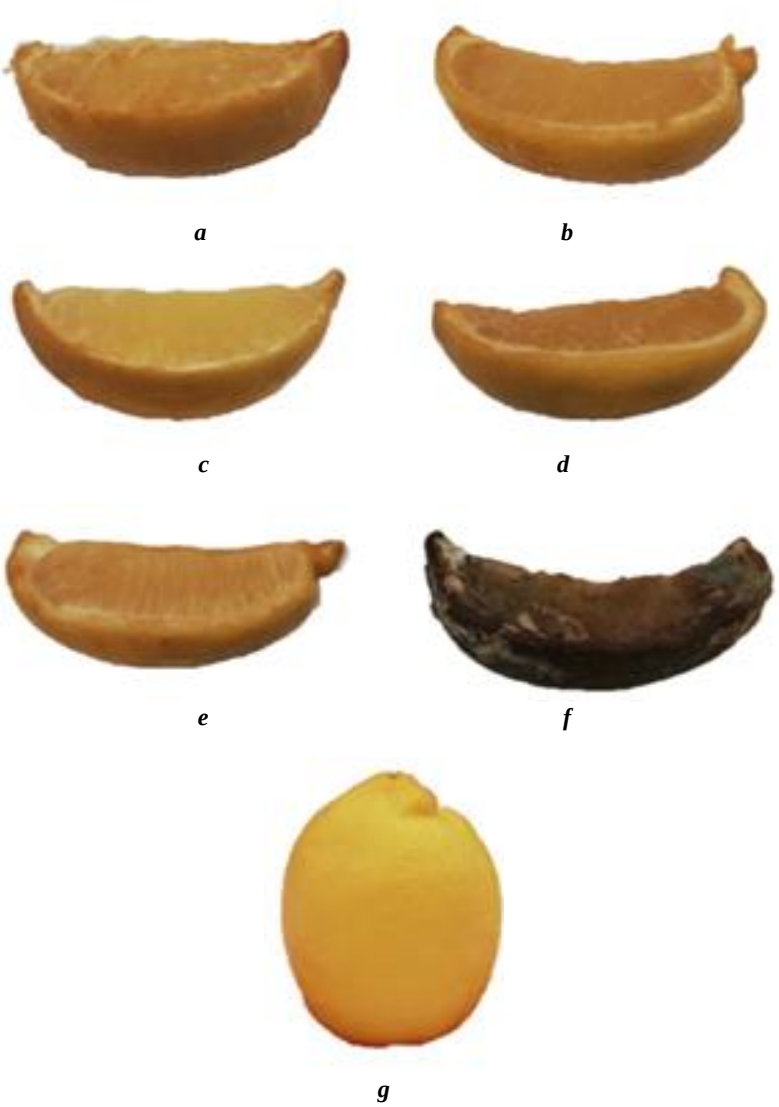

Figure 2. Captured images of lemon samples covered by chitosan-based films 9 days after packaging.
a: Ch, **b**: Ch+FSO,
c: Ch+AKO, **d**: Ch+PKO,
e: Ch+Triple, **f**: Control,
g: Status of the material in the beginning (Any post-image processing was not applied on the raw images except cropping)

Molecular Dynamics Simulation of Candidate Active Substance In Seed Oils and Chitosan

Benzaldehyde is an aromatic aldehyde of plant metabolites as flavouring agent and one of the mostly found active organic compound in apricot, plum (Verma et al., 2017; Vyviurska et al., 2017) and also in fig (Russo et al., 2017; Mujić et al., 2014). It has also been reported that benzaldehyde is one of the constituent that gains antibacterial, antifungal and antioxidant properties to the certain essential oil extracts including apricot kernel oil (Eissen et al., 2015; Alamri et al., 2012; Geng et al., 2016). At this point we have speculated and thus examined computationally whether benzaldehyde, a common ingredient in tested oils, could contribute effects of chitosan by interacting in films anywise. In order to test this capability, we performed Molecular Dynamics (MD) simulation.

The configuration of the benzaldehyde molecule on the chitosan polymer chain is represented in Figure 3. It can be seen that hydrogen bonding build up between –NH2 functional groups of the chitosan and carbonyl group of the benzaldehyde molecule. It can be expected that the interaction of the benzaldehyde molecules with the polymer chain have the ability to increase the antimicrobial activity of the chitosan due to the introduction of hydrophobicity to the polymer chain (Goy et al 2009). However, chitosan is an amphiphilic polymer (Sahariah and Másson, 2017), certain studies demonstrated that the antibacterial activity of the chitosan had improved with an increasing on the chain length of the alkyl substituent (Rabea et al., 2003; Inta et al., 2014). In these studies, researchers found that the activity could be attributed to the contribution of the hydrophobic portions on the polymer. Tamer et al. has also reported that two derivatives of chitosan prepared by 4-chloro benzaldehyde and benzophenone structures had improved the antibacterial activity of the polymer against both Gram-positive and Gram-negative strains (Tamer et al., 2016). Additionally, it was shown that N-benzylidene chitosan, synthesized by chitosan-benzaldehyde interaction, had better adsorption properties to metals and higher chemical resistance than that of chitosan (Thien et al., 2017). N-acylated chitosan was also assessed to be favorable for the interaction of polymer molecule and bacterial cell as a result of enriched hydrophobicity in polymer (Kong et al., 2010; Hu et al., 2007). On the other hand, it was investigated that antimicrobial activity of chitosan in lipid emulsions was higher than in aqueous solutions (Jumaa et al., 2002). Hereby, we have proposed that benzaldehyde might be one of the compound in the fig and other fruits which improves the chemical properties of chitosan and that could possibly be one of the reasons of upgrading anti-spoilage properties of films enriched with fig oil alone and in combination but not a significant bactericidal effect in water-based culture conditions.

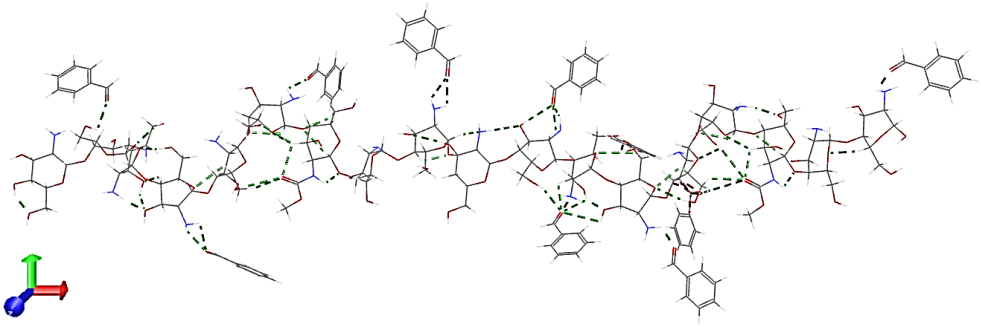

Figure 3. Hydrogen bond formation between benzaldehyde and –HN2 groups of chitosan

Conclusion

Herein, apricot kernel oil, fig seed oil and plum kernel oil in order showed a fine anti-spoilage activity by inhibiting the growth of saprophyte microorganisms such as bacteria and mold on the food. Moreover cooperation of three oil extracts had a more and synergistic protective effect contrary to deterioration of both fruits. With this study, we have demonstrated the anti-spoilage effect and usage potential of fig seed oil singly and together with other tested oils in the chitosan-based films for antimicrobial packaging, for the first time. Hence, we have concluded that fig seed oil can contribute antimicrobial effect of chitosan by alone and/or in combination with certain oils and thus it can be used as an alternative natural additive in chitosan films to produce more effective antimicrobial packages. However improvement of antimicrobial effect of chitosan films could be resulted by benzaldehyde in oil extracts. In conclusion, addition of fig seed oil into food packages may extend the shelf life of a food by keeping it fresh along days.

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Antioxidant characteristics of non-traditional spicy-aromatic vegetable raw materials for restaurant technology

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Abstract

Introduction. The aim of the study is to determine the antioxidant capacity of non-traditional for restaurant technology spicy-aromatic vegetable raw materials.

Materials and methods. Antioxidant capacity of spicy-aromatic plant raw materials: *Hyssopus officinalis L.*, *Dracocephalum moldavica L.*, *Agastache foeniculum L.*, *Melissa officinalis L.*, *Ocimum basilicum L.*, *Foeniculum vulgare Mill.*, *Glebionis coronaria L.*, *Satureja hortensis L.* were determined by redoxmetry and pH-metry of water-alcohol infusions; sensory indicators – by expert method; the results of mathematical and statistical processing – by the method of linear Pearson correlation.

Results and discussion. The hydrogen index for water-alcohol infusions from spicy-aromatic raw materials has a value of 5.28 units pH (*Hyssopus officinalis L.)* to 6.69 units pH (*Agastache foeniculum L.*). The minimum theoretical value of redox potential (*RP*) for plant water-alcohol infusions, which varies from 258.6 mV (*Agastache foeniculum L.*) to 343.2 mV (*Hyssopus officinalis L.*), was obtained. The actual measured *RP* of infusions was established – from 93 mV (*Hyssopus officinalis L.*) to 148 mV (*Glebionis coronaria L.*). Water-alcohol infusions from plant raw materials and a volume fraction of ethanol of 40% have a value of reduction energy (*RE*) in the range from 120.6 mV (*Agastache foeniculum L.*) to 250.2 mV (*Hyssopus officinalis L.*). Water-alcohol infusions from spicy-aromatic raw materials have values of sensory evaluation (*S.e.*) from 9.50 to 9.68 points. The greatest value of *S.e.* – 9.68 points characteristic of *Melissa officinalis L.*: color – light brown; taste – moderately burning, grassy; aroma – herbal, lemon.

The use of spicy-aromatic vegetable raw materials for restaurant technology is promising. Studies have confirmed the biological value of aromatic herbs for enriching tea and herbal compositions, white and red basic sauces, compotes and improving sensory evaluation.

Conclusion. The use of spicy-aromatic vegetable raw materials from *Hyssopus officinalis L.* and *Melissa officinalis L.*, which received increased antioxidant characteristics *RE* – 250.2 mV and *RE* – 184.6 mV, respectively, and positive sensory evaluation S.e. – 9.53 and S.e. – 9.68 points.

Introduction

Currently, the use of vegetable raw materials (Andreou et al., 2018; Belemets et al., 2016; Chandrasekara, Shahidi, 2018; Fotakis et al., 2016; Halliwell, Gutteridge, 1990; Hrabovska et al., 2015, 2018; Iannitti, Palmieri, 2009; Kawa-Rygielska et al., 2019; Kochubei-Lytvynenko et al., 2017) in restaurant business is very relevant (Gnytsevych et al., 2018; Gubskyi et al., 2015; Deinychenko et al., 2020; Ianchyk et al., 2016, 2018; Niemirich et al., 2015–2017; Sylchuk et al., 2016, 2017).

Current demand for high-quality spicy-aromatic vegetable raw materials involves the development of new technological methods for its preparation, with increased quality control, environmental friendliness, higher energy efficiency, lower cost and safer operation (Dainelli et al., 2008; Mujumdar, Law, 2010). These methods will allow to preserve biologically active substances (Swasdisevi et al., 2009) – volatile aromatic substances, phenolic compounds, reduce their losses (Ruan et al., 2021; Pavlyuk et al., 2018), increase organoleptic properties (Mayor, Sereno, 2004).

These biologically active substances are very sensitive to the conditions of preparation, especially to solvents – water (Tülek et al., 2020), ethyl alcohol, water-alcohol mixture (Kuzmin et al., 2020). Therefore, the conditions necessary for the efficient extraction of these compounds are specific to each plant, and this is an important issue in the process of their extraction (Tülek et al., 2020). Despite some achievements, a number of issues remain related to the preparation of spicy-aromatic plant raw materials (Priecina et al., 2018), which has antioxidant capacity. These are rare plant crops in Ukraine that are unconventional for restaurant technology (Kuzmin et al., 2020).

Spicily-aromatic vegetable raw materials contain different chemical substances that display a broad spectrum of biological activities (Frolova et al., 2019; Gerolis et al., 2017; Imark et al., 2000; Kamdem et al., 2013; Pyrzynska, Sentkowska, 2019; Sentkowska, Pyrzynska, 2018; Siddiqui et al., 2018; Steenkamp et al., 2004; Wong et al., 2020).

They have gained growing interest among scientists and consumers due to their antioxidant properties (Breiter et al., 2011; Dube et al., 2017. The ability of plant phenolics to act as free radical scavengers has led to increased interest in their ability to act as antioxidants (Herrera et al., 2018; Humia et al., 2020; Keating et al., 2014; Oh et al., 2013). Antioxidants are able to reduce the output of oxidation products: hydroperoxides, alcohols, aldehydes, ketones, fatty acids.

Spicy-aromatic raw material that exhibits antioxidant and tonic properties (Kurylo et al., 2018; Vergun et al., 2018; Vergun et al., 2019). At present, the antioxidant characteristics of all prescription components, food additives, biologically active substances and their combinations have not been sufficiently studied (Buglass et al., 2012; Grunert et al., 2018; Gullón et al., 2018; Gulua et al., 2018; Joubert, Beer, 2012).

These circumstances determine the relevance of this work, which is to develop water-alcohol extracts of vegetable raw materials in the technology of restaurants. Creating restaurant products with reduced toxicity through the introduction of spicy-aromatic infusions with antioxidant properties, allows restaurant establishments to create new products, which favorably differentiates them from competitors, creating a favorable image of the institution, which cares for the protection of consumers.

The aim of the study is to determine the antioxidant capacity of non-traditional for restaurant technology spicy-aromatic vegetable raw materials.

To achieve this goal, the following research objectives were set:

– To confirm the prospect of using non-traditional for restaurant technology spicy-aromatic vegetable raw materials;

- To establish the value of antioxidant capacity of spicy-aromatic vegetable raw materials;
- To carry out the mathematical and statistical analysis of indicators of antioxidant ability of spicy-aromatic vegetable raw materials and to establish internal correlation;
- To identify the richest sources of natural antioxidants due to spicy-aromatic plant raw materials for use in restaurant technology;
- To investigate the compositions of spicy-aromatic vegetable raw materials for restaurant technology.

Materials and methods

Materials

The study used plant raw materials that are allowed to be used in the production of restaurant products. In the *M.M. Gryshko National Botanic Garden of NAS of Ukraine* was created new cultures of spicy-aromatic plants, which became the subject of these studies (Rakhmetov, 2011).

The studies used spicy aromatic vegetable raw materials: *Hyssopus officinalis L.; Dracocephalum moldavica L.*; *Agastache foeniculum L.*; *Melissa officinalis L.*; *Ocimum basilicum L.*; *Foeniculum vulgare Mill.*; *Glebionis coronaria L.*; *Satureja hortensis L.* For preparation of extracts used the following basic raw materials: ethanol rectified, water, cardboard filtering.

Description of research procedure

Drying of spicy-aromatic vegetable raw materials was carried out in natural conditions for 6-8 days to constant humidity – not more than 14 %. Collected and inspected raw materials were laid out on clean white paper, each type separately.

The first stage – the preparation of infusions. Plant raw materials were minced into a size of 3x3 mm, suspensions of 4 g were placed into the glass bottles, were filled by 100 ml of alcohol solvent with volume fraction of rectified ethyl alcohol 40 %. The resulting infusions were cooled to 20 °C for 7 days, stirring periodically.

Next, the infusions were filtered and studies were performed to determine the indicators of active acidity, which was measured on a *pH* meter in the mode of *pH* measurement with a combined glass electrode. The *RP* was measured in the potential measurement mode with a combined redoxmetric platinum electrode.

Description of methods

Expert method of sensory evaluation

The expert method of determination of values of indexes of quality is based on the account of opinions of group highly skilled specialists-experts. (The expert of − it a specialist on the certain type of object which owns the increased sensitiveness to properties of this object) (Kuzmin et al., 2016).

Methods for determining antioxidant capacity

RP is an important indicator of the biological activity of solutions (Kuzmin O. et al., 2016; Merwe et al., 2017). It characterizes the deviation from the ionic balance of free electrons in a liquid medium. Changing the concentration of free electrons leads to a change in its electron charge and, accordingly, the *RP*. If the *RP* is positive, it indicates the oxidizing ability of the solution, negative indicates recovery ability. The value of *RP* allows to estimate the energy of processes, that is, characterizes the activity of ions in redox reactions (Bahir, 1999; Priluckij, 1997). Therefore, in order for the human body to optimally use in the exchange processes water-alcohol solutions and food, the *RP* values must correspond to the *RP* values of the internal environment of the organism, or have more negative values (Bahir, 1999).

To evaluate the antioxidant properties of the obtained water-alcoholic plant extracts, the method (Priluckij, 1997), based on the difference of *RP* in inactivated inorganic solutions and complex biochemical media. The main criteria of this method were its clarity, simplicity, specificity, reproducibility of results and efficiency. A number of researchers also emphasize that method allows to determine the total antioxidant activity of liquid products, including in total in a complex mixture, and multifunctional antioxidants (Kuzmin et al., 2016).

Formula (1) holds for inactivated inorganic solutions in equilibrium. This formula links the active acidity of the *pH* and the *RP* (Priluckij, 1997):

$$Eh_{min}=660–60\cdot pH, \text{ mV} \quad (1)$$

where Eh_{min} – the minimum theoretically expected value of the *RP*;
pH – active acidity of the test solution.

Acquired *RP* values were compared with actual measurements of Eh_{act} solution. The change of the *RP* toward the recovery energy (*RE*) was determined by the formula (Priluckij, 1997):

$$RE = Eh_{min}–Eh_{act}, \text{ mV} \quad (2)$$

where *RE* – the shift of *RP* to the side of recovered meanings (resilence);
Eh_{min} – minimal theoretically expected meaning of *RP*;
Eh_{act} – actual measured *RP*.

Mathematical and statistical methods

Pearson correlation coefficient measures the strength of the linear association between variables. Each variable should be continuous, random sample and approximately normally distributed. There are many rules of thumb on how to interpret a correlation coefficient, but all of them are domain specific. For example, here is correlation coefficient (Table 1) interpretation for behavioral sciences offered by Hinkle et al., 2003.

The correlation coefficient can take a range of values from +1 to -1. Positive correlation coefficient means that if one variable gets bigger, the other variable also gets bigger, so they tend to move in the same direction. Negative correlation coefficient means that the variables tend to move in the opposite directions: If one variable increases, the other variable decreases, and vice-versa. When correlation coefficient is close to zero two variables have no linear relationship (Hinkle et al., 2003; Shendrik et al., 2019).

Table 1

Correlation coefficient interpretation

Absolute value of coefficient (*r*)	Strength of correlation
0.90–1.00	Very high
0.70–0.90	High
0.50–0.70	Moderate
0.30–0.50	Low
0.00–0.30	Little, if any

Results and discussions

Sensory evaluation

The results of sensory evaluation (*S.e.*, points) of the obtained infusions on the extractant are presented in the Table 2 and Figures 1.

Table 2

Quality indicators of extracts on extractant

Plant raw materials	*t*, °C	*pH*	Eh_{min}, mV	Eh_{act}, mV	*RE*, mV	*S.e.*, points
1. Hyssopus officinalis L.	20	5.28	343.2	93	250.2	9.53
2. Dracocephalum moldavica L.	19	6.14	291.6	113	178.6	9.57
3. Agastache foeniculum L.	18	6.69	258.6	138	120.6	9.65
4. Melissa officinalis L.	18	6.09	294.6	110	184.6	9.68
5. Ocimum basilicum L.	19	6.22	286.8	105	181.8	9.62
6. Foeniculum vulgare Mill.	18	6.51	269.4	115	154.4	9.50
7. Glebionis coronaria L.	19	6.38	277.2	148	129.2	9.51
8. Satureja hortensis L.	19	6.03	298.2	108	190.2	9.63
9. Extractant – water-alcohol mixture	19	7.96	182.4	180	2.4	9.57
min	**18**	**5.28**	**258.6**	**93**	**120.6**	**9.50**
max	**20**	**6.69**	**343.2**	**148**	**250.2**	**9.68**

where: *t* – temperature of infusion; *pH* – active acidity of the test solution; Eh_{min} – minimal theoretically expected meaning of *RP*; Eh_{act} – actual measured *RP*; *RE* – recovery energy; *S.e.* – sensory evaluation of extracts

Antioxidant capacity

Physicochemical studies, namely determination of the *pH* level and *RP* (Nicoli et al., 2004; Prévost, Brillet-Viel, 2014), were performed according to the method (Priluckij, 1997) and calculations given above (Kuzmin et al., 2016). As a result of extraction received infusions (Andreou et al., 2018; Chandrasekara, Shahidi, 2018; Halliwell, Gutteridge, 1990; Iannitti, Palmieri, 2009; Kawa-Rygielska et al., 2019), physicochemical indicators (Breiter et al., 2011; Dube et al., 2017) of which are presented in the Table 2.

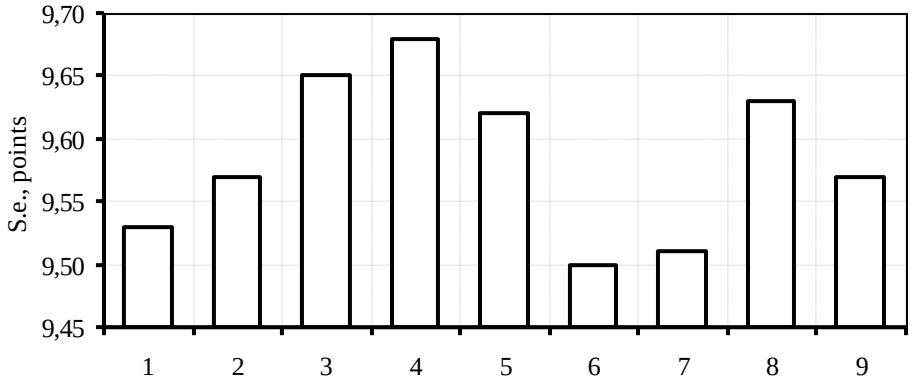

Figure 1. Sensory evaluation indicators of extracts on the extractant:
1 – Hyssopus officinalis L.; 2 – Dracocephalum moldavica L.; 3 – Agastache foeniculum L.; 4 – Melissa officinalis L.; 5 – Ocimum basilicum L.; 6 – Foeniculum vulgare Mill.; 7 – Glebionis coronaria L.; 8 – Satureja hortensis L.; 9 – Extractant – water-alcohol mixture

Figures 2–5 show graphically the change in the physicochemical indicators of the quality of extracts of spicy-aromatic raw materials on the extractant.

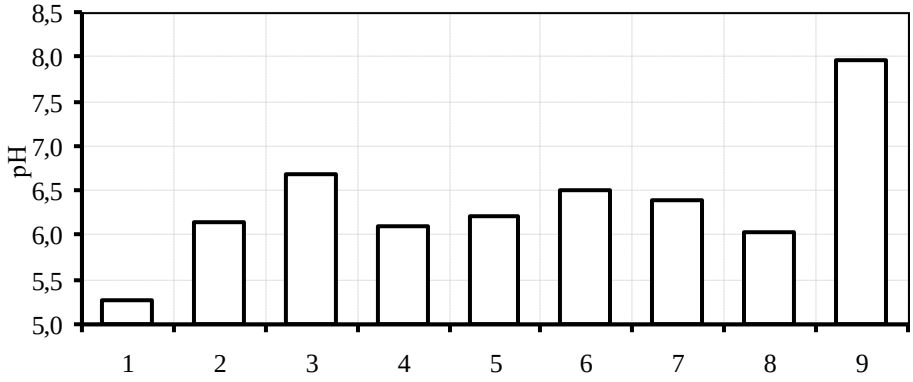

Figure 2. Hydrogen index (*pH*) of infusions of the investigated raw material:
1 – Hyssopus officinalis L.; 2 – Dracocephalum moldavica L.; 3 – Agastache foeniculum L.; 4 – Melissa officinalis L.; 5 – Ocimum basilicum L.; 6 – Foeniculum vulgare Mill.; 7 – Glebionis coronaria L.; 8 – Satureja hortensis L.; 9 – Extractant – water-alcohol mixture

The minimum theoretical value of *RP* (Eh_{min}) for plant water-alcohol infusions (Priluckij, 1997) was obtained, which has a value from 258.6 mV (*Agastache foeniculum L.*) to 343.2 mV (*Hyssopus officinalis L.*). The actual measured *RP* of infusions (Eh_{act}) was established – from 93 mV (*Agastache foeniculum L.*) to 148 mV (*Glebionis coronaria L.*). The hydrogen index for water-alcohol infusions from spicy-aromatic raw materials has a value of 5.28 units *pH* (*Hyssopus officinalis L.*) to 6.69 units *pH* (*Agastache foeniculum L.*).

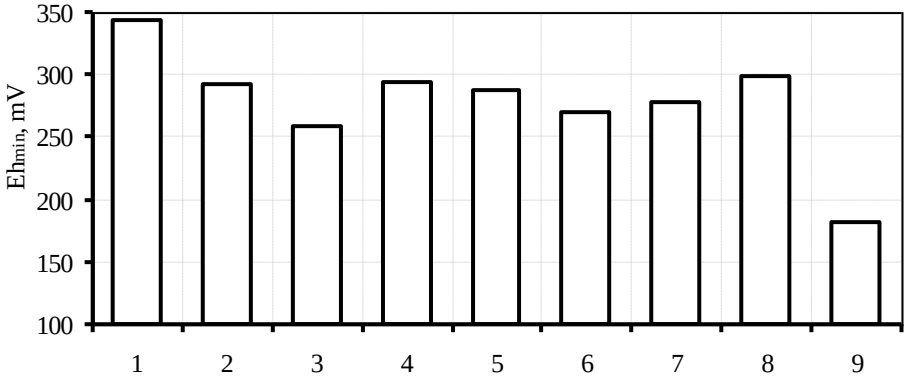

Figure 3. The minimum theoretical value of *RP* (Eh_{min}) of infusions of the investigated raw material:
1 – Hyssopus officinalis L.; 2 – Dracocephalum moldavica L.; 3 – Agastache foeniculum L.; 4 – Melissa officinalis L.; 5 – Ocimum basilicum L.; 6 – Foeniculum vulgare Mill.; 7 – Glebionis coronaria L.; 8 – Satureja hortensis L.; 9 – Extractant – water-alcohol mixture

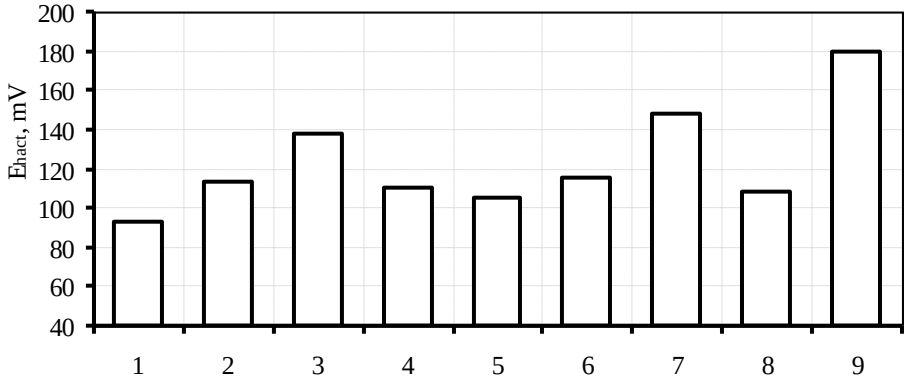

Figure 4. The actual measured *RP* of infusions (Eh_{act}) of infusions of the investigated raw material:
1 – Hyssopus officinalis L.; 2 – Dracocephalum moldavica L.; 3 – Agastache foeniculum L.; 4 – Melissa officinalis L.; 5 – Ocimum basilicum L.; 6 – Foeniculum vulgare Mill.; 7 – Glebionis coronaria L.; 8 – Satureja hortensis L.; 9 – Extractant – water-alcohol mixture

Water-alcohol infusions from vegetable raw materials and a volume fraction of ethanol of 40% have the value of regenerative capacity (recovery energy – *RE*) in the range from *RE* – 120.6 mV (*Agastache foeniculum L.*) to *RE* – 250.2 mV (*Hyssopus officinalis L.*). For the restaurant business in the manufacture of beverages are promising water-alcohol infusions of *Hyssopus officinalis L.* and *Melissa officinalis L.*, which received increased antioxidant characteristics *RE* – 250.2 mV and *RE* – 184.6 mV, respectively, and positive sensory

evaluation (*S.e.*) – 9.53 and *S.e.* – 9.68 points.

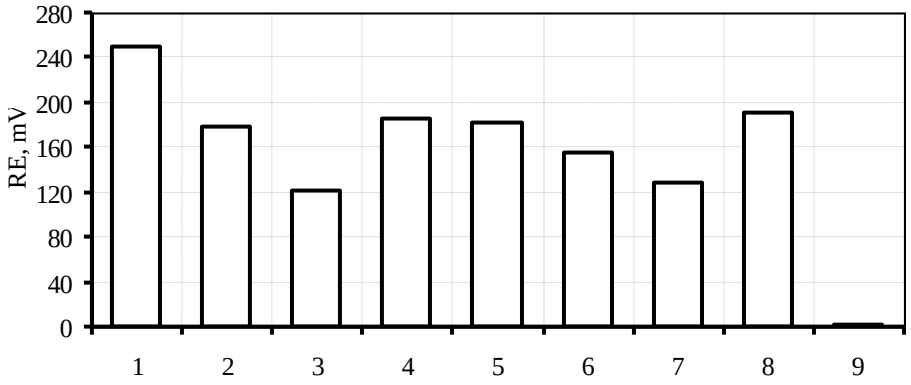

Figure 5. Recovery energy (RE) of infusions of the investigated raw material:
1 – Hyssopus officinalis L.; 2 – Dracocephalum moldavica L.; 3 – Agastache foeniculum L.; 4 – Melissa officinalis L.; 5 – Ocimum basilicum L.; 6 – Foeniculum vulgare Mill.; 7 – Glebionis coronaria L.; 8 – Satureja hortensis L.; 9 – Extractant – water-alcohol mixture

The prescription composition of alcoholic beverages may include water-alcohol infusions.

Determination of Pearson's linear correlation

According to the physicochemical and sensory evaluation, mathematical and statistical analysis (Hinkle et al., 2003; Shendrik et al., 2019) was performed in the Pearson correlation matrix (Table 3).

Table 3

Marked correlations (*r*) are significant at *p*<0,05; *N*=9

	t	***pH***	***Eh$_{min}$***	***Eh$_{act}$***	***RE***	***S.e.***
t	1.00	-0.3	0.3	-0.16	0.27	-0.38
pH	-0.33	1.0	**-1.0**	**0.91**	**-0.99**	0.00
Eh$_{min}$	0.33	**-1.0**	1.0	**-0.91**	**0.99**	-0.00
Eh$_{act}$	-0.16	**0.9**	**-0.9**	1.00	**-0.96**	-0.13
RE	0.27	**-1.0**	**1.0**	**-0.96**	1.00	0.05
S.e.	-0.38	0.0	-0.0	-0.13	0.05	1.00

where: *t* – temperature of infusion; *pH* – active acidity of the test solution; *Eh$_{min}$* – minimal theoretically expected meaning of *RP*; *Eh$_{act}$* – actual measured *RP*; *RE* – recovery energy; S.e. – sensory evaluation

According to the obtained matrix 6*6, it was found that of the 6 indicators (*t*, *pH*, *Eh$_{min}$*, *E$_{hact}$*, *RE*, *S.e.*), only 4 indicators are statistically significant. As a result of research it was

found that physicochemical parameters (*t*, *pH*, Eh_{min}, Eh_{act}, *RE*) are statistically insignificant for sensory evaluation (*S.e.*), because the correlation coefficient is very weak (*r* 0.0–0.3) and weak (*r* 0.3–0.5). Also, a weak (*r* 0.3–0.5) and very weak (*r* 0.0–0.3) relationship is observed between temperature (*t*) and other physicochemical and sensory evaluation. The range of values with very high correlation (*r* 0.9–1.0) includes the following indicators: *pH*, Eh_{min}, E_{hact}, *RE*.

Figure 6 shows the graphical dependence of *pH* on Eh_{min}. It was found that the *pH* is in the range of 5.28–7.96, and Eh_{min} – 182.4–343.2 mV. According to the obtained equation, at a *pH* value of 6.00 Eh_{min} is 300 mV. When the *pH* value changes by 1 (*pH* 7.00), the Eh_{min} decreases by 60 mV (Eh_{min} 240 mV). That is, the relationship between Eh_{min} and *pH* is very high, because *r*=-1, because it is inversely correlated, which leads to an increase in *pH* to a decrease in the level of Eh_{min}.

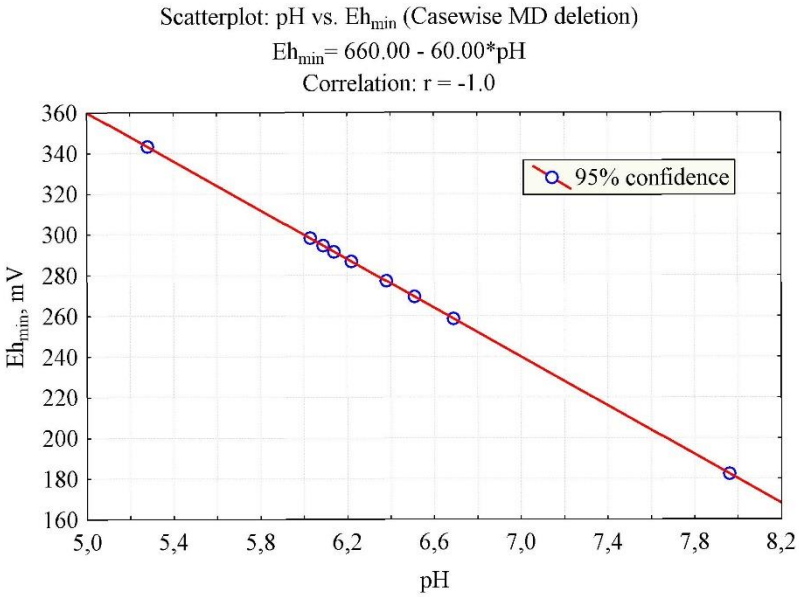

Figure 6. Dependence of *pH* level on the *RP* (Eh_{min})

Figures 7–11 show the graphical dependence of the *pH* level, Eh_{act}, Eh_{min}, *RE*.

It was found that Eh_{act} is in the range from 93 to 180 mV, and the *pH* is 5.28–7.96. At the value of Eh_{act} 111 mV, the *pH* level is 6.0. If you increase the *pH* to by one to 7.0 then the value of Eh_{act} will be 145 mV, i.e. Eh_{act} will increase by 34 mV. This is due to the fact that there is a very strong interdependence between the variables Eh_{act} and *pH* (*r*=0.9). As the *pH* value increases, the Eh_{act} index increases.

It was found that *RE* is in the range from 2.4 to 250.2 mV, and the *pH* is 5.28–7.96. When the value of *RE* 190 mV, the *pH* level is 6.0. If you increase the *pH* by one to 7.0, the value of *RE* will be 95 mV. Increasing the *pH* per unit from 6.0 to 7.0 leads to a decrease in *RE* by 94 mV. This is due to the fact that there is a very strong interdependence between the variables *RE* and *pH* (*r*=-1.0). As the *pH* value increases, the *RE* decreases.

The general graph of the three most correlation-significant physicochemical parameters is shown in Figure 12. In volumetric form, it is seen that some points (Eh_{act}, *RE*, Eh_{min}) are as close as possible to the surface, i.e. there is a very strong correlation between them. The

farther the points are from the surface, the weaker the relationship.

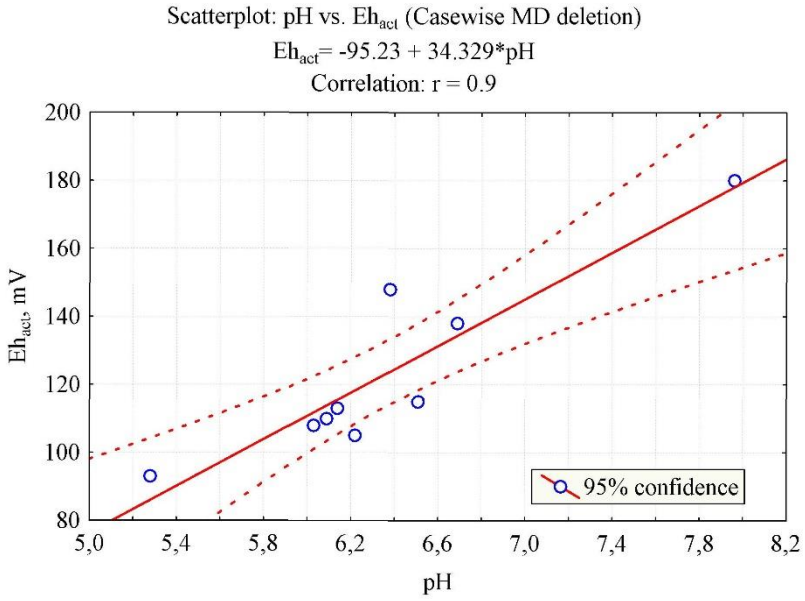

Figure 7. Dependence of *pH* level on the *RP* (*Eh$_{act}$*)

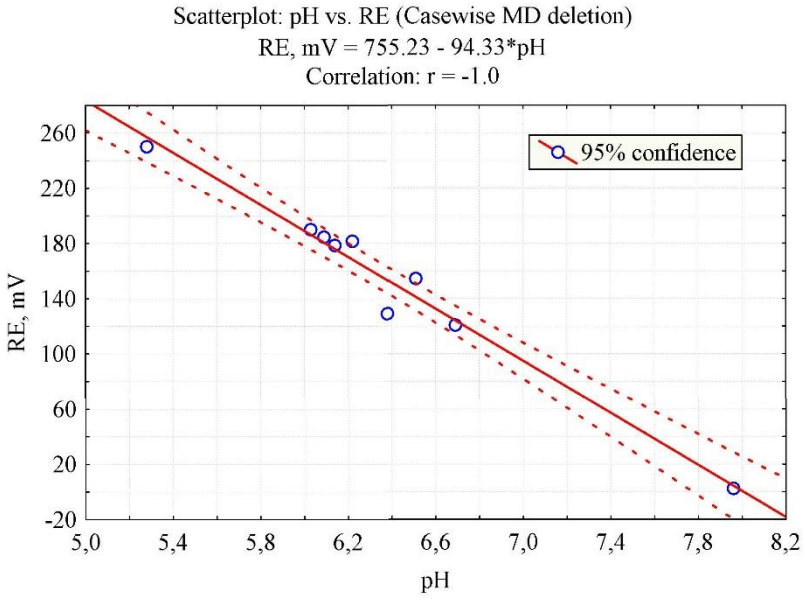

Figure 8. Dependence of *pH* level on reduction energy (*RE*)

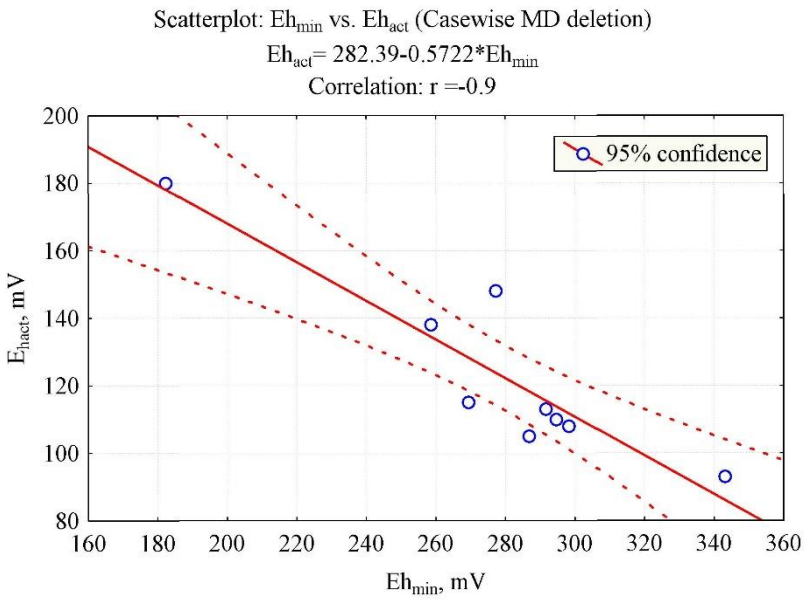

Figure 9. Dependence of *RP* (*Eh_{min}*) on *RP* (*Eh_{act}*)

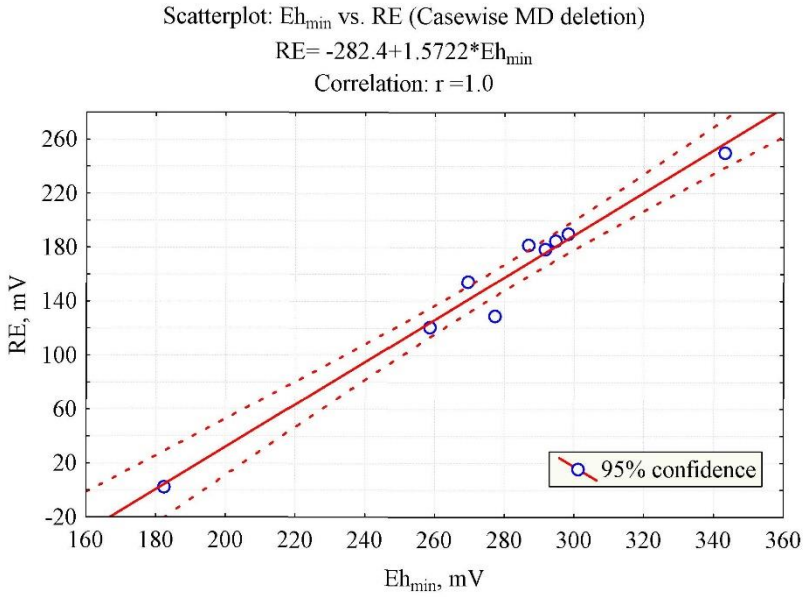

Figure 10. Dependence of *RP* (*Eh_{min}*) on reduction energy (*RE*)

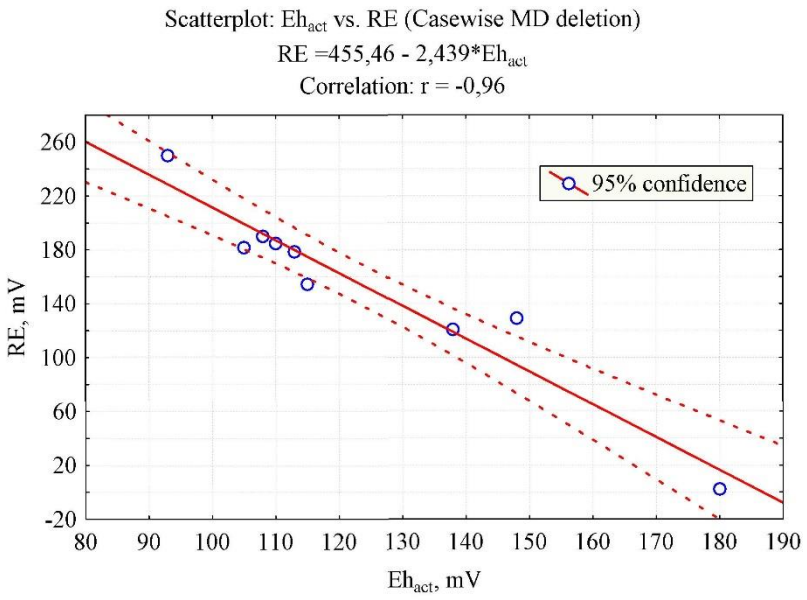

Figure 11. Dependence of *RP* (*Eh$_{act}$*) on reduction energy (*RE*)

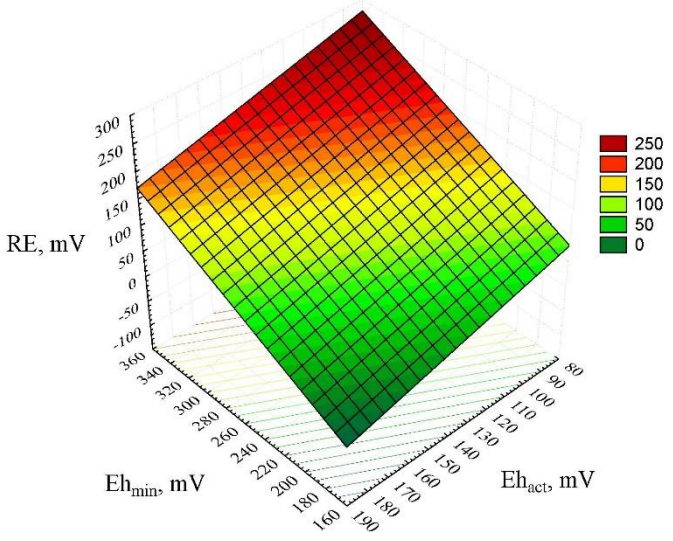

Figure 12. Response surface of *RP* (*Eh$_{act}$*) from reduction energy (*RE*) and *RP* (*Eh$_{min}$*)

Based on mathematical and statistical analysis, it was found that physicochemical parameters (*pH, Eh_{min}, Eh_{act}, RE*) are statistically insignificant for sensory evaluation (*S.e.*) and infusion temperature (*t*). The range of values with very high correlation (*r* 0.9–1.0) includes the following indicators: *pH, Eh_{min}, Eh_{act}, RE*.

Sensory evaluation of tea-herbal compositions with the addition of spicy-aromatic vegetable raw materials

The results of sensory evaluation of tea-herbal compositions are shown in table 4.

Table 4

Sensory evaluation of tea-herbal compositions

Compositions	Appearance, points	Aroma, points	Color, points	Taste, points	*S.e.*, points
Long black tea	5.0	5.0	4.9	4.8	4.925
Melissa officinalis L.	5.0	5.0	5.0	4.9	4.975
Satureja hortensis L.	4.9	4.8	4.6	4.6	4.725
Agastache foeniculum L.	5.0	5.0	4.9	4.9	4.950
Melissa officinalis L. + Satureja hortensis L.	4.9	4.9	4.9	4.7	4.850
Melissa officinalis L. + Agastache	5.0	5.0	5.0	4.9	4.975
Melissa officinalis L.+ Hyssopus officinalis L.+ Dracocephalum moldavica L.	5.0	5.0	4.9	4.6	4.875
Melissa officinalis L.+Agastache foeniculum L.+ Hyssopus officinalis L. + Dracocephalum moldavica L.	5.0	5.0	4.7	4.9	4.900

The highest score was obtained by tea-herbal compositions based on *Melissa officinalis L.* and a mixture of *Melissa officinalis L.+Agastache foeniculum L.* (1:1) – S.e. 4.975 points. The prospects of creating tea-herbal compositions based on ready-made dried mixtures have been confirmed by many authors (Alaşalvar, Çam, 2019; Tülek et al., 2020).

Sensory evaluation of white main sauce with the addition of spicy-aromatic vegetable raw materials

Evaluation of spicy-aromatic plants in mixtures showed that unsurpassed in taste, aroma and overall tasting evaluation of white main sauce based on meat broth with the addition of spicy spices (bay leaf, black peas, ground black pepper) and experimental samples of plants (broth) from beef + bay leaf + black pepper peas + ground black pepper + greens + *Hyssopus officinalis L. + Ocimum basilicum L. + Dracocephalum moldavica L.*) (Table 5).

The undoubted advantage of non-traditional for restaurant spicy-aromatic vegetable raw materials is its biological value. The presence in the composition of substances that have antimicrobial, antioxidant, hepatoprotective properties, improve digestion when used daily in small doses. They do not cause allergies, have a positive effect on the psychophysical state of man. Studies have confirmed the value and availability of spicy-aromatic vegetable raw materials to culinary dishes and can be successfully used instead of the traditional set of spices or supplement them.

Table 5

Sensory evaluation of white main sauce

Compositions	Appearance, points	Aroma, points	Color, points	Taste, points	*S.e.*, points
White sauce (main) on beef broth (control)	5.0	5.0	4.8	4.6	4.850
White sauce + bay leaf + black pepper peas + ground black pepper + greens	5.0	5.0	4.8	5.0	4.950
White sauce + *Hyssopus officinalis L.*	4.6	4.6	4.2	4.1	4.375
White sauce + *Ocimum basilicum L.*	4.9	5.0	4.6	4.6	4.775
White sauce + *Dracocephalum moldavica L.*	5.0	4.8	4.3	4.3	4.600
White sauce + *Hyssopus officinalis L.+ Ocimum basilicum L. + Dracocephalum moldavica L.*	5.0	5.0	4.3	4.8	4.775
White sauce + bay leaf + black pepper peas + ground black pepper + greens + *Hyssopus officinalis L.+ Ocimum basilicum L. + Dracocephalum moldavica L.*	5.0	5.0	5.0	5.0	5.000

Sensory evaluation of red main sauce with the addition of spicy-aromatic vegetable raw materials

The addition of spicy-aromatic vegetable raw materials to the red main sauce significantly enriched it and increased the tasting score (Table 6).

Table 6

Sensory evaluation of red main sauce

Compositions	Appearance, points	Aroma, points	Color, points	Taste, points	*S.e.*, points
Sauce red (main) on beef broth (control)	5.0	4.2	5.0	4.0	4.550
Sauce red + *Ocimum basilicum L.*	5.0	5.0	5.0	5.0	5.000
Sauce red + *Agastache foeniculum L.*	5.0	5.0	5.0	4.8	4.950
Sauce red + *Hyssopus officinalis L.*	5.0	5.0	5.0	4.2	4.800
Sauce red + *Satureja hortensis L.*	5.0	5.0	5.0	4.7	4.925
Sauce red + Dracocephalum moldavica L.	5.0	5.0	5.0	4.5	4.875
Sauce red + Glebionis coronaria L.	5.0	5.0	5.0	4.7	4.925

For the production of red main sauce used a hybrid of tomatoes «Maximato F_1», which have an increased dry matter content – 5.2%; total sugar – 3.0%; vitamin C content – 20.9 mg/100 g; total acidity – 0.51 %, which significantly exceeds other tomato hybrids.

To enrich the aroma and taste of the red main sauce, it is recommended to add spicy-aromatic herbs, especially *Ocimum basilicum L. S.e.* – 5,000 points.

Sensory evaluation of compotes with addition of spicy-aromatic vegetable raw materials

Traditionally, the healing drink in Ukraine is dried fruit compote. The aroma and taste of compotes were improved by adding spicy-aromatic vegetable raw materials (Table 7).

Table 7

Sensory evaluation of dried fruit compotes

Compositions	Appearance, points	Aroma, points	Color, points	Taste, points	S.e., points
Dried fruit compote (control)	5.0	5.0	5.0	5.0	5.000
Dried fruit compote + *Hyssopus officinalis L.*	5.0	4.9	5.0	4.9	4.950
Dried fruit compote + *Dracocephalum moldavica L.*	5.0	4.8	5.0	4.9	4.925
Dried fruit compote + *Agastache foeniculum L.*	5.0	5.0	5.0	5.0	5.000
Dried fruit compote + *Melissa officinalis L.*	5.0	4.9	5.0	4.9	4.950
Dried fruit compote + herbal mixture (*Hyssopus officinalis L., Dracocephalum moldavica L., Agastache foeniculum L., Melissa officinalis L.*)	5.0	4.8	5.0	4.8	4.900

The samples with the addition of spicy-aromatic herbs in aroma, color and taste were at the level of control (compote made from dried apples), slightly lower scores on aroma and taste can be considered insignificant compared to the fact that herbs significantly enriched the biochemical composition of the product. The specimen with the addition of *Agastache foeniculum L.* (S.e. – 5,000 point), which was better than the control, was especially distinguished.

The data obtained are correlated with the basic scientific concepts which are displayed in the works (Buglass et al., 2012; Frolova, Ukrayinets, 2018; Frolova, Korablova, 2016; Gerolis et al., 2017; Grunert et al., 2018; Gullón et al., 2018; Gulua et al., 2018; Imark et al., 2000; Joubert, Beer, 2012; Kamdem et al., 2013; Naithani et al., 2006; Naumenko et al., 2015, 2017; Pyrzynska, Sentkowska, 2019; Ruiz-Ruiz et al., 2020; Sentkowska, Pyrzynska, 2018; Siddiqui et al., 2018; Silka et al., 2016; Steenkamp et al., 2004; Wong et al., 2020), regarding the processes of extracting of plant materials.

Improving restaurant technology (Andreou et al., 2018; Chandrasekara, Shahidi, 2018; Fotakis et al., 2016; Halliwell, Gutteridge, 1990; Iannitti, Palmieri, 2009; Kawa-Rygielska et al., 2019; Kurylo et al. , 2018; Vergun et al., 2018; Vergun et al., 2019) due to the addition of spicy-aromatic raw materials. It allows to increase the antioxidant properties of the product (Breiter et al., 2011; Dube et al., 2017; Herrera et al., 2018; Humia et al., 2020; Keating et al., 2014; Kurylo et al., 2018; Oh et al., 2013; Vergun et al., 2018; Vergun et al., 2019), will help to increase the immunity of the human body, improve the metabolism, positively affect the cardiovascular system, in addition it increases the consumer properties and will allow to reduce the cost of the finished product (Kumar et al., 2018; Peschel et al., 2006; Tan et al., 2020; Wang et al., 2019).

Conclusions

1. The expediency of using non-traditional for restaurant technology spicy-aromatic vegetable raw materials in the creation of tea-herbal compositions, sauces of white and red main, compotes, in order to increase the biological value and improve sensory characteristics.
2. Experimental studies show that all aqueous-alcoholic extracts of aromatic origin contain antioxidant systems. It was found that the recovery value of all the tested extracts is positive and ranges from (*RE*) 120.6 (*Agastache foeniculum L.*) to 250.2 mV (*Hyssopus officinalis L.*).
3. Based on mathematical and statistical analysis, it was found that the infusion temperature (*t*) has a statistically insignificant effect on physicochemical parameters (*pH*, Eh_{min}, Eh_{act}, *RE*), which have a statistically insignificant effect on sensory evaluation (*S.e.*). The range of values with very high correlation (r=0.9–1.0) includes the following indicators: *pH*, Eh_{min}, Eh_{act}, *RE*.
4. Improvement of restaurant technology by adding spicy-aromatic raw materials allows to increase the redox properties of the product, increases consumer properties and reduces the cost of the finished product.
5. Compositions of spicy-aromatic vegetable raw materials for restaurant technology in the production of tea-herbal compositions, white and red main sauces, compotes of high biological value and improved sensory characteristics have been studied.

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Enzymatic destruction of protopectin in vegetable raw materials to increase its structuring ability in ice cream

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Abstract

Introduction. The expediency of protopectin enzymatic hydrolysis of vegetable raw materials as a functional-technological semi-finished product for ice cream production is proved.

Materials and methods. Rheological characteristics of fermented and unfermented vegetable purées, as well as milk-vegetable mixtures were studied. The efficiency of enzymolysis was determined by the calcium pectate method, the effective viscosity – by rotational viscosimetry, and the active acidity – potentiometrically.

Results and discussion. The purpose of the work is to study the influence of the degree of protopectin enzymatic hydrolysis of vegetable purées of different types on their structuring ability in the composition of ice cream.

The advantage of protopectin enzymatic hydrolysis in vegetable purées, compared with acid hydrolysis, is to increase the yield of soluble pectin by 8–12% at lower energy consumption. Parameters of the process of protopectin enzymolysis by the degree of protopectin hydrolysis (not less than 90%): for different types of vegetable raw materials with a pectin content of 0.22 to 2.56% were optimized. For carrots and beets, the need for the enzyme is highest (0.1–0.2%) with a prolonged duration of the fermentation process (from 120–180 minutes to 240 minutes). For zucchini, broccoli and tomatoes, the duration of process is reduced to 60-120 minutes while reducing the dose of the enzyme – up to 0.05-0.10%. At the prolongation of the enzymolysis process due to excessive hydrolysis of pectin substances, the effective viscosity of vegetable purees decreases somewhat. The thixotropic capacity of these systems is also reduced. Partial loss of functional and technological properties of vegetable raw materials due to excessive hydrolysis of pectin substances has a negative impact on the rheological characteristics of milk-vegerable mixtures for the ice cream production. Vegetable raw materials increase the effective viscosity of milk-vegetable mixtures for ice cream production under the recommended fermentation conditions, which can be explained by the formation of structuring complexes between polysaccharides and milk proteins.

Conclusions. Protopectin of enzymatic hydrolysis is more efficient than acid hydrolysis and depends on the physicochemical characteristics of vegetables. Fermented vegetable purées are structuring systems and show thixotropy in the composition of mixtures for the production of ice cream.

Introduction

In dispersed food systems, pectins as protective colloids have a stabilizing and emulsifying ability, form a creamy consistency, increase whipping and control syneresis (Voragen et al., 2009), which is important for the formation of ice cream quality indicators. Highly purified pectin preparations are usually used in ice cream. At the same time, significant theoretical and practical interest is pectin-containing raw materials, the technological properties of which are activated by hydrolytic conversion of protopectin to the active state (Polishchuk et al., 2013). Exactly soluble pectin that has the ability to form gels in an acidic environment and in the presence of sugar (Ivashhenko, 2015). This property confirms the feasibility of using pectin to form and stabilize the structure of ice cream with fruits, berries, and vegetables.

Pectin-containing vegetable raw materials have not yet been widely used in technologies of different types of ice cream, due to the specific organoleptic properties of certain types of vegetable raw materials and its relatively low structural ability, which depends on the conditions of pre-processing (Torres and Canet, 2001; Pavlyuk et al., 2018; Syed et al., 2018; Hassan and Barakat, 2018).

Activation of pectin-containing raw materials by increasing the content of soluble pectin as a product of protopectin hydrolysis increases its ability to bind water and structure food systems. Thus, the content of soluble pectin in fruit and vegetable purees can reach 1.5-2.0% (Yovbak et al., 2013; Müller-Maatsch et al., 2016).

But traditional methods of acid and alkaline activation of pectin-containing fruit and vegetable raw materials are energy-intensive and characterized by too harsh processing conditions, which leads to a partial loss of its structural ability (Canteri et al., 2012; Deynychenko et al., 2016).

Instead, the hydrolytic action of pectolytic enzymes, which is aimed at the connection of protopectin with cellulose and hemicellulose, allows to more effectively carry out its transition to soluble form with maximum preservation of native properties (Zapata A. D et al., 2017). The kinetics and dynamics of the enzymolysis process can be easily regulated (Abbès et al., 2011), which is also a significant advantage of this method of destruction of protopectin.

Pectinase is effectively used to increase juice yield and improve filtration during juice extraction, as well as to clarify juices and wine materials. The enzyme preparation also allows to obtain fruit purees and jellies (Zapata A. D. et al., 2009; Habibrahmanova et al., 2018). At the same time, protopectin enzymolysis should be carried out only to a technologically appropriate level, which requires additional research.

The main way to inactivate enzyme preparations is high temperature. But the thermal effect on fermented vegetable raw materials should be short-lived for maximum preservation of thermally unstable biologically active compounds (vitamin C, B vitamins, etc.) (Gonzalez and Rosso, 2011). Vegetables that are promising for ice cream production have different pulp strength, different content, and properties of pectin substances (Voragen et al., 2009), which requires further detailed study in each case.

Thus, the possible study of enzymatic processing of vegetable raw materials of different types in order to purposefully influence its structuring ability in the composition of ice cream is an urgent scientific task.

The *purpose* of the work is to study the effect of protopectin enzymatic hydrolysis in vegetable purées on their structuring ability in ice cream.

The following research *tasks* were formed:

- Choose the most promising types of vegetable raw materials for ice cream production;

- To conduct a comparative analysis of the acid and enzymatic hydrolysis effectiveness of vegetable raw materials by the degree of protopectin destruction;
- To establish the optimal modes of protopectin enzymatic hydrolysis in vegetable raw materials;
- To investigate the influence of the hydrolysis degree of the protopectin on the structuring ability of vegetable purees and milk-vegetable mixtures for ice cream production.

Materials and methods

Materials

The most promising and little-studied vegetables for use in ice cream were selected for the research: table beets, zucchini, broccoli and tomatoes. Carrots were studied as a traditional vegetable for ice cream production with a maximum content of pectin (Hassan and Barakat, 2018).

It was selected the most affordable and relatively cheap varieties of vegetables grown in Ukraine: table beets "Delicatessen", broccoli "Jaguar", carrots "Queen of Autumn", tomatoes "Asterix F1", zucchini "Cavili".

As a hydrolyzing preparation enzyme Pectolad (SE "Enzyme", Ukraine) with a pectolytic activity of at least 30 units/g. Pectolad belongs to pectinases. The preparation contains a complex of pectolytic enzymes (endo-α1,4-polygaclacturonase, exo-α1,4-polygalacturonase) and catalyzes the hydrolysis of internal 1-4-linked α-D-galacturonoside bonds in the main chain of polygalacturonates and pectin substrates with low degree of esterification. The enzyme preparation is obtained by targeted deep fermentation of the strain *Aspergillus foetidus*. The recommended dosage of pectolad, depending on the type of raw material and technological process, is on average 50–100 g/100 liters. The operating temperature range of the enzyme at an acidity of pH 2.0–5.2 is 25–55 °C.

Methods

Quantitative pectin content was determined by calcium pectate method (Podkorytova and Kadnikova, 2009).

The viscosity characteristics of fermented and unfermented vegetable purées and milk-vegetables blends were determined on a rotary viscometer with a measuring system cylinder-cylinder by removing the curves of deformation kinetics (flow). The measurements were performed at a temperature of 20 °C. Shear stress measurements τ (Pa) were performed at twelve values of the shear rate gradient D in the range from 3 to 1312.2 s^{-1} at forward and reverse (Bass O et al., 2017).

The active acidity was measured potentiometrically (Tomovska et al., 2016).

Optimization of the process of vegetable protopectin enzymatic hydrolysis was performed using the mathematical package MathCad 15 (Breus et al., 2019; Dayong et al., 2020). The regression equations were obtained in order to identify the optimal modes of fermentation of vegetable purées of different species to ensure protopectin hydrolysis at least 90% of its total amount.

Organization of the study

To obtain homogeneous purée, vegetables were washed, cleaned, cut into pieces measuring 20x20 mm, blanched until soft for no longer than 5 minutes, ground at a temperature of 50-55 °C to a homogeneous mass using a homogenizer with cutting knives at a speed of 1000 minutes -1 for 3 minutes to a particle size not larger than 1-2 mm.

Ice cream mixtures were pasteurized (85±2 ºC, 3 min), homogenized (12±1 MPa), cooled, vegetable puree was added, mixed and incubated (4±2 ºC, 2 h). The mixtures were heated to 20 °C before measuring the effective viscosity. The content of vegetable purée was 35%, sugar – 15%, fat – 3.5%, dry skimmed milk residue – 10%.

At the first stage of the study to compare the effectiveness of different methods of destruction of protopectin in vegetable purées studied the yield of soluble pectin in the case of acid and enzymatic hydrolysis.

Acid hydrolysis was performed at the average values of the recommended modes – temperature 90 ºC, duration 45 min, active acidity pH = 2.0 (Canteri et al., 2012). Citric acid was used to regulate the acidity of vegetable purées.

Fermentolysis of vegetable purées was carried out at the average values of the technological modes recommended by the manufacturers: mass fraction of enzyme – 0.1%, temperature 40 ºC, processing time – 2 h, active acidity – 4.0 units. pH. After fermentation, the pectinase was inactivated by heating the samples to 90 ºC without exposure. The redistribution of pectin substances was studied in samples cooled to a temperature of 20 ºC. The content of pectin substances (PS), protopectin (PP) and soluble pectin (SP) was determined in fresh and processed vegetable purée.

At the second stage of the study to optimize the process of protopectin enzymolysis of different vegetables to the prepared samples of purée with a given acidity pH = 4.0 was added enzyme in an amount of from 0.05 to 0.25% in increments of 0.05% – from 60 to 240 minutes. Soluble pectin content and effective viscosity of vegetable purée samples were determined.

At the third stage, the effect of soluble pectin content in vegetable purées on their structuring ability, including in the composition of mixtures for ice cream production, was studied.

Results and discussion

Comparative analysis of the different methods' effectiveness of protopectin destruction in vegetable purées

The most promising and lesser known vegetables for use in ice cream were selected for the study: table beets, zucchini, broccoli and tomatoes. Carrots were studied as a traditional vegetable for ice cream production with a maximum content of pectin. The average chemical composition of vegetables in descending order of pectin content is given in Table 1 (Golub et al., 2013; Velichko et al., 2018; Machulkina et al., 2014; Sagar et al., 2018).

According to Table 1, it should be noted that the mass fraction of pectin in selected vegetables varies from 0.03–0.23% to 1.57–2.93%, depending on the variety. Also, these vegetables are very strong pulp, and can perform various functions in the composition of ice cream, including coloring this product due to the content of β-carotene, anthocyanins, chlorophyll.

Table 1

Chemical composition of vegetables

Kind of vegetables	Contents of						
	water, %	dry matter, %	ash, %	organic acids, %	pectin substances, %	proteins, %	vitamin C, mg%
Table carrot	86–8.8	13–14	0.6	0.2	1.57–2.93	1.0–2.2	5
Table beet	86.0–87.0	18.6	1.35	0.08	1.15–1.5	1.5	17.92
Zucchini	93.0–96.0	4.6–4.8	0.49	0.05–0.1	1.0–1.4	0.4–0.6	17
Broccoli	88.68–91.46	10.5–13.1	0.76–0.8	0.8	0.76–0.87	2.74	89.2
Tomatoes	88.0–92.0	4.0–6.0	5.6–5.8	0.3–0.7	0.03–0.23	0.6–1.6	20–40

Available and relatively cheap varieties of vegetables grown in Ukraine were selected for the study: table beets "Delicatessen", broccoli "Jaguar", table carrots "Queen of Autumn", tomatoes "Asterix F1", zucchini "Cavili".

The nature of the change in the content of PS, PP and SP in vegetable purees under stable conditions of the hydrolysis process is given in Table 2.

According to Table 2, for all vegetables, the increase in the content of PS was accompanied by a decrease in the content of PP. The maximum yield of SP, compared with its content in fresh purees, was observed in the case of enzymolysis – 5.0–5.3 times for beet and carrot, 5.6–5.9 times for zucchini and broccoli and 6.7 times for tomatoes. This figure was slightly lower in the case of acid hydrolysis with an increase in SP yield – 4.6–4.8 times for beets and carrots, 5-5.4 times for zucchini and broccoli and 6 times for tomatoes. Thus, the largest changes in the SP content were found for vegetables with low pulp strength (zucchini, broccoli, tomatoes), and slightly smaller – for vegetables with a stronger structure (carrots and beets). This is probably due to the fact that stronger fibers of carrots and beets, even in a mechanically destroyed state, partially shielded the access of hydrolytic agents to protopectin. It should also be noted that the total amount of pectin after hydrolysis is slightly reduced, which indicates the formation of intermediate products of their destruction.

The role of pre-blanching of vegetables was quite noticeable, during which the content of soluble pectin increased on average three times, which correlates with the data on blanching of pectin-containing fruits (Levi et al., 2006). This confirms the need for pre-thermal softening of vegetables not only to facilitate their grinding into homogeneous purees, but also to ensure the gradual and more efficient hydrolysis of protopectin.The advantage of enzymatic hydrolysis over acid hydrolysis of pectin substances is obvious not only at higher SP yield, but also due to lower heat consumption (40 ºC vs 90 ºC). Enzymolysis occurs at a relatively moderate acidity of vegetable purees (pH = 4.0), which is more acceptable for the production of ice cream than acid hydrolysis at pH = 2.0–2.5. Thus, enzymatic hydrolysis allows to increase the yield of soluble pectin by an average of –12% at lower energy consumption, maximum preservation of biologically active compounds of vegetable raw materials, better organoleptic and physico-chemical characteristics of vegetable purees for further use in ice cream.

The advantages of using enzymatic hydrolysis over acid hydrolysis were also noted by Liew S.Q. etc. according to the results of comparative analysis of both methods on the example of passion fruit peel processing (Liew et al., 2016).

Table 2

Distribution of pectin substances in vegetable purees (P≥0,95, n=3)

Model samples	Mass fraction of pectin substances, g/100 g		
	PS	PP	SP
Table carrot			
Fresh puree	0,41±0,01	2,15±0,09	2,56±0,11
Puree after blanching	1,25±0,04	1,30±0,04	2,55±0,10
Puree after blanching and acid hydrolysis	1,98±0,05	0,55±0,02	2,53±0,09
Puree after blanching and enzymolysis	2,17±0,07	0,36±0,01	2,53±0,09
Table beet			
Fresh puree	0,21±0,01	1,02±0,05	1,23±0,04
Puree after blanching	0,61±0,02	0,62±0,02	1,23±0,03
Puree after blanching and acid hydrolysis	0,96±0,03	0,26±0,01	1,22±0,02
Puree after blanching and enzymolysis	1,06±0,06	0,16±0,01	1,22±0,01
Zucchini			
Fresh puree	0,20±0,01	0,98±0,02	1,18±0,03
Puree after blanching	0,62±0,03	0,55±0,02	1,17±0,04
Puree after blanching and acid hydrolysis	1,00±0,03	0,17±0,01	1,17±0,02
Puree after blanching and enzymolysis	1,12±0,04	0,05±0,002	1,17±-,02
Broccoli			
Fresh puree	0,14±0,01	0,73±0,03	0,87±0,02
Puree after blanching	0, 45±0,02	0,41±0,02	0,86±0,02
Puree after blanching and acid hydrolysis	0,76±0,02	0,09±0,01	0,85±-0,03
Puree after blanching and enzymolysis	0,82±0,03	0,03±0,01	0,85±0,02
Tomatoes			
Fresh puree	0,03±0,001	0,19±0,01	0,22±0,01
Puree after blanching	0,10±0,005	0,11±0,01	0,21±0,01
Puree after blanching and acid hydrolysis	0,18±0,01	0,03±0,002	0,21±0,01
Puree after blanching and enzymolysis	0,20±0,01	0,01±0,001	0,21±0,01

Determination of optimal parameters of protopectin enzymatic hydrolysis process in vegetable purées

To determine the efficiency of enzymatic hydrolysis of vegetable protopectin by variable parameters of this process, regression equations were obtained in the form of multidimensional polynomials of the second degree. The equations describe the dependence of soluble pectin content (%) on the amount of enzyme preparation (0.05–0.25%) and the duration of biotechnological treatment (60–240 min) of vegetable purées obtained from the blanched pulp.

In coded form, the regression equations have the following form:

$$Z1(x,y) := 0.60534 + 8.77724x - 24.90331x^2 + 0.01508y - 0.00004y^2 - 0.01172xy$$

$$Z2(x,y) := 0.44088 + 1.89409x - 4.06224x^2 + 0.00648y - 0.00002y^2 - 0.00354xy$$

$$Z3(x,y) := 0.44165 + 2.82174x - 7.32555x^2 + 0.00779y - 0.00002y^2 - 0.0094xy$$

$$Z4(x,y) := 0.49817 + 2.06334x - 5.35853x^2 + 0.00681y - 0.00002y^2 - 0.00783xy$$

$$Z5(x,y) := 0.10596 + 0.1903x - 0.82497x^2 + 0.0014y - 0.0000049y^2 - 0.00127xy$$

where: Z1, Z2, Z3, Z4, and Z5 – a mass fraction of soluble pectin in mashed carrots, beets, zucchini, broccoli, and tomatoes, %;X is the mass fraction of the enzyme, %; Y is the duration of enzymolysis, min

The accuracy of the approximation is $\delta Z = \pm 0.01\%$.

To ensure the degree of protopectin hydrolysis not less than 90%, the optimal parameters for vegetable purees with a pectin content of 0.22 to 2.56% are as follows:

- for carrots and beets with an enzyme content of 0.1–0.15%, the duration of enzymolysis – 180–240 minutes, with a content of 0.2% – 120–180 minutes;
- for zucchini, broccoli and tomatoes for doses of the enzyme – up to 0.05–0.10%, the duration of enzymolysis is 60–120 minutes.

The results of the study show a significant difference between the optimal parameters of the fermentation process for two groups of vegetables, which differ in the content of protopectin and the strength of plant cell walls (Gonzalez and Barrett, 2010; Sagar et al., 2018). Therefore, it can be argued that the physicochemical characteristics of the selected vegetable raw materials significantly affect the technologically appropriate parameters of the pectin substances fermentation process.

For all vegetables it should be noted that in excess of the optimal dose of the enzyme and the duration of enzymolysis, there is a slight decrease in the content of SP. This is probably due to the partial depolymerization of pectin compounds, which are not identified by the calcium pectate method (Podkorytova and Kadnikova, 2009). Partial destruction of pectin compounds can lead to deterioration of their structural ability, which must be experimentally verified.

Structuring ability study of the vegetables purées of different degrees of processing

At the next stage of the study on the example of carrot purée, which contains the most pectin, determined the effective viscosity:

- Fresh purée (control);
- Purée fermented at a content of 0.1% enzyme for 180 minutes (sample 1);
- Purée fermented at a content of 0.25% of the enzyme for 240 min (sample 2).

The effective viscosity of the practically intact structure (η_0) at the shear rate $\gamma=3s^{-1}$ in the direct course of measurement, the effective viscosity of the extremely destroyed structure (η_m) at the shear rate $\gamma=1312{,}2s^{-1}$ and the effective viscosity of the reduced structure (η_n) at the speed $\gamma=3\ s^{-1}$ shear at the reverse of the measurement is shown in Figure 1.

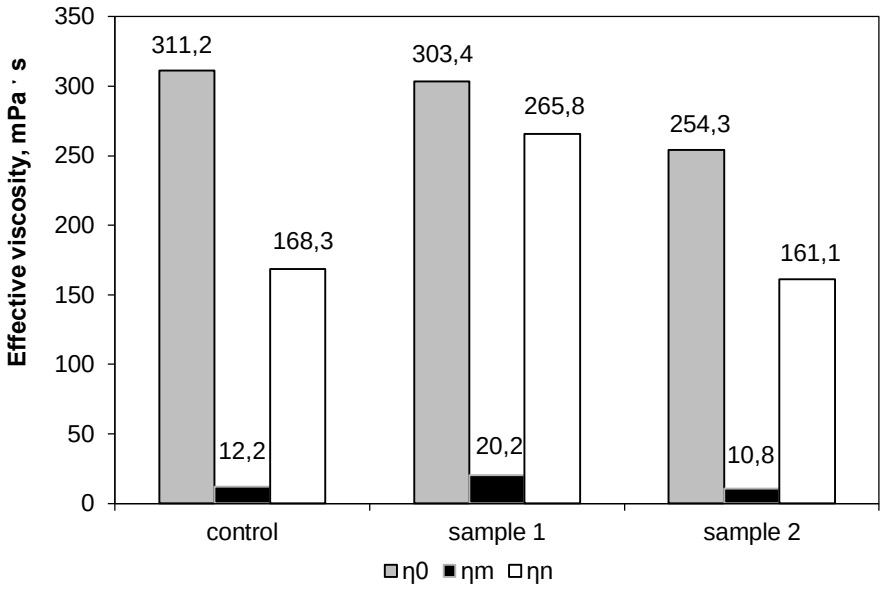

Figure 1. Effective viscosity of fresh and fermented vegetable purée samples at different stages of measurement using a rotary viscometer

Figure 1 confirms the previous assumption regarding the possible reduction of the structural ability of vegetable puree in the case of excessive hydrolysis of pectin substances. It is also interesting that the protopectin hydrolysis under optimal modes of this process even slightly reduces the initial effective viscosity of carrot puree (311.2 mPa·s vs 303.4 mPa·s), but significantly increases its thixotropic capacity. The difference between the values of the effective viscosity of the samples fermented under rational regimes (sample 1) and their excess (sample 2) was 104,7 mPa·s with a decrease in the shear rate to γ=3 s^{-1} in the reverse course of the measurement.

According to the authors, the ticotropic ability of pectin-containing raw materials in ice cream mixes that is extremely important. Too much thickening of mixtures in ice cream technology is undesirable, as their foaming ability may be reduced (Milliatti and Lannes, 2018).

Initial viscosity is not the main rheological characteristic of mixtures, as their ability to rapidly destroy the structure during freezing of mixtures and to quickly recover over time under static portions of soft ice cream portions after molding before hardening is more significant.

Therefore, in the last stage of the experiment, the effective viscosity of vegetable ice cream mixtures with the content of unfermented and fermented under optimal modes of carrot puree in the amount of 35% was investigated. The effective viscosity of mixtures with vegetable puree, in comparison with the control mixture without vegetables, is shown in Figure 2.

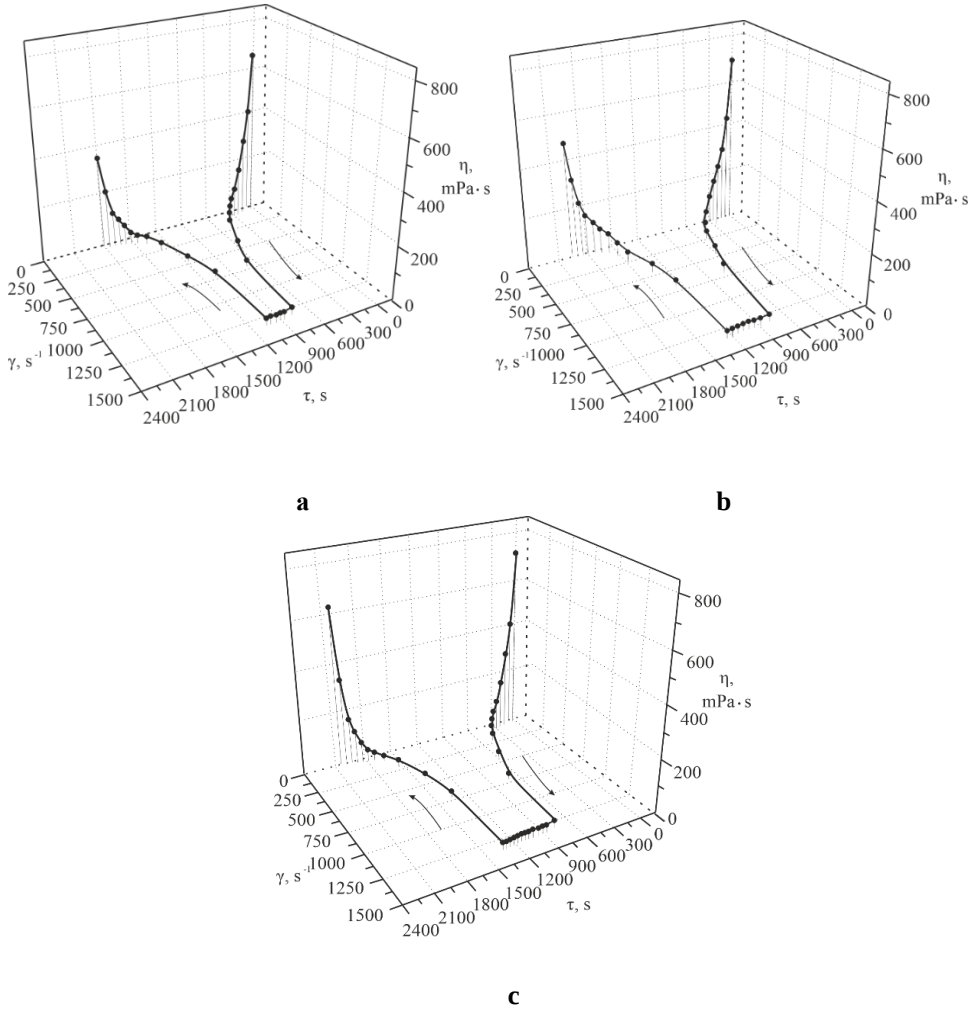

Figure 2. Effective viscosity of milk mixtures with a variable shear rate gradient: a – control; b – milk-vegetable mixture with unfermented vegetable purée; c – milk-vegetable mixture with fermented vegetable purée

The results of the last stage of the study confirmed the increased thixotropic capacity of pectin-containing fermented raw materials compared to unfermented in the composition of milk mixtures .

Also, important is the ability of the mixture with fermented purée to a significant and relatively rapid destruction of the structure over time, with its subsequent recovery by reducing the destructive force. Aeration of low-viscosity ice cream mixes is greatly facilitated during freezing.

It is known that excessive thickening of ice cream mixes is undesirable, as it may reduce their foaming ability (Milliatti and Lannes, 2018). The positive effect of the presence of

pectic substances in milk formulas is also obvious, which allows to obtain structured systems within the recommended limits of effective viscosity (Liew et al., 2016; Akalın et al., 2008).

It should be noted the increase in time during which the shear rate $\gamma = 1312\ s^{-1}$ obtained the lowest viscosity of the extremely destroyed structure for a mixture with fermented vegetable purée, compared with the sample with unfermented purée and the control sample. The observed effect indicates the presence of more low-energy coagulation-type bonds and surface contacts between the particles of enzymatically treated plant material.

A fundamentally new result of the study is the significant influence of fermented vegetable purée on the rheological characteristics of milk-vegetable mixtures. The high content of soluble pectin and the formation of complexes "protein-polysaccharide" mixtures with fermented purée show high structuring ability and thixotropic properties. Spontaneous restoration of the structure of milk-vegetable mixtures by reducing the shear rate will increase the stabilization of the structure of the formed portions of ice cream in static conditions.

The prospect of further research is to study the degree of preservation in vegetable hydrolyzed purees of biologically valuable compounds (vitamins, pigments, phenolic compounds, etc.) and the development of scientifically sound recipes for vegetable and milk ice cream.

Conclusions

1. Enzymatic hydrolysis of protopectin in vegetable purees with a pectin content of 0.22 to 2.56% is more efficient than acid hydrolysis and allows to increase the yield of soluble pectin by 8–12% at lower energy consumption.
2. To ensure the hydrolysis of at least 90% of protopectin, it is necessary to use from 0.05 to 0.2% of the enzyme preparation for 60–120 minutes to 240 minutes, depending on the type of vegetable and the strength of its tissues.
3. The use of fermented vegetable purées in milk formulas allows obtaining structured systems with the recommended values of effective viscosity and pronounced thixotropic ability.
4. Excessive enzymolysis reduces the structuring ability of vegetable purees, which negatively affects the rheological characteristics of milk-vegetable mixtures for ice cream production.

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Effect of grape skin powder extract addition on functional and physicochemical properties of marshmallow

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Abstract

Introduction. The present research evaluated the effect of grape skin powder extract addition on functional and physicochemical properties of marshmallow.

Materials and methods. To asses the effect of grape skin on marshmallow quality, alcoholic grape skin extracts (GSE) were prepared and introduced in different amounts in marshmallow recipes. The functional properties of zephyrs were estimated by determining the total polyphenol content and antioxidant activity. The microbiological stability of the product was assessed by using agar meat broth. Molds proliferation and morphology of cells from single colonies was studied under microscope.

Results and discussion. The effects of grape skin extract (GSE) on marshmallow quality were evaluated. The marshmallow physicochemical properties in terms moisture and sugar content were affected by GSE incorporation. A directly proportional relationship was observed between the addition of GSE and the moisture content of the marshmallow samples, registering an increase from 15.02 to 15.58% for the samples with 1% and 3% GSE respectively. The sugar content varied in the limits of 14.05–14.21%, being higher for the samples with an increased amount h GSE. Total phenolic content of GSE and marshmallow samples with added GSE was determined as 27.39, 5.11 (1% GSE marshmallow), 6.46 (2% GSE marshmallow) and 7.89 (3% GSE marshmallow) mg/g gallic acid (GAE), respectively. Hydrogen peroxide inhibition capacity and DPPH radical scavenging of marshmallows had increased in proportion to rising GSE level. Antioxidant activity of marshmallows containing 3% GSE was found to be higher (35.72%) than others. The addition of GSE significantly affected the marshmallows color parameters, as the amount of grape skin increased, a more intense purple coloration was observed. The marshmallow containing 2% GSE was most appreciated in terms of sensorial properties. The GSE addition had inhibitory effects on mold population during storage, a higher degree of mold growth reduction ($p < 0.05$) being observed in the sample with 3% GSE after 7 days of storage.

Conclusions. The addition of grape skin extract in marshmallow formulation increased the biological value in terms of antioxidant activity and total phenol content, and the consumers' acceptancy.

Introduction

It is known that the main wine making by-product is represented by grape pomace, that constitutes about 15/20% of the total grape weight (around 7 million tons) (*Oiv-Statistical-Report-on-World-Vitiviniculture-2018.Pdf*, n.d.). The nutritional quality of grape pomace appears rich in sugars, vitamins, minerals, polyphenols and other macromolecules which are of high interest for the food industry (Bordiga et al., 2019). Due to the rich nutritional compositions and beneficial effects of grape skin on human health demonstrated by numerous researches (Abarghuei et al., 2010; Iora et al., 2015), the interest for the development of new foods based on its use as cookies, various fillings for pastries, drinks, etc. has increased (Acun et al., 2014). Numerous researches have been carried out related to the capitalization of these industrial wastes, including as animal feed, additions in flour mixes for pastry products, ingredients for candy production, sources of biologically active substances with antioxidant potential, etc. (Cappa et al., 2015; Otero-Pareja et al., 2015). The positive effect of grape skin flour on total fibre and ash contents in yogurts and other fermented analogs has been demonstrated (dos Santos et al., 2017). Use of grape skin powder in functional cookies increased their that protein and fiber content and showed higher antioxidant potency, higher total phenol and higher retention of hardness during storage (Theagarajan et al., 2019).

The research carried out particularly emphasizes the content of polyphenols in grape skins and their antioxidant capacity. Depending on the grape variety, the total polyphenol content varies within > 20 mg GAE/g of grape skin dry matter for white grapes and >70 mg GAE/g of grape skin dry matter for red grapes (Cvjetko Bubalo et al., 2016; Katalinić et al., 2010), flavonols, catechins and anthocyanins being the main phenolic compounds detected (Antoniolli et al., 2015; Caldas et al., 2018). *Sun and others* have investigated the antioxidant effects of these compounds, and their antitumor and antimetastatic role in preventing breast cancer (Sun et al., 2012), and *Jariyapamornkoon and colab.* showed the positive effect of red grape skin extract on protein glycation (Jariyapamornkoon et al., 2013).

Often to give color and to extend the products shelf life, especially sweet products, manufacturers use dyes and antioxidants of synthetic origin such as carmuazine and tartrazine, etc. which are more intense and stable during products storage (Caleja et al., 2017). However taking into account current trends in the food industry, the need of this researches results from the FAO/WHO strategy of substituting synthetic substances (additives, texture agents, etc.) with natural bioactive components, which is a way to increase food safety and quality, a benchmark in optimized nutrition (McGuire, 2013; Taghvaei et al., 2015) and of diminishing the food waste level (Gustavsson et al., 2011).

Withal, the replacement of synthetic substances with natural ones resulting from the vines processing is a strategic problem, because, unlike synthetic ones, natural substances have fragile molecules, sensitive to food matrix, storage conditions, etc. However, the antiradical and microbiostatic activity of grape skin extracts, rich in polyphenols, is a promising source of alternative solutions for their use in order to replace certain synthetic food preservatives (Katalinić et al., 2010; Shin et al., 2010).

However, as far as is known, the grape variety, food matrix, ingredients and investigated quality parameters in each study are different from those presented in the current study. In the present study the marshmallow prototype was chosen as the food matrix. Marshmallow is a sweet product preferred by all categories of the population, but especially children have always preferred them (Ungure et al., 2013). Due to the fact that many researchers have demonstrated the positive effects of incorporating grape skin into various food products, but less in marshmallows, hence the *purpose* of the present research is to evaluate the effect of grape skin powder extract addition on functional and physicochemical properties of marshmallow.

Materials and method

Materials and chemicals

A standard recipe of marshmallow was used. The ingredients for the marshmallow production were as (Yurchenko et al., 2020): egg whites, agar-agar, sugar, citric acid and grape skin powder.

Grape skin extracts

In order to obtain grape skin extract (GSE), pomace resulting from the production of Merlot wine was used. The grape pomace was initially dried at 50±2 °C until its moisture content reached 5% value. The pomace was then blown to separate the skin from the seeds. The skin of the grapes was minced and hydroalcoholic extracts (50% EtOH, 1:10) were prepared.

Marshmallow production

The marshmallow was prepared using agar-agar, egg whites, sugar, apple puree and water (Yurchenko et al., 2020), with the addition of grape skin hydroalcoholic extract (1.0% and 2.0%, 3.0% of total weight).

The technological process of preparing marshmallows begins with the preparation of apple puree, which involves peeling apples, cutting them into pieces and boiling them with a little water, over a moderate heat until the consistency of the apple pieces is soft. The apple pieces are minced with the a blender and an amount of 30% of sugar is then added. The mixture is put on a moderate heat and mixed until the sugar crystals are completely dissolved.

The apple puree, GSE and the egg whites are placed in the bowl of the mixer and the mixture is foamed starting from the low speed to the higher speed of the mixer. The mixture is foamed until the hard peaks are obtained.

Separately the sugar syrup with agar is prepared: the agar is mixed with water and sugar and put on the fire, stirring constantly until boiling. From the moment of boiling, the syrup is kept on the fire for another 5 minutes until the thin thread syrup is obtained. The syrup then is added to the mixture of egg white and apple puree while the mixer continues to foam the composition. The mixture is beaten until a shiny and firm composition is obtained. Using a pastry bag, the marshmallows are shaped and then allowed to dry for 3-6 hours.

Grape skin powder and marshmallow properties

Moisture content

The moisture content of grape skin powder and marshmallow samples was determined by oven drying, according to the AOAC, 2005; method 930.15 (Horwitz, 2005).

Sugar content

The reducing sugars concentration of marshmallows was analyzed by Benedict's, Bertrand's and Fehling standard procedures (Kumar et al., 2014).

Titratable acidity (TA)

The TA was determined by titration to pH 8.1 with 0.1 M NaOH. Phenolphthalein (0.1%) was used as an indicator (Mutlu et al., 2018).

Total Polyphenol content (TPC)

The total polyphenol content in grape skin powder and marshmallow samples was determined by Folin Ciocalteu method described by *Makkar et al.* (2003) (Makkar, 2003).

Antioxidant activity (AA)

1,1-Diphenyl-2-picryl-hydrazyl (DPPH) solutions were prepared in methanol. The sample solutions (Grape skin and marshmallow hydroalcoholic extracts 100 µL) at varying concentrations (0.1–10 mg/mL) was added to a 0.1 mM methanolic solution of DPPH (3.9 ml) and shaken vigorously. The reaction tubes, in triplicates, were kept in dark, at 30 C for 30 min. Spectrophotometric measurements were done at 517 nm using Hach Lange DR 5000 spectrophotometer. The data are mean±SD (Sharma et al., 2009).

Hydrogen peroxide scavenging activity assay

Hydrogen peroxide inhibition capacity of the GSE and marshmallow samples was determined by replacement titration. Aliquot of 1 ml of sample and 1 ml of hydrogen peroxide solution (0.1 mM) were mixed. Then 2 drops of ammonium molybdate (3%) solution were added, followed by.10 ml of sulfuric acid (2M) and 7 ml of potassium iodide (1.8M). The obtained solution was left to interact for 20 minutes, then titrated with sodium thiosulphate (5.09 mM) until the yellow color disappeared. In parallel, the control sample (without extract) was analysed. The thiosulphate volume expended for titration were record (V1-for sample with GSE, and V0 – for control sample). Percentage of hydrogen peroxide inhibition was calculated as (Nagulendran et al., 2007):

$$\%\ H_2O_2 Inhibition\ =\ \frac{(V_0 - V_1)}{V_0 \times 100} \quad (1)$$

Color parameters assessment

In the food science and technology color is traditionally represented using the CIE 1976 L*a*b* or CIELAB color space (Goñi et al., 2017). The influence of the addition of GSE on the chromatic parameters of the marshmallow was evaluated with using the tristimulus CieLab colorimeter. For each sample, individual color parameters L *, a * and b * were quantified.

Color changes was measured as the modulus of the distance vector between the initial color values and the actual color coordinates. This concept is called total color difference (ΔE). The total color difference indicates the magnitude of the color difference between the control samples and those investigated (Ly et al., 2020).

Microbiological analysis

For each sample, marshmallows were analyzed after 1, 3 and 7 days of storage at 6±2 °C. Appropriate dilutions were made and pour-plated onto selective media. In order to determine the microbiological stability of the product, determinations were performed on agar meat broth for molds proliferation. Each sample was inoculated in triplicate. The selectivity of the growth conditions was confirmed by morphology of cells from single colonies under microscope (Jung et al., 2016).

Sensory evaluation

In order to perform the organoleptic test, marshmallows were prepared one day in advance. Twelve panelists (aged 24 to 69 years old), participated in this study and appreciated the quality of marshmallows based on the 9-point hedonic scale from "dislike extremely" to "like extremely". For the present research, first was discussed the main quality characteristics of marshmallow. The marshmallow samples were investigated for appearance, texture, taste, flavor, color and overall acceptance (Cano- Lamadrid et al., 2018).

Statistical analysis

All experiments were carried out in triplicate. The results are given as mean standard deviation (SD). Student's t-test was used for comparison between two means. A difference was considered statistically significant when $p \leq 0.05$.

Results and disscution

Physico-chemical parameters

Marshmallow samples showed different values of the studied parameters depending on the added GSE concentration.

The following physico-chemical indices were investigated in the experimental samples: moisture content, reducing sugars content and total titratable acidity. The obtained results are illustrated in table 1.

The obtained results (Table 1) indicate that with an increase in the mass fraction of grape skin extract, the moisture content of marshmallows increases as well from 15.05% for the control sample to 15.68% for the sample with 3% GSE addition. This increase in moisture can be explained by the increase, in the same time, of reducing sugars (glucose, fructose) and dietary fibers amount that lead to the increase of marshmallow water binding capacity (Ergun et al., 2010).

Table 1

Physico-chemical characteristic of the marshmallow

Sample	Moisture,%	Reducing sugar,%	Titratable acidity, mEq/l
Control sample	15.02±0.19	14.05±0.24	7.63±0.11
Marshmallow – 1% GSE	15.21±0.21	14.11±0.16	7.80±0.09
Marshmallow – 2% GSE	15.45±0.15	14.15±0.18	7.82±0.13
Marshmallow – 3% GSE	15.68±0.23	14.21±0.22	8.01±0.08

The fact that the titratable acidity of marshmallow samples increases from 7.63 to 8.01 with increasing GSE concentration is confirmed by the presence of three major acids in grape skin (Le Moigne et al., 2008). The presence of organic acids from natural plant extracts has been associated with the possibility of extending the shelf life of sugar confections like fruit paste, jelly candy and others (Anand et al., 2013).

Total Polyphenol content and Antioxidant activity

In the context of today's nutrition tendency, when people opt for healthier products with a high biological value (Cucinotta, 2018) it is proposed to determine the total polyphenol content and antioxidant effect of obtained marshmallow with added GSE using the value of the ability to inhibit hydrogen peroxide and the DPPH assay.

The values obtained are indicated in figure 1.

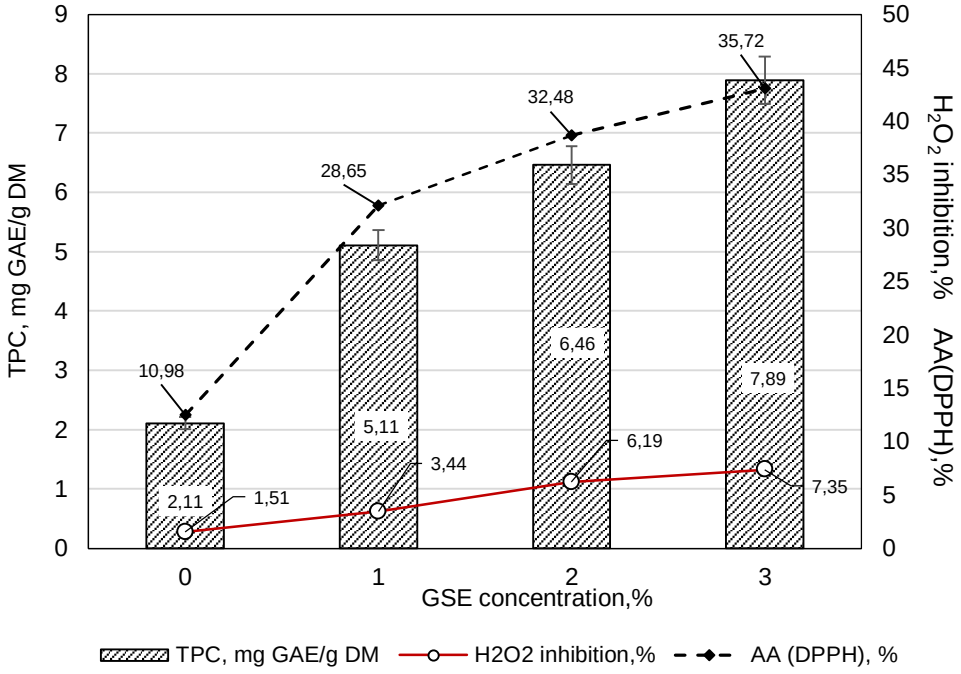

Figure 1. Influence of the total polyphenol content on the antioxidant activity of marshmallow samples

The total content of polyphenols in grape skin powder is 27.39±0.41 GAE/g DM, data which are similar to those in the literature, which ranges between 22.2±9.95 mg GAE/g and 45.0±26.3 mg GAE/g depending on grape variety, number of extractions and used solvent (Katalinić et al., 2010; Negro et al., 2003). The incorporation of grape skin in the marshmallow formulation has the effect of increasing the polyphenol content in the investigated samples from 2.11±0.29 mg GAE / g DM for the control sample to 7.89±0.23 mg GAE / g DM for the marshmallow sample with 3% GSE. The same effect of the addition of grape skin on food matrices has been demonstrated in the case of its use in formulations of candies (Cappa et al., 2015; Mutlu et al., 2018), sausages (Ryu et al., 2014), cookies (Acun et al., 2014), etc. In the case of the mentioned products, the positive effect on the total polyphenol content was higher due to the incorporation of GS in the form of powder and not of extract.

In the present study, tests were also performed with the direct use of grape skin powder, but these samples did not have a uniform color distribution, their surface being with purple

dots, and the consistency was also negatively affected, respectively they have were rejected from a sensory point of view.

The antiradical activity of plants is largely due to polyphenols (Pulido et al., 2000). From the data presented in figure 1 it can be seen that the amount of polyphenols increases with increasing GSE concentration in the marshmallow samples. At the same time, the amount of polyphenols is directly proportional to the antioxidant activity of the products.

It is known that grape skin has an enormous antioxidant potential (Dordoni et al., 2019). As presented in figure 1, marshmallow samples with added GSE showed hydrogen peroxide inhibition activity. Many common and life threatening human diseases have free radical reactions as an underlying mechanism of injury. Hydrogen peroxide can cross cell membranes rapidly and form hydroxyl radical and this may be the origin of many toxic effects (Rani et al., 2015). The inhibition of H_2O_2 by marshmallow samples with added GSE may at least partly result from its antioxidant and free radical scavenging activity. The H_2O_2 inhibition level of marshmallow with 3% GSE rose to 7.35% comparing to 1.51% for control sample.

DPPH is another free radical which is reduced in the presence of an antioxidant molecule. Based on results presented in figure 1, we can say that as a rule the addition of ethanol grape extract to marshmallow showed higher antioxidant activity values than the control sample. Based on the DPPH assay, the radical scavenging activity of the marshmallow samples with added GSE were 28.65±0.21% for the sample with 1% GSE, 32.48±0.19% and 35.72±0.14% for the sample with 2% and 3% GSE respectively comparing to 10.98% for the control sample. It worths mentioning that the DPPH radical scavenging activity of the GSE was 89.71%. Rockenbach et al., 2011 mentioned values of 16,925 – 3640 μmol Trolox equivalents /100 g for the DPPH assay for different grape varieties (Rockenbach et al., 2011).

Color parameters

Results concerning the color analysis showed that as a result of the different rates grape skin addition, significant difference was noticed between L*, a* and b* values. Data on the color parameters values (L, a, b) of marshmallow are shown in Table 2.

Table 2

Color parameters values of marshmallow with added grape skin extract

Sample / Parameter	Control sample	Marshmallow – 1% GSE	Marshmallow – 2% GSE	Marshmallow – 3% GSE
L*	97.56±1.12	91.00±0.83	81.27±0.41	77.13±0.65
a*	1.21±0.04	4.32±0.11	10.44±0.13	11.52±0.21
b*	-1.34±0.03	-4.27±0.09	-9.31±0.23	-10.37±0.17
ΔE	-	7.83±0.17	20.35±0.09	24.60±0.11
C*	-	6.07±0.06	13.99±0.13	15.50±0.09
H (°)	-	-0.78±0.01	-0.73±0.01	-0.73±0.01

As the amount of grape skin increased, a more intense purple coloration of the marshmallow samples was observed. The control sample was white, with L* value 97, and tends to decrease by L*=77 for the 3% GSE sample, this can be explained that at pH higher than 5, brightness slightly decreases, indicating that other colored forms are being formed (Chi et al., 2020). On one hand the red – green parameter (a*) also shows an increasing

tendency, towards a redder shade. This is due to the anthocyanins present in the grape skin, whose color varies in shades of red – blue (Khoo et al., 2017). The same is demonstrated by the decrease of the b*value (blue-yellow), the addition of grape skin giving the products a slightly purple shade. The GSE addition had an influence and on ΔE of marshmallow samples, a directly proportional relationship was observed between the increase in GSE addition and the ΔE increase. The (C*) – chroma parameter shows the color saturation of the marshmallow samples, i.e. indicates the intensity of the purple color relative to white (Milla´n, 2002). The marshmallows with 3% GSE had a more saturated color. The hue angle (H), is expressed in the scale 00 -3600, considered the qualitative attribute of the color, is the attribute according to which the colors have been traditionally defined as reddish, greenish, etc., and is used to define the difference of a certain color with reference to gray with the same lightness. As for GSE marshmallows' hue angle, the addition of GSE did not significantly influence the parameter values which ranged from -0.78 to -0.73°.

Microbiological stability

The microbial cells present in the samples to be analyzed on solidified nutrient media formed visible colonies. The total number of mold colonies are shown in figure 2.

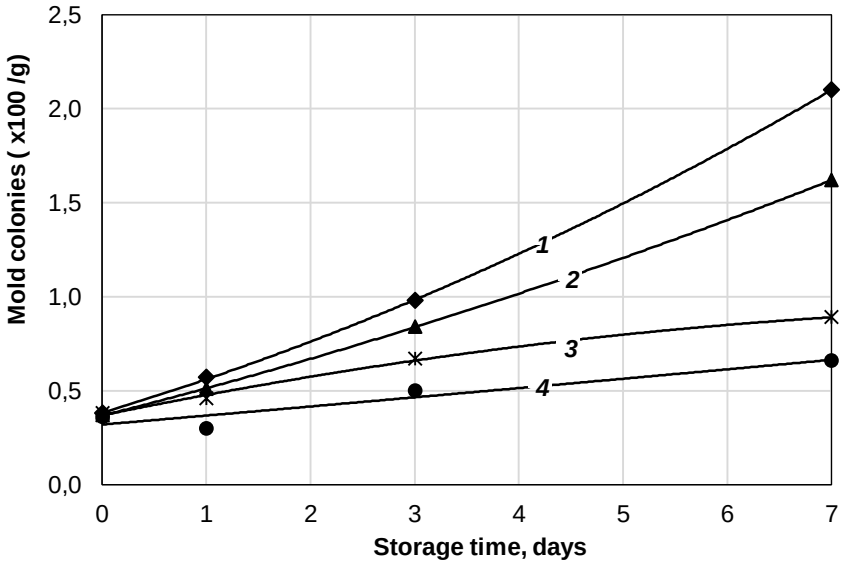

Figure 2. Effect of storage time and grape skin extract addition on microbiological stability of marshmallow.
% GSE:
1 – 0; 2 – 1; 3 – 2; 4 – 3.

Addition of grape skin extract to marshmallow had inhibitory effects on mould population during storage, a higher degree of mould growth ($p < 0.05$) being observed in the control samples ($2.1 \cdot 10^2$ colonies/g) as compared to the sample with 3% added grape skin extract ($0.66 \cdot 10^2$ /g) i.e. 0.5 log reduction in growth in the sample with 3% GPE after 7 days

of storage. The bacteriostatic effect is due to the polyphenols present in the grape skin. The bacteriostatic effect of phenolic compounds has also been demonstrated for Ocimum basilicum Leaves Extracts (Ababutain, 2019), cocoa powder (Pina-Pérez et al., 2017, p.), green tea (Zhang et al., 2020), red raspberry (Nikitina et al., 2007), etc. Concerning grape phenols, their showed an antibacterial effect even at at 1 and 2.5% concentrations (Furiga et al., 2009; Özkan et al., 2004). Several studies explained the phenols antibacterial activity by the modification of cell membranes permeability (Cushnie et al., 2011), the modification of some intracellular functions induced by hydrogen binding of the phenolic compounds to enzymes (Taguri et al., 2006) or by the modification of the cell wall rigidity with integrity losses due to different interactions between phenols and cell membrane (Negi, 2012).

Length of storage time, showed a concomitant increase in mould population. Seven days storage of control samples increased mould contamination to 0.23 log (Figure 2), indicating an essential effect of storage time on mould growth. The increase of GPE addition to marshmallow formulation showed also a visible effect on mould growth reduction. The main detected mold especies were *Aspirgillus versicolor* and *Penicillium islandicum*. The microbiostatic effects of grape skin are also confirmed by previous studies (Hassan et al., 2019).

Sensory evaluation

Sensory analysis was performed to evaluate the sensory profile of the marshmallow with added grape skin powder extract. The effect of grape skin addition on marshmallow formulation was studied, and 5 quality attributes were evaluated.

Mean scores for liking of color, appearance, flavor (grape notes), taste, texture attributes, and overall liking of samples are presented in Table 3.

Table 3

Mean scores for marshmallow color, appearance, flavor, taste, texture, and overall liking

Sample / Parameter	Marshmallow – 1% GSE	Marshmallow – 2% GSE	Marshmallow – 3% GSE
Color	8.2±0.23	9.2±0.24	7.3±0.32
Appearance	7.9±0.31	9.5±0.36	7.1±0.29
Taste	8.1±0.27	8.9±0.45	8.7±0.37
Flavor	7.7±0.26	8.9±0.46	8.1±0.28
Texture	8.4±0.28	8.4±0.27	8.3±0.41
Overall acceptability	8.06±0.27	8.98±0.41	7.9±0.69

Parameters that were most influenced by the addition of grape skin were color and flavor. Due to the anthocyanin content of the grape skin, the marshmallow samples acquired a shade of purple, which in the case of using 3% of GSE was too intense, similar to synthetic dyes, for this reason the panelists gave this parameter 7.3 points. In terms of flavor, color and appearance, the most optimal amount of GSE addition would be 2%, in this case the products had a pleasant color and slightly perceived notes of grape flavor. In general, all the tested marshmallow samples obtained scores above 7.0, on a nine-point hedonic scale, and can be considered as acceptable.

Conclusion

- The development of marshmallow with grape skin extract is a good strategy to promote the valorization of an industrial waste that has high biological value.
- The formulation of marshmallow with 3% grape skin extract addition led to the best results in terms of total phenol content, antioxidant capacity, color, texture and general acceptability.
- The fortification with GSE increased the total phenol content and antioxidant activity.
- The addition of grape skin extract also achieved other advantages: the reduction of mold growth during the storage and the delivery of beneficial compounds for human health, promoting in the same time the efficient use of a plant material used to be considered an industrial waste.

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Improving the efficiency of mass-exchange between liquid and steam in rectification columns of cyclic action

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Abstract

Introduction. The purpose of the work was to determine the optimal time of residence of the liquid on the plates, the grade of extraction and concentration ratio of volatile impurities of alcohol and the specific consumption of heating steam in rectification columns of cyclic action.

Materials and methods. The studies were carried out in a rectification column, equipped with flaky plates with a variable free cross-section. Concentration of alcohol volatile impurities was determined by chromatographic method, the grade of their extraction and concentration ratio – by calculation method, other indicators – by commonly known methods.

Results and discussion. The maximum extraction of volatile impurities was being achieved in a rectification column, equipped with flaky plates containing turnaround sections connected to drive mechanisms, the action of which is occurred according to a given algorithm. The optimal parameters of operating the column were: vapor velocity in the orifices of the flakes during the period of liquid retention on the plates 12–14 m/s; during liquid pouring 1–1.5 m/s; time of residence of the liquid on the plates 40 s, pouring time 1.7 s; pressure in the lower part of the column 12 kPa; the concentration of ethyl alcohol in the still residue 3–4% vol. In order to provide the cycles, the free sectional area of the plates must change instantaneously from 5.5 to 51.7%. This technical solution allows to provide complete disposal of ethers, methyl acetate and isopropyl alcohol, to increase the grade of extraction of higher alcohols of fusel alcohol and methanol by 38%, the concentration ratio of aldehydes by 25%, higher alcohols by 38%, methanol by 37%, and to reduce specific consumption of heating steam by 40% compared to a typical column operating in stationary mode.

Conclusion. The innovative technology of cyclic rectification allows to increase the grade of extraction and the concentration ratio of volatile impurities of alcohol by 25–38% and reduce energy consumption by 40% compared with the known ones.

Introduction

Technical progress in the alcohol industry is inextricably linked to the development and implementation of highly efficient column apparatuses (Shyian et al., 2009; Kyziun et al., 2006) and energy-saving ways of mass transfer between the liquid and steam on their plates (Martseniuk et al., 2019). One of the ways of solving the mass transfer process problem is the use of cyclic mode of phase motion, which is based on alternate change of two periods: the steam passing up the column period and the period of liquid pouring on its plates (Maleta, et al., 2011; Kiss et al., 2012). Implementation of controlled cycles of liquid retention on the plates allows to prolong the time of its contact with steam, to create conditions in order to achieve a phase state close to equilibrium and to bring the efficiency of each real plate closer to the theoretical one (Buliy et al., 2019). This significantly reduces the specific consumption of heating steam, decreases the volume of alcohol-containing waste and minimizes the cost of equipment (Kiss, 2015).

There are well-known ways of increasing the residence time of liquid on the plates by organizing the flow of separate steam–liquid jets with their mutual collision (Pătruț et al., 2014) or additional installation of baffles and reflectors, directing the steam through the appropriate bypass pipelines, etc. (Krivosheev et al., 2015). Despite the obtained positive results in reducing energy costs, the known methods and apparatuses of cyclic operation have not found wide practical application due to the lack of mass exchange in the steam period (Lita et al., 2012), the steam pressure dependence of pouring devices' operation (Toftegard et al., 2016), the fluctuations of the steam pressure in the collector, the inability to stabilize the hydrodynamic mode of plates (Flodman et al., 2012), the mixing of liquid on adjacent plates during its pouring, the low apparatuses' steam and liquid throughput capacity, and the complexity of constructive solutions (Bastian et al., 2018).

The authors proposed an innovative rectification technology, which excludes earlier mentioned disadvantages (Buliy et al., 2016) and provides periodic liquid pouring from one plate to another at continuous supply of liquid and heating steam into the column (Ukrainets et al., 2018). To implement the technology, the design of a rectification column equipped with plates with variable free cross-section was developed (Bulii et al., 2019). For stable operation of plates in the column hydrodynamic regimes were maintained, providing effective mass transfer between liquid and steam without entrainment of liquid on upper plates during the fluid retention period and its intensive pouring through pouring and barbotage holes after the end of the retention time.

The aim of the work was to study the efficiency of mass-exchange between liquid and steam in column apparatuses of cyclic action: to determine the grade of extraction and the concentration ratio of volatile impurities of alcohol during its extraction from alcohol-containing fractions and to identify the specific rate of heating steam in the studied rectification column.

Research objectives:

1. To determine the grade of extraction and the concentration ratio of alcohol impurity concentrations under conditions of typical and cyclic rectification (in columns equipped with moving valves and turning plate sections);
2. To determine the optimal technological parameters of the studied column and the residence time of the liquid on its plates, by which the maximum extraction of volatile impurities is provided without reducing the liquid throughput of the column;
3. To determine the specific rate of heating steam in a rectification column of cyclic action.

Materials and methods

Research objects

Rectification columns of cyclic action with moving valves (RC)

The RC is made of stainless steel AISI 304, equipped with flaky plates of arched type. Technical characteristics: diameter – 426 mm; number of plates – 30; distance between the plates – 300 mm; the cross-sectional area of flakes' holes – 19,42 mm^2; thickness of the plate fabric – 2 mm; free cross-section of the plate: 5,5% – during the residence of the liquid on the plates – 5,5%; during the liquid pouring – 51,7%.

A fragment of the RC with movable rods, valves and hydraulic shutters is shown in Figure 1a (patent UA 116565. Rectification column with controlled cycles). The operation of the column provided the conducting of the adjustable in time cycles of liquid residence on the plates and its synchronous pouring from one plate to another over the entire height of the column in two successive stages, repeating periodically in time, alternately, according to the specified algorithm without interrupting the liquid and steam supply (patent UA 89874. Method of liquid pouring on plates of column apparatus in the process of mass transfer between steam and liquid). The interval of liquid retention was being determined experimentally depending on the grade of extraction of volatile alcohol impurities and their concentration ratio.

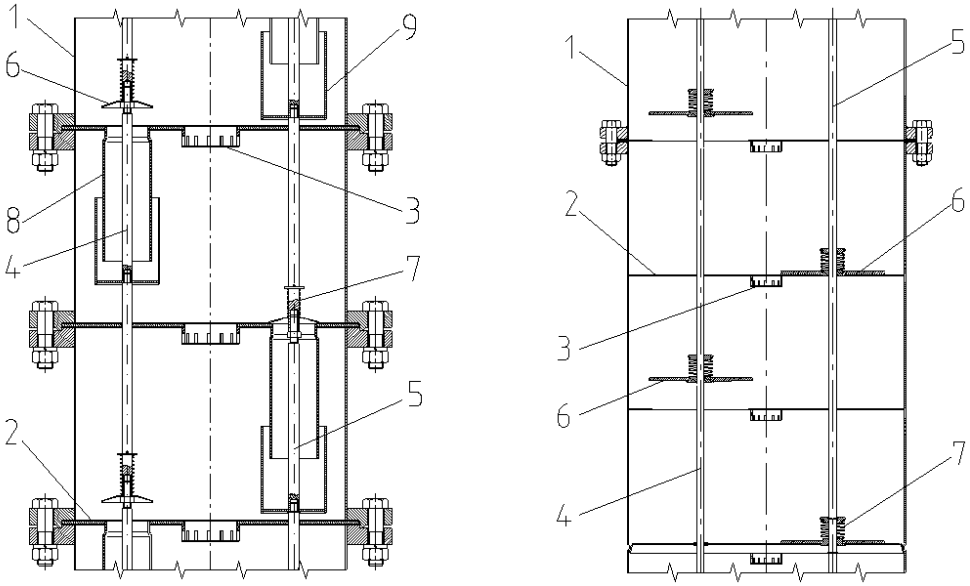
The experimental RC was included in the scheme of the bragorectificational plant (BRP). The column contained corps *1*, plates *2* with contact elements *3*, movable rods *4* and *5*, on which valves *6* and springs *7* were mounted. The rods moved up and down under the action of drive mechanisms (double-acting pneumatic cylinders of DNT type manufactured by FESTO). At that, valves *6* closed and opened the holes of pouring pipes *8* alternately. The operation of pneumatic cylinders was managed in accordance with the M340 controller program of 'Schneider Electric' company. Pipes *8* were inserted into sleeves *9* and together with them served as water traps, which prevented steam breakthrough through all the holes during liquid pouring.

Figure 1b shows an experimental RC with movable rods and valves without hydraulic shutters (patent UA 139228. Column mass-exchange apparatus of cyclic action). The technical solution allowed one-stage (full) and two-stage methods of liquid pouring on plates (Figure 2). The one-step method involved pouring all the liquid from one plate to another (Figure 2a). According to the two-stage method (patent UA 141245. Method of pouring the liquid on the plates of mass-exchange column apparatus) part of the liquid had been pouring from the upper plate to the lower one (30–70% of its volume), and after a specified delay time, its remnants were poured (Figure 2b).

Plant for ethyl alcohol extraction from alcohol-containing fractions

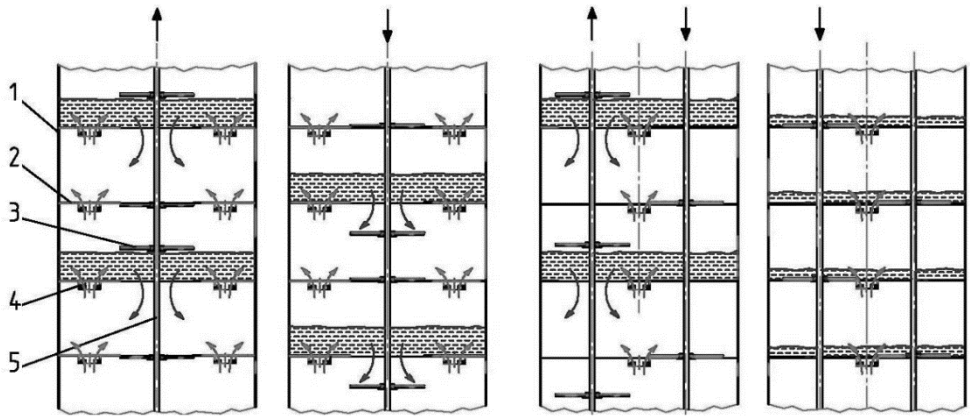
The scheme of the implementation of the studied RC into the BRP one is shown in Figure 3.

The plant included the experimental column 6, the upper and lower parts of which are connected to the vacuum breakers *4*, evaporator *5*, dephlegmator *7*, condenser *8*, alcohol-collecting vessel (trap) *9*, softened water collector *1*, intermediate collectors of still residue *15* and alcoholic fractions *18*, flow-meters *3*, *11*, *12* and *13*, centrifugal pumps *2*, *16*, *17* and decantator *10*.



a *b*

Figure 1. Fragments of the studied RC with movable valves: with hydraulic shutters (*a*) and without hydraulic shutters (*b*): 1 – body; 2 – plates; 3 – contact devices; 4, 5 – rods; 6 – valves; 7 – springs; 8 – pouring pipes; 9 – sleeve.



a *b*

Figure 2. One-stage and two-stage methods of liquid pouring on the plates of the cyclic action RC: 1 – body; 2 – plate; 3 – valve; 4 – contact element; 5 – moving rod.

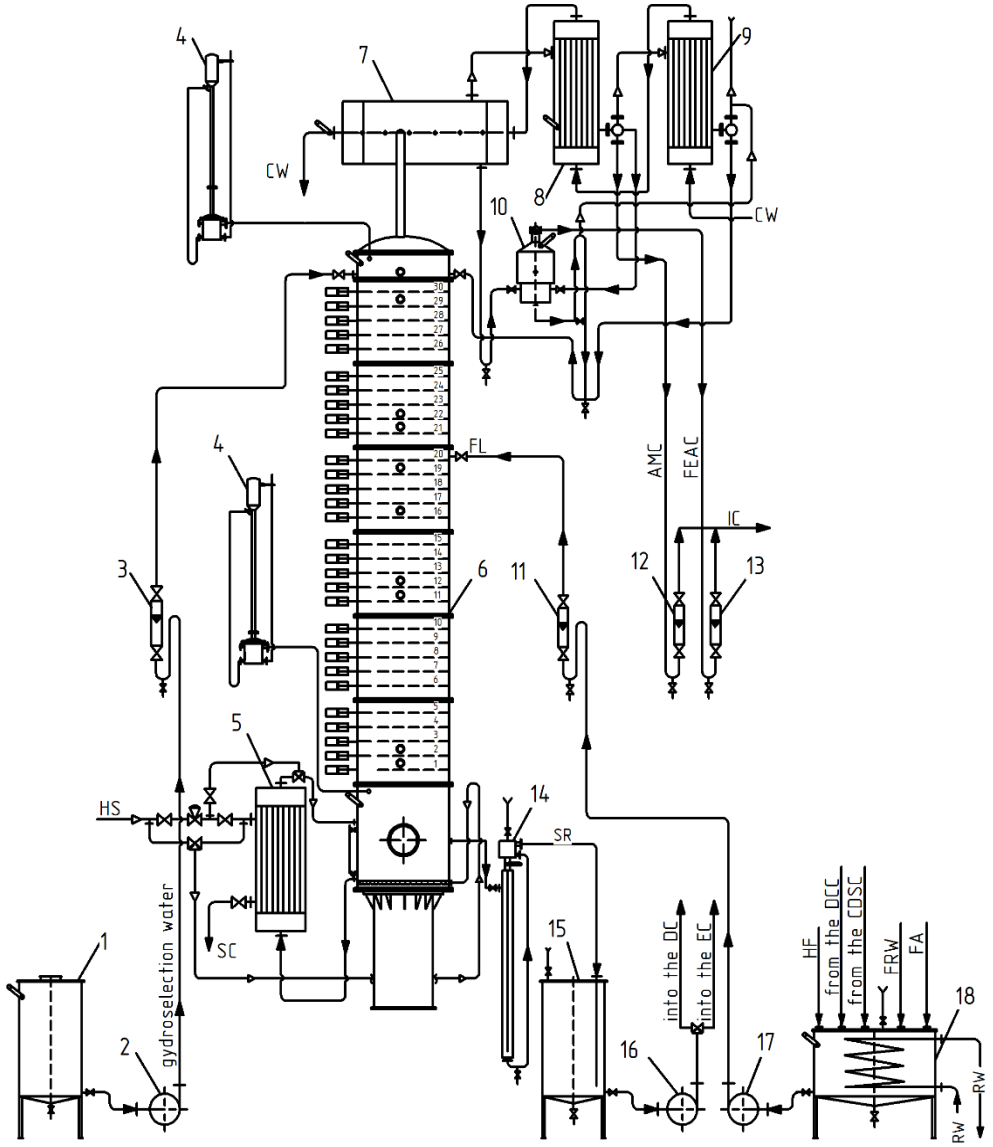


Figure 3. The technological equipment scheme of the cyclic action column

1 – softened water container; 2, 16, 17 – centrifugal pumps; 3, 11, 12, 13 – flow-meters; 4 – vacuum breakers; 5 – evaporator; 6 – rectificational column; 7 – dephlegmator; 8 – condenser; 9 – alcohol-collecting vessel (trap); 10 – decanter; 14 – hydraulic shutter; 15 – still residue container; 18 – alcohol-containing fraction collector.

Notation keys: AMC – aldehyde-methanol concentrate; DC – distillation column; EC – epyurating column; IC – impurities concentrate; DCC – distillation column condenser; CDSC – carbon dioxide separator condenser; SR – still residue; CW – cooling water; FA – fusel alcohol; FEAC – fusel-ester-aldehyde concentrate; SC – steam condensate; FRW – fusel rinse water; HS – heating steam; HF – head fraction; FL – feed liquid; RW – residue water.

The 950 mm diameter RC was equipped with flaky plates with pivoting sections connected to the pneumatic cylinders and modern computer-integrated means (patent UA 136561. Mass-exchange contact plate) (Figure 4).

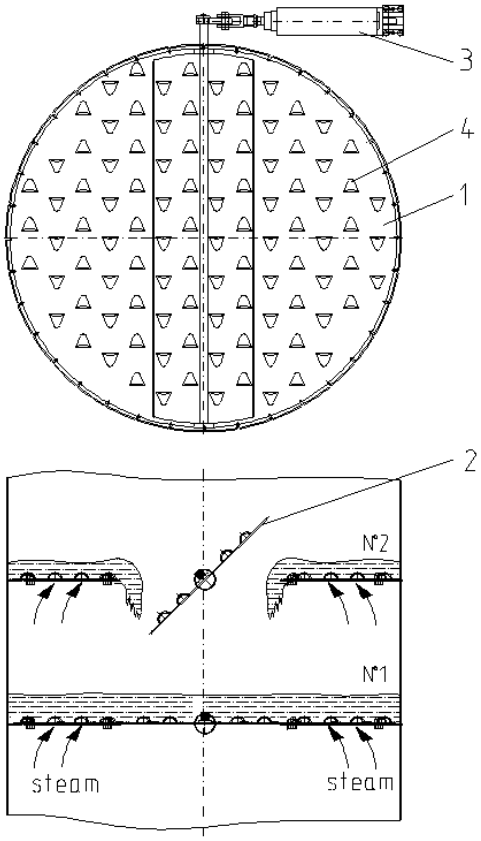


Figure 4. Flaky plate of cyclic action with coaxial placement of flakes and variable free cross-section:
1 – plate fabric; 2 – moving section; 3 – drive mechanism (pneumatic cylinder); 4 – flakes.

The moving sections opened and closed the pouring holes of the plates so that the liquid pouring occurred periodically. The coaxial placement of the flakes made it possible to eliminate the 'one-way' steam and liquid flow and the chance of forming stagnant zones. Pneumatic cylinders and technological parameters operation control (i. e. temperature, pressure) was carried out with the help of automatic sensors, the signal from which was transmitted to the microprocessor controller.

The head fraction of ethyl alcohol, steam condensate from the condensers of the distillation column and carbon dioxide separator, as well as fusel alcohol and fusel rinse water were served on the feeder plate of the column in total amount of 688.3 dm^3/h (250 dm^3/h in terms of anhydrous alcohol (a.a.). The aldehyde-methanol concentrate from the condenser and the fusel-ester-aldehyde concentrate from the upper part of the decanter were sorted to the impurity concentrate collector.

Research methods

Liquid consumption. The consumption of alcohol-containing fractions, water for hydroselection, the still residue and rectified alcohol was monitored using PM flow-meters (Yarovenko et al., 1981).

Concentration of ethyl alcohol in water-alcohol solutions. The concentration of ethyl alcohol in the still residue of the RC was determined by areometric method (Polygalina, 1999).

Concentration of volatile alcohol impurities. The concentration of volatile impurities in the head fraction, in the condensate steam from the condensers of the distillation column and carbon dioxide separator, in fusel alcohol, in fusel rinse water and in the feed of the column were implemented on a gas chromatograph with an HP FFAP 50 m × 0.32 m column (Plutowska et al., 2008; Polyakov, 2007). Three-time repetition samples were taken for chromatographic analysis. The mean values were chosen as the determining ones.

Grade of extraction and concentration ratio of volatile alcohol impurities. The grade of extraction *(α)* and concentration ratio *(β)* of key organic alcohol impurities were calculated as follows:

$$\alpha = \frac{X_{fp}}{X_{sr}}; \quad \beta = \frac{X_{ic}}{X_{fp}};$$

where X_{fp}, X_{ic}, X_{sr} – the concentration of volatile alcohol impurities on the feed plate, impurities concentrate and still residue, mg/dm^3 in terms of a.a. (Shyian et al., 2009).

Studied modes

It is known, that for flaky plates the lower critical speed of steam in holes, at which liquid spilling stops, is 6,5–7,5 m/s, linear speed in free cross-section of the column in barbotage mode is 0,5–0,9 m/s, in transitional 0,9–1,3 m/s and in jet 1,3–2,0 m/s. Upper critical speed of steam is 15–16 m/s (Stojkovic et al., 2018). Intensive liquid pouring through the holes of the plates occurs at steam velocities of 1.5–1 m/s (Gerven et al., 2009).

Considering the above, the velocity of steam in the holes of flakes during the liquid residence on the plates of the studied RC was maintained within 12–14 m/s.

The extraction of ethyl alcohol from alcohol-containing fractions was carried out under the circumstances of moderate and deep hydroselection. Therefore, the upper plate of the column was provided with steam condensate, the temperature of which was 90–92 °C. The condensate consumption was being increased from 2000 to 4500 m^3/h. Yet the concentration of ethyl alcohol in the still residue of the column varied from 2.8 to 8% vol. Depending on the quantity of liquid, the residence time on the plates was being varied from 20 to 60 s, the pouring time — from 7 to 1.7 s. The height of the liquid layer on the plates was 35–40 mm. Depending on the quantity of alcohol-containing fractions and water for hydroselection the pressure at the bottom of the column was being varied between 12 and 18 kPa. For an effective separation of the heterogeneous mixture, the decanter temperature of the RC was being maintained around 30–35 °C (Shyian et al., 2009). The aldehyde-methanol concentrate and the fusel-ester-aldehyde concentrate were being changed from 12 to 1 dm^3/h, while controlling the quality parameters of the RC still residue and rectified ethyl alcohol.

Stages of research

At the first stage, the efficiency of the mass transfer process in the typical (Mishchenko et al., 2020) and cyclic (Maleta et al., 2015) rectification in the existing and experimental RC with hydraulic gates was investigated (Figure 1a). The head fraction of ethyl alcohol, steam condensate from the condensers of the distillation column and carbon dioxide separator, as well as fusel alcohol were served on the feeder plate of the column in total amount of 96 dm^3/h in terms of anhydrous a.a. Heating steam was continuously provided to the lower part of the column and hot softened water – to the upper plate in order to hydroselect the impurities, which ranged the concentration of ethanol in the still residue from 4-5% vol. The residence time of the liquid on the plates was 23 s and the pouring time through the hydraulic shutters was 7 s.

At the second stage the efficiency of mass exchange between liquid and steam in an experimental RC of cyclic action without hydraulic shutters was investigated (Figure 1b). The technical solution suggested by the authors provided time-controlled cycles of liquid residence on the plates and its pouring from the upper plates to the lower ones, thanks to instantaneous change of steam velocity in the holes from 12–14 to 1,5–1 m/s by changing free cross-section of the plates from 5,5 to 51,7 %. While the valves were being lifted at the moment the pouring holes were opened, the steam velocity in the holes became lower than critical and the liquid was pouring simultaneously through all the holes to the underlying plates.

At the third stage of the research the optimal parameters of mass exchange process of the experimental RC operation (Figure 3), equipped with flaky plates with turnaround sections, presented in Figure 4. The research included liquid sampling at the feeder plate (FL) as well as samples of the head fraction (HF), fusel alcohol (FA), fusel rinse water (FRW), fractions from the distillation column condenser (DCC) and carbon dioxide separator condenser (CDSC). To determine the efficiency of processing alcohol–containing fractions in a given hydrodynamic mode, the concentration of volatile impurities of alcohol in the still residue (SR), impurities concentrate (IC), epyurate (E), and rectified ethyl alcohol (REA) were studied. The results of the chromatographic analysis of the studied samples are shown in Tables 1 and 2.

Results and discussion

Study on the efficiency of mass exchange between liquid and steam in RC, equipped with moving valves and plates with hydraulic shutters.

Studies have shown that in the experimental column the esters were completely removed. The grade of extraction of higher alcohols of fusel alcohol and methanol in the cyclic mode increased by 25%, the concentration ratio of head impurities – by 21%, higher alcohols and methanol – by 30% in comparison with the column operating in the stationary mode. That said, it reduced the specific heating steam consumption by 38% and 1.2 kg/dal of a.a. introduced into the column. This is explained by the fact that when the phase contact time had been prolonged from 13 to 23 s, the difference in concentration of volatile impurities in steam and liquid decreased, thus increasing the grade of phase equilibrium (Bozeya et al., 2013).

The disadvantages were the low liquid capacity of the column (750 dm^3/h), its mixing on adjacent plates during pouring and a 15% reduction in the working area of the plate due to the presence of the hydraulic shutters.

Study on the efficiency of mass exchange between liquid and steam in RC, equipped with moving valves and plates without hydraulic shutters

Design changes allowed to increase the liquid throughput by 34% (750 to 1000 dm^3/h) without reducing the liquid retention time by lessening the pouring time from 7 to 2 s. Due to the absence of hydraulic shutters, the contact area of the phases on each plate has increased by 15%, which has improved the performance of the plates and the efficiency of the mass exchange: the grade of extraction of higher alcohols of fusel alcohol and methanol was increased by 29%, the concentration ratio of aldehydes was increased by 23%, higher alcohols – by 33 % and methanol by 34 % compared to a column operating in stationary mode.

One-stage (full) and two-stage pouring methods

The one-stage (full) pouring method did not provide an even distribution of liquid on the plates due to a lack of liquid on the paired plates while it being held on the unpaired plates and vice versa (Figure 2a). This technical decision made it impossible to maintain a stable hydrodynamic regime along the height of the column (Chu et al., 2013).

In order to optimize the operation of the RC and to increase the efficiency of mass exchange, the pouring of liquid from plate to plate was carried out in two stages (Figure 2b). The method allowed to operate all the plates simultaneously, to ensure that the liquid level on the plates is the same throughout the height of the column and to stabilize the hydrodynamic mode of their operation. At that, the RC liquid throughput has increased by 20% (from 1000 to 1200 dm^3/h), the grade of extraction of higher alcohols of fusel alcohol and methanol – by 38%, the concentration ratio of head impurities has increased by 25%, higher alcohols – by 38%, methanol – by 37% compared to a column operating in stationary mode.

The disadvantage of the one- and two-stage methods of liquid pouring is the impossibility of autonomous regulation of liquid residence time on each individual plate, because moving elements of pouring devices of paired and unpaired plates were set in motion by one drive mechanism.

Studies on the efficiency of mass exchange between liquid and steam in RC, equipped with plates with rotary sections

To eliminate the disadvantages mentioned above, the authors have proposed a method of processing alcohol-containing fractions in a column equipped with plates with rotary sections (patent UA 136560. Method of mass-exchange between liquid and steam in a column apparatus). The results of chromatographic analysis of alcohol–containing fractions entering the column and the distribution of impurities in its still residue, concentrate, epyurate and rectified alcohol are presented in Tables 1 and 2.

The criterion for the RC optimization was the concentration of acetaldehyde, higher alcohols of fusel alcohol (including isopropyl alcohol) and methanol in the still residue and in the rectified ethyl alcohol. The determinants of mass exchange efficiency between liquid and steam were the grade of extraction and concentration ratio of volatile alcohol impurities in the studied RC.

Table 1

Results of the chromatographic analysis of alcohol-containing fractions

Impurity name	Concentration, mg/dm^3					
	HF	**DCC**	**CDSC**	**FA**	**FRW**	**FL**
Ethanol, % об.	92,5	48,8	60	89	17,5	30,5
Aldehydes	1135,2	37,2	126,2	4,9	7,0	318,7
Acetaldehyde	926,1	37,2	90,9	4,9	7,0	242,3
Methylacetate	209,1	traces	35,3	traces	traces	76,4
Esters	2394,9	186,4	39,7	20,2	68,3	40,5
Ethylacetate	2223,6	165,8	traces	2,1	traces	trace
Isobutylacetate	23,0	13,0	7,9	10,1	traces	11,1
Isoamylacetate	90,6	7,6	31,8	8,0	68,3	29,4
Ethylbutyrate	57,7	traces	traces	traces	traces	traces
Methanol, %	0,49	0,025	0,1445	0,013	0,0032	0,18
Fusel alcohol	3113,1	18820	12583	48824	197726	105883
Isopropanol	4,9	4,9	1,7	1,1	traces	1,2
n-propanol	1186,4	1403	699,6	14741	36681	20002
Isobutanol	1640	606,1	4082	27557	36826	20297
n-butanol	2,7	6,4	16,5	35	705,2	362
Isoamylol	279,1	1863,5	7783	6485,3	123514	65221

Table 2

Concentration of impurities in the cube liquid, impurities concentrate, epyurate and rectified ethyl alcohol

Impurity name	Concentration, mg/dm^3			
	SR	**IC**	**E**	**REA**
Ethanol, % об.	3,7	67	30,1	96,5
Aldehydes	2,8	2302,2	0,3	0,18
Acetaldehyde	2,8	1396,7	0,3	0,18
Methylacetate	traces	905,5	traces	–
Esters	traces	446615	traces	–
Isobutylacetate	traces	3234,8	traces	–
Isoamylacetate	traces	494,4	traces	–
Ethylbutyrate	traces	442886	traces	–
Methanol, %	0,004	2,69	0,0023	0,0003
Fusel alcohol	721,7	726464	1179,8	0,88
Isopropanol	traces	22,4	0,4	0,88
n-propanol	677,5	220,6	121,4	–
Isobutanol	4,9	357247	326,0	–
n-butanol	2,7	1003,8	2,0	–
Isoamilol	13,8	367970	728,5	–

According to the results of the study, optimal technological parameters of RC operation were:
– the liquid retention time on the plates is 40 s;
– the time of liquid pouring from the upper plate to the lower one is 1.7 s;
– the pressure at the bottom of the column is 11.5–12 kPa;
– pressure at the top of the column is up to 0.03 kPa;
– the temperature at the bottom of the column is 100.5–101.5 °C;
– the temperature in the steam phase above the upper plate is 93.5–94 °C;
– temperature in the steam phase on the plate of feed is 93.2–94 °C;
– the water temperature for hydroselection is 95–98 °C;
– the temperature of the mixture in the decanter is 30–35 °C;
– water consumption for hydroselection is 4050–4500 dm^3/h;
– the temperature in the tube space of the condenser is 45–50 °C;
– water temperature for cooling after the dephlegmator is 85–87 °C;
– concentration of ethyl alcohol in the still residue is 3–4 % vol.;
– withdrawal of aldehyde-methanol concentrate (AMC) from the RC is 7–9 dm^3/h;
– concentration of ethyl alcohol in the AMC is 70.5% vol.;
– withdrawal of the fusel-ester-aldehyde concentrate (FEAC) from the decanter is 2–3 dm^3/h.

The calculated values (α) and (β) at RC operation in the selected hydrodynamic mode and the specified optimal technical parameters are shown in Table 3.

Table 3

Calculated values of the grade of extraction (α) and concentration ratio (β) of volatile alcohol impurities

Name of impurities	Typical rectification		Cyclic rectification	
	α	β	α	β
Aldehydes	85,4	5,3	113,8	7,2
Acetaldehyde	63,7	4,3	86,5	5,8
Methylacetate	∞	8,8	∞	11,9
Esters	79,7	8163,7	∞	11027
Isobutylacetate	57,8	214,7	∞	291,4
Isoamylacetate	∞	12,3	∞	16,8
Methanol	27,6	9,3	45	14,9
Fusel alcohol	89,8	4,1	146,7	6,9
Isopropanol	87	10,9	∞	18,7
n-propanol	17,9	0,005	29,5	0,01
Isobutanol	2414,4	10,5	4142,2	17,6
n-butanol	82,9	1,6	134,1	2,8
Isoamylol	3953,2	3,3	4726,2	5,6

Result analysis

Analysis of the obtained results showed that by increasing the contact time of steam and liquid on the RC plates to 40 s the grade of extraction and concentration ratio of volatile alcohol impurities increased by 25-38%. At the same time, complex esters, methylacetate and isopropyl alcohol are completely extracted – those are the impurities that significantly

degrade the quality of rectified alcohol in small amounts. This can be explained by the fact that there was more of a complete steam saturation with it volatile components on the plates of the column and liquid with volatile steam components, the mixing of liquid on adjacent plates during its pouring was excluded, so that the grade of phase equilibrium achievement was increased (Chen et al., 2010; Shyian et al., 1991). The prolonging in the residence time of the liquid on the plates longer than 40 s proved to be impractical due to an increase in the specific heating steam consumption without a significant increase in the grade of impurity extraction.

Specific consumption of heating steam in experimental RC decreased by 40% (from 20 to 12 kg/dal of a.a. injected to the feed plate) compared to the column operating in the stationary mode. This is explained by the fact that the free cross-sectional area of the plates in the column of cyclic action was 50–75% smaller than that of the column operating in the stationary mode, and was 2.5–5.5% (Bausa et al., 2001).

After the experimental distillation column for concentrating impurities was put into operation, the yield of rectified ethyl alcohol increased by 3.8% due to its extraction from the head fraction and other alcohol-containing waste without deteriorating its qualitative indicators. The use of the RC liquid purified from volatile impurities for carrying out hydroselection in the epyurating column made it possible to reduce the consumption of hot softened water by 2000 dm^3/h (patent UA 119277. Method of producing rectified alcohol; Ukrainets et al., 2006).

Conclusion

1. To increase the efficiency of mass exchange between liquid and steam in rectification columns the expediency of using a cyclic rectification technology that provides periodic pouring of liquid from plate to plate at continuous supply of alcohol-containing fractions and steam in the column is proved.
2. To implement the technology, the plates have to be equipped with moving sections connected to driving mechanisms (e.g., pneumatic cylinders), which are controlled according to the program of the controller in consonance with a predetermined algorithm.
3. Equipping the columns with flaky plates allows to increase their capacity by 34% due to intensification of liquid pouring by doing so simultaneously through the pouring and barbotage holes.
4. At the moment of liquid pouring, steam velocity in barbotage holes should be 1,5–1 m/s. At this speed the pouring occurs within 1.7 s.
5. To ensure stable operation of the plates during the period of liquid retention and in order to intensify its pouring, their free cross-section area should instantly change from 5.5 to 51.7%.
6. In working environment, the optimal technological parameters of column operation of cyclic action were established. It is experimentally proven, that prolonging the contact time of steam and liquid up to 40 s allows to increase the grade of extraction and concentration ratio of volatile impurities of alcohol by 25–38% compared to a column operating in stationary mode. In doing so, the complete extraction of esters, methylacetate and isopropyl alcohol is provided.
7. The coaxial placement of the flakes on the plate fabric allows to eliminate the possibility of formation of stagnant zones and intensify the mass transfer between steam and liquid.

8. The use of innovative technology makes it possible to reduce the specific consumption of heating steam during processing of alcohol-containing fractions by 40% compared to the known ones.
9. It is advisable to use the results of the research to design column mass exchange apparatuses of cyclic action.

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Sorption properties of bread based on oatmeal

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Abstract

Introduction. For improving the quality of gluten-free bread, it is offered to use hydrocolloids. The aim of the study is to determine the sorption characteristics of bread from oat flour and optimize its storage conditions.

Materials and methods. To determine the sorption characteristics, an Autosort apparatus was used. To control humidity, the method of high performance liquid chromatography was used. The relative humidity conditions were varied from 0 to 90% at a temperature of 25 °C.

Results and discussion. The efficiency of joint use of glucano-delta-lactone and food casein for bread production with the use of oatmeal has been proved. The regularities of the influence of oatmeal on the properties of the dough and the qualitative characteristics of bread are determined. It was found that the introduction of glucano-delta-lactone in the amount of 1.0% by weight of oatmeal in the dough in combination with food casein causes an increase in the specific volume and porosity of the finished products. According to the results of determining the change in the specific volume of dough during fermentation, it was found that the increase in the quality of gluten-free bread with the introduction of glucan-delta-lactone and food casein is associated with increased gas-holding capacity of dough semi-finished products. The positive effect of the use of glucano-delta-lactone in the technology of rice bread on the elastic properties of the crumb of the products has been clarified. The identified patterns make it possible to predict the extension of the shelf life of finished products.

In the studied products, adsorption begins as polymolecular and ends with capillary condensation with developed hysteresis, in which the amount of absorbed moisture and removed does not match. The adsorption process is influenced by the shape and radius of the capillaries.

The introduction of oatmeal in combination with casein and glucan-delta-lactone affected the redistribution of pores by radius, increasing the total volume of larger pores with a radius within $(50–55)\times10^{-10}$ m almost twice, compared to the structure of the product exclusively on oatmeal.

Conclusions. Addition of oatmeal in the amount of 100% with glucan-delta-lactone in the amount of 1% and natural protein of animal origin – casein in the amount of 5% improves bread quality: increases porosity, increased specific volume, which has a positive effect on the sorption properties of finished products.

Introduction

It is considered that oats and oat products are not suitable for baking dough or bread, because it does not contain gluten, although not many studies have been conducted to study new applications. Differences in oat varieties regarding the potential for making gluten-free bread recipes based on a pancake-like system (known as dough) (Thompson, 2003). However, it is not known what factors are important for bread making and whether, for example, a high content of β-glucan will be suitable for bread procurement (Ronda et al., 2015). The same goes for the fat content, which is extremely high in oats compared to wheat. This high fat content in oats requires an additional processing step to avoid damaging the sensory quality of the grains (Hoffenberg et al., 2000).

The quality of most foods largely depends on their physical, chemical and microbiological stability. This stability is mainly due to the relationship between the equilibrium moisture content (EMC) of the food material and its corresponding water activity (a_w) at a certain temperature (Demirkesen et el., 2010). The sorption isotherm describes the thermodynamic relationship between water activity and food moisture balance at constant temperature and pressure (Torbica et al., 2010). Knowledge and understanding of sorption isotherms are extremely important in food science and technology for the design and optimization of drying equipment, packaging design, quality prediction, stability, shelf life and for calculating moisture changes that may occur during storage (Wehrle et al., 1998). The sorption isotherm can be used to study the structural features of a food product, such as surface specific surface area, pore volume, pore size distribution, and crystallinity (Ramanathan, 1994). Sorption isotherms can be obtained by adsorption or desorption; the difference between these curves is defined as hysteresis (Ajisegiri et al., 2007).

The most common equations used to describe sorption in foods include the Langmuir equation, the Brunauer equation, the Emmett, Teller (BET) model, the Oswin model, the Smith model, the Helsey model, the Henderson model, the Iglesias Kirife equation, the GAB model, and the Peleg model et al. (Paderewski et al., 1999). Thus, the net isosteric heats of sorption for beta-glucan rich biscuits, calculated using Clausius-Clapeyron equation, showed an exponential relationship with moisture content (Adeseye et al., 2019). Based on the results of the study of sorption by scientists, it is confirmed that the quality of bread flour is significantly affected by storage conditions (Panjagari et al., 2014).

Knowledge and understanding of sorption isotherms are critical for equipment design, prediction of food quality, stability and shelf life, packaging design, and for calculating moisture changes that may occur during storage (Gray et al., 2003). The MSI of most foods are nonlinear, usually S-shaped, and are classified as type II isotherms (Paderewski et al., 1999). Several attempts have been made to describe sorption isotherms using mathematical models and to confirm the effectiveness of such models using statistical methods (Sciarini, 2017). It has been scientifically proven and established that the quality of products deteriorates due to changes in sensory properties, which is accompanied by an increase in the number of harmful microbes, and these deteriorating changes increase with increasing water activity. Water activity (aw) is the ratio of water vapor pressure over a given product to vapor pressure over pure water at the same temperature (Nascimento et al., 2013).

The *aim* of the study is to determine the sorption characteristics of bread from oat flour and optimize its storage conditions. It is studied the bread, prepared with the use of oatmeal enriched with biologically valuable prescription components – casein and glucan-delta-lactone.

Materials and methods

Methods of sorption measurements

To estimate the parameters of the porous structure of the materials used the method of static interval isothermal sorption. The weight version of the sorption method allows you to simultaneously measure the amount of steam absorbed by the sorbent and the equilibrium pressure of the same steam over the created system. The principle of the weight method of sorption study is to determine the amount of sorbed substance by the difference in weight of the sample of sorbent before and after sorption (Ramanathan et al., 1994).

To do this, we used a sorption unit (Figure 1), which consists of two main parts: a vacuum, which serves to create a residual air pressure of 10^{-3} Pa, and a working one, in which sorption measurements performed directly. The working *part A* is placed in an air thermostat equipped with a heater and a contact thermometer.

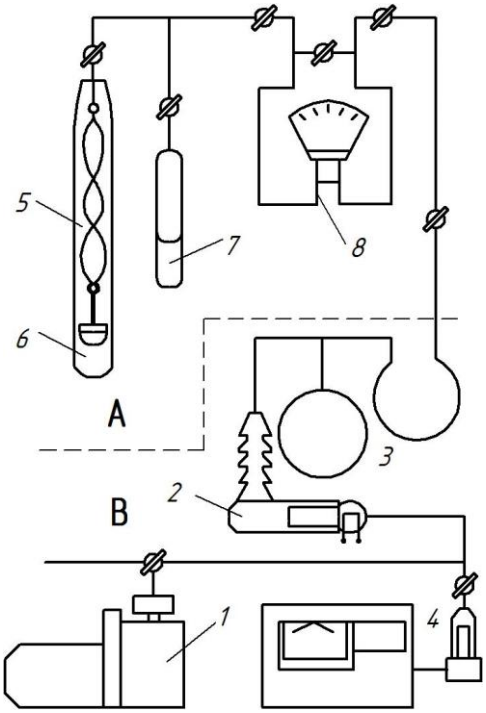

Figure 1. High-vacuum sorption installation scheme

The working part of the installation is a detachable cylindrical tank (5), to the head of which are suspended spiral scales of McBen. A thin-walled glass cup with a sample (6) is attached to the lower end of the spiral. Pre-calibrated quartz spirals should have a sensitivity of about $0{,}5\times10^3$ m/kg and have no hysteresis. The ampule (7) contains a liquid whose vapors sorbed. Sorption experiments performed at a temperature of 298 K.

Prior to the determination, the test sorbent and sorbate subjected to vacuum at a residual pressure of 10^{-3} Pa to constant weight, in order to remove volatile substances.

The B-630 cathetometer used to supply steam, after which the stretching of the quartz spirals monitored and the kinetic curve of steam absorption by the sorbent constructed. Measurements performed with an accuracy of 5×10^{-5} m.

Experimental data were used to calculate the equilibrium number of grams of sorbed steam (x/m) per gram of sorbent by the equation:

$$\frac{x}{m}=\frac{\gamma\times\Delta n}{m}\ , \tag{1}$$

where Δn – spiral elongation, mm; m – the amount of polymer, g; γ – the price of spiral division, g/mm, and to calculate the amount of adsorption a (mmol) per 1 g of sorbent:

$$a=\frac{x\times1000}{M\times m}, \tag{2}$$

After equilibrating, the equilibrium pressure of the sorbate vapor is measured. The obtained data was presented in the form of a sorption isotherm, which was constructed in the coordinates x/m (a) depending on the relative vapor pressure p/p_s, where p_s is the saturated vapor pressure of the sorbate at the study temperature.

The Autodesk system used to determine the sorption characteristics. The method of high performance liquid chromatography used to control the humidity. Relative humidity conditions ranged from 0 to 90% at a temperature of 25 °C (9).

Sorption characteristics systematize data on equilibrium and monomolecular moisture levels, based on which it is possible to correctly determine the methods for processing, storage, and packaging of finished products (Ramanathan et al., 1994).

The relationship between the equilibrium humidity M and the activity of water aw was determined experimentally and the results depending on the temperature built sorption isotherms (Ajisegiri et al., 2007).

Calculation of the specific surface area S_s

Adsorption isotherms make it possible to determine the capacity of a monolayer of a test sample, which can be used to calculate its specific surface area S_s. The capacity of a monolayer is the amount of adsorbate that can be accommodated in a fully filled adsorption layer 1 molecule thick – a monolayer – on the surface of a unit mass (1 g) of a solid. From the capacity of a monolayer a_m, expressed in moles of adsorbate per gram of adsorbent, the specific surface area as the surface area of 1 g of a solid S_s is calculated by the simple equation:

$$S_s=a_m\cdot\omega\cdot N_A\,, \tag{3}$$

where ω is the average area occupied by an adsorbate molecule in a filled monolayer, N_A is Avogadro's number.

The value of a_m was determined using the equation of the sorption isotherm obtained by Brunauer, Emmett and Teller (*BET equation*) (Paderewski et al., 1999),

$$a=\frac{a_m\cdot c\cdot p/p_S}{(1-p/p_S)\cdot\left[1+(c-1)\cdot p/p_S\right]}\ , \tag{4}$$

where a – the equilibrium number of moles of sorbed substance per gram of sorbent, p/ps – relative vapor pressure, c – a constant.

To calculate the value of a_m, the BET equation modified:

$$\frac{p/p_S}{a\cdot\left(1-p/p_S\right)}=\frac{1}{a_m\cdot c}+\frac{c-1}{a_m\cdot c}\cdot p/p_S \quad , \tag{5}$$

Equation type 4 is derived for sorbents, in the pores of which the sorption process accompanied by the phenomenon of capillary condensation. For such sorbents, the sorption isotherm has a typical S-shape with sorption hysteresis. Similarly, this equation formally is used to calculate the values S_s of non-porous adsorbents.

The calculation S_s was performed on the basis of experimental data, constructing the sorption isotherm in coordinates $a=\int\left(p/p_S\right)$.

Calculation of the total pore volume W_0

Depending on the nature of the porous structure of the sorbent, there are different methods of calculating W_0. In our case, the total pore volume calculated from the maximum amount of sorbed substance, because the sample belongs to the group of mesoporous sorbents characterized by S-shaped sorption isotherms.

The absorption of the sorbed substance a_{max} occurs at $p/p_s = 1$, according to the equation:

$$W_0 = a_{\max}\cdot V_{\max}\,, \tag{6}$$

The right-hand side of this equation is the maximum volume of sorbed matter, which is essentially equal to the volume of pores available to the molecules of this sorbate.

Results and discussion

Effect of glucan-delta-lactone (GDL) on the change of technological characteristics and quality of bread

The study of the effect of glucan-delta-lactone on the change of technological characteristics and quality of bread depending on its dosage is presented in Table 1.

Table 1

Influence of GDL on dough properties and bread quality

Quality indicators	Control sample	Sample with the addition of GDL,%		
		0,5	1,0	1,5
Semi-finished dough				
Titrated acidity	2,7	2,7	2,8	2,8
Active acidity	4,3	4,4	4,4	4,5
Gas formation, cm^3/100 g of dough	420	408	400	398
Specific volume, cm^3/g	1,60	2,3	2,4	2,1
Bread				
Specific volume, cm^3/g	1,86	2,55	2,70	2,18
Acidity, deg	3,1	3,1	3,1	3,2
Porosity,%	52,3	68,1	69,7	66,4

The data obtained indicate that during fermentation of the dough from glucano-delta-lactone there is a slight decrease in the intensity of acid accumulation, which is apparently associated with a decrease in the activity of lactic acid bacteria and yeast Saccharomyces during maturation of dough semi-finished products.

Thus, the introduction of glucano-delta-lactone in the amount of 0,5–1,5% by weight of flour leads to a decrease in the amount of carbon dioxide released by 1,9–3,8% relative to the control. Obviously, a colloidal solution of cellulose ether wraps a thin film of yeast cells, limiting access to nutrients. The study of quality indicators of finished products showed that their specific volume in the case of application of glucan-delta-lactone in the amount of 0,5% and 1,0% by weight of flour increases by 35,0 and 37.1%, respectively, compared with the control. The porosity of the crumb is improved by 30,0 and 32,5% relative to the control sample. The obtained results are explained by the improvement of the gas holding capacity of the dough, as a result of which, despite the reduction of gas formation in the semi-finished product, the losses of carbon dioxide formed during fermentation are insignificant.

As a result of experimental researches it is established that increase in dosage of GDL above 1.0% to weight of flour is inefficient as at the same time the crumb of products is condensed, has worse developed porosity, bread has smaller specific volume, and on its surface there are cracks.

The increase in the specific volume of the dough with the combined application of glucano-delta-lactone and casein is more intense compared to the control sample without hydrocolloid. This is due to the high water holding capacity of GDL, which reduces the content of free moisture in the system, that is, the layers of intermicellar fluid between the colloids of the dough are reduced, which leads to the formation of a homogeneous framework of the semi-finished product, resulting in improved structural and mechanical properties.

The results of the research became the basis for further determinations of the sorption properties of finished bread products.

Optimization of the prescription composition of oatmeal bread

The study of the chemical composition of alternative raw materials – oatmeal, allows understanding and explain the main processes of dough formation, and most importantly to choose the winning combinations of improvers in order to eliminate possible shortcomings (Paderewski et al., 1999).

Given the fact that oat products do not contain gluten (Thompson et al., 2003), thus questioning the use of oats as a main component in baking, an effective measure to mimic the gluten skeleton in the technology of gluten-free bakery products based on oatmeal is the use of various improvements.

Our results are promising, because even without process optimization and without understanding the functionality of all oat components except beta-glucans, the overall appearance of bread products and their specific volume lead us to think that there is room for much greater improvement. Our oat bread is similar to the bread that consumers are familiar with (Figure 2). We believe that the lack of knowledge is the main reason for the limited availability of oat bread. Oats, as an agricultural crop and as a raw material for the food industry, has features that must first be understood in order to be able to deal with them in the process of product development.

Nutritionists have confirmed that it is advisable to use glucan-delta-lactone (GDL) as a structurant of food systems (Li et al., 2016).

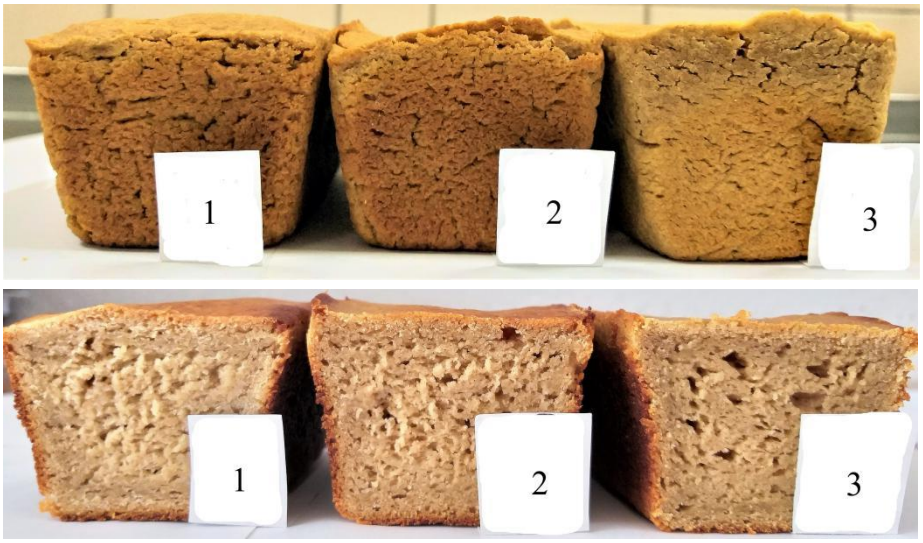

Figure 2. Picture of test baking results
1 – bread with 100% oatmeal; 2 – bread with 100% oatmeal and 1% GDL;
3 – bread with 100% oatmeal, 5% casein and 1% GDL

In the course of trial laboratory baking, it was found that the addition of only gdl in the technology of production of bread products is not sufficient. This technology needs the improvement.

To improve the viscoplastic properties of oat raw materials, we have selected a combination of a structuring agent and a protein-containing component of animal origin, taking into account the research of specialists in this field (Deora et al., 2014; Stathopoulos et al., 2008; Ziobro et al., 2016).

The next stage of our research was to conduct a trial baking of bread with the introduction of GDL and casein at the established optimal ratio.

During the study it was found that the introduction of oatmeal in the amount of 100% with a structuring substance (glucan-delta-lactone) in the amount of 1% and natural animal protein (casein) in the amount of 5% improves the quality of bread: increases porosity, increases specific volume. The humidity and acidity of all baked samples remain within the norm set by the standard. This is because the introduction of oatmeal into the dough intensifies the fermentation process due to sugar, vitamins, minerals, which are an additional source of nutrition for yeast.

According to the results of the study, it was found that the best quality indicators are bread with a complete replacement of wheat flour with the addition of a structural component and protein of animal origin.

Effect of temperature and relative humidity on equilibrium moisture content

The next step was to study the sorption characteristics of the obtained samples.

The experimentally obtained values of equilibrium humidity M, % dry weight, and standard deviation s_d of the investigated product at different values of water activity a_w and

at a temperature of 25 °C for adsorption and desorption processes is shown in Table 2. The equilibrium moisture content at each a_w represents the mean value of three replications (Adeseye et al., 2019).

Table 2

Comparative characteristics of research results

a_w	M	s_d
0,106	3,24959	0,0881
0,249	5,46128	0,0787
0,348	6,88063	0,1304
0,443	8,39007	0,2291
0,538	10,0220	0,2342
0,629	12,0427	0,2482
0,737	15,6526	0,3046
0,818	19,9702	0,240

Based on the obtained data, it is confirmed that the equilibrium moisture content increases with increasing in temperature at constant relative humidity and increases with increasing in relative humidity at constant temperature.

To compare sorption and desorption, the figures show the isotherms of all the studied samples (Figures 3, 4).

The sorption-desorption isotherms of water (Figures 3, 4) are given in the coordinates: the amount of adsorbed water (a) – its activity (a_w), which is directly related to the relative equilibrium vapor pressure ($a_w = P/R_S$).

To compare sorption and desorption are given isotherms in Figures 3: a sample of bread based on oatmeal with the addition of 1% GDL (Figure 3, a), a sample of bread based on oatmeal from a combination of 1% GDL and 5% casein (Figure 3, b) and a sample of bread based on oatmeal without additives (Figure 3, c).

Analysis of isotherms showed that the characteristic S-shaped configuration of the lines corresponds to polymolecular adsorption. In the works (Adeseye et al., 2019; Panjagari et al., 2014; Gray et al., 2003) note that in the adsorption accompanied by capillary condensation, hysteresis is often observed, so we can conclude that in the studied products adsorption begins as polymolecular and ends with capillary condensation with developed hysteresis, in which the amount of absorbed moisture and coincides.

It is known (Gomez et al., 2013) that the hygroscopicity of raw materials depends on its porosity. It is obvious that the main part of moisture in the investigated raw material condenses in small pores of the adsorbent – capillary condensation. To test this hypothesis, the distribution of pore volume by radius is analyzed (Figure 3) and the pore volume (V) in the studied raw material is determined (Paderewski et al., 1999):

$$V = \int_a^b f(R)dR, \tag{7}$$

The results of calculations of pore volume with a certain radius for the studied bread samples are shown in Table 3.

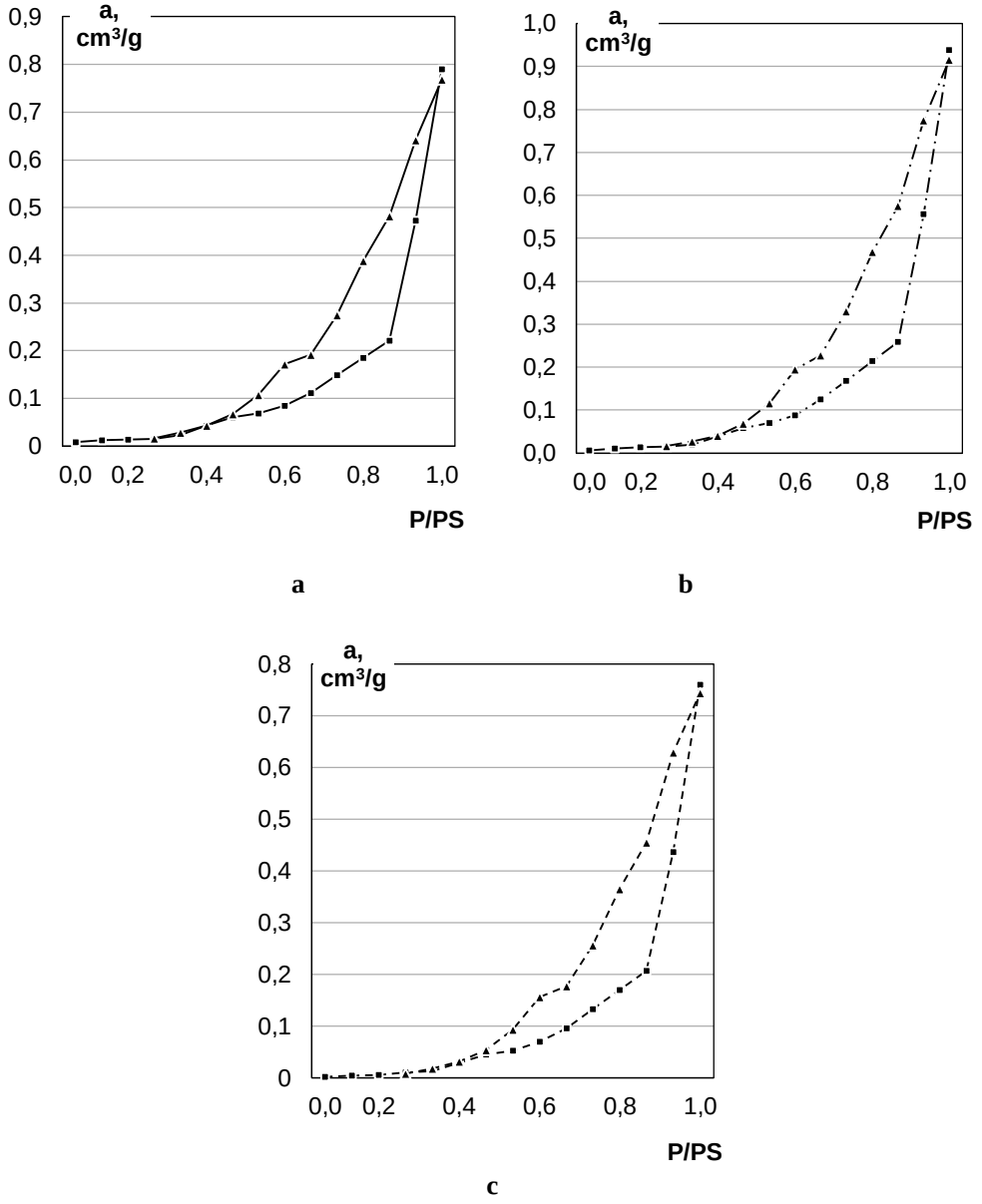

Figure 3. Sorption isotherms of adsorption and desorption processes
a – bread with 100% oatmeal and 1% GDL;
b – bread with 100% oatmeal, 5% casein and 1% GDL;
c – bread with 100% oatmeal.

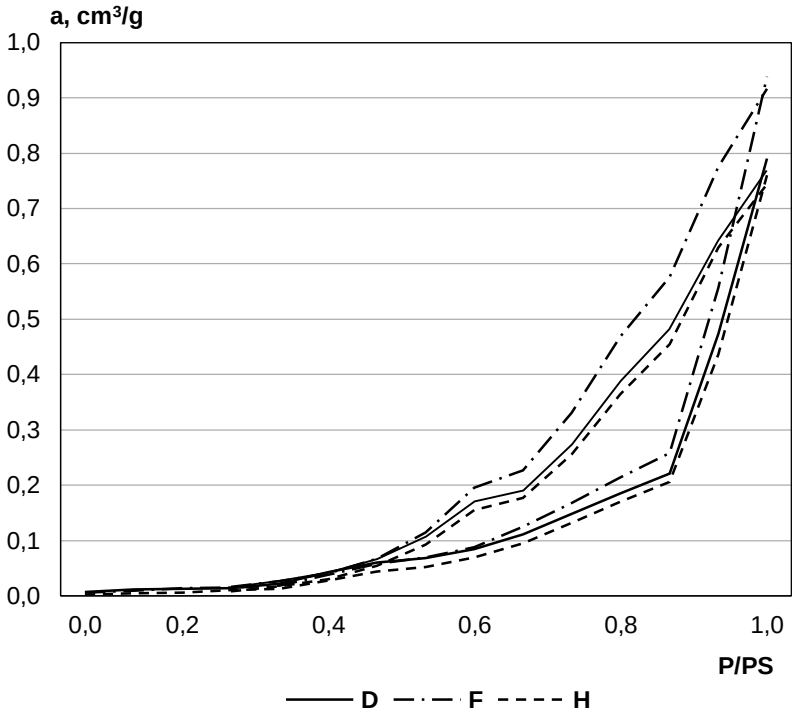

Figure 4. Sorption isotherms of adsorption and desorption processes. Generalized results for the three studied samples:
D – bread with 100% oatmeal and 1% GDL;
F – bread with 100% oatmeal, 5% casein and 1% GDL;
H – bread with 100% oatmeal.

Table 3

Structural characteristics of samples

№	Sample name	S, m^2/g	R^2	V_S, cm^3/g	D, A
1.	100% oatmeal, 1% GDL	54	0,8619	0,79	585
2.	100% oatmeal, 5% casein, 1% GDL	53	0,9788	0,94	696
3.	100% oatmeal	56	0,4964	0,76	543

Based on the first graph of moisture adsorption, we can say that the samples adsorbed moisture to a pressure of P/P_s = 0,3 is not active, because there was a so-called adsorption of the surface layer of samples, and then – penetration into the internal volume, and they were activated. After all, under the pressure of moisture, the samples loosened and absorbed vapors, because the pore volume of the samples is quite high.

The sample with the type "100% oatmeal, 5% casein, 1% improver" gained the most moisture (Figure 5, b), the pore volume was equal to: V_s = 0,94 cm^3/g, and the sample with the type "100%" gained the least moisture. Oatmeal (Figure 5, c), as evidenced by its pore volume: V_s = 0,76 cm^3/g (from the Table of structural characteristics).

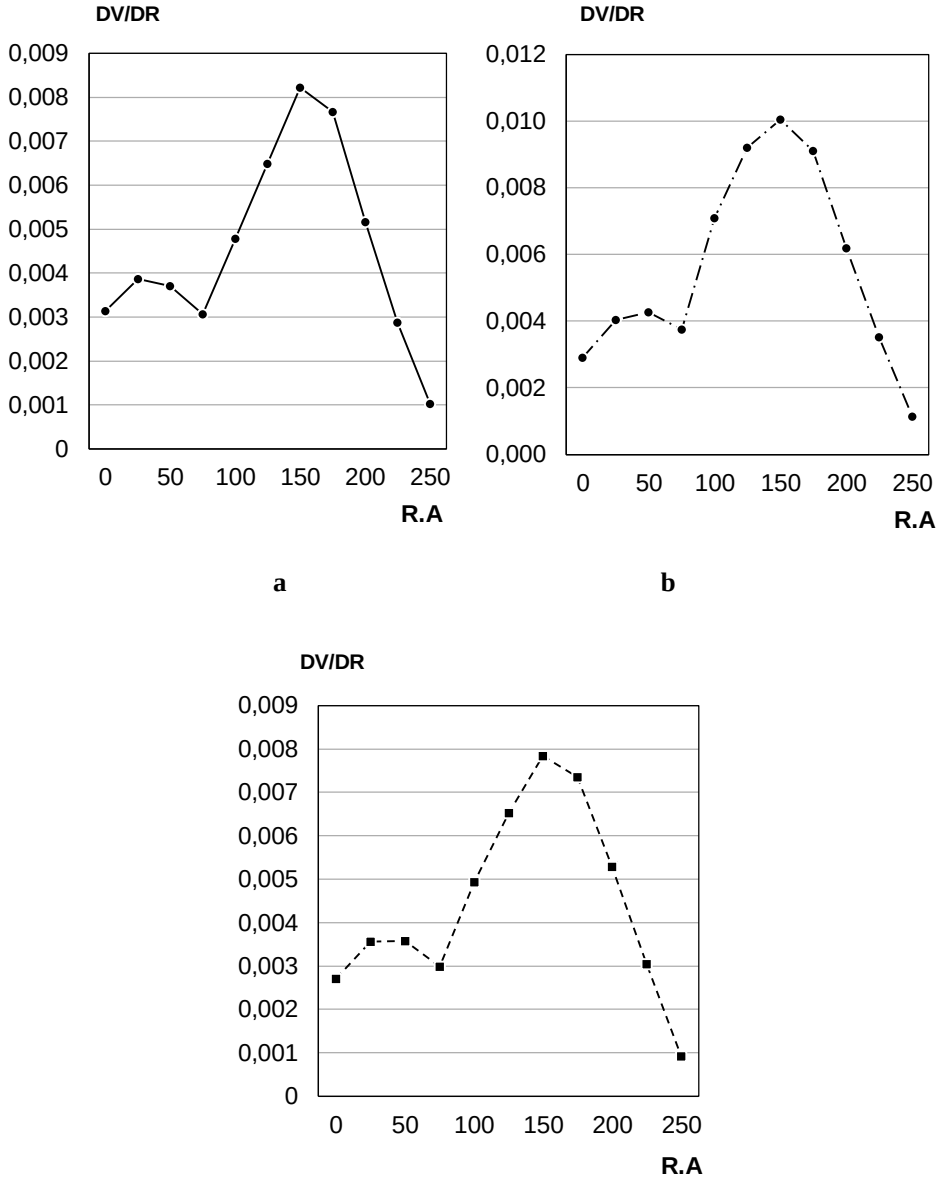

Figure 5. Distribution of pore volume by their radius in the raw material:
a – bread with 100% oatmeal and 1% GDL
b – bread with 100% oatmeal, 5% casein and 1% GDL
c – bread with 100% oatmeal

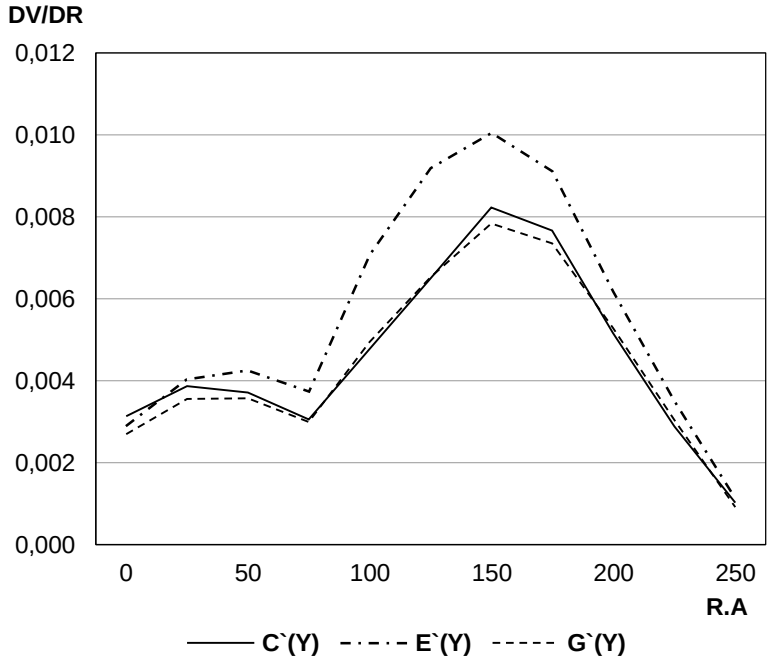

Figure 6. Distribution of pore volume by their radius in the raw material. Generalized results for the three studied samples:
C` – bread with 100% oatmeal and 1% GDL;
E` – bread with 100% oatmeal, 5% casein and 1% GDL;
G` – bread with 100% oatmeal

From the data in Figures 5, 6 and Table 2 we can conclude that the addition of a composition with 1% GDL and 5% casein to the recipe of oatmeal bread affected the redistribution of pores by radius, increasing the total volume of larger pores with a radius within $(50–55)\times10^{-10}$ m compared to other samples.

The hysteresis loops (the area between the adsorption curve going from zero to the top and the desorption going from top to zero) in the samples are almost the same in shape, indicating similar structural data.

The radius distribution curve shows that the sample type "100% oatmeal, 5% casein, 1% improver" has the highest peak, is it has the largest large pores with a diameter of 64,8 angstroms. The smallest peak in a sample of the type "100% oatmeal", it has few large pores, so it has the smallest pore volume.

All samples have almost a similar adsorption structure, because their adsorption curves coincide in shape. Although in the sample type "100% oatmeal, 5% casein, 1% improver" the curve is the largest in the volume of the hysteresis loop, and in the sample type "100% oatmeal" – the smallest.

From the graph of pore distribution by radii you can see clearly and calculate the number of pores. This is defined as follows: the perpendicular lowered from the end of the rounding to the abscissa on each side and the area under the perpendiculars gives the number of pores.

All samples have excellent convex hysteresis, but then the desorption line does not fall on the adsorption line completely and does not end at zero, because the samples have chemisorption, which determines their residual removal of sorbate.

Conclusions

1. For all studied samples there was a significant effect of temperature on the equilibrium sorption of moisture in the range of studied temperatures.
2. Sorption isotherms of the tested bread samples had an S-shape, this type is inherent in food systems.
3. Research and analysis of sorption properties confirmed our theory of improving the viscoplastic characteristics of oatmeal in bread production technology by including glucan-delta-lactone (1%) and casein (5%) in the prescription components.
4. The analysis of obtained results to determine the effect of the studied raw material on the change in the specific volume of oatmeal during fermentation shows that the joint application of casein and glucano-delta-lactone has a synergistic effect on the gas holding capacity of dough semi-finished products high-volume yield.

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Influence of water activity on the properties of wheat flour

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Abstract

Introduction. The purpose of the research is to determine the influence of water activity indicator on the quality of wheat flour and modified wheat flour.

Research methodology. Samples of wheat flour obtained from wheat grain of different starch polymer composition and component composition were using. The water activity index for wheat flour and modified according to ISO 18787:2017 was determined. In the bread recipe, 10% of flour was replaced by modified wheat flour. Determined the effect of improved formulation on the shelf life of loaves.

Research results. The highest value of enthalpy and water activity is for flour, which has a base ratio of amylose and amylopectin. Softening of the structure and weakening of the starch-protein matrix bonds reduces the enthalpy by 2.3 J/g and the activity of water. The lowest value of enthalpy and water activity has flour, which contains only amylopectin in the starch granules. It should be noted, that the increased composition of micro- and macronutrients flour leads to a decrease in enthalpy of 3.7 J/g. Studies have shown that the physical modification of wheat flour leads to a change in water activity of the samples. In particular, is reduced from 0.619 to 0.591. At the same time, the change in the structure of starch granules leads to an increase in water activity for flour samples: confectionery – from 0.477 to 0.585, type "waxy" – from 0.542 to 0.570, enriched with micro- and macronutrients – from 0.491 to 0.597.

The use of ingredients with a reduced rate of aw shows an inhibitory ability to develop potato disease and micromycetes, which prolongs the storage of bread loaves for 1–2 days. The change in the component composition of the flour after its modification significantly affected its technological quality. The use of such a product in the production of food products helps to extend their shelf life, and, accordingly, for a long time to promote the preservation of organoleptic properties and to show stability to the action of microorganisms.

Samples of flour differ in the composition of carbohydrates ($p < 0{,}001$), proteins ($p < 0{,}01$), fats ($p < 0{,}05$). Therefore, with temperature modification there are structural changes of the main components with the formation of simpler ones.

Conclusions. The water activity index for native wheat flour is in the range of 0.491–0.619 and for modified wheat flour in the range of 0.570–0.597.

Introduction

The ingredients, such as wheat fiber, modified wheat flour, etc. are being introducing into the recipe of bakery and flour confectionery products to improve the quality and extend the shelf life (Venturi C et. al., 2016; Birch AN et. al., 2013). Modified flour is produce in a sufficiently full volume and can meet the needs of the food industry (Abbas K.A., et. al., 2010). At the same time, the influence of modified flour on the quality of bakery products remains unexplored; one of the criteria influencing the quality and storage of products is the indicator of water activity. In our opinion, the method of modification of starch in raw materials also has an effect on the change of water activity index (Rodel W., 2001).

In this regard, one of the key issues is the indicator that affects the structural properties of the product, shelf life and depends on the nature and amount of components soluble in the aqueous phase of the product (Scott WJ et. Al., 1957), as well as methods of processing raw materials, semi-finished products, etc. (Rodel W., 2001). Accordingly, the value of the indicator "water activity" (aw) is crucial, on which depends not only the forming indicator of the product but also the development of microorganisms (Rodel W., 2001). Since the gradient of values water activity of the product and relative humidity is the driving force of mass moisture exchange in various thermal and hydrothermal processes (Kataoka, Y. et. al., 2011), as well as storage (Chervenka L. et al., 2006), data on water activity of treated products are relevant to justify optimal processes (Kataoka, Y. et. al., 2011). The change in the indicator affects the possibility and intensity of development of microorganisms in bakery products (Sereno A. M., 2001).

Water activity (Červenka L. et al., 2006) was determined in such food products as: fruits (0.97), eggs (0.97), flour (0.80), jam (0.82–0.94) meat products. Foods such as milk powder, crackers and instant products have water activity (a_w) values in the range of 0.35–0.5, which usually show a property such as the fragility of the structure (Sereno A. M., 2001). Affects a_w on the development of microorganisms in food (Červenka L. et al., 2006; Schmidt S.J., 2004) and defined the following limits for: bacteria a_w = 0.75–0.98; yeast a_w = 0.62–0.90; micromycetes a_w = 0.60–0.88. Thus, the indicator of water activity affects the quality and storage of food. Thus, it is necessary to study the replacement of part of wheat flour in the recipe for the production of loaves of bread with modified flour, respectively, to study the change in the shelf life of the obtained loaves.

The so-called barrier technologies (Leistner L. and Gorris, L.G.M., 1995) have been developing for the production of a number of products aimed at ensuring the safety and quality of products with extended shelf life. Given that the water activity index was determined for a number of food products, it is important to determine this indicator for modified wheat flour, which is already produce by some starch companies (Chervenka L. et al., 2006), but little is studied.

The purpose of the research is to determine the influence of water activity indicator on the quality of wheat flour and modified wheat flour.

Materials and methods

Materials

The studies used wheat flour, which had a different structural composition of starch:

- **Sample 1** – flour with a ratio of starch granules of amylose: amylopectin as 30:70;
- **Sample 2** – wheat flour from soft confectionery wheat has a low protein content (10.5–11.8%), easily digestible, starchy flour and by weight 20–25% lighter than baking wheat flour, which provides looseness flour confectionery. Flour obtained from such grain does not lose its whiteness when the yield is above 70%, has a low water absorption capacity (54-55%) (Rybalka OI and Axelrud DV, 2004);
- **Sample 3** – wheat flour from soft wheat type "waxy" white color and has an extra high water absorption capacity (72% and above) (Rybalka OI et. al., 2005);
- **Sample 4** – wheat flour from soft wheat with an amylose: amylopectin ratio of 30:70 and enriched with micro- and macroelements. Flour obtained from such grain has a grayish-white color, black or dark brown bran, and has excellent food flavoring properties of cereals (Rybalka OI, 2008).

The samples used in the study had the following physicochemical parameters, which are shown in Table 1 (Kuznietsova I.V. et al., 2020).

Table 1

Physic-chemical parameters of samples of wheat flour (n=3, p≤0.05)

Indicator	Wheat flour			
	Sample 1	Sample 2	Sample 3	Sample 4
Content of hygroscopic water,%	10,54	13,78	9,52	10,19
Starch content,%	68,42	65,80	61,90	56,01
Ash content,% (550 °C)	1,78	1,86	1,93	1,94
Protein content,%	11,60	10,66	9,96	11,84
Fat content, %	1,9	1,4	2,0	2,1
Fiber content,%	6,5	6,2	6,8	7,8

The control sample for modified samples of wheat flour is obtained in industrial conditions extruded wheat flour (Table 2) (Kuznietsova I.V. et al., 2020).

Table 2

Physic-chemical parameters of the control sample (n = 3, p≤0.05)

Indicator	Extruded flour (control)
Content of hygroscopic water,%	9,13
Starch content,%	64,91
Ash content,% (550 °C)	1,80
Protein content,%	11,68
Fat content, %	2,1
Fiber content,%	6,7
Fiber content,%	

Preparation of prototypes

We have improved the method of obtaining physically modified flour. Which is the use of convective drying:

Modifications (Khomichak L.M. et al., 2019) of wheat flour samples with different structural composition of starch were performed. Modification of wheat flour samples was carried out by preparing them by brewing, followed by drying in a convective dryer at a temperature of 110–120 ºC for 30–45 min and at a temperature up to 60–65 ºC to obtain the product – flour modified with a dry matter content of 6–10%, grinding and sifting (Khomichak L.M. and others, 2020).

The obtained samples of modified wheat flour with different starch composition were used to measure the water activity index.

According to the physical modification of the flour will be obtained appropriate samples of the modified flour:

With sample 1M – modified flour obtained from sample 1;
With sample 2M – modified flour obtained from sample 2;
With sample 3M – modified flour obtained from sample 3;
With sample 4M – modified flour obtained from sample 4.

Description of methods and installation

Determination of water activity of wheat flour samples and after their physical modification (Schmidt S.J., 2004).

The water activity index was determined according to the method described in ISO 18787:2017 for four samples of wheat flour and four samples of modified wheat flour, a sample of extruded wheat flour (control for samples of modified flour).

Measurement of thermodynamic parameters of all flour samples was carried out on the device Hygrolab-2 (Rotronic, Switzerland) at a temperature of 18–20 °C with a measurement accuracy of 1.5%, a_w + 1.5% of the value.

Determination of the effect replacing part of wheat flour with modified in the recipe on the shelf life of loaves of bread

Determination of the effect of aw on the shelf life of the product using a modified sample 1 of flour was carried out by obtaining samples of loaves: on sourdough with wheat flour; on leaven with replacement of 10% of extruded flour; on leaven with replacement by 10% of the 1M sample. The obtained samples of loaves were stored for 12 days.

Determination of structural and mechanical properties, crumbliness and microbiological spoilage during storage of bread loaves.

Investigations of the structural and mechanical properties of breadcrumbs were performing on an automated penetrometer. The method consists in measuring the amount of immersion (penetration) of a body of a certain shape and size under the influence of a certain load for a certain time.

Determination of breadcrumbs is an indicator that characterizes the freshness of bread or the degree of its hardening. From the crumb cut two pieces in the shape of a parallelepiped of 5 g each and transfer to a conical flask with a volume of 250 cm^3. Shake the flask for 5

minutes. The crumb formed as a result of friction of two pieces is collected and weighed on scales with an accuracy of 0.01 kg.

To determine the effect of yeasts on wine yeast and flour types on the microbiological stability of loaf samples were studied as follows. Products manufactured by intensive technology (Pico J., et. al. 2015; Torrieri E., et. al. 2014) were placed at a temperature of 35-40 ºC in provocative conditions to observe the appearance of signs of potato disease and the development of micromycetes.

Results and discussion

It was studied the change a_w for wheat flour and after its modification, which will show the effect of physical modification on this indicator and, accordingly, on product quality and allow to predict the shelf life of food obtained with modified wheat flour in the recipe.

Determination of a_w for wheat flour samples

Table 3 presents the indicators. For which the measurement of water activity was performed. It should be noted that the enthalpy values are different for flour samples, which indicates the effect of the ratio of the concentrations of the components, which, accordingly, affects the activation energy of the compounds.

Table 3

Thermodynamic characteristics of soft wheat flour (n = 3, p≤0.05)

№	Indicator	Sample 1	Sample 2	Sample 3	Sample 4
1	Enthalpy, J/g	40,06	37,70	35,63	36,28
2	Specific moisture content, g/kg	8,33	7,36	6,51	6,74
3	Ratio of the concentrations of the components of the mixture, g/kg	8,40	7,41	6,55	6,78
4	Vapor concentration at saturation, g/m^3	15,97	16,12	16,21	16,28
5	Partial water vapor pressure, hPa	13,33	11,77	10,42	10,78
6	Saturated vapor pressure, hPA	21,52	21,70	21,86	21,96
7	Water activity (aw)	0,619	0,542	0,477	0,491

The highest enthalpy is for sample 1, which has a base ratio of amylose and amylopectin. Any change in the structure of starch granules leads to a decrease in enthalpy. In particular, softening the structure and weakening the bonds of the starch-protein matrix (sample 2) reduces the enthalpy by 2.3 J/h. The lowest value of enthalpy is sample 3, which contains only amylopectin in the starch granules. At the same time, the influence of other components of flour should be noted. Thus, the increased composition of micro- and macroelements (sample 4) leads to a decrease in enthalpy by 3.7 J/g accordingly, aw has a similar nature of reduction. This indicates the dependence of this indicator on the energy of the reactions in the flour and on the influence of other components such as: the structure of the starch granule, the influence of micro- and macronutrients.

Determination of aw for samples of modified wheat flour

As studies have shown (Table 4), the physical modification of wheat flour leads to a decrease in aw in the samples.

Table 4

Thermodynamic characteristics of modified wheat flour (n = 3, p≤0.05)

№	Indicator	Control	Sample 1M	Sample 2M	Sample 3M	Sample 4M
1	Enthalpy, J/g	43,89	45,26	44,57	45,20	45,12
2	Specific moisture content, g/kg	8,72	9,33	9,04	9,28	9,34
3	Ratio of the concentrations of the components of the mixture, g/kg	8,80	9,42	9,12	9,37	9,43
4	Vapor concentration at saturation, g/m^3	18,78	18,57	18,64	18,64	18,40
5	Partial water vapor pressure, hPa	13,95	14,91	14,45	14,83	14,92
6	Saturated vapor pressure, hPA	25,54	25,24	25,34	25,34	24,99
7	Water activity (aw)	0,546	0,591	0,570	0,585	0,597

In particular, for sample 1 is reduced from 0.619 to 0.591. At the same time, the change in the structure of the starch granule leads to an increase in the water activity index for flour samples: sample 2 from 0.477 to 0.585, sample 3 from 0.542 to 0.570, sample 4 from 0.491 to 0.597. It should be noted that the value of aw is 0.546, which indicates an in-depth thermal modification due to extrusion. Thus, the structure of the starch granule affects the value of water activity.

The water activity of the product determines its ability to evaporate from the product relative to the ability to evaporate pure water at the same temperature (Tsukanov MF and Chernomorets AB, 2010). Accordingly, the change in the aw between the native flour and the modified is influenced by the structure of the starch-protein matrix, in which due to ionic bonds, hydrophobic and hydrogen bonds and Van der Waals forces, the interaction of product moisture with compounds such as starch and protein (Miyazaki MR et.al., 2006).

The value of the water activity index for samples of modified wheat flour is in the range above 0.5, which indicates low fragility (Hazelton JL et. Al., 2003) and the effect of fat (Červenka L. et al., 2006) on this indicator, which practically does not bind water (Rodel W. et. al., 2001). At the same time, the change in the values of water activity indicators for native flour samples and their modifications indicates the influence of the quantitative content of protein and sugars and their qualitative composition (Tanaka M. et. Al., 2011).

The value of ethalpy for all samples of modified flour increases, on J/g: sample 1 – 5.2; sample 2 – 6.87 sample 3 – 9.57; sample 4 – 5.84. Therefore, the energy increases to change the structure of the compounds under the action of temperature.

In addition to influencing chemical reactions and the development of microorganisms, water activity affects the texture of products (Schmidt S.J. et. al., 2004). At the maximum value of the water activity index allowed in dry products and without loss of the specified properties, the indicator varies in the range of 0.35 – 0.5, and for softened products, which should not have brittle properties, the indicator should exceed 0.5 (Chervenka L. et al., 2006).

Correlation coefficients of a_w for wheat flour

According to the obtained results (Tables 3 and 4), correlation coefficients were obtained (Table 5).

Table 5

Statistical parameters of the regression dependence of the water activity index on the component composition of wheat flour (n = 3, p≤0.05)

Indicator	Correlation coefficients			Determination coefficient R^2
	A*	B**	C***	
Native	0,0227	0,1586	0,7583	0,9819
Modified	0,0083	0,0380	0,6188	0,8112

p <0,001*, p <0,01**, p <0,05***.

With a constant value of aw and an increase in the processing temperature of the flour, the amount of adsorbed water increases. Samples of flour differ in the composition of carbohydrates (p <0.001), proteins (p <0.01), fats (p <0.05). Therefore, with temperature modification there are structural changes of the main components with the formation of simpler ones.

Influence of aw of modified flour samples on suitability for storage

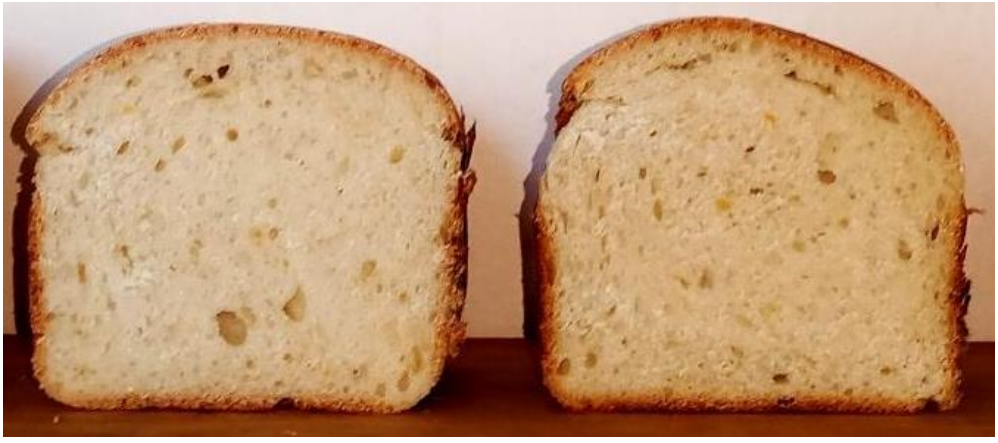
The influence of the use of sample 1M in the recipe of bread by intensive technology on the shelf life of the loaf was studying. The main problem of accelerated technology is the rapid hardening of loaves, which begins to be observed after cooling. This is manifeste in increased tidiness, reduced elasticity of the crumb and the ability to restore shape after pressing. Traditionally, the method of extending the shelf life of products, based on which the reduction of moisture content is achieve by concentration or dehydration (Sereno A. M. et al., 2001). The range of moisture-binding additives is constantly expanding, offering compounds or ingredients such as (Rodel W. et al., 2011): salts, polysaccharides, amino acids, proteins, polyhydric alcohols and more. It is known (Tsukanov M.F. and Chernomorets A.B., 2010), substances such as sugar and salt reduce the a_w in the product. In particular, in a saturated solution of sugar at 20 °C, the activity of water is 0.864, and table salt – 0.753 (Červenka L. et al., 2006). The influence of the method of processing raw materials and semi-finished products on the value of the moisture activity index, is known (Sereno A. M. et al., 2001). In this direction, the modification of flour plays a role as a moisture-retaining component in the food product.

At a value of a_w = 0.6–0.8, which corresponds to a moisture content of the product up to 40% (Hazelton JL et al., 2003) for a long time there are no processes associated with the

deterioration of product quality (Scott WJ et. Al., 1957). Such a_w (0,619) has a sample 1, that usually flour with such a component composition is used for the manufacture of bakery products. Obviously, the presence and amount of hydrophilic substances such as starch and sugars, which modify the flour interact with water in the flour particles and thus form new ionic and hydrogen bonds, which reduces the amount of available moisture in the product (Hazelton JL et al., 2003).

The effect of the use of modified flour in the recipe of bread by intensive technology on the shelf life of the loaf was studied. The main problem of accelerated technologies is the rapid hardening of loaves, which begins to be observed after cooling. This is manifested in increased lidiness, reduced elasticity of the crumb and the ability to restore shape after pressing.

Received bread according to usual recipe and bread in which 10% of flour was replaced by modified flour. The taste of the products is different: when used in this technology, sourdough is allowed in the products, which is noticeable in the control samples. However, with increasing replacement of wheat flour, the sour taste is almost not felt. The taste is typical of wheat bread, in products with the replacement of wheat flour is wetter and more pleasant to the taste. In products with wheat flour replacement, the state of the crumb differs (Figure 1) by a denser thick-walled porosity.



a **b**

Figure 1. Bread crumb:
a – according to the usual recipe;
b – with the replacement of 10% wheat flour with modified wheat flour.

This is increased by 1% humidity of the product compared to the control sample. However, in the case of replacing 30% of extruded flour, the crumb has a slight stickiness.

According to physicochemical parameters (Table 6), it is noted that the products have an underestimated volume, which is due to the complete exclusion of compressed yeast from the recipe.

Table 6

Quality indicators of the obtained loaves

Quality indicators	Control	Replacement 10%
Weight of the dough, g	250	250
Weight of hot bread, g	232	224
Weight of cold bread, g	224	215
Volume, cm^3	750	660
Specific volume, cm^3/g	3,35	3,29
Humidity, %	40	41
Acidity, deg	1,2	1,6
Porosity, %	69	68
Baking, %	7,2	10,2
Drying, %	3,4	4,3

The moisture content of the products increases with the replacement of wheat flour, which is due to the greater water absorption capacity of the modified flour. The brightly colored crust of the products indicates a sufficient accumulation of sugars involved in the reaction of melanoid formation.

Change of deformation of loaves at introduction in a compounding of 10% of flour of the modified (table 7)

Table 7

Evaluation of changes in the quality of bread during storage

Structural and mechanical properties of crumb	Control	replacement 10% flour	
		modified	extruded
General deformation H gen, units ave			
in 4 hours	61	78	48
in 48 hours	33	48	32
Plastic deformation of H pl, units ave.			
in 4 hours	51	58	28
in 48 hours	27	40	21
Elastic deformation of H el.d., units ave.			
in 4 hours	10	23	12
in 48 hours	6	9	9
Covering, %			
in 4 hours	4,9	1,7	0,1
in 48 hours	6,5	2,1	0,5
Wetting,% to DM			
in 4 hours	271	276	164
in 48 hours	227	221	148

The main problem of accelerated technologies is the rapid hardening of finished products (Pico J., et. al., 2015), which begins to be observed immediately after cooling. This is manifested in increased crunchiness, reduced elasticity of the crumb. Therefore, one of the main tasks in the improvement of accelerated technologies is on the one hand to improve the taste and aroma of finished products, and on the other – to improve quality during storage.

It was found that for samples of loaves obtained with the replacement of 10% by modified flour, there are signs of better safety and stability of quality during storage for 48 hours. In the samples with the replacement of flour with extruded flour there was a decrease in the total deformation of the crumb after cooling from 48 to 32 units, with the replacement of the modified flour there was a decrease in the total deformation from 78 to 48 units etc. Crispiness, as one of the main signs of loss of freshness (Torrieri E., et. al., 2014), was the highest in the control, and in samples of loaves with the replacement of modified flour, this figure was lower by 65–80%. Slowing down of hardening of samples made with extruded or modified flour is confirmed by wetting indicators.

Effect of adding 10% of modified flour to the bread recipe for storage of loaves

The obtained samples of bread loaves obtained using sourdough on wheat flour and replacement of wheat flour with modified flour were evaluated. One of the problems with the quality of bread is the frequent cases of microbiological spoilage – mold and potato disease (especially in summer). To determine the effect of yeast on wine yeast and different types of flour on the microbiological stability of the finished products, studies were performed at a temperature of 35–40 °C in provocative conditions to observe the appearance of signs of potato disease and mold.

The mold was observed on day 8 in the bread sample with the replacement of 10% of wheat flour with iodized, in the control sample – on the 9th day of storage. According to the signs of potato disease, it was found in loaves: control sample on the 7th day of storage, with modified flour – on the 9th day of storage. The development of micromycetes was observed in loaf samples made using sourdough on a loaf sample with modified flour on the 12th day of storage and with extruded flour on the 10th day.

The use of ingredients with low aw shows an inhibitory ability to develop potato disease and micromycetes, which prolongs the storage of bread loaves for 1–2 days. The change in the component composition of the flour after its modification significantly affected its technological quality. The use of such a product in the production of food products helps to extend their shelf life, and, accordingly, for a long time to promote the preservation of organoleptic properties and to show stability to the action of microorganisms.

Conclusions

1. The indicator of water activity for wheat flour and its modifications is determined, which is in the range of 0.491–0.619 for native wheat flour and in the range of 0.570–0.597 for modified wheat flour. According to the general classification, modified flour belongs to products with low humidity.
2. The indicator of water activity is influence not only by the content of such compounds as protein, fat, sugar, but also the structure of the starch polymer. Using samples of wheat flour with different structure of starch polymer, there is also a pattern of reducing the water index after modification of the flour.

3. Modified wheat flour for the value of water activity can be used as a moisture-retaining component of flour products, which will increase the shelf life of loaves produced on sourdough by accelerated technology.
4. Correlation coefficients are calculated and a significant impact of the modification process on the quality of the finished product is showing – flour samples have become more stable and suitable for a longer period to maintain organoleptic characteristics.

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Investigation of frying process of meat sausages in glued casings from intestinal raw materials

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Abstract

Introduction. The purpose of the work is to specify the peculiarities of heat and mass transfer processes during the frying of meat sausages in glued shells obtained in different ways from intestinal raw materials.

Materials and methods. Samples of meat sausages in different intestinal casings were studied: pork bellies; glued intestinal sausage casings from pork bellies with local thermal coagulation reinforcement, with local tanning reinforcement, with continuous tanning and plasticization.

Results and discussion. The temperature kinetics of different layers of meat sausages in glued intestinal casings during their frying were studied. The temperature of raw material inside the sample is uniformly distributed in the range from 80 to 90 °C in the investigated frying modes. This eliminates the negative impact of high temperatures on product quality due to the formation of substances of pyrogenetic fat breakdown with an unpleasant taste and odor.

The kinetics of the mass of meat sausages in glued intestinal casings during their frying is studied. The maximum rate of weight loss is obtained for the control sample – sausage in the traditional intestinal shell. The smallest value of the rate of weight loss has a sample which uses a sausage casing glued by continuous tanning followed by plasticization with glycerin. This is due to the lower permeability of the developed shells to the transmission of fat and water vapor in relation to the control sample. Glued intestinal membranes have less permeability in relation to the transmission of fat and vapor of system water than the traditional intestinal membrane, which is a more acceptable functional and technological property.

Thermophysical properties of the studied samples are determined to a greater extent by thermophysical properties of minced meat, the mass fraction of which is much larger compared to the mass fraction of the sausage casing.

Conclusions. Regularities of the course of heat and mass transfer process during frying of sausages in casings from intestinal raw materials are revealed.

Introduction

The main technological peculiarities of fried meat sausages are frying on the surface at a temperature of 140–200 °C (Sukhareva et al., 2021) and use of mostly natural casings (Heinz et al., 2007). This is the cause of significant losses during the technological production process, which is about 40% (Toldrà et al., 2007).

The peculiarities of the process of meat products' frying compared to other methods of heat treatment are that the product is heated not in water or steam, but on the special surface for frying with the addition of vegetable oil or fat at a temperature of 140–200 °C. During the use of this method of heating a specific crust is formed on the surface of the product. The heating of minced meat during frying is characterized by temperature changes at different points and at different time, i.e. the temperature is a function of coordinates and time. It is worth noting that the heat and moisture transfer in this process are determined by the heating regime and the forms of system moisture connection with the material (Zayas, 1981).

On the other side, special attention should be paid to the formation of the protective properties of casings, as their level determines the degree of technological losses and economic efficiency of fried meat sausages production (Sidorova et al., 2011).

From the standpoint of resource conservation, an effective way to rationalize the technological process of preparing fried meat sausages is to use glued casings from intestinal raw materials remaining after obtaining gut products.

Literature analysis indicates the presence of scientific reports on improving the technology of glued casings (Domin et al., 2020), known regularities of heat and mass transfer during meat frying (Skrypnyk, 2015). Quantitative and qualitative indicators of fried sausages technology in modified shells (Shubina et al., 2015) are determined.

In order to increase the strength and reduce the degree of reversibility of the gluing-delamination process during the contact with minced meat containing a significant amount of water, methods of their additional reinforcement using thermal coagulation, tanning and plasticization have been proposed. It is shown that thermal coagulation, tanning and plasticization change physical and mechanical properties of shells (Onishchenko et al., 2021). At the same time, the regularities of heat and mass transfer processes during frying of meat sausages in glued intestinal casings, obtained in different ways, remain uncertain, which is a necessary scientific basis for comparing the efficiency of their use. These data are currently absent in the scientific and practical literature.

Thus, the study of the kinetics of temperature and mass of meat sausages in the intestinal casings glued in different ways during their frying is crucial. Their results will scientifically substantiate the feasibility of using the proposed technical and technological solutions and increase the resource efficiency of the technology.

The aim of the work is to establish the peculiarities of heat and mass transfer processes during the frying of meat sausages in the traditional intestinal shell and in the glued shells obtained in different ways from intestinal raw materials.

To achieve the desired goal, the following tasks were solved:

- To study the temperature kinetics of meat sausages layers in glued intestinal casings during their frying;
- To study the kinetics of meat sausages mass in the glued intestinal casings during their frying;
- To compare the processes of heat and mass transfer and to determine regularities of their passing during the frying of meat sausages in the traditional intestinal shell and in the glued shells obtained in different ways from intestinal raw materials.

Materials and methods

Materials

Samples of meat sausages in different intestinal casings were studied:

control: pork bellies with a diameter of 35–37 mm;

experiment: glued intestinal sausage casings (in one row) with a diameter of 35–37 mm, made of pork bellies:

- With a local reinforcing seam using thermal coagulation;
- With a local reinforcing seam using tannage;
- Reinforced with continuous tannage and plasticized with glycerin.

Separated pork bellies, which were previously cleaned of serous, muscular and mucous membranes, washed, sorted by quality, salted and stored as a salty product were used as outward raw material. The outward raw material was freed from salt, then washed and kept in water. Methods of obtaining glued sausage casings are described in detail in [9].

As a control, intestinal casings – pork belly products used in traditional technologies of making fried sausages – were used. That is, the raw material was the separated pork bellies, which were cleaned in advance from serous, muscle, and mucous membranes, washed, sorted by quality, salted, and stored in the form of salted products. The raw materials were desalted, washed, and aged in water (Hyro et al., 2007).

Preparation of semi-finished meat sausages in glued casings

Meat sausages were prepared by the following recipe (Rohov et al., 1993), kg/100 kg: lean pork semi-fat – 100; table salt – 1,8; white sugar – 0,2; ground black pepper – 0,25; fresh peeled crushed garlic – 1,0. In order to bring the moisture state of the minced meat to modern technological conditions, 30% water was added to the minced meat composition that is mixed. In order to form a monolithic structure of the finished product, 1/2 of the semi-fat skimmed pork was crushed on a meat comminutor with a lattice hole diameter of 14–20 mm, and the other 1/2 with a lattice hole diameter of 3–4 mm. The obtained minced meat was mixed with spices in a mixer, water was added, mixed again and left for pickling for 8–10 h at a temperature of 6–10 °C.

After loose filling and knitting, the sausage loaves were blanched in water at a temperature of 85–90 °C for 5 minutes, then placed on a greased surface and fried at a temperature of 140–150 °C for 20 minutes on each side. Cooling was carried out to a temperature in the loaf thickness of 8ºC.

Samples of sausages, for which the frying process was studied (Božiková et al., 2017), were made using a parallelepiped frame (Figure 1). The framework is made of a steel wire with plastic isolation. The dimensions of the frame $a \times b \times c$, respectively, were 30×30×100 mm. 5 thermocouples were placed between the frame guides at a distance of 7,5 mm from each other, as shown in Figure 1.

Next, a sausage casing, tied on one side, stuffed with minced meat and tied on the other end, was stretched on the frame along the guides.

The test sample was placed on a frying surface preheated to a temperature of 140–150 °C and lubricated with vegetable oil (Naghavi et al., 2018). The sample was placed on the surface for frying with the plane $a \times b$ (Figure 1).

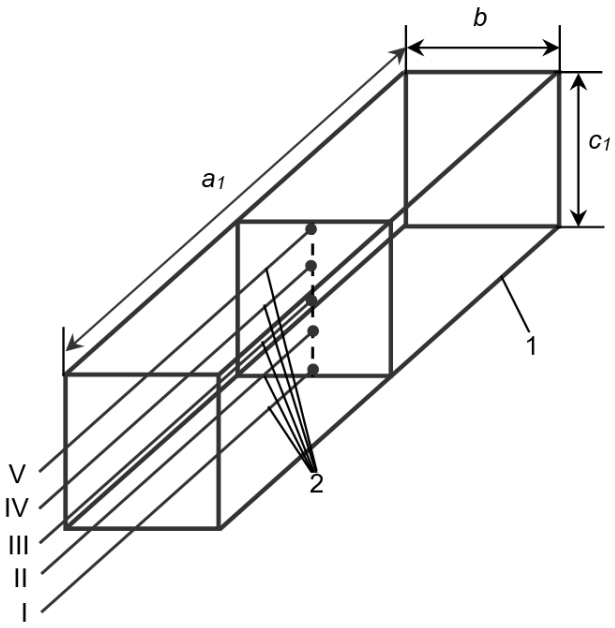

Figure 1. Parallelepiped frame for making samples of sausages used in the study of the frying process, and the layout of thermocouples in them

Methods of research of heat and mass transfer processes during the frying of sausages in glued intestinal casings and in control sample

Thermograms obtained during the frying of the control sample are shown in Figure 2. They are signals from thermocouples placed as shown in Figure 1. Thermocouple number VI is fixed on the surface for frying.

Frying from side 1 was performed until the temperature inside the sample (thermocouple III) reached the value of 70 °C. Duration of frying side 1, as can be seen from Figure 2 (first dotted line), is 20 minutes.

The sample was then inverted and fried for 1, i.e. 20 minutes.

After frying the sample for 40 minutes, heating of the frying surface was stopped (second dotted line). The product was cooled to room temperature. The cooling process to room temperature took 44 minutes. During the frying and cooling of the samples, their current weight and time were recorded.

Extensive evidence of various parameters obtained in the course of research. Their mathematical processing was carried out in order to check the degree of probability of the obtained data, to establish certain relationships between them, to present the results of processing in graphical form. Processing was performed using the Mathcad software package, which contains a wide range of procedures for solving problems of statistical analysis, interpolation, data smoothing, regression and correlation analysis (Snezhkin et al., 2019).

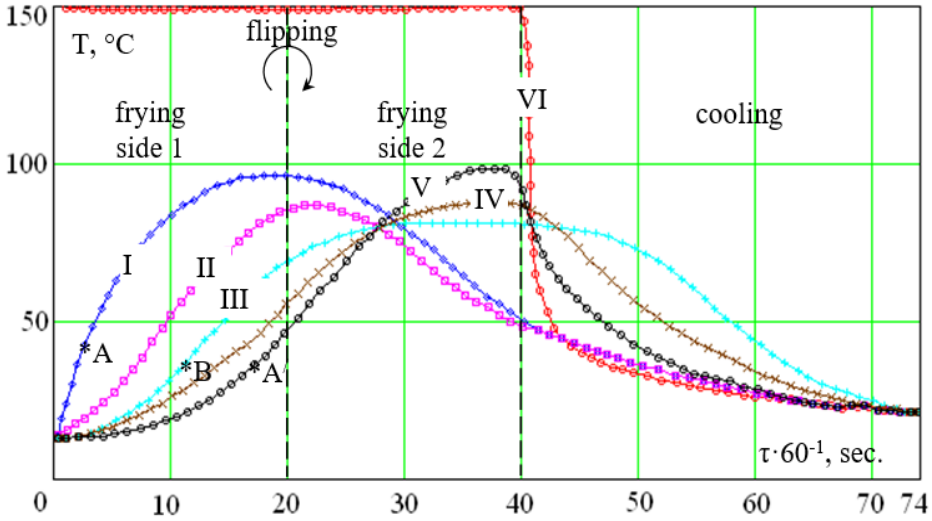

Figure 2. Thermograms obtained during frying and cooling of the control sample: I, II, III, IV, V – thermocouples (from Figure 1); VI – thermocouple mounted on the surface for frying.

Results and discussion

Temperature kinetics of different layers of meat sausages in glued intestinal casings during their frying

At the time of flipping, the temperature at evenly distributed points of thermocouples I, II, III, IV, V is 98 °C, 86 °C, 70 °C, 56 °C and 48 °C, respectively, due to their distance from the frying surface. Flipping and additional frying, on the other hand, mostly gives opposite results concerning peripheral thermocouples. There is a slight increase in temperature (by 2–3 °C), associated with an increase in the duration of thermal contact. The direction and the nature of temperature changes in the cooling phase are close to all thermocouples. The curves are finally smoothed in the interval (70–74)·60 s, with the temperature values of about 20°C.

The nature of thermograms for the samples of meat sausages in glued intestinal casings is similar to the thermograms for the control sample (Figure 2). It is assumed that the analysis of thermograms by characteristic points for the control sample is valid for thermograms for other test samples. The differences are in the position of these characteristic points, which is reflected in the nature of the dependence of the moisture content of the studied samples in the process of frying and cooling.

Obviously, the nature of thermograms is determined by how close a certain layer of the test sample is to the heat source, the function of which is performed by the surface for frying. The most sensitive are the layers with thermocouples: I – before turning the sample on the surface for frying; V – after inversion of the sample on the surface for frying. These layers are heated the fastest, respectively, the first before inversion of the sample, and the second – after. After inversion of the sample, the layer with thermocouple I is cooled, as it is found the furthest from the heat source and borders on the environment. As for the layer with

thermocouple V, its heating is the slowest for the same reasons before inverting the sample. Accordingly, after turning – the layer heats up at the highest speed.

The layer for which the distance from the heat source is constant and which is a kind of «indicator» of the product's readiness, is the central layer of raw materials (thermocouple III). It heats up at an intermediate rate before turning over. After inversion, its temperature rises to 75–80°C and is kept constant due to the heat accumulated by the layers with thermocouples I and II and due to heat supply from the heat source through the layers with thermocouples IV and V.

Let us consider the characteristic points of the obtained thermograms.

As noted above, the most sensitive to changes in surface temperature for frying (thermogram VI) is the surface layer (layers with thermocouples: I – before inversion of the sample on the surface for frying; V – after inversion of the sample on the surface for frying), i.e. the layer in direct contact with heat source. The thermograms of these layers have characteristic inflection points A and A/, which correspond to the beginning of intense evaporation from the surface of the sample. At these points begins intensive loss of the sample mass to be fried.

The thermogram of the central layer (thermocouple III) has an inflection point B. The presence of this point indicates that at a temperature of 35–40 °C internal endothermic processes are completed and more intense heat accumulation begins by the inner layers of the sample.

To analyze the temperature distribution over the volume of the sample, the created temperature fields are shown in Figure 3.

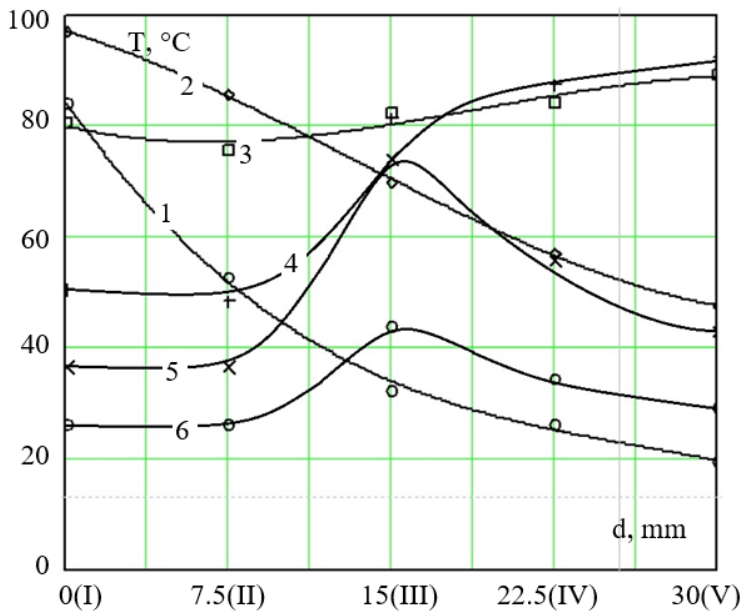

Figure 3. Temperature fields inside the control sample during frying and cooling at different times, min.:
1 – 10; 2 – 20; 3 – 30; 4 – 40; 5 – 50; 6 – 60.

Temperature fields were constructed according to the data obtained from thermocouples placed inside the sample, as shown in Figure 1, at intervals of 10 minutes. The start time and end time (last 14 minutes) are not given, because the temperature values from all thermocouples are close to room temperature at these time points.

Figure 3 shows that the largest temperature gradient occurs in the first 10 minutes (1) because thermocouple I is the closest to the heating surface. As a result, a convective heat transfers to the sample through the shell. Next, the temperature gradient decreases (2) due to the heating of the inner layers of the raw material. After inversion of the sample, the temperature gradient changes its sign, but its value is insignificant. It should be noted that 30 minutes after the beginning of the frying process at a surface temperature for frying 140–150 °C, the temperature of the raw material inside the sample is uniformly distributed in the range from 80 to 90 °C. This, firstly, indicates the readiness of the product (Vujadinović et al., 2014), and, secondly, eliminates negative influence of high temperatures on product quality due to the formation of pyrogenetic cleavage of fat with an unpleasant taste and odor (Grujić et al., 2014).

The differences between the temperature fields during frying for the test samples and for the control sample are within the error. The temperature values between the respective layers of raw materials inside the samples differ by no more than 3– 7%. The mass fraction of sausages, i.e., minced meat, is much higher (more than 98%) compared to the mass fraction of sausage casings (Marcus et al., 2013). Thus, thermophysical properties of the studied samples are determined mostly by thermophysical properties of minced meat, that is the same for all samples (Akpan et al., 2017). The indicated allows to use glued intestinal casings in the technology of making meat sausages without any changes in the relevant technological schemes.

Kinetics of the meat sausages mass in glued intestinal casings during their frying

Kinetics of the mass of the studied samples of sausages during frying and cooling is shown in Figure 4. On the y-axis of the graph the mass of the sample is plotted and normalized to its original value.

These dependences were determined by approximating the obtained experimental data by a polynomial function of the form:

$$m(\tau) = \sum_{n=0}^{P} a_n \cdot \tau^n ,$$

where m is the current relative weight, *kg/kg*; τ – current time, s; P – the degree of the polynomial.

The optimal degree of the polynomial was determined by the minimum ratio

$$\cdot \sigma^2_{\min} = \frac{\sum_{i=1}^{N} \left(m_i - \sum_{n=0}^{P} a_n \cdot \tau^n \right)}{N - P - 1}$$

The values of the approximation coefficients for different samples are given in the Table 1.

Kinetics of the studied samples mass are of the same nature: the mass decreases monotonically during the process of frying and subsequent cooling. Obviously, the decrease in mass is due to the evaporation of system water and the leakage of fat through the used shells (Behailu et al., 2020).

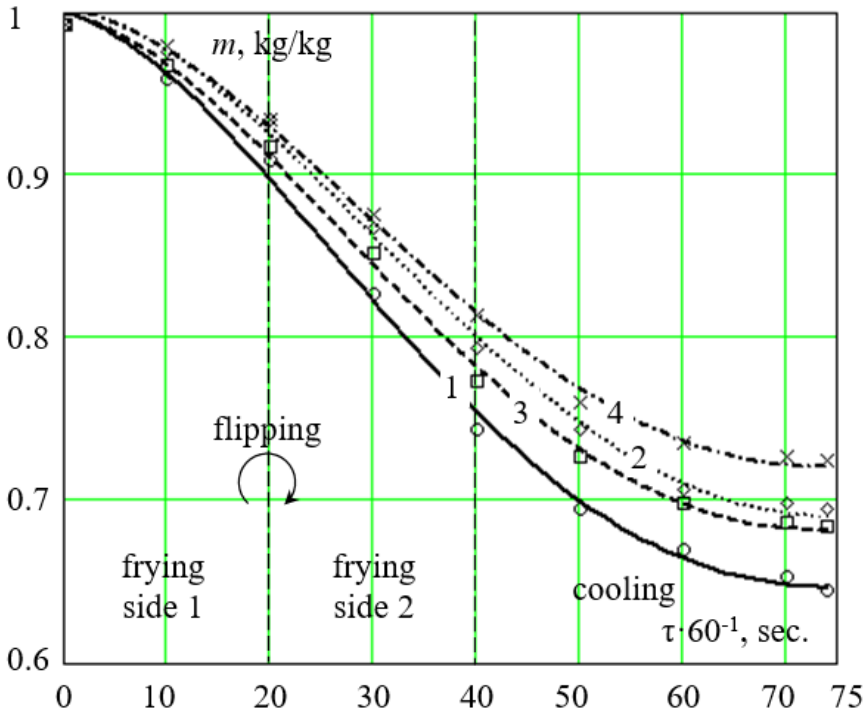

Figure 4. Kinetics of the control sample (1) mass and samples in the shells:
2 – glued sausage casings, reinforced with the use of local thermal coagulation;
3 – glued sausage casings, reinforced with the use of local tannage with a solution of tannin;
4 – sausage casings glued by continuous tannage followed by plasticization with glycerin.

Table 1

Values of approximation coefficients for different samples

Samples in the shells	a_0	a_1, $\times 10^3$	a_2, $\times 10^4$	a_3, $\times 10^6$	a_4, $\times 10^8$
Control sample	1.210	-2.091	-3.020	5.142	-2.344
Glued sausage casings, reinforced with the use of local thermal coagulation	1.213	-1.044	-2.500	3.621	-1.331
Glued sausage casings, reinforced with the use of local tannage with a solution of tannin	1.210	-1.670	-2.261	4.310	-1.838
Sausage casings glued by continuous tannage followed by plasticization with glycerin	1.212	-9.975	-2.381	3.617	-1.429

However, the given kinetics of mass differ in the different angle of inclination to the abscissa axis, on which time is set, and in the different final mass. The angle of inclination to the axis on which time is set, is determined by the rate of weight loss by the samples under research. It is possible to determine kinetics of the rate of mass loss by finding the time derivative of the approximation function. Kinetics of the rate of weight loss obtained in this way for the studied samples are shown in Figure 5.

The nature of these dependences is similar, i.e., there is a monotonic increase in the rate of weight loss, reaching the maximum speed and a monotonic decrease in this characteristic. Obviously, due to the heating of the sample, the evaporation rate of system water increases, respectively, reaching the maximum value at the investigated temperatures. The same concerns the leakage of fat, which melts at temperatures above 39–42 °C (Kameník et al., 2018).

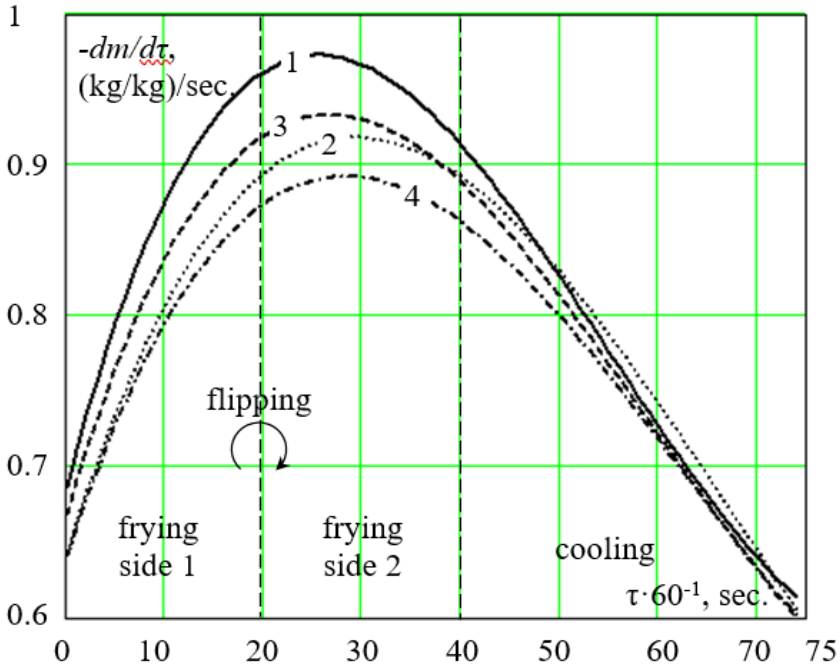

Figure 5. Kinetics of the rate of weight loss of the control sample (1) and samples in the shells: 2 – glued sausage shells, reinforced with the use of local thermal coagulation; 3 – glued sausage casings, reinforced with the use of local tannage with a solution of tannin; 4 – sausage casings glued by continuous tannage followed by plasticization with glycerin.

Due to the reduction in the amount of system water that can evaporate at the temperatures under study and the amount of fat that leaks out, the rate begins to decrease in the process of frying (Hwang et al., 2020). And during cooling, the rate of weight loss tends to zero, due to a decrease in the intensity of these processes (meaning the evaporation of system water, melting and leakage of fat) and their gradual cessation (Huebner-Keese et al., 2016).

It should be noted that the maximum rate of weight loss was obtained for the control sample (curve 1 in Figure 5). For samples of glued reinforced sausage casings, it has an intermediate value among the studied samples (Mellema et al., 2003). The smallest value of the rate of weight loss has a sample that uses a sausage casing glued by continuous tanning followed by plasticization with glycerin.

This happens due to lower permeability of the developed shells to the transmission of fat and water vapor in relation to the control sample. It should be assumed that this is the reason for different final mass of the samples under research.

Thus, the study proves that the developed shells have less permeability to the transmission of fat and vapor of systemic water compared to the traditional intestinal membrane, which is a more acceptable functional and technological property.

Conclusions

Based on the analysis and comparison of the obtained results, the regularities of heat and mass transfer processes during frying of meat sausages in the traditional intestinal shell (pork belly) and in glued shells obtained in different ways from intestinal raw materials were specified.

1. Research of the temperature kinetics of different layers of meat sausages in glued intestinal casings during their frying showed that the temperature of raw materials inside the sample for the studied frying modes is uniformly distributed in the range from 80 °C to 90 °C. It is noted that this eliminates the negative impact of high temperatures on product quality due to the formation of substances of pyrogenetic breakdown of fat with an unpleasant taste and odor.
2. Research of the kinetics of meat sausages mass in the glued intestinal casings during their frying showed that the maximum rate of weight loss was obtained for the control sample – sausage in the traditional intestinal shell (pork belly). It was found that the lowest value of the rate of weight loss has a sample that uses sausage casing glued by continuous tannage followed by plasticization with glycerol, due to lower permeability relatively to the control sample of the developed casings to the transmission of fat and water vapor. The weight loss in the glued intestinal membranes was reduced by 6–11% compared to the control sample made in pork bellies.
3. It is proved that thermophysical properties of the studied samples are determined to a greater extent by thermophysical properties of minced meat, the mass fraction of which is much larger (exceeds 98%) compared to the mass fraction of the sausage casing. It is proved that thermophysical properties of sausages in the intestinal membrane differ from sausages in glued intestinal casings by no more than 3–7%, i.e., within the error. It was found that glued intestinal membranes have less permeability to the transmission of fat and steam of systemic water in relation to the traditional intestinal membrane, which is a more acceptable functional and technological property.

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Анотації

Харчові технології

Вплив температури зберігання на текстуру сиру «Кашкавал»

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Вступ. Мета цієї статті – дослідити вплив температури зберігання на параметри текстури сиру з коров'ячого молока «Кашкавал».

Матеріали і методи. Зразки сиру «Кашкавал» готували за класичною технологією і зберігали при різних температурах (4,0±1,0 °C; 1,0±1,0 °C; 7,5±0,5 °C та -18,0±1,0 °C). Аналіз текстури проводили аналізатором StableMicroSystems TA-XT2i, оснащеним завантажувальним елементом 50 кг.

Результати і обговорення. Спостерігалася значну різниця у значеннях показника твердості між зразками сиру, що зберігаються в охолодженому стані, та тими, що зберігаються в переохолодженому і замороженому станах. Зі збільшенням температури зберігання сиру спостерігалася тенденція до зниження ($p < 0,05$) значень показника згортання. Температура зберігання мала вирішальний вплив на зміни пружності сиру. Більш високі температури зберігання (4,0±1,0 ° C) супроводжувались значним зниженням пружності сиру. Зберігання сиру «Кашкавал» в охолодженому стані супроводжувалось значним збільшенням ($p < 0,05$) його адгезивності. Ця тенденція посилювалася з підвищенням температури зберігання. Зі збільшенням температури зберігання сиру спостерігалося більш значне зниження ($p < 0,05$) значень індексу клейкості. Оскільки всі три показники знижувались у процесі зберігання в умовах охолодження, це суттєво вплинуло на клейкість.

Висновок. Більш інтенсивні зміни текстури досліджуваних зразків сиру «Кашкавал» спостерігалися з підвищенням температури зберігання.

Ключові слова: *сир, кашкавал, зберігання, сир-паста, текстура.*

Харчова цінність білка в пшенично-житньому хлібі, виготовленому з додаванням борошна з низькоалкалоїдних сортів люпину

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Вступ. Мета дослідження – оцінити харчову цінність білка в пшенично-житньому хлібі, виготовленому з додаванням борошна з низькоалкалоїдних сортів люпину.

Матеріал і методи. Для випікання пшенично-житнього хліба використовували борошно із сортів жовтого люпину Юнона, Поло, Легат і Маркіз. У хлібі визначали: вміст білка, засвоюваність білка, амінокислотний склад, ефективний вміст білка (EP),

хімічний показник (CS), індекс незамінних амінокислот (EAAI), справжню засвоюваність білка (TD), засвоюваність білка, скоригований показник амінокислот (PDCAAS) та коефіцієнт ефективності білка (PER).

Результати і обговорення. Спостерігається тенденція збагачувати зернові продукти, що виготовляються з пшениці та пшенично-житніх сумішей, борошном, отриманим з інших рослин, таких як люпин, зелений горошок, квасоля, конопля та гречка. Цінність такого борошна підтверджується функціональними властивостями, зокрема розчинністю, емульгувальними, піноутворювальними та желювальними властивостями, здатністю утримувати воду. Додавання люпинового борошна та білкових ізолятів з насіння люпину не впливає на смак кінцевого продукту. Додавання люпинового борошна призвело до підвищення вмісту загального білка і перетравного білка в хлібі. Найбільший приріст EP, CS, PDCAAS та EAAI спостерігався під час збагачення хліба борошном сорту Поло. Суттєвих змін у PER не зафіксовано. Білок у продуктах з люпиновим борошном містить більше лейцину, лізину, аспарагіну й аргініну порівняно з контрольним зразком. Додавання люпинового борошна до пшеничного хліба значно покращує якість і кількість білка та харчових волокон у кінцевому продукті. Високі значення індексів PDCAAS та PER дають змогу порівнювати білки люпину і білки бобових рослин з білками тваринного походження.

Висновки. Білок низькоалкалоїдних сортів люпину підвищує харчову цінність хлібного білка, тому його слід рекомендувати для виробництва хліба.

Ключові слова: *хліб, люпин, білок, засвоюваність*

Обґрунтування раціонального способу очищення соку сорго цукрового в технології отримання харчового сиропу

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Вступ. Метою дослідженя є встановлення закономірностей видалення високомолекулярних і барвних сполук при різних способах очищення соку сорго цукрового для отримання харчового сиропу.

Матеріали і методи. Для отримання харчового сиропу використовували гібрид сорго цукрового Мамонт. Нативний сік сорго підлягав ферментативному обробленню з метою гідролізу крохмалю. Для вилучення із соку сорго цукрового розчинних нецукрів, зокрема, високомолекулярних сполук (ВМС) і барвних речовин застосовували катіонний флокулянт полігексаметиленгуанідин гідрохлорид (ПГМГ ГХ) та природний мінеральний сорбент – цеоліт-клиноптилоліт. Для інтенсифікації технологічного процесу очищення застосовували мембранні методи фільтрування та іонообмінне очищення.

Результати і обговорення. Завдяки використання цеоліту за оптимальних витрат 0.8–1,0% до маси соку досягається ефект знебарвлення на рівні 41–46%, а ефект видалення ВМС – 20–22%.

Використання цеоліту в очищенні соку сорго у поєднанні з мембранними методами фільтрування, такими як механічне фільтрування і ультрафільтрування, призводить до покращення технологічних показників соку сорго. За цих умов очищення отримано сік сорго цукрового із чистотою 90.72% та забарвленням 245.8 од. ICUMSA, а ефект очищення, видалення ВМС та білкових речовин склав 46.1, 82.3 і 69.5%, відповідно. За умов доповнення способу іонітним очищенням отримаємо

підвищення ефекту очищення, видалення ВМС та білків відповідно до величин 51,9, 98.5 і 89.2%.

Запропоновані способи очищення соку сорго цукрового є ефективними щодо вилучення ВМС, білків та барвних сполук і забезпечують отримання харчових сиропів, у яких збалансовано оптимальне співвідношення вуглеводів сахарози та глюкози і фруктози (65:35)% до маси загальної кількості цукрів.

Висновки. Найкращі показники якості мали сиропи, отримані з використанням адсорбційного очищення цеолітом, мембранного фільтрування та іонітного очищення.

Ключові слова: *сорго цукрове, сироп, флокулянт, цеоліт, ультрафільтрація.*

Ферментативний гідроліз лактози в концентратах відновленої демінералізованої сироватки, призначених для виробництва морозива

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Вступ. Доведено доцільність застосування ферментованих концентратів відновленої демінералізованої підсирної сироватки як джерела сироваткових білків і моноцукрів у складі морозива.

Матеріали і методи. Досліджено фізико-хімічні показники відновлених ферментованих і неферментованих концентратів демінералізованої підсирної сироватки з вмістом сухих речовин від 10 до 40%. Вміст лактози визначали прискореним колориметричним методом, активність води – на аналізаторі активності води.

Результати і обговорення. Раціональні режими ферментативного гідролізу лактози в концентратах відновленої демінералізованої підсирної сироватки з вмістом 10-40% сухих речовин: за температури 40–43 °С і pH 6,1–6,6 рекомендована доза рідкого ферментного препарату GODO-YNL2, одержаного з дріжджів *Kluyveromyces lactis*, для концентратів з вмістом лактози 7,7–30,8% – в діапазоні від 0,1 до 0,4 %. Тривалість ферментативного гідролізу за вказаних умов упродовж 4±2 год забезпечує ступінь гідролізу лактози не нижче 70%. З метою підвищення ступеня гідролізу лактози одночасно з ферментним препаратом у визначених кількостях застосовано одноштамову ліофілізовану пробіотичну культуру «*L. acidophilus LYO 50 DCU-S*». Впродовж перших 4 год сквашування активна кислотність зразків сироваткових концентратів досягає значень, не нижчих pH=5,7–5,9. За вказаної кислотності ферментний препарат GODO-YNL2 проявляє достатню активність, а присутність продуктів гідролізу лактози незначно стимулює розвиток *L. Acidophilus.* За рахунок спільної гідролізуючої дії ферментного та заквашувального препаратів впродовж 6–8 год можна досягти ступеня гідролізу лактози 80–85%. Перспективою подальших досліджень є розробка науково-обґрунтованих рецептур морозива на основі гідролізованих концентратів відновленої ферментованої сироватки. Морозиво, збагачене сироватковими білками та пробіотичною культурою *L. acidophilus*, також буде відрізнятися зниженим вмістом дицукрів – цукрози та лактози.

Висновки. Доведено можливість підвищення ефективності ферментолізу лактози в концентратах відновленої демінералізованої підсирної сироватки до 80–85% за спільної специфічної дії ферментного препарату GODO-YNL2 і закваски на основі *L.*

Acidophilus. Продукт ферментолізу являє технологічний інтерес як багатофункціональний інгредієнт у складі морозива.

Ключові слова: *сироватка, лактоза, ферментоліз, β-галактозида, Lactobacillus acidophilus, морозиво.*

Антимікробні властивості олії з насіння інжиру як компоненту плівок на основі хітозану

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Вступ. Мета дослідження – обґрунтування синергетичного збільшення протимікробної здатності полімеру хітозану, застосовуючи за різних умов олію з насіння інжиру, олію з ядер сливи та абрикосової кісточки.

Матеріали і методи. Протимікробну дію олій проти певних видів бактерій оцінювали з / без хітозану за допомогою агарового диска / лунки та спектрофотометричного вимірювання. Виготовляли тонкі плівки хітозану, збагачені оліями, для перевірки як протигрибкових, так і антибактеріальних ефектів, а також у повсякденному застосуванні.

Результати і обговорення. Незважаючи на те, що ми не змогли досягти значного ефекту в умовах культури, олія з насіння інжиру окремо або в поєднанні з олією кісточок абрикоса і сливи змогла покращити властивості проти псування плівки хітозану. Хоча скибочки свіжого лимона і банана, обгорнуті лише звичайною харчовою плівкою, демонстрували повне погіршення стану, плівка хітозану могла суттєво припинити псування. Для цих продуктів, особливо упакованих плівкою хітозану, збагаченою олією з насіння інжиру, спостерігався майже повний захист від мікробного псування. Проведено моделювання для оцінки передбачуваних взаємодій між олійними сполуками та хітозаном. Ми припустили, що найбільш потенційною сполукою у всіх олійних екстрактах є бензальдегід. За допомогою Н-зв'язку було визначено взаємозв'язок між функціональними групами молекули хітозану та бензальдегіду шляхом обчислювального аналізу. Це може бути одним із можливих факторів, що спостерігається в олії з насіння інжиру, а також в інших екстрактах олії, до ефекту псування пакувальної плівки на основі хітозану.

Висновки. Додавання олії з насіння інжиру окремо або в поєднанні з різними екстрактами в харчові упаковки є перспективним з точки зору продовження терміну придатності харчових продуктів на декілька днів.

Ключові слова: *антимікробний, плівка, упаковка, насіння інжиру, олія.*

Антиоксидантна характеристика нетрадиційної пряно-ароматичної овочевої сировини для технології ресторанного господарства

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Вступ. Метою дослідження є визначення антиоксидантної здатності нетрадиційної для технології ресторанного господарства пряно-ароматичної рослинної сировини.

Матеріали і методи. Антиоксидантну здатність пряно-ароматичної рослинної сировини: гісопу лікарського (*Hyssopus officinalis L.),* змієголовнику молдавського (*Dracocephalum moldavica L.*), лофанту анісового (*Agastache foeniculum L.*), меліси лимонної (*Melissa officinalis L.*), васильків справжніх (*Ocimum basilicum L.*), фенхелю овочевого (*Foeniculum vulgare Mill.*), хризантеми овочевої (*Glebionis coronaria L.*), чаберу садового (*Satureja hortensis L.*), визначали за методом редоксметрії та *pH*-метрії водно-спиртових настоїв; сенсорні показники – за експертним методом; результати математико-статистичної обробки – за методом лінійної кореляції Пірсона.

Результати і обговорення. Водневий показник для водно-спиртових настоїв з пряно-ароматичної сировини має значення від 5,28 од. *pH* (*Hyssopus officinalis L.*) до 6,69 од. pH (*Agastache foeniculum L.*).

Отримано мінімальне теоретичне значення окисно-відновного потенціалу (*RP*) для рослинних водно-спиртових настоїв, яке змінюється від 258,6 мВ (*Agastache foeniculum L.*) до 343,2 мВ (*Hyssopus officinalis L.*). Актуальне значення *RP* настоїв становило від 93 мВ (*Hyssopus officinalis L.*) до 148 мВ (*Glebionis coronaria L.*).

Водно-спиртові настої з рослинної сировини та об'ємною часткою етанолу 40% мають величину енергії відновлення (*RE*) в межах від 120,6 мВ (*Agastache foeniculum L.*) до 250,2 мВ (*Hyssopus officinalis L.*).

Водно-спиртові настої з пряно-ароматичної сировини мають значення сенсорних показників (*S.e.*) від 9,50 до 9,68 бала. Найбільше значення *S.e.* – 9.68 бала, характерне для *Melissa officinalis L.*: колір – світло-коричневий; смак – помірно пекучий, трав'янистий; аромат – трав'янистий, лимонний.

Перспективним є використання пряно-ароматичної рослинної сировини для технології ресторанного господарства. Дослідження підтвердили біологічну цінність пряно-ароматичних трав для збагачення чайно-трав'яних композицій, соусів білого та червоного основного, компотів і поліпшення органолептичних показників.

Висновки. Для технології ресторанного господарства запропоновано застосування пряно-ароматичної рослинної сировини з *Hyssopus officinalis L.* та *Melissa officinalis L.*, яка отримала підвищені антиоксидантні характеристики *RE* – 250,2 мВ та *EB* – 184,6 мВ відповідно, та позитивні сенсорні показники *S.e.* – 9,53 та *S.e.* – 9,68 бала.

Ключові слова: *пряно-ароматичний, рослинна сировина, антиоксидант, окисно-відновний потенціал, ресторанні технології.*

Ферментативна деструкція протопектину в овочевій сировині для підвищення її структурувальної здатності у складі морозива

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Вступ. Доведено доцільність ферментативного гідролізу протопектину овочевої сировини як функціонально-технологічного напівфабрикату для виробництва морозива.

Матеріали і методи. Досліджено реологічні характеристики ферментованих і неферментованих овочевих пюре, а також молочно-овочевих сумішей. Ефективність ферментолізу визначали кальцій-пектатним методом, ефективну в'язкість – за допомогою ротаційної віскозиметрії, активну кислотність – потенціометрично.

Результати і обговорення. Мета дослідження – вивчити вплив ферментативного гідролізу протопектину в овочевих пюре на їхню структурувальну здатність у складі морозива.

Перевага ферментативного гідролізу протопектину в овочевих пюре, порівняно з кислотним гідролізом, полягає в підвищенні виходу розчинного пектину на 8–12% за менших витрат енергоресурсів. Оптимізовані параметри процесу ферментолізу протопектину за ступенем гідролізу протопектину (не нижче 90%): для різних видів овочевої сировини з вмістом пектинових речовин від 0,22 до 2,56%. Для моркви та буряку потреба у ферменті є найвищою (0,1–0,2%) за подовженої тривалості процесу ферментації (від 120–80 хв до 240 хв). Для кабачків, броколі і томатів тривалість процесу скорочується до 60–120 хв за одночасного зниження дози ферменту – до 0,05–0,10%. За подовження процесу ферментолізу внаслідок надмірного гідролізу пектинових речовин ефективна в'язкість овочевих пюре дещо знижується. Також знижується тиксотропна здатність цих систем. Часткова втрата функціонально-технологічних властивостей овочевої сировини за надмірного гідролізу пектинових речовин негативно відбивається і на реологічних характеристиках молочно-овочевих сумішей для виробництва морозива. За рекомендованих умов ферментації овочева сировина підвищує ефективну в'язкість молочно-овочевих сумішей для виробництва морозива, що можна пояснити утворенням структурувальних комплексів між полісахаридами та молочними білками.

Висновки. Ферментативний гідроліз протопектину більш ефективний, порівняно з кислотним гідролізом, і залежить від фізико-хімічних характеристик овочів. Ферментовані овочеві пюре є структурувальними системами і виявляють тиксотропність у складі сумішей для виробництва морозива.

Ключові слова: *морозиво овочеве, пектин, пектиназа, гідроліз, в'язкість.*

Вплив екстракту порошку шкірки винограду на функціональні та фізико-хімічні властивості зефіру

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Вступ. Оцінено вплив додавання екстракту порошку шкірки винограду на функціональні та фізико-хімічні властивості зефіру.

Матеріали і методи. Для оцінки впливу шкірки винограду на якість зефіру були виготовлені спиртові екстракти виноградної шкірки (GSE), які введені в різних кількостях у рецепти зефіру. Функціональні властивості зефірів оцінювали шляхом визначення загального вмісту поліфенолу і антиоксидантної активності. Мікробіологічну стійкість продукту оцінювали за допомогою м'ясного бульйону з агару. Розмноження цвілі і морфологію клітин з окремих колоній вивчали під мікроскопом.

Результати і обговорення. На фізико-хімічні властивості зефіру щодо вмісту вологи та цукру вплинуло включення екстракту виноградної шкірки (GSE). Прямопропорційний зв'язок спостерігався між додаванням GSE та вмістом вологи у зразках зефіру, реєструючи збільшення з 15,02 до 15,58% для зразків з 1 та 3% GSE відповідно. Вміст цукру варіював у межах 14,05–14,21%, найвищий – у зразках із збільшеною кількістю GSE. Загальний вміст фенолів у зразках GSE і зефіру з

додаванням GSE склав, відповідно, 27,39 і 5,11 (1% зефіру GSE), 6,46 (2% зефіру GSE) та 7,89 (3% зефіру GSE) мг/г галової кислоти (GAE) в. Інгібувальна здатність пероксиду водню і поглинання DPPH радикалів у зефірі зросли пропорційно підвищенню рівня GSE. Встановлено, що антиоксидантна активність зефіру, що містить 3% GSE, вища (35,72%), ніж інші. Додавання GSE суттєво вплинуло на параметри кольору зефіру. Оскільки кількість шкірки винограду збільшувалася, спостерігалося більш інтенсивне фіолетове забарвлення. Зефір, що містить 2% GSE, був найбільш оцінений з точки зору сенсорних властивостей. Додавання GSE мало інгібувальну дію на популяцію цвілі під час зберігання, більш високий ступінь зменшення росту цвілі ($p < 0,05$) спостерігався у зразку з 3% GSE через 7 днів зберігання.

Висновки. Додавання екстракту виноградної шкірки у зефір збільшило біологічну цінність з точки зору антиоксидантної активності та загального вмісту фенолу, а також прийнятності серед споживачів.

Ключові слова: *виноград, шкірка, зефір, антиоксидант, фенол.*

Підвищення ефективності масообміну між рідиною і парою в ректифікаційних колонах циклічної дії

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Вступ. Мета дослідження – визначити оптимальний час перебування рідни на тарілках, ступінь вилучення і кратність концентрування летких домішок спирту і питому витрату гріючої пари в ректифікаційній колоні циклічної дії.

Матеріали і методи. Дослідження проводили в розгінній колоні, оснащеній лускоподібними тарілками із змінним вільним перерізом. Концентрацію летких домішок спирту визначали хроматографічним методом, ступінь їх вилучення і кратність концентрування – розрахунковим методом, інші показники – загальновідомими методами.

Результати і обговорення. Максимальне видалення летких домішок досягалось у ректифікаційній колоні, оснащеній лускоподібними тарілками, що містять поворотні секції, з'єднані з приводними механізмами, дія яких відбувається за заданим алгоритмом. Оптимальні параметри роботи колони: швидкість пари в отворах лусок у період затримки рідини на тарілках 12–14 м/с, у період переливу рідини 1–1,5 м/с, час перебування рідини на тарілках 40 с, час переливу 1,7 с; тиск у нижній частині колони 12 кПа, концентрація етилового спирту в кубовій рідині 3–4 % об. Для забезпечення циклів площа вільного перерізу тарілок повинна миттєво змінюватись від 5,5 до 51,7 %. Технічне рішення дає змогу забезпечити повне видалення естерів, метилацетату та ізопропилового спирту, збільшити ступінь вилучення вищих спиртів сивушного масла і метанолу на 38%, кратність концентрування альдегідів – на 25%, вищих спиртів – на 38%, метанолу – на 37%, а питомі витрати нагрівальної пари зменшити на 40 % порівняно з типовою колоною.

Висновки. Інноваційна технологія циклічної ректифікації дає змогу підвищити ступінь вилучення і кратність концентрування летких домішок спирту на 25–38 % і зменшити енерговитрати на 40 % порівняно з відомими.

Ключові слова: *спирт, ректифікація, тарілка, колона, домішки.*

Вплив показника активності води на властивості борошна пшеничного

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Вступ. Метою дослідження є визначення впливу показника активності води на якість борошна пшеничного та борошна модифікованого пшеничного.

Матеріали і методи. Використовували зразки борошна пшеничного, отриманого із зерна пшениці різного крохмального полімерного складу та компонентного складу. Визначено індекс активності води для пшеничного борошна та модифікованого відповідно до ISO 18787:2017. У рецептурі хліба 10% борошна замінювали борошном модифікованим пшеничним. Визначено вплив покращеної рецептури на термін придатності буханок хліба.

Результати і обговорення. Найвище значення ентальпії та активності води має борошно з базовим співвідношенням амілози й амілопектину. Пом'якшення структури й ослаблення матричних зв'язків крохмаль-білок зменшує ентальпію на 2,3 Дж/г та активність води. Найменше значення ентальпії та активності води має борошно, яке містить лише амілопектин у гранулах крохмалю. Слід зазначити, що підвищений склад мікро- та макроелементів борошна призводить до зменшення ентальпії на 3,7 Дж/г. Дослідження показали, що фізична модифікація пшеничного борошна призводить до зміни активності води у зразках. Зокрема, для звичайного борошна зменшено показник активності води з 0,619 до 0,591. Водночас зміна структури гранул крохмалю призводить до збільшення показника активності води для зразків борошна: кондитерського напряму – з 0,477 до 0,585, типу «ваксі» – з 0,542 до 0,570, збагаченого мікро- та макроелементами – з 0,491 до 0,597.

Використання інгредієнтів із зниженою швидкістю a_w показує здатність до розвитку картопляної хвороби та мікроміцетів, що подовжує зберігання хлібних хлібів на 1–2 дні. Зміна компонентного складу борошна після його модифікації суттєво вплинула на його технологічну якість. Застосування такого продукту у виробництві харчових продуктів сприяє продовженню терміну їх зберігання і, відповідно, на тривалий час сприяє збереженню органолептичних властивостей та виявленню стійкості до дії мікроорганізмів.

Зразки борошна відрізняються складом вуглеводів ($p < 0,001$), білків ($p < 0,01$), жирів ($p < 0,05$). Тому при модифікації температури відбуваються структурні зміни основних компонентів з утворенням більш простих.

Висновки. Індекс активності води для пшеничного борошна знаходиться в межах 0,491–0,619, а для борошна модифікованого пшеничного – у межах 0,570–0,597.

Ключові слова: *пшениця, борошно, модифікація, активність, ентальпія, хліб, зберігання.*

Процеси і обладнання

Особливості процесу смаження м'ясних ковбасних виробів у склеєних оболонках з кишкової сировини

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Вступ. Мета дослідження – встановлення особливостей перебігу процесів тепло- та масообміну під час смаження м'ясних ковбасних виробів у склеєних оболонках, отриманих різними способами із кишкової сировини.

Матеріали і методи. Досліджувались зразки м'ясних ковбасних виробів у різних кишкових оболонках: черева свинячі; склеєні кишкові ковбасні оболонки зі свинячих черев з армуванням локальною тепловою коагуляцією, з армуванням локальним дубленням, із суцільним дубленням і пластифікацією.

Результати і обговорення. Досліджено кінетику температури різних шарів м'ясних ковбасних виробів у склеєних кишкових оболонках під час їх смаження. Для досліджуваних режимів смаження температура сировини всередині зразка однорідно розподілена в діапазоні від 80 до 90 °С. Це усуває негативний вплив високих температур на якість продукції через утворення речовин пірогенетичного розщеплення жиру з неприємним смаком і запахом.

Досліджено кінетику маси м'ясних ковбасних виробів у склеєних кишкових оболонках під час їх смаження. Максимальна швидкість втрати маси отримана для контрольного зразка – ковбасного виробу у традиційній кишковій оболонці. Найменше значення швидкості втрати маси має зразок, в якому використано ковбасну оболонку, склеєну способом суцільного дублення з подальшою пластифікацією гліцерином. Це обумовлено меншою щодо контрольного зразка проникністю розроблених оболонок до пропускання жиру та пари води. Склеєні кишкові оболонки мають меншу щодо традиційної кишкової оболонки проникність до пропускання жиру та пари системної води, що є більш прийнятною функціонально-технологічною властивістю.

Встановлено, що теплофізичні властивості досліджуваних зразків визначаються переважно теплофізичними властивостями м'ясного фаршу, масова частка якого значно більша порівняно з масовою часткою оболонки ковбаси.

Висновки. Виявлено закономірності перебігу процесу тепломасообміну під час смаження ковбасних виробів в оболонках з кишкової сировини.

Ключові слова: *ковбаса, смаження, оболонка, армування, тепломасообмін.*

.

Instructions for authors

Dear colleagues!

The Editorial Board of scientific periodical
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invites you for publication of your research results.

Requirements to all texts:
Language – English.
Recommended size of the article – 15–20 pages.
Font – Times New Roman, font size – 14, line intervals – 1, margins on both sides – 2 cm.

The structure of the article:

1. The title of the article
2. Authors (full name and surname)
3. Institution, where the work has been performed.
4. Abstract (2/3 of a page). The structure of the abstract should correspond to the structure of the article (Introduction – 2–3 lines, Materials and methods – 3-5 lines, Results and discussion – a half of page, Conclusion – 2 lines).
5. Keywords.
6. The main body of the article should contain the following parts:
   - Introduction
   - Materials and methods
   - Results and discussion
   - Conclusion
   - References

   If you need you can add another parts and/or divide them into subparts.
7. The information about the author (Name, surname, scientific degree, place of work, email and contact phone number).

All figures should be made in graphic editor, the font size 14.

The background of the graphs and charts should be only in white color. The color of the figure elements (lines, grid, text) – in black color.

Figures and EXCEL format files with graphs additionally should be submitted in separate files.

Photos are not recommended to be used as graphical materials.

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Шановні колеги!

Редакційна колегія наукового періодичного видання «**Ukrainian Food Journal**» запрошує Вас до публікації результатів наукових досліджень.

Вимоги до оформлення статей

Мова статей – англійська.

Мінімальний обсяг статті – **10 сторінок** формату А4 (без врахування анотацій і списку літератури)**.**

Для всіх елементів статті шрифт – **Times New Roman**, кегль – **14**, інтервал – 1.

Всі поля сторінки – по 2 см.

Структура статті:

1**.** УДК**.**
2. **Назва статті.**
3. Автори статті (ім'я та прізвище повністю, приклад: Денис Озерянко).
4. *Установа, в якій виконана робота*.
5. Анотація. **Обов'язкова** структура анотації:
   - Вступ (2–3 рядки).
   - Матеріали та методи (до 5 рядків)
   - Результати та обговорення (пів сторінки).
   - Висновки (2–3 рядки).
6. Ключові слова (3–5 слів, але не словосполучень).

Пункти 2–6 виконати англійською і українською мовами.

7. Основний текст статті. Має включати такі обов'язкові розділи:
   - Вступ
   - Матеріали та методи
   - Результати та обговорення
   - Висновки
   - Література.

За необхідності можна додавати інші розділи та розбивати їх на підрозділи.

8. Авторська довідка (Прізвище, ім'я та по батькові, вчений ступінь та звання, місце роботи, електронна адреса або телефон).
9. Контактні дані автора, до якого за необхідності буде звертатись редакція журналу.

Рисунки виконуються якісно. Скановані рисунки не приймаються. Розмір тексту на рисунках повинен бути **співрозмірним** (!) тексту статті. **Фотографії можна використовувати лише за їх значної наукової цінності.**

Фон графіків, діаграм – лише білий. Колір елементів рисунку (лінії, сітка, текст) – чорний (не сірий).

Рисунки та графіки EXCEL з графіками додатково подаються в окремих файлах.

Скорочені назви фізичних величин в тексті та на графіках позначаються латинськими літерами відповідно до системи СІ.

У списку літератури повинні переважати англомовні статті та монографії, які опубліковані після 2010 року.

Оформлення цитат у тексті статті:

Кількість авторів статті	Приклад цитування у тексті
1автор	(Arych, 2019)
2 і більше авторів	(Bazopol et al., 2021)

Приклад тексту із цитуванням: It is known (Bazopol et al., 2006; Kuievda, 2020), the product yield depends on temperature, but, there are some exceptions (Arych, 2019).

У цитуваннях необхідно вказувати одне джерело, звідки взято інформацію.
Список літератури сортується за алфавітом, літературні джерела не нумеруються.

Правила оформлення списку літератури

В Ukrainian Food Journalвзято за основу загальноприйняте в світі спрощене оформлення списку літератури згідно стандарту Garvard. Всі елементи посилання розділяються **лише комами**.

1. Посилання на статтю:

Автори А.А. (рік видання), Назва статті, *Назва журналу (курсивом)*, Том (номер), сторінки.

Ініціали пишуться після прізвища.

Всі елементи посилання розділяються комами.

1. Приклад:
   Popovici C., Gitin L., Alexe P. (2013), Characterization of walnut (Juglans regia L.) green husk extract obtained by supercritical carbon dioxide fluid extraction, *Journal of Food and Packaging Science, Technique and Technologies,* 2(2), pp. 104–108.

2. Посилання на книгу:

Автори (рік), *Назва книги (курсивом)*, Видавництво, Місто.

Ініціали пишуться після прізвища.

Всі елементи посилання розділяються комами.

Приклад:

2. Wen-Ching Yang (2003), *Handbook of fluidization and fluid-particle systems*, Marcel Dekker, New York.

Посилання на електронний ресурс:

Виконується аналогічно посиланню на книгу або статтю. Після оформлення даних про публікацію пишуться слова **Available at:** та вказується електронна адреса.

Приклади:

(2013), *Svitovi naukovometrychni bazy*, Available at: http://www.nas.gov.ua/publications/q_a /Pages/scopus.aspx

Cheung T. (2011), *World's 50 most delicious drinks*, Available at: http://travel.cnn.com/explorations/drink/worlds–50-most-delicious-drinks–883542

Список літератури оформлюється лише латиницею. Елементи списку українською та російською мовою потрібно транслітерувати. Для транслітерації з українською мови використовується паспортний стандарт.

Зручний сайт для транслітерації з української мови: http://translit.kh.ua/#lat/passport

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