ISSN 2313–5891 (Online)
ISSN 2304–974X (Print)

Ukrainian Food Journal

Volume 14, Issue 2
2025

Kyiv **Київ**
2025

Ukrainian Food Journal is an international scientific periodical journal that publishes articles by specialists in the field of food science, engineering and technology, chemistry, economics and management.

Ukrainian Food Journal – міжнародне наукове періодичне видання для публікації результатів досліджень фахівців у галузі харчової науки, техніки та технології, хімії, економіки і управління.

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Ukrainian Food Journal індексується наукометричними базами:

Index Copernicus (2012)
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Ukrainian Food Journal включено у перелік наукових фахових видань України з технічних наук, категорія А (Наказ Міністерства освіти і науки України № 358 від 15.03.2019)

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of Food Technologies
68 Volodymyrska str.
Kyiv 01601, **Ukraine**

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Ukrainian Food Journal is an open-access, peer-reviewed scientific journal published by the National University of Food Technologies (Kyiv, Ukraine). The journal accepts original research articles, short communications, review papers, news, and literature reviews across all areas of food science, including food technology, engineering, nutrition, food chemistry, economics, and management.

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Editorial

In the last days of June, as universities and colleges across Ukraine were holding diploma defenses and graduation ceremonies, russia launched a missile strike on the college of the National University of Food Technologies in the city of Smila. The educational and laboratory building was destroyed.

The aggressor state continues its acts of violence and destruction. Ukrainian educators and scientists are working under these challenging conditions, without ceasing to prepare for the future. Innovations are being developed, scientific research is ongoing, and the educational process remains uninterrupted.

The terrorists destroyed the building, but they will never break the spirit of enlightenment or extinguish the thirst for knowledge. The college endures as does Ukraine, and is already preparing to accept a new generation of students. We shall overcome this evil through hard work and a deep desire for freedom.

We express our deep gratitude and sincere respect to our colleagues for their continued support of Ukrainian students, scientists, and educators.

We are also thankful for your valuable contributions to the Journal through high-quality scientific content.

Sincerely,
on behalf of the publisher of the Ukrainian Food Journal
Rector of the National University of Food Technologies
Oleksandr Shevchenko

DOI: 10.24263/2304-974X-2025-14-2-3

Effect of cream-plant mixtures on properties of multicomponent albumin-based products

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Keywords:

Albumin
Dairy
Cream
Cocoa fiber
Bird cherry
Normalization

Article history:

Received
25.10.2024
Received in revised form 28.02.2025
Accepted
30.06.2025

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DOI:
10.24263/2304-974X-2025-14-2-4

Abstract

Introduction. The aim of the research is to determine the effect of adding cream-plant mixtures on the properties of multicomponent albumin-based products.

Materials and methods. The research utilized albumin mass, bird cherry flour, and cocoa fiber. Solubility was determined using the Shoha method. The fat- and moisture-holding capacities were measured by centrifugation. Thermogravimetric analysis was performed using a derivatograph. The moisture-holding capacity was assessed by the modified gravimetric method.

Results and discussion. The solubility of cocoa fiber and bird cherry flour in water is higher compared to their solubility in whey by 1.6±0.1% and 1.5±0.1%, respectively; in cream with 10% fat content, solubility increases by 4.9±0.1% and 4.5±0.1%; and in cream with 20% fat content, by 6.5±0.1% and 5.3±0.1%, respectively. This increase is attributed to the high content of water-soluble carbohydrates and certain dietary fibers present in the plant-based ingredients. The optimal amounts of plant ingredients were found to be 3.0–5.0% for bird cherry flour and 2.0–3.0% for cocoa fiber. The addition of bird cherry flour had a slight impact on the active acidity (pH) of the test samples, while also contributing to a reduction in moisture content. To investigate the forms of moisture binding in the albumin product containing bird cherry flour, a thermogravimetric analysis was conducted. It was established that incorporating this plant ingredient into the albumin product increases its hydrophilicity and raises the specific heat required for product dehydration. Adding bird cherry flour directly to the albumin mass with 77±2% moisture content enables binding of free moisture, which is then normalized by the subsequent addition of cream. This approach ensures stable quality indicators of the product, enrichment with flavonoids, phytoncides, and essential amino acids (leucine, valine, and phenylalanine). A potential extension of shelf life is possible due to the presence of antioxidants in bird cherry flour. Cocoa fiber binds and retains moisture due to its hydrophilic properties, moisture-retaining, and primarily fat-retaining capacities.

Conclusions. The use of cocoa fiber and bird cherry flour in the composition of cream-plant mixtures helps to normalize multicomponent albumin products.

Introduction

Albumin-based snack and dessert products have a wide assortment range (Bintsis and Papademas, 2023; Kindar, 2022). Whey cheeses are currently produced worldwide under various local names. In Italy, they are known as ricotta, ricotta salata, ricottone, and ricotta fresca; in Greece – anthotyros, myzithra, manouri, xynomyzithra, and urda; in Turkey – lor; in Cyprus – anari; in Croatia and Serbia – skuta; in Norway – gjetost and brunost; in Sweden – mesost and messmör; in Iceland – mysuostur; in Denmark – myseost; in Portugal – requeijão; and in Spain and Mexico – requesón (Bintsis and Papademas, 2023).

One of the well-established technologies for ricotta cheese production involves the incorporation of dietary fibers at varying concentrations—1%, 3%, and 5% of the raw material mass. The sample with the addition of 3% citrus dietary fiber demonstrated the most harmonious sensory characteristics (Zinina et al., 2021). Other studies explore the addition of various herbs and/or plant extracts to diversify the range of whey cheeses and extend their shelf life (Akpinar et al., 2022). Specifically, black cumin, thyme, and rosemary are frequently used as additives that enhance sensory characteristics and impart antioxidant properties to the cheese (Ramírez-Rivas et al., 2022). Another direction in the improvement of such technologies involves the use of quality prediction methods for albumin-based products during storage. The possibility of predicting the quality indicators of albumin paste using potato fiber "Potex" as a moisture regulator during storage has been substantiated (Grek et al., 2017)

In modern albumin product technologies, functional additives are widely used (Grek et al., 2023). The technology for Ricotta cheese enriched with spirulina powder at levels of 0.25, 0.5, 0.75, and 1.0 g/100 g of cheese is known. Enrichment with spirulina powder led to an increase in the content of proteins, fats, ash, fiber, carbohydrates, and minerals, phenolic compounds, β-carotene compared to the control (Ismail et al., 2023). Furthermore, Kaminarides et al. (2019) determined the effect of fat content in whey on the technological and consumer properties of whey cheeses. Four cheese variants were studied. It was found that increasing the fat content in rennet whey contributes to a higher yield of the final product and improves its sensory and textural characteristics. There is a limited amount of studies concerning the enrichment of albumin-based products standardized for fat. For the effective use of plant ingredients in such multicomponent albumin products, selection criteria have been developed, including functional and technological properties, regional availability, and industrial processing methods. It is advisable to give preference to plant raw materials that have undergone special thermal treatment, exhibit stable characteristics, and are microbiologically safe.

The increasing use of plant-based ingredients in food formulations is a notable trend in the food industry (Stabnikova et al., 2021). Fruits of bird cherry (*Prunus padus* L.) are rich in bioactive compounds possessing antioxidant activity, while bird cherry flour could serve as a useful ingredient in the preparation of functional foods (Stabnikova et al., 2024). Bird cherry flour is a raw material obtained from bird cherry fruits by drying and grinding using specialized equipment. The flour has a brown color and an almond-like flavor, which can impart original sensory properties to the final product (Telichowska et al., 2020). Bird cherry flour is used in the production of bakery and confectionery products, including gluten-free items, as well as a component in reduced-calorie sauces (Dziundzia and Zvaholska, 2021; Niemirich, 2018).

Bird cherry flour has the following chemical composition: protein – 7.6%; carbohydrates – 21.8%; dietary fiber – 4.7%; ash – 2.7%; iron – 0.2 mg; potassium – 13.4 mg; calcium – 2.2 mg; manganese – 0.9 mg; zinc – 0.3 mg. It also contains vitamins B_1 (0.39 mg), B_2 (0.07 mg), E (1.7 mg), PP (0.9 mg), ascorbic acid (0.45%), malic acid (0.63%), and citric acid (0.28 mg) (Dziundzia and Zvaholska, 2021; Niemirich et al., 2018). Bird cherry flour contains tannins (6–

8%) and flavonoids (1.5–2.0%), including hyperoside, quercetin 3-galactoglucoside, and astragalin. It also contains anthocyanins (5.4–16.5% of total flavonoids), primarily cyanidin 3-rutinoside (9.13%) and cyanidin 3-glucoside (7.35%). Additional bioactive components include carotene; cyanogenic glycosides such as amygdalin, prunasin, and amygdonitril glycoside; carbohydrates including fructose (4.42–6.44%), glucose (5.33–6.35%), sucrose (0.6–6.2%); pectin substances (1.1%); and benzaldehyde with essential oils (0.26%). The flour also contains trace elements such as Mg, Cr, Al, Se, Ni, Sr, Pb, B, I, Cu, and Co (Telichowska et al., 2020).

Cocoa fiber is a modern functional ingredient derived from the husk and kernel of cocoa beans, ground during the production of cocoa butter or cocoa powder. It is non-alkalized and contains no flavorings, preservatives, or added sugars. Cocoa fiber contains higher levels of antioxidants and magnesium than any other plant product and has a low fat content. It can be considered a carbohydrate-protein complex with valuable technological functionalities (Anoraga et al., 2024).

Cocoa fiber was used for enrichment (Certificate NC-3413, COID: POL-1-9445-658511) with the following composition, %, per 100 g of product: protein – 17.0, fat – 2.5, carbohydrates – 0.7, fiber – 72.0% (including insoluble – 61.0, soluble – 11.0), salt – 0.1. The energy value of the product is 249 kcal per 100 g. According to sensory indicators, the fiber is a brown powder with a uniform texture, a pronounced chocolate aroma and taste, and no foreign flavours (Rojo-Poveda et al., 2020). In addition to its functional properties, dietary fiber possesses specific technological capabilities that enable the development of products with a moderately viscous consistency (Anoraga et al., 2024).

In the production of multicomponent dessert products based on dairy protein concentrates, including high-albumin content mass, normalization is achieved by adding cream (Gawande et al., 2023). Similarly, in the production of sour milk cheese using the separated method, it is essential to normalize the protein base with cream of varying fat contents. This cream was heat-treated at (78±2) °C for 15–20 seconds, and then cooled to (8±2) °C before mixing.

For albumin products with a fat content of up to 3.0%, it is relevant to normalize with cream of various fat contents up to 20% (Freitas et al., 2024). When enriching albumin products with normalization, it is necessary to determine the most effective methods for incorporating bird cherry flour and cocoa fiber, considering their functional and technological properties.

It is advisable to determine the effect of cream-plant mixtures on the characteristics of multicomponent albumin products.

Materials and methods

Materials

The basis for the production of experimental samples of multicomponent albumin products was albumin mass obtained from rennet whey by the thermo-acid coagulation method, which had a moisture content of 77±2 %, titratable acidity of (60±3) °T, protein content at the level of 18.0±1.0%, and lactose content of 1.7±0.1%.

The whey had the following physicochemical characteristics: titratable acidity – 15±1 °T, fat content – (0.2±0.1) %, and dry matter – (6.30±0.02) %.

Plant components – bird cherry flour and cocoa fiber were used for enrichment of multicomponent albumin products (Figure 1).

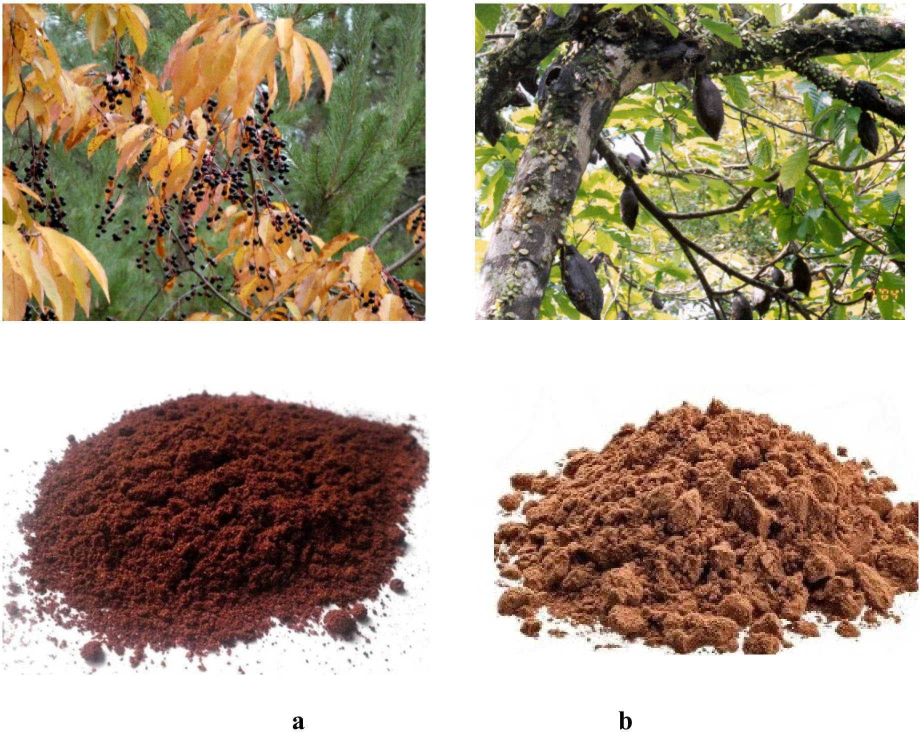

Figure 1. Bird cherry fruits and bird cherry flour (a); cocoa pods and cocoa fiber (b) used for enrichment of multicomponent albumin products

Preparation of cream-plant mixtures for normalization of multicomponent albumin products

Cream-plant mixtures for the normalization of multicomponent albumin products consisted of dairy cream, cocoa fiber or bird cherry flour, and sugar. Before being added to the albumin mass, the plant ingredients were soaked in pasteurized dairy cream at a temperature of 60-65 °C with a holding time of 10-15 minutes, followed by cooling to a temperature of 4±2 °C. Sugar was added for 15%.

The cream had the following physicochemical characteristics: fat content – 10% or 20%, protein – 2.8-2.9%, carbohydrates – 3.7-4.3%, titratable acidity – 17±1 °T, density – 1005 kg/m³. The cream had a liquid, homogeneous consistency without fat lumps or protein flakes; a sweetish, clean flavor and odor, with no foreign or uncharacteristic notes for fresh cream.

Production of model samples of multicomponent albumin products

For the research, model samples were produced, normalized with cream and supplemented with cocoa fiber or bird cherry flour in amounts ranging from 2.0% to 6.0% of the final product mass.

To prepare the multicomponent products, albumin mass at a temperature of 10–15 °C, the cream-plant mixture, and sugar were mixed, and then sent for packaging and further cooling to a temperature not exceeding 8 °C.

The control was an albumin product (including fat-standardized) obtained using the aforementioned technology without the addition of plant ingredients.

At the first stage, the functional and technological properties of cocoa fiber and bird cherry flour were determined for their use as components of multicomponent albumin products, including the identification of optimal environments and methods of incorporation (cream or directly into the albumin mass). The parameters studied included solubility, water- and fat-binding capacities, which are associated with the carbohydrate and protein composition of the ingredients.

The solubility of dry plant ingredients in an aqueous medium is determined by the presence of soluble compounds – certain proteins and carbohydrates, including some pectic substances and starch; insoluble compounds include fiber, and lignin.

Methods

Research procedure. Standardized and validated methods for analysing techno-functional properties and quality indicators were applied during the research process, ensuring the reliability of the obtained results and achievement of the stated objective.

Functional and technological properties of bird cherry flour and cocoa fiber

The solubility of plant ingredients of various origins was determined using the Shoha method (Onopriichuk et al., 2022). This method is based on the ability of carbohydrates to swell and dissolve in water depending on the strength of their structure.

T*he fat-binding capacity* of cocoa fiber or bird cherry flour mixed with cream with fat contents of 10% and 20% was determined by the centrifugation method (Delgado-Ospina et al., 2021).

The method for determining the water-holding capacity of plant ingredients is similar to that of fat-binding capacity but is conducted in water (control) and whey. The result is expressed in grams of absorbed moisture per gram of plant ingredient sample (Grek et al., 2020).

Physicochemical and sensory indicators of multicomponent albumin products

The water-holding capacity (WHC) of multicomponent albumin products was determined by the gravimetric method of Grau-Hamm as modified by A.A. Alekseev (Grek et al., 2023).

The sensory indicators of multicomponent albumin products were assessed according to ISO standard 22935-3:2009 and individual sensory attributes (Elsamani, 2016).

Thermogravimetric parameters of multicomponent albumin products

Derivatography allows determination of the stages and temperature intervals of decomposition, type of thermal effects, and depth of structural-chemical transformations (Lammerskiten et al., 2019).

Thermogravimetric studies were conducted using a modernized Q–1000 derivatograph produced by MOM.

Information on changes in T, TG, DTG, and DTA is transmitted to an analog-to-digital converter and then via interface converter to a personal computer, where it is stored and processed.

Conditions for thermal analysis:

– Sample heating rate	3.6 K/min;
– Temperature interval	from 20±2 °C to 250 °C;
– Weight scale	100 mg.

Samples (control and albumin product with bird cherry flour) were placed in an open conical platinum crucible from the derivatograph set. The atmosphere was static air. Aluminium oxide was used as the inert substance in the comparison crucible. The DTA scale of the derivatograph was calibrated using stearic acid (ΔH_{ref} = 198.9 J/g), a reference substance used for calibration of DTA and DSC instruments. The temperature deviation did not exceed ±0.5 K (Ovsiienko et al., 2021).

Determination of the rheological characteristics of cream-plant mixtures

The dynamic viscosity of cream-plant mixtures with varying amounts of cocoa fiber was determined using a Hepler viscometer equipped with a set of balls of different diameters and a cylindrical gauge. Absolute (dynamic) viscosity (η) of cream-plant mixtures with cocoa fiber was calculated using the formula, Pa·s × 10^{-3}:

$$\eta = \tau \cdot (d - d_1) \cdot k, \qquad (1)$$

where τ is a time of ball descent, s; d is a density of the material from which the ball is made at 20 °C, kg/m^3; d_1 is a density of cream-plant mixtures with cocoa fiber at 20 °C, kg/m^3; k is a ball constant. The density of the ball material and the constant are specified in the certificate supplied with the device (Sapiga et al., 2019).

Statistical analysis

All experiments were conducted in triplicate or more, and the results were expressed as mean ± standard deviation. Data were analysed using one-way ANOVA and Tukey's HSD tests by the SPSS statistical package. Significant difference was defined at $p \leq 0.05$.

Results and discussion

Functional and technological properties of bird cherry flour and cocoa fiber

The results of determining the solubility of plant ingredients in water, whey, and cream with fat content of 10 % and 20% are presented in Figure 2. The process was conducted at a temperature of 20±2 °C.

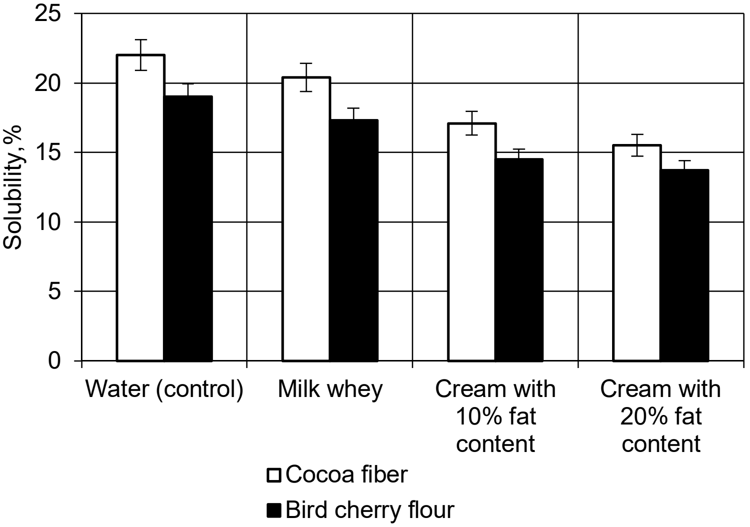

Figure 2. Solubility of cocoa fiber and bird cherry flour in different media

The solubility of cocoa fiber in water is higher by 1.6±0.1%, 4.9±0.1%, and 6.5±0.1% than in whey and cream with fat content of 10 % and 20 %, respectively, while for bird cherry flour these values are higher by 1.47±0.1%, 4.5±0.1%, and 5.3±0.1% respectively. This is explained by the presence of surface-active substances in the dispersive media (whey and cream), which adsorb onto the surface of plant ingredients, form a protective layer, and hinder fluid access. These indicators are due to the content of nearly 11.0 % soluble fiber, proteins, and other substances in cocoa fiber that ensure high solubility (Chico, 2022; Younes et al., 2023), as well as the ability to increase mixture viscosity, and its high dispersibility (99 % < 40 µm) (Soares et al., 2022). Bird cherry flour contains a large amount of water-insoluble carbohydrates and dietary fibers (21.8 % and 4.7 %) (Dziundzya et al., 2021), which reduce the solubility level of this plant ingredient to 19.0±0.2 % (in water). Thus, it has been found that for normalization of cream-plant mixtures, it is appropriate to use cocoa fiber, while bird cherry flour should be added directly into the albumin mass, which contains 77±2 % moisture (whey), followed by cream-based normalization. This technological technique is effective.

At the next stage, the water- and fat-binding capacities of cocoa fiber and bird cherry flour in water, whey, and cream with fat content of 10 and 20% were studied. The process was conducted at a temperature of 20±2 °C. The research results are presented in Figure 3.

According to Figure 3, the highest fat-binding capacity was recorded for cocoa fiber: in cream with 10% and 20% fat content, the indicator was 3.3±0.1 g/g and 2.7±0.1 g/g respectively. At the same time, this dietary fiber shows high water-binding capacity: in water – 4.6±0.1 g/g and in whey – 4.01±0.1 g/g. This effect in cocoa fiber results from its chemical composition and is similar to the values reported by other authors in similar systems (Delgado-Ospina et al., 2021). The fat- and water-binding capacity values of bird cherry flour are lower than those of cocoa fiber by 1.1±0.1 g/g and 0.9±0.1 g/g in cream with 10 % and 20 % fat content, respectively, and by 1.3±0.1 g/g in whey and 1.11±0.1 g/g in water.

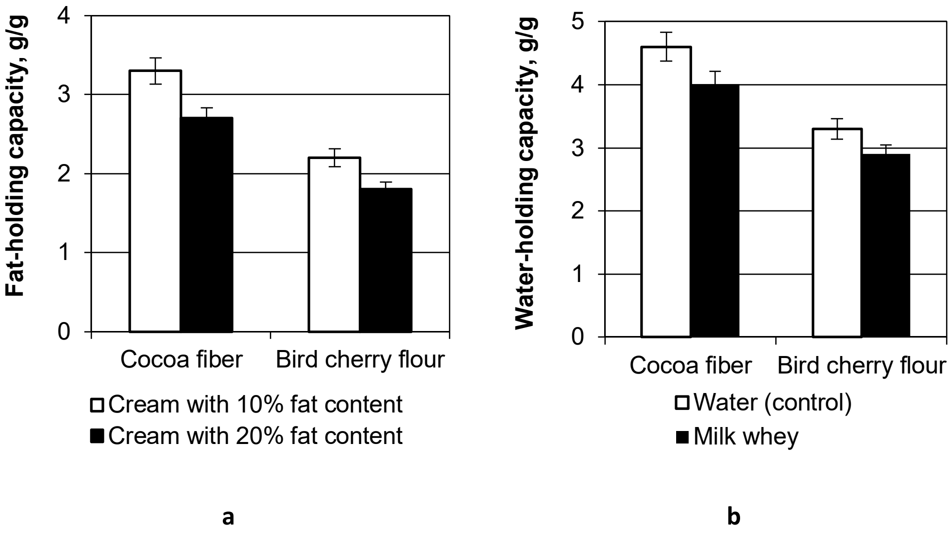

Figure 3. Fat- (a) and water-holding (b) capacities of cocoa fiber and bird cherry flour in different media

Considering the results of the solubility, fat- and water-binding capacity studies of cocoa fiber and bird cherry flour; it is advisable to use 10% fat cream for normalization by fat content in albumin products.

Physicochemical and sensory characteristics of multicomponent albumin-based products

The selection of the optimal level of plant ingredient incorporation was based on maintaining the standard sensory properties characteristic of albumin-based products. Table 1 presents the results of the sensory evaluation of multicomponent albumin-based products with varying levels of bird cherry flour addition.

Appropriate taste properties and consistency of the multicomponent albumin product were achieved with the addition of 3.0–5.0% bird cherry flour. The samples had stable quality indicators, increased content of biologically active components, original sensory characteristics, and a brown color evenly distributed throughout the mass with inclusions of the plant ingredient.

Adding more than 5% bird cherry flour results in excessive changes in the sensory properties of the multicomponent albumin product – an intensely pronounced almond aroma and dark brown color with significant inclusions of bird cherry flour appear. Adding less than 3% of the plant ingredient has no noticeable positive effect on sensory properties and minimizes the enrichment of the albumin product with biologically active nutrients. The almond aroma of the multicomponent albumin product is due to the content of glucosides (amygdalin, prulaurasin, and prunasin) in bird cherry flour (Telichowska et al., 2020).

An albumin product containing 3.0–5.0% bird cherry flour can be used as a base for dairy-protein products with a plastic structure (creams, desserts, sauces).

Table 1

Sensory characteristics of multicomponent albumin products with varying amounts of bird cherry flour added

Parameters	Amount of bird cherry flour added,%				
	2	3	4	5	6
Consistency and appearance	Homogeneous, with inclusions of bird cherry flour	Homogeneous, moderately dense, does not change during storage			Homogeneous, moderately dense
Taste and aroma	Clean, creamy, with a slight almond aroma	Clean, creamy, with an almond aroma			Clean, creamy, with a strongly pronounced almond aroma
Color	Light brown, uneven throughout the mass with occasional inclusions of bird cherry flour	Brown, even throughout the mass with inclusions of bird cherry flour			Dark brown, even throughout the mass with significant inclusions of bird cherry flour

At the next stage, the main physicochemical indicators of normalized albumin products with 3.0–5.0% bird cherry flour content were studied. The respective values are presented in Table 2.

Table 2

Physicochemical indicators of normalized albumin products with different bird cherry flour content

Amount of bird cherry flour added,%	Physicochemical indicators of the albumin products			
	Content,%		Moisture retention capacity,%	Active acidity, pH units
	Dry matter	Moisture		
Control	23.0 ± 0.3^{a}	77.0 ± 2.0^{a}	47.5 ± 0.2^{a}	5.00 ± 0.02^{a}
3	25.8 ± 0.2^{b}	74.2 ± 1.0^{ab}	51.2 ± 0.1^{b}	4.89 ± 0.03^{ab}
4	26.7 ± 0.3^{bc}	73.3 ± 0.5^{b}	53.4 ± 0.3^{bc}	4.85 ± 0.01^{b}
5	27.8 ± 0.3^{c}	72.2 ± 1.0^{b}	55.3 ± 0.2^{c}	4.83 ± 0.01^{b}

Note: Values are the means ± standard deviation. Means within the same column with different superscripts are significantly different at $p \leq 0.05$.

With the addition of 3% to 5% bird cherry flour, the active acidity of the experimental samples changes slightly. For the control, this indicator is 5.00±0.02 pH units, while for the sample with the maximum amount of bird cherry flour it is 4.83±0.01 pH units. The moisture content decreased as the amount of bird cherry flour added increased. The change in moisture content and moisture retention capacity of albumin products with plant ingredients can be explained by the chemical composition of bird cherry flour – a complex of linear and branched biopolymers with hydroxyl (cellulose, hemicellulose), phenolic (lignin), and carboxyl (hemicellulose, pectic substances) groups, which over time strengthen hydrogen bonds and the ability to sorb water and other polar molecules and ions, thereby increasing moisture retention capacity.

Effect of bird cherry flour addition on thermogravimetric parameters of multicomponent albumin products

The quality of food products, appearance, taste, and stability during storage directly depend on the moisture content. One of the methods for determining the moisture content of food products is thermogravimetric analysis. When setting research objectives in this area, the analysis of the kinetics of mass loss under differences in the internal heat content of the system during exothermic and endothermic transformations is important; as well as comparing the thermal stability of food systems under specific values of mass loss.

Qualitative and quantitative assessment of the processes occurring during sample heating is carried out using curves of temperature change (T), sample mass (TG), rate of mass change (DTG), which is the derivative of the TG curve, and DTA. The DTA curve represents the temperature difference between the sample and an inert material in the form of the difference in thermoelectromotive forces of thermocouples. DTA allows the identification of thermal processes occurring in the sample during heating. Due to the thermal resistance of the sample material, the temperature peaks of thermal effects (DTA) are higher than the corresponding maxima in the rate of mass change (DTG).

Figures 4 and 5 show the derivatograms of the multicomponent albumin product with bird cherry flour (experimental sample) and the albumin product (control sample).

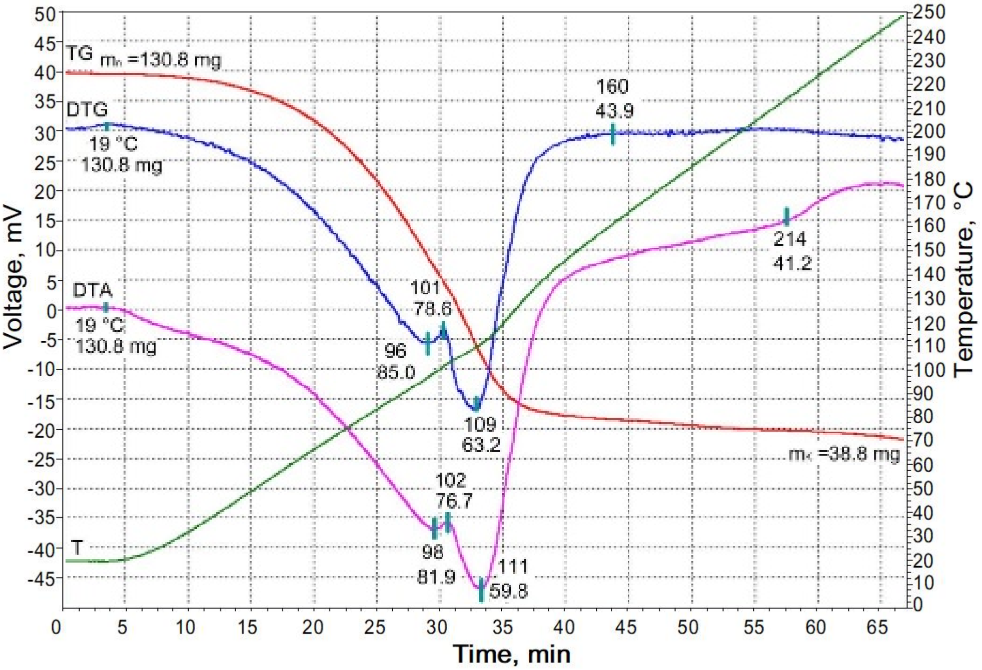

Figure 4. Derivatograms of multicomponent albumin product with bird cherry flour

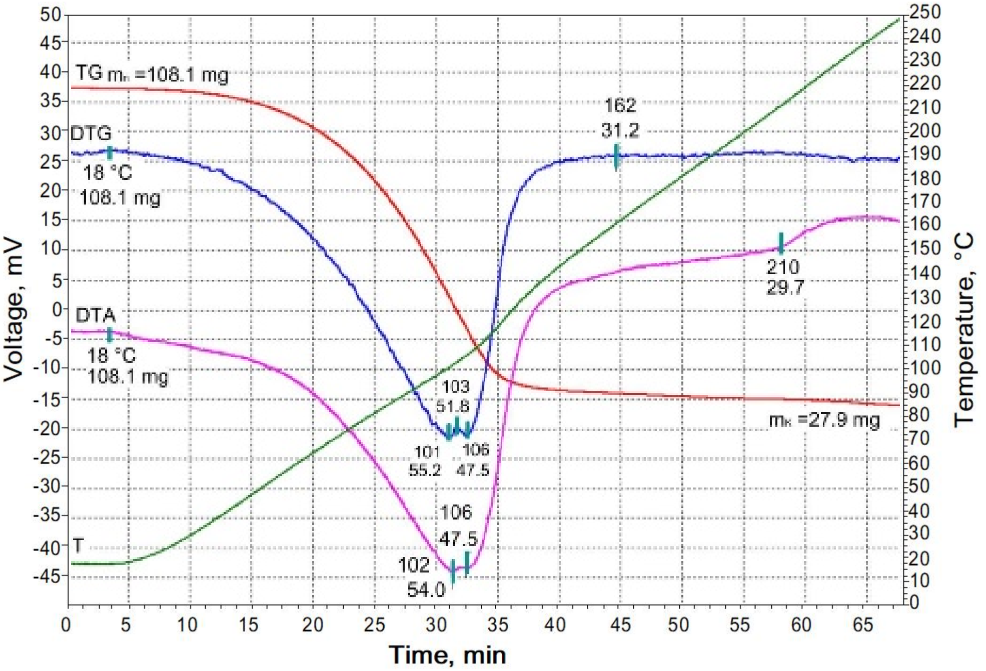

Figure 5. Derivatograms of the albumin product

The nature of both derivatograms is similar and shows the feature that dehydration occurs in two stages. Mass loss of the multicomponent albumin product samples with and without bird cherry flour (TG curves) begins at 18 and 19 °C (for control and experimental samples respectively) and is accompanied by heat absorption, as the DTA curves deviate downward. As the temperature increases, the dehydration rate (DTG curves) rises, reaching a maximum during the first stage of dehydration for the experimental sample of the multicomponent albumin product with bird cherry flour at 109 °C (Figure 4), and for the control sample of the albumin product at 106 °C (Figure 5).

A slight increase in dehydration rate is recorded for both the control and experimental samples, with maxima at 101 °C and 96 °C, respectively. During the first stage of dehydration, 77.79% and 78.80% of water is removed from the multicomponent albumin products with and without bird cherry flour, with an average rate of 1.57% and 1.72%, respectively.

The temperature ranges of dehydration, average rate, and share of removed water relative to the total water mass are presented in Table 3.

According to the method (Ovsiienko et al., 2021), based on DTA curves, the integral heat used for dehydration of the multicomponent albumin product samples with and without bird cherry flour during heating was determined and the specific heat of dehydration was calculated (Table 4).

Table 3

Amount of water removed during dehydration and loss of dry matter during thermal decomposition

Material	Dehydration							
	Firs stage			Second stage			Total	
	τ, °C	RW, %	AR, %/min	τ, °C	RW, %	AR, %/min	τ, °C	AR, %/min
Albumin product	18–106	78.80	1.72	106–162	21.20	1.23	18–162	1.59
Multicomponent albumin product with bird cherry flour	19–109	77.79	1.57	109–160	22.21	1.36	19–160	1.53

Note: τ is temperature; RW – removed water; AR – average rate.

Table 4

Mass and moisture of samples, temperature range and specific heat of dehydration, interval and dry matter loss during thermal decomposition

Material	Moisture content, %	Dehydration				Thermal decomposition	
		τ, °C	RW, mg	IH, J	SH, kJ/kg	τ, °C	DML,%
Albumin product	71.14	18–162	76.9	208.062	2706	162–250	3.05
Multicomponent albumin product with bird cherry flour	66.44	19–160	86.9	238.521	2745	160–250	3.90

Note: RW – removed water; IH – integral heat; SH – specific heat; DML – dry matter loss.

The second stage of dehydration of the multicomponent albumin product with bird cherry flour begins at 109 °C, while that of the control sample starts at 106 °C, and differs from the first stage by a significant increase in the dehydration rate, which reaches a maximum at 117.7 °C and 114.4 °C, respectively, for the experimental and control samples. At the final stage of dehydration, slight increases in the rate are recorded, with maximum values at 162 °C for the experimental sample and 160 °C for the control.

The amount of water removed during the second stage of dehydration of the multicomponent albumin product with bird cherry flour (experimental sample) is 22.21% of the sample's mass at an average dehydration rate of 1.36%/min. The control sample loses 21.20% of water with an average rate of 1.23%/min. The higher dehydration rate of the control albumin product sample compared to the experimental one is associated with its higher moisture content. Dehydration of the multicomponent albumin product samples is completed at practically identical temperatures (160.0–162.0 °C) and at the same temperature, the thermal decomposition process begins.

After complete dehydration of the samples, mass loss is observed on the TG and DTG curves along with heat generation, which is reflected in the rise of the DTA curves (Figures 5 and 6). Such process behavior results from the onset of thermal decomposition, which occurs in the intervals of 162–250 °C and 160–250 °C. Before reaching 250 °C, the samples lose different portions of dry matter. The experimental sample loses 3.9% of dry matter, while the control loses 3.05%. These results are likely due to differences in the initial content of dry matter in the products under the same decomposition conditions.

According to the method described by (Mykhailyk et al., 2015), the integral heat consumed for dehydration of the samples of multicomponent albumin products with and without bird cherry flour during heating was determined from the DTA curves, and the specific heat of dehydration was calculated. The DTA scale of the derivatograph was calibrated using stearic acid (ΔH_{ref} = 198.9 J/g) – a reference substance used for calibrating DTA and DSC instruments (Lammerskiten et al., 2019).

According to the method described by Ovsiienko et al. (2021), the integral heat consumed for sample dehydration during heating was determined from the DTA curves, and the specific heat of dehydration was calculated (Table 4).

The obtained values of the specific heat of dehydration of the multicomponent albumin products with and without bird cherry flour exceed the average specific heat of evaporation of pure water from a free surface, which is approximately 2360 kJ/kg, in the studied temperature ranges, characterizing the studied objects – multicomponent albumin products – as systems with a high degree of hydration.

A comparison of the specific heat values during dehydration of the studied samples showed that the multicomponent albumin product containing bird cherry flour exhibited a 1.5% higher specific heat compared to the control albumin sample. The addition of bird cherry flour to the composition of the albumin product increased the content of hydrophilic centers in the composition and thus increased the amount of bound water, which caused an increase in the specific heat of dehydration.

Effect of cocoa fiber addition on the quality characteristics of multicomponent albumin products

The profile chart of sensory evaluation of the multicomponent albumin product with varying amounts of cocoa fiber is presented in Figure 6.

When 2.0–3.0% cocoa fiber is added, the multicomponent albumin product exhibits the best taste and texture. A smaller amount (less than 2.0%) has an insufficient effect on the properties of the final product, whereas an excess (more than 3.0%) makes the structure heterogeneous, dense, with an intensely pronounced color and aroma of cocoa fiber. The influence of the optimal amount of cocoa fiber addition on the change in dynamic viscosity of the cream-plant mixture was determined, as well as the change of this indicator depending on the temperature of the mixtures.

To determine the effect of the studied dietary fibers on the change in dynamic viscosity of cream-plant mixtures at different temperatures, mixtures with the addition of 2% and 3% cocoa fiber were prepared under the parameters identified in previous studies – the profile chart of sensory evaluation of the multicomponent albumin product. The heating temperature of the mixtures ranged from 20 °C to 80 °C. This temperature range was chosen due to the further development of technologies for multicomponent albumin products and for the purpose of destroying unwanted microflora. As the temperature rises, denaturation of the cream proteins occurs, leading to a decrease in the biological value of the cream-plant mixtures.

The change in dynamic viscosity of cream-plant mixtures with different amounts of cocoa fiber at temperatures from 20 °C to 80 °C is graphically presented in Figure 7.

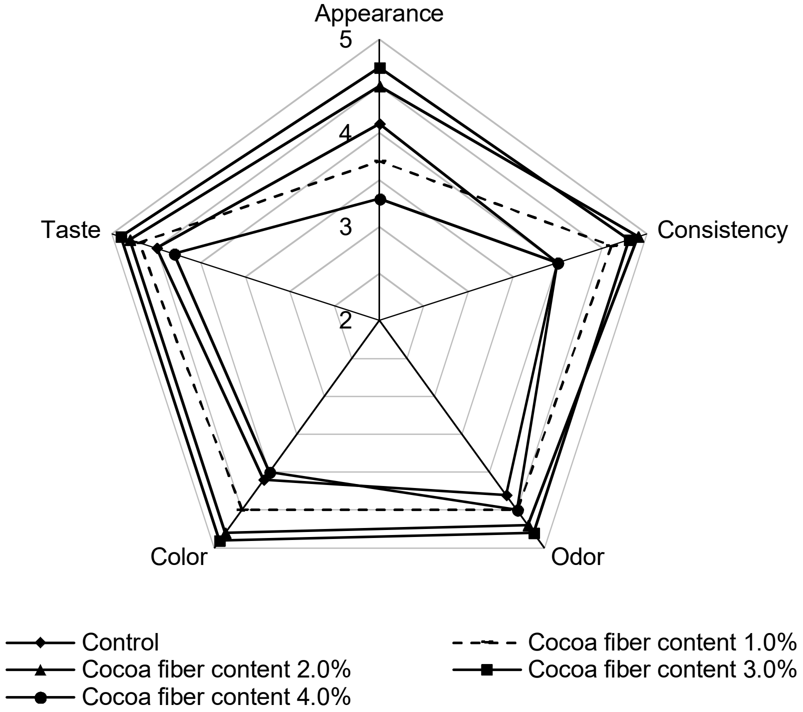

Figure 6. Sensory evaluation profile diagram of multi-component albumin products with different cocoa fiber contents

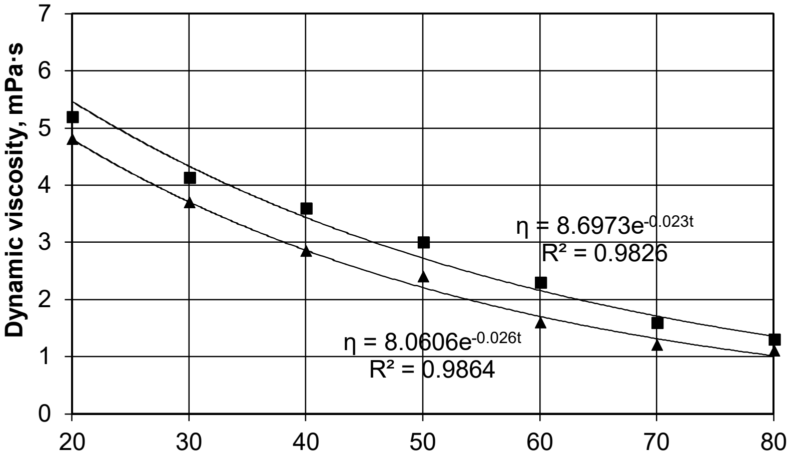

Figure 7. Change in dynamic viscosity of cream-plant mixtures with different contents of cocoa fiber at temperatures from 20 to 80 °C

As seen from Figure 8, the highest dynamic viscosity – 5.20±0.01 mPa·s at a temperature of 20 °C – was recorded for the cream-plant mixture with 3.0% cocoa fiber. With the addition of 2.0% fiber, this indicator at the aforementioned temperature was 4.80±0.01 mPa·s. Creams with a fat content of 10–20% approach Newtonian fluids, but when the fat content exceeds 30%, they become significantly structured and acquire non-Newtonian fluid properties (Kyazze et al., 2004; Šestan et al., 2016). As the temperature of cream-plant mixtures with cocoa fiber increases, dynamic viscosity decreases by 3.7-3.9 mPa·s, respectively, due to the increased Brownian motion of solution molecules and the weakening of interaction forces between them. The viscosity of the studied cream-plant mixtures significantly depends on the content of milk fat and proteins, particularly whey proteins, which correlates with the data of Biglarian et al. (2022). With further influence of high temperature on milk proteins due to the increase in the size of casein particles and complex formation between whey protein fractions and casein (Sutariya et al., 2017), the viscosity of cream-plant mixtures decreases. The decrease in viscosity of cream-plant mixtures during prolonged heat treatment can also result from partial destabilization of the milk fat emulsion. This pattern is observed in all samples of mixtures with plant-based ingredients. The presence of carbohydrates, including soluble (pectins) and insoluble fiber, as well as mineral compounds in the cocoa fiber composition, additionally binds free water and improves the structuring of cream-plant mixtures.

The effect of varying amounts of cream-plant mixtures with cocoa fiber on the physicochemical indicators of multicomponent albumin products was also studied. The corresponding values are presented in Table 5.

Table 5

Main physicochemical indicators of multicomponent albumin products with cocoa fiber

Amount of added cream-plant mixture,%	Mass fraction,%		Moisture retention capacity,%	Active acidity, pH units
	Dry matter	Moisture		
10	25.6±0.2^{a}	74.4±0.2^{a}	50.11±0.10^{a}	5.16±0.01^{a}
15	26.5±0.1^{ab}	73.5±0.3^{ab}	49.20±0.15^{ab}	5.24±0.02^{ab}
20	27.4±0.1^{b}	72.7±0.2^{b}	48.60±0.13^{b}	5.32±0.01^{b}

Note: Values are the means ± standard deviation. Means within the same column with different superscripts are significantly different at $p \leq 0.05$.

It was found that with an increase in the content of cream-plant mixtures, the active acidity of the multicomponent albumin products slightly increased – from 5.00±0.02 pH units to 5.32±0.01 pH units.

From the obtained values of water-holding capacity (Table 5), it can be seen that with an increase in the content of cream-plant mixtures, this indicator changes for all multicomponent albumin products, with the growth dynamics varying. This is due to the amount of added soluble fiber, protein, and other substances that ensure high solubility (Chico, 2022; Younes et al., 2023) and indicates the ability to increase the viscosity of cream-plant mixtures, as well as having high dispersibility (99% <40 μm) (Soares et al., 2022). When cream-plant mixtures were added at levels between 15% and 20%, the difference in water-holding capacity among the experimental samples was less pronounced, amounting to just 0.6%. The highest WHC values were observed in samples with the addition of 10% cream-plant mixtures. In addition, these multicomponent albumin product samples were characterized by a dense consistency.

Conclusions

The possibility of effective use of cocoa fiber and bird cherry flour in the composition of cream-plant mixtures for normalization in the technology of multicomponent albumin products has been proven.

The solubility, water- and fat-holding capacities of cocoa fiber and bird cherry flour were determined. The solubility in water, whey, and cream of different fat content was recorded for cocoa fiber at the level of: in water – 22.0±0.3%, in whey – 20.4±0.2%, in cream with 10% fat – 17.1±0.2%, and in cream with 20% fat – 15.5±0.1%. For bird cherry flour, the corresponding values were: in water – 19±0.1%, in whey – 17.3±0.1%, in cream with 10% fat – 14.5±0.1%, and in cream with 20% fat – 13.7±0.1%, respectively. The highest fat-holding capacity was shown by cocoa fiber in cream with 10% fat content (3.3 g/g) compared to cream with 20% fat. For bird cherry flour, the mentioned indicators were recorded at the level of 2.8±0.1 g/g and 1.8±0.1 g/g in cream with 10% and 20% fat content, respectively, and the water-holding capacity was sufficiently high – 3.2±0.1 g/g in milk whey.

The physicochemical and sensory parameters of multicomponent albumin products depending on the amount of cocoa fiber or bird cherry flour added were studied. The appropriate sensory indicators were observed in products with the addition of 3.0-5.0% bird cherry flour and 2.0-3.0% cocoa fiber. The samples had stable quality indicators, an increased content of biologically active components, and original sensory properties.

By means of thermogravimetry and differential thermal analysis, it was determined that the dehydration of multicomponent albumin product samples with bird cherry flour and albumin product samples occurs in two stages due to the composite composition and structural changes. These stages differ in temperature intervals, the proportion of removed water, and the rate of removal. Due to the sufficiently high content of bound water in the materials of both multicomponent albumin product samples, the specific heat of their dehydration significantly exceeds the average heat of water evaporation from a free surface. The addition of bird cherry flour to the composition of the albumin product increases the product's hydrophilicity, which leads to an increase in the specific heat of dehydration.

The physicochemical parameters of multicomponent albumin products depending on the amount of cream-plant mixtures with cocoa fiber were studied. It was established that with an increase of the amount of cream-plant mixtures from 10% to 20% in multicomponent albumin products, the content of dry substances increases from 23.0±0.3% (for control) to 27.4±0.1% and the active acidity up to 5.32±0.01%. At the same time, the moisture content and water-holding capacity decrease to 72.7±0.2% and 48.60±0.13%, respectively.

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Cite:

UFJ Style

Grek O., Tymchuk A., Bogachuk A., Soloviov N., Shumylo O. (2025 Effect of cream-plant mixtures on properties of multicomponent albumin-based products, *Ukrainian Food Journal*, 14(2), pp. 220–237, https://doi.org/10.24263/2304-974X-2025-14-2-4

APA Style

Grek, O., Tymchuk, A., Bogachuk, A., Soloviov, N., & Shumylo, O. (2025). Effect of cream-plant mixtures on properties of multicomponent albumin-based products. *Ukrainian Food Journal, 14*(2), 220–237. https://doi.org/10.24263/2304-974X-2025-14-2-4

Meat cutlets with iron oxide nanoparticles and alga *Laminaria japonica*

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Abstract

Introduction. The aim of the study was to produce beef cutlets with enhanced biological value using a complex food additive based on iron oxide nanoparticles and dried powder of the brown alga *Laminaria japonica* (kelp).

Materials and methods. A complex food additive was incorporated at levels of 0.1, 0.2, and 0.3% by weight into the beef cutlet formulation. Physicochemical, structural-mechanical, functional, and technological properties of the minced beef and the cutlets made from it were evaluated using standard methods.

Results and discussion. The complex food additive (CFA) was incorporated into minced beef in powder form during the mixing process. It was found that adding the CFA at a level of 0.2% significantly improved the sensory characteristics of the cutlets compared to the control: appearance (shape) improved by 1.15 times, consistency by 1.18 times, color intensity increased by 6%, and the overall sensory score rose by 17.6%. Simultaneously, the structural and mechanical properties of the minced meat improved: the instantaneous elastic modulus increased by 1.20 times, and the high-elastic modulus by 1.16 times. Functional and technological properties were also enhanced, with water-holding capacity increasing by 1.33 times; water- and fat-binding capacities by 1.30 and 1.27 times, respectively; and fat-holding and fat-emulsifying capacities by 1.29 and 1.27 times, respectively. An improvement in emulsion stability by 1.25 times, along with reduced moisture and fat losses during heat treatment, led to a 5.5% increase in the yield of the final product. The overall digestibility of cutlet proteins increases by 1.12 times, while the acid value decreases by 2.2 times and the peroxide value by 1.6 times. These changes, along with the bacteriostatic properties of the complex food additive, contribute to the stability of the product qualities during storage. The content of protein, starch, dietary fiber, some mineral elements and vitamins increased in the beef cutlets. Based on sensory, structural-mechanical and physicochemical analyses, the optimal concentration of the complex food additive was determined as 0.2% of the total recipe mass. It provides a meat product enriched with iodine to a level of 75±10 mcg per 100 g, which covers more than 50% of the recommended daily allowance for an adult. In addition, it increases the iron content by 27.8% compared to the control sample, which allows it to satisfy more than 10% of the daily requirement for this trace element.

Conclusions. A complex food additive based on iron oxide nanoparticles and kelp powder can be used in the formulation of minced meat products to stabilize their structure and improve consumer properties. Due to the presence of valuable nutritional components, it also enhances the biological and nutritional value of beef cutlets.

Keywords:

Meat
Iron
Nanoparticles
Laminaria japonica
Iodine

Article history:

Received
30.08.2024
Received in revised form
12.02.2025
Accepted
30.06.2025

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DOI:
10.24263/2304-974X-2025-14-2-5

Introduction

There has been a growing interest in balanced nutrition and a healthy lifestyle, which has led to an active search for new, healthy food sources, among which seaweed is attracting particular attention. According to the Food and Agriculture Organization (FAO) of the United Nations, approximately 600 species of macroalgae are used in human nutrition worldwide, while in Europe, approximately 200 species have proven food and commercial potential (FAO, 2022; Lähteenmäki-Uutela et al., 2021). The growth of consumer interest in algae has led to the formation of a new concept in the food industry – "phyco-gastronomy" (Milinovic et al., 2021). Among the most popular species in global food production, algae of the genera *Laminaria* and *Saccharina* hold leading positions, accounting for over 34.6% of total production. They are mainly used in the preparation of salads, sauces, and seasonings (Peñalver et al., 2022). In addition to its high nutritional value, seaweed consumption is associated with positive health effects, including reduced blood pressure and prevention of cardiovascular disease (Cotas et al., 2022). In the context of global population growth and increasing scarcity of natural resources, seaweeds are considered a sustainable and promising resource that can enhance food security. They can be grown in seawater, which does not compete with arable land or freshwater resources, making their widespread use possible in both the food industry and aquaculture (Costa et al., 2021, 2024; Farghali et al., 2021).

Improving the nutritional value of meat products is a pressing issue in research and development in food science and nutrition, driven by the need to promote sustainable and efficient protein consumption by the population, as well as the possibility of increasing the biological value of meat products (Maikova et al., 2022; Masliychuk et al., 2024). One promising solution is the addition of non-traditional components to the meat products, in particular seaweeds such as a brown alga *Laminaria* (Choi et al., 2012; Kryzhova et al., 2021; Stabnikova et al., 2025; Vognivenko and Kachur, 2024). Thanks to its rich mineral and amino acid profile, laminaria enhances minced meat products by adding biologically active compounds, improving both their nutritional value and consumer appeal (Alisha and Aisha, 2019; Bondar et al., 2019; Gullón et al., 2019; Moroney et al., 2013; 2015; Tagliapietra and Clerici, 2023).

Laminaria, commonly known as kelp, is a rich source of several essential minerals, including iodine, phosphorus, calcium, and iron. It contains up to 1000 µg of organically bound iodine per 100 g of dry matter, which is readily absorbed from seawater. Additionally, it provides approximately 45 mg of phosphorus, 170 mg of calcium, and 3.0 mg of iron per 100 g of dry matter.

The polysaccharides found in algae are characterized by high hydration and adsorption capacities, enabling them to effectively bind and remove toxins from the human body. The sulfated heteropolysaccharide fucoidan acts as a cholesterol antagonist, helping to reduce cholesterol deposits in blood vessels. Additionally, laminaria exhibits anticoagulant properties and has a positive effect on the restoration of digestive system functions as well as on skin health (Costa et al., 2021; Milinovic et al., 2021; Moroney et al., 2013, 2015; Salido et al., 2024).

The inclusion of algae in meat and other food products helps to enrich them with biologically active compounds, in particular proteins, dietary fiber, carbohydrates, vitamins and minerals (Cotas et al., 2024; Stabnikova et al., 2021). The use of algae as food additives imparts functional properties to finished products, including sorption, hydration, radioprotective, antihypertensive, antidiabetic, antioxidant, anti-inflammatory, antitumor, antiviral, antimicrobial activity, as well as the ability to form complexes (Babich et al., 2022; Biancarosa et al., 2018; Cotas et al., 2022; Rogel-Castillo et al., 2023; Salido et al., 2024).

This makes the use of algae as an innovative, non-traditional raw material scientifically and technologically feasible for developing new types of food products, particularly minced meat products. Despite existing studies that have examined the use of edible seaweeds in meat dishes, in particular: laminaria (*Laminaria* sp.), wakame (*Undaria pinnatifida*), nori (*Porphyra umbilicalis*) and sea spaghetti (*Himanthalia elongata*) (Gohara-Beirigo et al., 2021; Cofrades et al., 2017; Peñalver et al., 2020), the specifics of the use of laminaria in meat product technologies, in particular cutlets, are insufficiently covered.

The complex food additive (CFA), consisting of dry kelp biomass powder and iron oxide ($FeO \times Fe_2O_3$) nanoparticles (IONPs), is a finely dispersed powder with a particle size of 0.2 mm, greenish-brown in color, with a characteristic taste and smell of algae (Figure 1) (Tsykhanovska et al., 2024a, 2025).

Figure 1. Complex food additive

The aim of the study was to produce beef cutlets with enhanced biological value using a complex food additive based on iron oxide nanoparticles and dried powder of the brown alga *Laminaria japonica*.

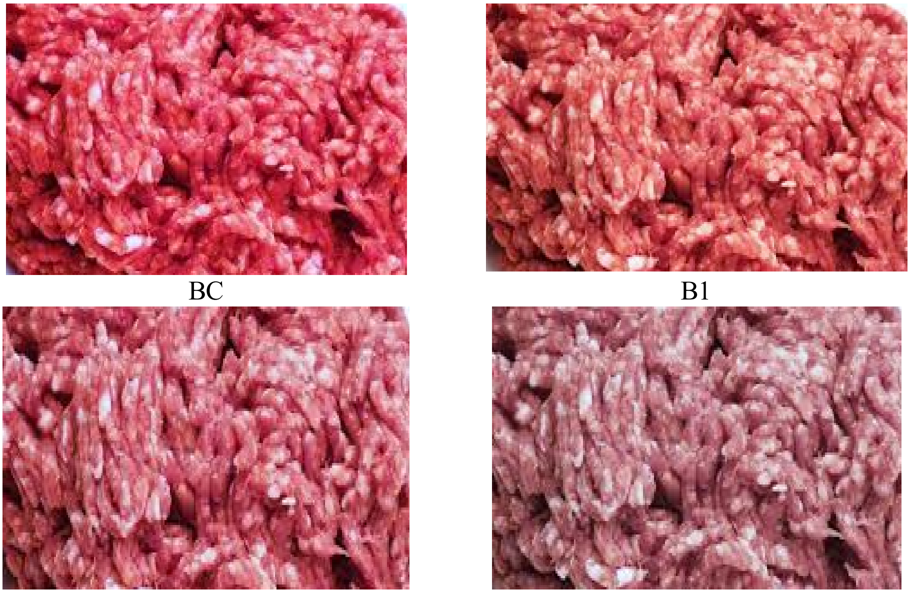
Materials and methods

Chemicals and raw materials. All reagents used for the synthesis of $FeO \times Fe_2O_3$ nanoparticles and for the analyses were provided by Merck (Darmstadt, Germany). Food powder of kelp (*Laminaria japonica*) with a moisture content of less than 12% and a crude protein content of 11.4% was purchased from Fuzhou Beautiful Agricultural Development Co., Ltd (Fujian, China). Premium or first grade beef, extra-fine cooking salt ("TM Dr. Igel", Ukraine), wheat bread (JSC "Kharkiv Bread Factory "Slobozhanskyi""), extra-class Grass-Fed beef rendered fat ("Steaks of the Carpathians"), and 2.5% fat milk (LLC "Agromol", Ukraine) were purchased at local markets in Kharkiv, Ukraine.

Preparation of a complex food additive. Preparation of a complex food additive (CFA) containing dry kelp biomass powder and iron oxide ($FeO \times Fe_2O_3$) nanoparticles is described in detail in (Tsykhanovska et al., 2024a). The CFA contained 15% (w/w) iron oxide nanoparticles (IONPs) and 85% (w/w) laminaria powder and was used as an ingredient in the recipe for beef cutlets.

Production of beef cutlets. To prepare the control sample of beef cutlets (designated as BC), a traditional basic recipe was used, consisting of: beef — 74.0 g; wheat bread — 18.0

g; breadcrumbs — 8.0 g; table salt — 1.0 g; cooking fat — 6.0 g; and water or milk — 24.0 g. The total mass of the minced mixture was 125.0 g, yielding 100.0 g of the finished product. In the developed versions of cutlets, a complex food additive (CFA) was added to the recipe in an amount of 0.1, 0.2, and 0.3% (w/w) of the total mass of the mixture. Accordingly, these samples were designated as B1, B2, and B3 (Figure 2).



BC B1

B2 B3

Figure 2. Minced beef with a complex food additive,% (w/w):
0 (BC); 0.1 (B1); 0.2 (B2), and 0.3 (B3)

Minced meat was obtained by grinding beef and wheat bread swollen in water/milk in an industrial meat grinder MIM-300 Arm-Eco (“Arm-Eco”, Ukraine) followed by mixing with salt, spices and CFA in a meat mixer Farshemis Hendi 282670 (“Hendi”, Netherlands) for 3-5 minutes and keeping the recipe mixture for 8-10 minutes. The minced meat samples were packed in airtight bags and stored at a temperature of (4±1)°C for further physicochemical and structural-mechanical studies.

The heat treatment of the samples was carried out by the method of basic frying at a surface temperature of 150–160°C until culinary readiness was achieved, which corresponded to a temperature of 85±1°C in the center of the product. After cooking, the cutlets were left at room temperature for 2 hours, and then packaged in airtight bags and stored at a temperature of (4±1)°C for further physicochemical analyses and sensory evaluation.

Minced meat characteristics

Functional and technological characteristics of minced beef

Water-binding capacity (WBC),*%,* was determined by pressing according to the Grau and Ham method: 0.3 g of minced meat was weighed on a polyethylene disc with a diameter of 15–20 mm, after which it was transferred to an ashless filter placed on a glass plate so that the disc with minced meat was under it. Another glass plate is placed on top of the minced meat, on which a load of 1 kg is placed, and kept for 10 minutes. After that, the load and the upper plate are removed, and then the contour of the spot of pressed minced meat is traced with a pencil. The content of bound moisture (in% of the total amount of water) is calculated by the formula (1):

$$WBC = \frac{(m - 8.4S)}{m} \times 100\% \tag{1}$$

where WBC is the content of bound moisture to total moisture,%; m is the total moisture content in the sample, mg; S is the area of the wet spot, mm^2; 8.4 is the empirical coefficient that reflects the ratio of the area of the wet spot to the amount of moisture that has passed into the spot.

Fat-binding capacity (FBC),%, was determined according to the method described in (Tornberg, 2005). Samples of minced meat weighing 10 g were placed in pre-weighed centrifuge tubes (m_0), after which the tubes were hermetically sealed and kept in a water bath at a temperature of 70°C for 30 minutes.

The tubes were cooled to room temperature and centrifuged for 10 minutes at 3000 rpm. After centrifugation, the separated fat was carefully drained, and the tubes with the residue were weighed again (m_1, g). The fat-binding capacity (FBC,%) was calculated using the formula (2):

$$FBC = \frac{(m_1 - m_2)}{(m_0 - m_2)} \times 100\% \tag{2}$$

where m_0 is the mass of the test tube with minced meat before heat treatment, g, m_1 is the mass of the test tube with minced meat after centrifugation, g, m_2 is the mass of the empty test tube, g.

Water-holding capacity (MHC), **Fat-binding capacity (FBC)**, **Fat emulsion capacity (FEC),** and **Fat emulsion stability (FES)** of minced meat were determined according to the methods described in (Tsykhanovska et al., 2018). Minced meat weighing 180–200 g is placed in hermetically sealed tin cans No. 3, weighed and subjected to heat treatment according to the production regime: cooked in a water bath at 78–80°C for 1 hour, and cooled under running water to 12–15°C. The cans are then opened and the separated liquid (broth and fat) is poured into pre-weighed aluminium bottles. The mince is dried with filter paper and weighed. The weighing bottles with broth are placed in a drying cabinet and dried to a constant weight at 103–105°C. Based on these data, the moisture released during heat treatment and the moisture-holding capacity of the mince are determined. From the weighing bottles with the remains of broth and fat, the fat is extracted with 10–15 ml of solvent (chloroform: ethanol=1:2), performing the extraction for 3–4 minutes with three to four repetitions. Having determined the content of fat remaining in the mince after processing, its fat-holding capacity (FRC) is calculated.

Water-holding capacity (WHC),% of the minced meat weight, was determined using the formula (3):

$$WHC = [W - (\frac{m_1 - m_3}{m_2 - m})] \times 100\% \quad (3)$$

where WHC is the water-holding capacity,% of the minced meat mass; W is the moisture content of the minced meat,%; m is the mass of the minced meat, g; m_1 is the mass of the separated broth with fat, g; m_2 is the mass of the liquid before drying (i.e., the total mass of the broth before drying in a box), g; and m_3 is the mass of moisture contained in the separated broth after drying.

The fat-holding capacity (FHC),% of the minced meat mass, was determined by the formula (4):

$$FHC = [(F_{mm} - (\frac{m_1 - m_3}{m_2 - m})] \times 100\% \quad (4)$$

where FHC is the fat-holding capacity of minced meat,% to the of minced meat mass; F_{mm} is the content of fat in minced meat, g; m is the mass of minced meat, g; m_1 is the mass of the separated broth with fat, g; m_2 is the mass of the liquid (broth with fat) before drying (i.e., the total mass in the box before drying), g; m_3 is the mass of fat released during heat treatment, g (determined after extraction from the box using a chloroform-ethanol mixture).

The fat emulsifying stability (FES) was determined according to the method described in (Tsykhanovska et al., 2018). Minced meat weighing 7 g is suspended in 100 ml of water in a homogenizer at a speed of 800 rpm for 1.0-1.5 minutes. 100 ml of refined sunflower oil is added and the mixture is emulsified in a homogenizer at a speed of 1500 rpm for 5 minutes. 100 ml of refined sunflower oil is added and emulsify the mixture in a homogenizer at a speed of 1500 rpm for 5 minutes is provided. After that, the emulsion is poured into 4 calibrated centrifuge tubes with a volume of 50 ml and centrifuged at a speed of 900 rpm for 10 minutes. The fat emulsifying capacity, FFC (%) was determined by the formula (5):

$$FEC = \frac{V_1}{V} \times 100\% \quad (5)$$

where V_1 is the volume of emulsified oil, ml; V is the total volume of oil, ml.

Emulsion stability (ES) was determined according to the method described in (Tsykhanovska et al., 2018). Minced meat, 10 g, was heating at 80°C for 30 minutes and cooling with water for 15 minutes. The resulting emulsion was poured into four calibrated centrifuge tubes with a volume of 50 ml and centrifuged at a speed of 500 s^{-1} for 5 minutes. The volume of the emulsified layer was measured. The emulsion stability, ES,%, was calculated by formula (6):

$$ES = \frac{V_1}{V_2} \times 100\% \quad (6)$$

where V_1 is the volume of emulsified oil, ml; V_2 is the total volume of the emulsion, ml.

Structural and mechanical properties of minced meat

The instantaneous elastic modulus and the highly elastic modulus were determined using the Tolstoy elastoplastometer (Tsykhanovska et al., 2018). Samples of cooked minced meat with a volume of no less than 50 cm³ (one-piece, without shaking) at a temperature of (4±1)°C were placed in a cuvette with a device, evenly distributing the samples and any visible excess behind a spatula. The specimen is pressed using a 2 mm diameter tip to a depth of 1 mm at a speed of 0.5 mm/s and the maximum resistance of the specimen during the first contact (peak pressure) is recorded on the load curve. The calculation of the instantaneous elastic modulus (E_0, Pa) is carried out according to formula (7):

$$E_o = \frac{F_{\max}}{(A \times \delta)} \tag{7}$$

where F_{max} is the maximum resistance force, N; A is the contact area, m^2; δ is the indentation depth, m.

When determining the high elastic modulus, after reaching an indentation depth of 1 mm, hold the sample in a stationary state for 30 seconds. Record the change in the resistance force over time while holding the load. In the relaxation region of the force (from 5 to 30 s), calculate the highly elastic modulus (E_h, Pa) using equation (8):

$$E_h = \frac{1}{(t_2 - t_1)} \int_{t_1}^{t_2} \frac{F_{(t)}}{(A \times \delta)} \times dt \tag{8}$$

where F(t) is the resistance force at time t, N; t_1 and t_2 are the boundaries of the relaxation interval, seconds.

Physicochemical parameters of minced meat. Acid number (AN, mg KOH/g fat) and peroxide number (PN, mmol ½O_2/kg fat) were determined titrimetrically using the methods of the Association of Official Analytical Chemists (AOAC, 2017). The acid number was determined by the formula (9):

$$AN = \frac{V \times C \times 56{,}1}{m} \tag{9}$$

where V is the volume of the titrant – KOH, ml; C is the concentration of the titrant – KOH, mol/l; 56.1 is the molar mass of KOH, g/mol; m is the mass of the sample, g.

The peroxide value was determined by the formula (10):

$$PN = \frac{(V_1 - V_0) \times C \times 1000}{m} \tag{10}$$

where PN is the peroxide number, mmol of active oxygen (½O_2) per 1 kg of fat; V_1 is the volume of $Na_2S_2O_3$ solution used for titration of the sample, ml; V_0 is the volume of $Na_2S_2O_3$ used for titration of the blank experiment (control), ml; C is the molar concentration of $Na_2S_2O_3$, mol/l; m is the mass of the sample,

Beef cutlets quality assessment

Sensory quality indicators. Sensory evaluation of the quality of beef cutlets (appearance – shape, consistency, color, taste, smell) was carried out on a 5-point scale using the profile method.

Physicochemical characteristics of beef cutlets. Total titrated acidity (TA), active acidity (pH units), moisture content (%) were determined by standard methods: the content of moisture was determined by the drying method; losses during heat treatment were determined by the calculation method after weighing the samples; pH was determined by the potentiometric method; total titrated acidity was determined titrimetrically: 0.1 M sodium hydroxide solution was used as the titrant, and phenolphthalein was used as the indicator. For analysis, 25.00±0.01 g of beef party were transferred to a 500 ml flask, 50 ml of distilled water was added and thoroughly ground to a pulp. Then another 200 ml of water was added, the flask was shaken for 5 min, after which the mixture was allowed to settle for 30 min and filtered. The supernatant was titrated with an alkaline solution, and the acidity was expressed in degrees. The yield of finished products was determined immediately after completion of the technological production process using formula (11):

$$X = \frac{A}{B} \times 100\% \tag{11}$$

where X is the yield of the finished product,%; A is the mass of the finished product, g; B is the mass of minced meat, g.

Proximate analysis of beaf cutlets. The protein content was determined by the Kjeldahl method (ISO 1871:2009, 2009), and a conversion factor of 6.25 was used to calculate the protein. The fat content was determined by the Soxhlet extraction method (ISO 11085:2015, 2015), the fat was extracted with petroleum ether with a boiling range of 40–60°C and determined gravimetrically.

The moisture and ash contents of the samples were determined according to official procedures 44-16.01 Moisture-Air-Oven (Aluminum Plate) and 08-01.01 Ash-Basic Method, respectively (AACC, 2000). Results are presented on a dry weight (dw) basis.

Vitamin content analysis was performed using a high-performance four-channel Agilent 1100 liquid chromatograph (Agilent Technologies, USA) in combination with a diode array detector (DAD) and mass spectrometry (MS) according to the method (Katsa et al., 2021; Sim et al., 2016).

The mineral composition of the cutlets was determined using an inductively coupled plasma atomic emission spectrometer Thermo iCAP 6300 Duo ICP-AES Spectrometer (Thermo Scientific, US) with operating parameters as in (Yurchenko et al., 2020).

Sample preparation was carried out according to (Bilgiçli and İbanoğlu, 2015) without modifications. Determination of iodine content in samples was carried out by galvanostatic coulometric titration using equipment (Gubskyi, 2023) according to the method (Gubskyi et al., 2015). Previously examined samples were subjected to mineralization by dry alkaline ashing to convert all chemical forms of iodine into iodide.

The heat resistance index (HRI), %, was determined by the iodine content in the experimental samples of beef cutlets according to the formula (12):

$$HRI = \frac{C_2}{C_1} \times 100\% \tag{12}$$

where C_1 is the iodine content before heat treatment (in minced meat), μg/g; C_2 is iodine content after heat treatment (in cutlets prepared from minced meat), μg/g.

Evaluation of the biological value of beef cutlet proteins in vitro. The biological value of beef cutlet proteins was assessed by the degree of hydrolysis of cutlet proteins under the action of proteolytic enzymes (pepsin and trypsin) according to the method (Brodkorb et al., 2019; Cutroneo et al., 2023; Minekus et al., 2014). A sample of 1.0 g of crushed meat cutlet was placed in a 50–100 ml test tube, 10 ml of 0.1 N HCl solution and 1 mg of pepsin/ml of solution were added (simulating the gastric phase). The mixture was then incubated at 37°C for 2 hours with occasional stirring. The pH was neutralized to ≈7.0 (by adding 0.1 N NaOH solution). The intestinal phase (trypsin) was simulated by adding 10 ml of buffer solution (pH 8.0) and 1 mg of trypsin/ml of solution, followed by incubation at 37C for another 2 hours. After that, the degree of protein hydrolysis was determined – according to the first method: the samples were centrifuged (3000 rpm, 10 minutes) and the optical density was measured in the supernatant at λ=280 nm (solution diluted 1:10); according to the second method (alternative): the method with biuret reagent or TNBS (trinitrobenzenesulfonic acid, TNBS) was used to quantitatively determine the released amino groups after hydrolysis. The calculation of biological value (hydrolysis index, HI, %) was carried out according to the formula (13):

$$HI = \frac{A_1}{A_2} \times 100\% \tag{13}$$

where A1 is the optical density of the test sample after hydrolysis; A2 is the optical density of a standard or fully hydrolysed sample (e.g., casein or albumin).

Microbiological analysis of beef cutlets

The following media were used to conduct microbiological studies: nutrient agar for counting the total number of microorganisms, namely the number of aerobic mesophilic and facultative anaerobic microorganisms (MAFAnM); potato-dextrose agar for the enumeration of yeast and mold; salmonella-shigellosis agar for the determination of *Salmonella* spp.; endo-agar for the determination of *Escherichia coli*; Kessler's medium for the determination of coliforms. Microbiological analysis was performed according to generally accepted methods of microbiological analysis of food products (Erkmen, 2022; Hasell et al., 2003; Olunlade et al., 2013).

Statistical analysis

A one-way analysis of variance (ANOVA) was used for a series of parallel measurements (at least 3). Data in the tables represent the mean ± standard deviation. A p value < 0.05 was considered statistically significant. Tukey's HSD test was used to determine significant differences between means. Basic statistics and ANOVA were performed using the Minitab statistical software package version 18.1 (Minitab Inc., USA).

Results and discussion

To select the optimal concentration of complex food additive (CFA) in the recipe for beef cutlets, a sensory assessment of cutlets with different amount of the additive, % (w/w): 0 (BC); 0.1 (B1); 0.2 (B2), and 0.3 (B3), was carried out (Table 1). The finished products were characterized by a color typical for split meat products, a soft and tender consistency, juiciness, a pleasant taste of fried meat and an expressive spicy-sea aroma.

Table 1

Sensory assessment of the quality of beef cutlets in points

Characteristics	Beef cutlets			
	BC	B1	B2	B3
Appearance (shape)	4.4	4.8	5.0	4.9
Consistency	4.3	4.9	5.0	4.9
Color	4.2	4.9	5.0	4.9
Taste	4.2	4.8	5.0	4.7
Smell	4.2	4.9	5.0	4.7
Overall score	21.3	24.3	25.0	24.4

The addition of the CFA improves the shape and consistency of the cutlets, enhances their color saturation, and gives them a pleasant, rich taste and aroma, resulting in higher sensory ratings for all experimental beef cutlet samples compared to the control. According to the results of the expert assessment, the highest scores were given to the sample with the addition of 0.2% of the complex food additive from the mass of the recipe mixture. This sample was identified as the optimal one for the subsequent incorporation of the additive. in the cutlet recipe.

During the formation of a semi-finished meat cross-section product, when the geometric shape of the culinary product is set, the cohesive properties of iron oxide nanoparticles (IONPs), which are part of the complex food additive (CFA), are manifested. This facilitates the cross-linking of biopolymer molecules – proteins, carbohydrates, and lipids – leading to the formation of a stable food spatial matrix. As a result, the ratio of meat raw materials to bread in the beef cutlets recipe can be adjusted when adding the CFA. At the stage of frying experimental samples of minced beef meat enriched with the CFA, better preservation of the shape of finished products and a decrease in weight loss were recorded, which is explained by the pronounced water- and fat-retaining properties of the additive.

An increase in the adhesion of the products to the frying surface was also observed, which can be attributed to the hydration, structure-forming, and stabilizing activities of the iron oxide nanoparticles (IONPs) and laminaria proteins present in the CFA, as well as the migration of moisture from the surface layers into the deeper layers of the product (Tsykhanovska et al., 2018; Yurchenko et al., 2018).

These conclusions are confirmed by the results of studies of the influence of a complex food additive incorporation on the functional and technological properties of minced beef (BC, B1, B2, B3, B4), namely: water-binding capacity (WBC),%, fat-binding capacity (FBC),%; the level of losses during its heat treatment (losses during heat treatment),%, and the yield of finished products (yield),%) (Fig.3).

It has been shown (Figure 1) that the introduction of a highly dispersed complex food additive into minced beef at levels of 0.1%, 0.2%, and 0.3% of the recipe mixture weight increases the product's ability to retain moisture and fat by an average of 1.30 times and 1.27 times, respectively, compared to the control. This, in turn, reduces losses during heat treatment and increases the yield of finished cutlets by 5.5%. The best results were achieved with the introduction of CFA at a concentration of 0.2%; further increasing the amount of the additive to 0.3% does not significantly improve the indicators.

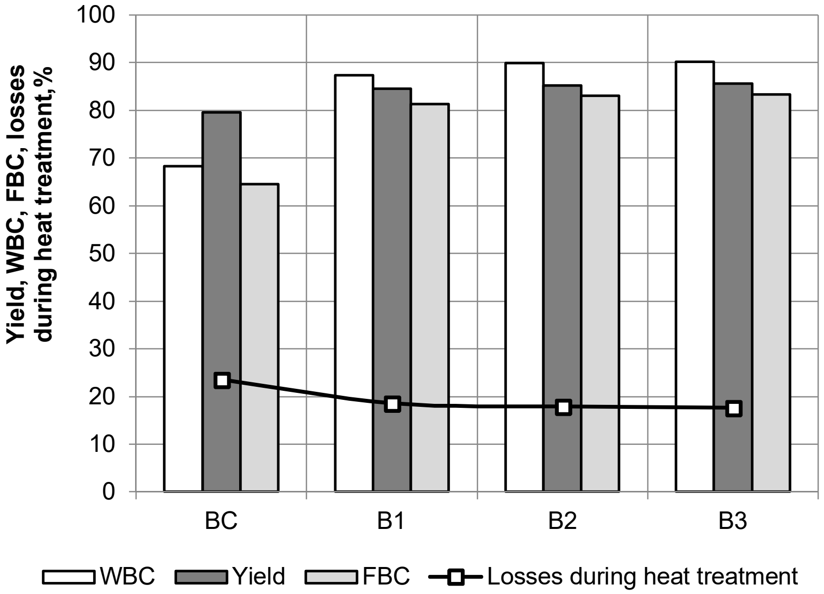
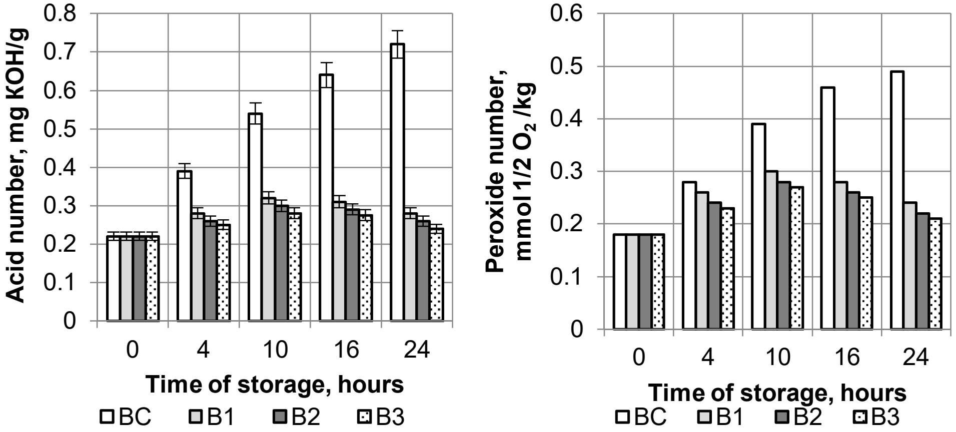


Figure 3. The effect of a complex food additive addition in minced beef, % (w/w): 0 (BC); 0.1 (B1); 0.2 (B2), and 0.3 (B3), on its functional and technological properties. WBC, water-binding capacity; FBC, fat-binding capacity

A study of oxidative processes in the fat component of minced beef showed the antioxidant effect of a complex food additive (CFA). CFA was added to the mince recipe in different quantities, the minced beef samples were stored for 24 hours at a temperature of (4±1)°C, and measurements of acid (AN, mg KOH/g of fat), and peroxide (PN, mmol $1/2O_2$/kg of fat), numbers were carried out during storage (Figure 4).

The values of acid number and peroxide number in all samples of minced beef during storage increase within the limits established by regulatory documentation: AN – no more than 3.5–4.0 mg KOH/g of fat (ISO 660:2020, 2020); PN – no more than 5 mmol $1/2O_2$/kg of fat (ISO 3960:2017, 2017), but increase more slowly in samples with the addition of CFA compared to the control: the AN was 2.2 times lower, and the PN was 1.6 times lower. This is due to the antioxidant properties of *Laminaria* (natural antioxidant content) and IONPs $FeO{\times}Fe_2O_3$. The formation of intermediate complexes between the iron atoms of IONPs and the oxygen atoms of peroxide radicals and hydrogen peroxides delays oxidative reactions. While, stabilization of fats by IONPs in a supramolecular ensemble slows down the hydrolysis of fats and reduces the accumulation of free fatty acids. In addition, due to the amphoteric nature of Fe ions, large specific surface area and high sorption activity, IONPs adsorb part of the fatty acids, which extends the shelf life and improves the quality of chopped meat products (Tsykhanovska et al., 2018).

Analysis of the physicochemical parameters of beef cutlets showed that the introduction of CFA slightly reduces the pH of finished products, i.e. increases their active acidity, which is due to the natural content of organic acids in laminaria (Table 2).



a b

Figure 4. Oxidative processes in minced beef with complex food additive, % (w/w): 0 (BC); 0.1 (B1); 0.2 (B2), and 0.3 (B3) during storage: a – acid number; b – peroxide number.

Table 2

Physicochemical characteristics of beef cutlets

Indicators	Beef cutlets			
	BC	**B1**	**B2**	**B3**
Active acidity, pH	6.50±0.01^{a}	6.40±0.01^{b}	6.37±0.01^{bc}	6.35±0.00^{cb}
Titrated acidity, °T	0.86±0.01^{a}	1.05±0.01^{b}	1.10±0.01^{bc}	1.12±0.01^{cb}
Moisture content,%	35.9±1.40^{a}	36.60±0.60^{b}	37.10±0.60^{bc}	37.30±0.60^{cb}

$^{a\text{-}c}$ The mean values in the same row with different superscripts differ significantly when $p<0.05$

The presence of amphoteric iron in the iron oxide nanoparticles (IONPs), the iron-containing component of the additive, slows the increase in the active acidity (pH changes) of minced meat by 1.11–1.18 times compared to the control, including during the storage of minced beef for 72 hours at 4±1°C (Figure 5). This occurs due to the chemisorption of organic acids on the surface of IONPs and can serve as a basis for extending the shelf life of minced meat.

The addition of the CFA significantly increases the titrated acidity of minced meat by 1.22–1.27 times compared to the control, while the taste properties of the cutlets remain unchanged. Increased acidity enhances the binding and retention of water by meat proteins, thereby increasing the water-holding capacity. As a result, the proportion of bound water in the minced meat rises, improving the juiciness of the cutlets and reducing losses during frying. With increasing acidity and due to the moisture-binding properties of laminaria and IONPs, the content of free water decreases, and the total moisture content of finished products increases by 1.02–1.04 times compared to the control. In addition, with an increase in the content of the complex food additive from 0.1% to 0.3% in the samples of minced beef (compared to the control), a gradual increase in the functional and technological indicators is observed: water-holding capacity (WHC), fat-holding capacity (FHC), fat emulsion capacity (FEC) and fat emulsion stability (FES) (Table 3).

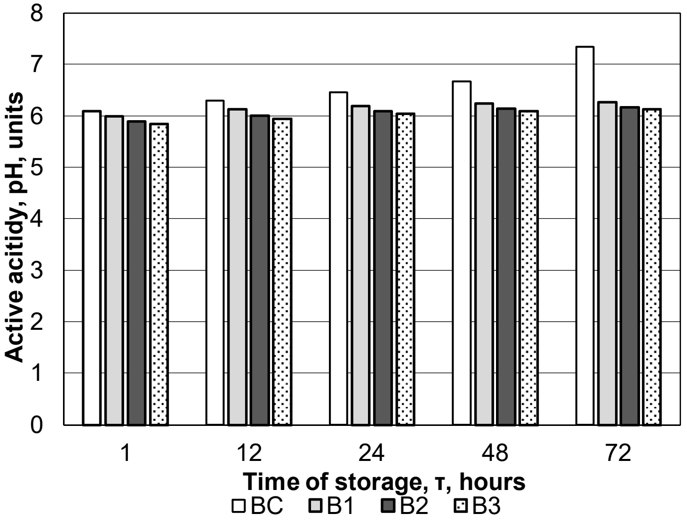


Figure 5. Change of active acidity in minced beef with complex food additive, % (w/w): 0 (BC); 0.1 (B1); 0.2 (B2), and 0.3(B3) during storage

Table 3
Functional and technological characteristics of minced beef samples

Minced beef samples	Characteristics			
	WHC,%	FHC,%	FEC,%	FES,%
BC	71.1±2.3^{a}	64.6±2.0^{a}	33.2±1.6^{a}	75.2±2.3^{a}
B1	88.9±2.5^{b}	81.2±2.4^{b}	41.2±1.8^{b}	91.9±2.7^{b}
B2	93.4±2.,8^{c}	83.5±2.4^{c}	43.6±1.8^{c}	93.4±2.9^{c}
B3	94.8±2.8^{dc}	84.9±2.5^{dc}	44.4±1.9^{dc}	94.2±3.0^{dc}

$^{a-d}$ The mean values in the same column with different superscripts differ significantly when $p<0.05$

It follows from results present in Table 3 that the functional and technological indicators increase as follows: WHC by 1.33 times, FHC by 1.29 times, EC by 1.27 times, ES by 1.25 times. This is explained by the sorption and structure-forming properties of laminaria proteins and $FeO \times Fe_2O_3$ nanoparticles (Costa et al., 2021; Kireeva et al., 2024; Mamat et al., 2014; Tsykhanovska et al., 2021, 2022a, b).

Consequently, compared to the control, positive changes in the physicochemical and functional-technological properties of the minced beef and the parties made from it are observed when a complex food additive (CFA) is introduced at levels of 0.1%, 0.2%, and 0.3% of the recipe mixture mass.

In particular, water loss during frying is reduced, which ensures a higher yield of the finished product; parties hold their shape well and retain their juiciness better due to an increase in the particle of bound water in the mince. The optimal content of CFA is determined to be 0.2% of the mass of the recipe mixture.

When studying the structural and mechanical properties of minced beef samples using an elastoplastometer, the instantaneous elastic modulus and the highly elastic modulus were determined (Table 4).

Table 4
Structural and mechanical properties of minced beef samples

Minced beef samples	Modulus of instantaneous elasticity, Pa	High-elastic modulus, Pa
BC	4428 ± 23^{a}	1596 ± 17^{a}
B1	4954 ± 28^{b}	1769 ± 17^{b}
B2	5202 ± 3^{c}	1827 ± 18^{c}
B3	5314 ± 32^{dc}	1852 ± 18^{dc}

$^{a-d}$ The mean values in the same column with different superscripts differ significantly when $p<0.05$

The addition of CFA improves these characteristics: the instantaneous elastic modulus, which reflects the ability of the minced meat to restore its original shape after loading, increases by 1.25 times compared to the control. This indicates an increased elasticity of the system due to modification by van der Waals interactions and coordination bonds with IONPs. In addition, the elastic modulus increased by 1.21 times compared to the control sample. So, the elasticity of the minced meat system is improved due to the high water-holding capacity of the complex food additive (algae-iron-containing carrier), which binds moisture and promotes the formation of solvate associates of IONPs with minced meat microparticles. As a result, a three-dimensional structural network is formed, capable of bending and returning to its original shape, which provides a soft texture and high shape-retention capacity of the beef mince. Thus, these studies provide a quantitative evaluation of the key rheological parameters required to optimize technological operations such as mixing, portioning, and forming in the production of minced meat products.

The biological value of the experimental samples of beef cutlets was assessed by the degree of digestion (hydrolysis index – HI,%) of the proteins of the beef cutlets (BC and B2) by proteolytic enzymes of the gastrointestinal tract in vitro (Figure 6).

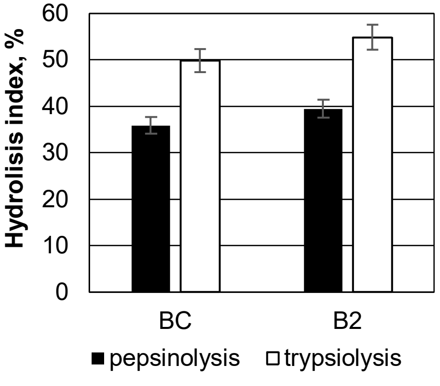


Figure 6. Enzymatic hydrolysis of proteins from beef cutlets

From Figure 6, it is evident that at both the pepsinolysis and trypsinolysis stages, the digestibility of proteins in the beef cutlets with the addition of 0.2% CFA (sample B2) exceeds that of the cutlets produced using traditional technology (control sample BC) by an average of 1.12 times. The overall digestion effect also increases by 1.12 times due to the activating effect of iron oxide nanoparticles on proteolytic enzymes of the gastrointestinal tract (pepsin and trypsin). This is due to their nano-size, significant specific volume, developed active surface, high affinity to proteins and the ability to form “protein-enzyme-nanoparticle $FeO \times Fe_2O_3$” complexes. Thus, the use of CFA in the minced meat system improves the digestion of finished proteins of meat products.

The introduction of CFA into minced meat also slows down the development of microorganisms. After 24 hours of storage at 4°C, the total number of aerobic mesophilic and facultative anaerobic microorganisms (MAFAnM) in minced B2 was 2.5×10^2 CFU/g, compared to 1.2×10^3 CFU/g in the control BC. Bacteria of the coliform group were not detected in 0.001 g; pathogenic microorganisms, including S*almonella* spp. and *Listeria monocytogenes*, were absent in 25.0 g; *Staphylococcus aureus* was not detected in 1.0 g; *Proteus* bacteria were absent in 0.1 g; and no yeast or mold fungi were detected.

Based on the results of comprehensive studies (sensory, physicochemical, structural-mechanical, microbiological), it was established that the introduction of a complex food additive in the amount of 0.1, 0.2, and 0.3% of the mass of the recipe mixture into minced beef contributes to the formation of new functional and technological properties of minced meat systems and the improvement of consumer characteristics of finished products. Similar conclusions are given in studies (Bondar et al., 2019; Cofrades et al., 2017; Espinosa-Ramírez et al., 2023; Kryzhova et al., 2021; Peñalver et al., 2020).

Analysis of the nutrient profile and nutritional value of beef cutlets (Figure 7) shows an improvement in the biological and nutritional value of B2 compared to the control BC (Table 5).

Figure 7. Beef cutlets: control (BC) and with 0.2% CFA (B2)

An increase in protein content is observed, as well as enrichment of minced meat products with starch, dietary fiber, individual mineral elements and vitamins. In addition, as stated above, the proposed complex food supplement is a natural source of iodine, while the control cutlets did not contain this microelement.

Table 5

Nutrient profile and nutritional value of beef cutlets

Nutrient profile	Beef parties	
	BC	B2
Macronutrients, g/100 g dry matter		
Water	54.1±0.4^a	55.6±0.40^b
Protein	25.6±0.2^a	27.4±0.20^b
Fats	18.4±0.01^a	18.62±0.01^b
Sugars	BDL	0.75±0.01
Starch	BDL	1.10±0.02
Cellulose	BDL	0.96±0.01
Orgainic acids	BDL	0.40±0.01
Ash	1.8±0.01^a	1.93±0.01^b
Minerals, mg/100 g dry matter		
Sodium (Na)	461.51±2.28^a	469.26±2.28^b
Potassium (K)	191.62±1.14^a	195.09±1.14^b
Calcium (Ca)	14.12±1.16^a	15.01±0.03^b
Magnesium (Mg)	24.46±1.04^a	25.54±1.04^b
Phosphorus (P)	150.08±1.03^a	151.16±1.03^b
Iodine (I)	BDL	0.075±0.010
Iron (Fe)	1.22±0.02^a	1.56±0.02^b
Sulfur (S)	BDL	0.06±0,01
Zinc (Zn)	BDL	0.05±0.01
Manganese (Mn)	BDL	0.002±0.0
Copper (Cu)	BDL	0.0011±0.0
Selenium (Se)	BDL	0.004±0.0
Cobalt (Co)	BDL	0.005±0.0
Bromine (Br)	BDL	0.01±0.0
Water- and fat-soluble vitamins, mg/100 g dry matter		
A (retinol)	BDL	0.16±0.02
E (tocopherol)	BDL	0.17±0.01
C (L-ascorbic acid)	BDL	0.15±0.02
B$_1$ (thiamine)	0.05±0.01^a	0.13±0.01^b
B$_2$ (riboflavin)	0.15±0.01^a	0.18±0.001^b
B$_3$ (PP) (niacin)	4.04±0.01^a	4.29±0.01^b
B$_6$ (pyridoxine)	0.07±0.002^a	0.09±0.002^b
B$_8$ (biotin)	0.07±0.002^a	0.21±0.02^b
B$_9$ (folic acid)	BDL	0.61±0.04
B$_{12}$ (cobalamin)	0.22±0.0^a	0.28±0.0^b
Nutritional value		
Biological value, BV,%	85.60±0.78^a	94.20±0.82^b
Heat resistance index,%	BDL	98.7±0.10
Approximate calorie content, kcal/100 g	268.62±1.02	287.02±1.02

$^{a\text{-}b}$ Mean values in the same row with different superscripts differ significantly at $p<0.05$; BDL – below detected limit.

The incorporation of the complex food additive into the beef cutlet formulation at a concentration of 0.2% (w/w) resulted in an iodine-enriched meat product containing 75±10 µg/100 g, meeting over 50% of the Recommended Dietary Allowance (RDA) for adults. Additionally, the product retained 27.8% more iron compared to the control, covering more than 10% of the adult RDA for iron. The biological value of the beef cutlets was determined by assessing the protein digestibility of the samples (BC and B2), measured as the degree of their hydrolysis (hydrolysis index, HI,%) under the action of gastrointestinal proteolytic enzymes in vitro. The obtained results indicate a high biological value of the samples: HI was 85.6% for sample BC and 94.2% for sample B2. The addition of a complex food additive also contributes to an increase in energy value by 18.4 kcal/100 g of beef cutlet with 0.2% (B2) compared to the control (BC). Therefore, enriching the beef cutlet with a complex food additive increases its nutritional value. The high heat resistance and iodine stability (98.7%) of the algal component in the complex food additive reflect the characteristics of the food matrix, which is stabilized by the formation of nanoassociates between IONPs and the biopolymers of the minced meat system at both the micro- and macro-levels.

Conclusions

The effect of the finely dispersed complex food additive (CFA), containing iron oxide nanoparticles ($FeO \times Fe_2O_3$) and dry kelp (*Laminaria*) biomass powder, on model minced beef and the cutlets made from it was studied. It was found that adding the CFA at a level of 0.2% improves (compared to the control) the sensory indicators of the cutlets — the overall sensory score increased by 17.6% — and reduces moisture and fat losses during heat treatment, increasing the yield of the finished product by 5.5%. Incorporation of 0.2% CFA into minced beef improved its functional and technological characteristics, increasing water-holding capacity by 1.33 times, fat-holding capacity by 1.29 times, emulsifying capacity by 1.27 times, and emulsion stability by 1.25 times. At the same time, the structural and mechanical properties of the minced meat improved: the instantaneous elastic modulus increased by 1.20 times, and the highly elastic modulus by 1.16 times. Oxidative processes in minced beef with the complex food additive were suppressed, as acid and peroxide numbers were 2.2 times and 1.6 times lower, respectively, after 24 hours of cold storage compared to the control sample without CFA. The addition of CFA also increased the protein content and enriched the cutlets with dietary fiber, specific minerals, and vitamins. A beef cutlet containing 0.2% CFA provides 75 ± 10 µg/100 g of iodine, satisfying more than 50% of the recommended daily intake (RDI) for an adult.

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Cite:

UFJ Style

Tsykhanovska I., Stabnikova O., Dorohovich V., Lytvyn O., Hetman P. (2025), Meat cutlets with iron oxide nanoparticles and alga *Laminaria japonica*, *Ukrainian Food Journal*, 14(2), pp. 238–258, https://doi.org/10.24263/2304-974X-2025-14-2-5

APA Style

Tsykhanovska, I., Stabnikova, O., Dorohovich, V., Lytvyn, O., & Hetman P. (2025), Meat cutlets with iron oxide nanoparticles and alga *Laminaria japonica. Ukrainian Food Journal, 14*(2), 238–258. https://doi.org/10.24263/2304-974X-2025-14-2-5

Effect of carrot pomace addition on the rheological and textural properties of soft wheat pasta dough

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Abstract

Introduction. The trend toward sustainable pasta production includes the incorporation of various unconventional ingredients, such as carrot pomace.

Materials and methods. Common wheat pasta dough supplemented with different carrot pomace varieties – Niagara, Belgrado, Sirkana, and Baltimore (0, 3, 6, 9, and 12%) was evaluated by dynamic rheological methods: frequency sweep, temperature sweep, and creep and recovery test. Dough texture was measured with a texturometer.

Results and discussion. The results revealed significant changes in the rheological and textural properties of soft wheat pasta dough when the amount of added carrot pomace exceeded 6%. A key finding is the consistent increase in dough strength with carrot pomace addition, indicated by notably higher values for both elastic (G′) and viscous (G″) moduli compared to the control. Furthermore, dynamic creep and recovery tests, effectively modeled by the Burgers model, demonstrated a clear reduction in various compliance parameters (J_{max} from $12.64 \cdot 10^5$ to $1.87 \cdot 10^5$ Pa^{-1}) as carrot pomace levels in common wheat pasta dough increased from 3 to 12%.

This also indicated dough strengthening and higher resistance to deformation, a conclusion supported by the general increase in steady-state viscosity (from $1.05 \cdot 10^{-6}$ to $9.25 \cdot 10^{-6}$ Pa·s).

The thermal properties were also affected, with carrot pomace delaying starch gelatinization, evidenced by higher initial and final gelatinization temperatures. Textural analysis highlighted significant modifications: dough hardness (from 1423.99 to 4579.25g) and gumminess (from 626.16 to 1498.21g) increased, while springiness (from 99.90 to 99.53%), resilience (from 1.04 to 0.88), and cohesiveness (from 0.40 to 0.31) generally decreased as the carrot pomace inclusion was higher.

Conclusions. The potential of carrot pomace as a functional ingredient for modifying dough properties is demonstrated. However, its addition affects the characteristics of pasta dough, indicating the need for further study of pasta quality for recipe optimization.

Keywords:

Carrot pomace
Wheat pasta dough
Rheology
Texture
Quality evaluation

Article history:

Received 01.12.2024
Received in revised form 23.03.2025
Accepted 30.06.2025

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DOI:
10.24263/2304-974X-2025-14-2-6

Introduction

The processing of plant materials generates a large amount of waste rich in valuable substances. Improper disposal of this waste often contributes to environmental pollution. Thus, the valorization of vegetable by-products to reduce the carbon footprint is a current priority aligned with the principles of the circular economy (Stabnikova et al., 2023a). Carrot pomace, a byproduct of juice production, contains a high concentration of bioactive compounds such as pigments and dietary fibers (McKee and Latner, 2000). This makes it a valuable additive for the development of functional foods, including commonly consumed products like pasta.

Semolina, made from durum wheat, which is rich in protein and gluten, is the most commonly used flour in pasta production. However, common wheat flour is also used in pasta production, particularly when durum wheat is less accessible. To enrich common wheat flour with fiber, carrot pomace can be incorporated as a functional ingredient. However, incorporation of fiber-rich ingredients like carrot pomace led to significant changes of dough rheology and texture. These features can predict dough behavior during pasta making, and represent useful tools for further recipe optimizations.

Dietary fiber is increasingly being incorporated into food products such as bread, pasta, and noodles to enhance their nutritional value and offer health benefits (Bashta et al., 2021; Lei et al., 2021; Stabnikova et al., 2023b; Tsykhanovska et al., 2024; Xu et al., 2021). However, the addition of fiber-rich ingredients significantly influences the rheological and textural properties of the dough (Xu et al., 2021). This impact is influenced by factors such as the type, structure, size, and amount of dietary fiber added, as well as its physical properties, including water holding, oil holding, swelling capacity, viscosity, or gel formation (Liu et al., 2019; Sivam et al., 2010).

A key mechanism behind the altered dough properties is the high water-binding capacity of dietary fiber (Bchir et al., 2014; Kultys and Moczkowska-Wyrwisz, 2022). Fiber competes with wheat components, primarily gluten and starch, for the available water during dough mixing. This competition can lead to reduced water availability for gluten hydration and development (Xu et al., 2021), potentially disrupting the gluten network (Asadi et al., 2024), and altering the distribution of moisture among dough constituents (Sivam et al., 2010). Upon hydration, dietary fiber swells and, depending on whether it remains as discrete particles or forms a viscous phase, it can physically disrupt the continuous gluten–starch matrix. The presence of other compounds such as polyphenols and enzymes can also play a role. Polyphenols can interact with gluten proteins through hydrogen bonding or covalent cross-linking, influencing viscoelastic properties (Girard and Awika, 2020; Han et al., 2021). Some fiber sources may contain enzymes (like proteases) that can degrade gluten proteins, weakening the network (Lu et al., 2018). Particle size also influences interaction, affecting hydration rate and gluten development (Liu et al., 2019).

Addition of carrot pomace powder to dough generally leads to increased water absorption, dough development time, and stability (Xu et al., 2021). It can also increase dough consistency and decrease viscosity, but it has been shown to decrease dough extensibility and reduce the mixing tolerance index (Bchir et al., 2014; Xu et al., 2021). Textural analysis of dough enriched with carrot pomace powder indicates decreased cohesiveness and elasticity, while gumminess and chewiness are increased (Asadi et al., 2024). The ratio of soluble to insoluble fiber in carrot pomace influences its effect, with a higher soluble fiber content potentially leading to higher water absorption and swelling (Dhingra et al., 2012; Kultys and Moczkowska-Wyrwisz, 2022). Using carrot puree or paste may yield different results compared to powder, potentially resulting in better quality pasta with lower cooking loss (Wang et al., 2022).

In summary, incorporating carrot pomace and other fiber-rich ingredients into dough formulations significantly alters their rheological and textural properties. These changes are complex and depend on the fiber source, amount, and processing, primarily mediated through water competition, physical interference with the gluten network, and interactions involving other co-present compounds like polyphenols and enzymes. To our knowledge, there are few papers regarding the impact of different carrot pomace varieties on common wheat pasta dough. Thus, the aim of this paper was to investigate the effects of carrot pomace (Niagara, Belgrado, Sirkana, and Baltimore varieties) at different levels (0, 3, 6, 9 and 12%) on common wheat pasta dough rheological and texture properties.

Materials and methods

Materials

This study utilized commercial common wheat flour (*Triticum aestivum*), obtained from Mopan, Suceava, and carrot pomace sourced from a farmer in Bacău, Romania. The carrot pomace came from four distinct varieties: Niagara, Belgrado, Sirkana, and Baltimore.

Common wheat flour was analyzed according to Romanian or International Cereal Chemistry methods, revealing the following characteristics: moisture content by ICC methods 110/1; ash content by ICC 104/1; protein content by ICC 105/2; fiber content by acid and alkaline digestion using a Fibertec 2010 automated analyzer; wet gluten by ICC 106/1. Gluten deformation index of wheat four was determined by SR 90:2007; falling number by ICC 107/1.

Carrot pomace from the four varieties displayed varying chemical characteristics: moisture content was determined by SR EN ISO 665:2020; fat content was measured by SR EN ISO 659:2009; protein levels was analyzed using SR EN ISO 20483:2014; ash content was determined according to SR EN ISO 2171:2023; the fiber content was measured by acid and alkaline digestion using a Fibertec 2010 automated analyzer.

Various wheat dough formulations were investigated. One sample of wheat flour served as a control (without carrot pomace). Other formulations included carrot pomace added to the common wheat flour at different incorporation levels (3%, 6%, 9%, and 12%) for each of the four carrot varieties. Dough was obtained by adding water to the flour mix to achieve 45% moisture and mixing for 15 min. The dough was rested for 15 min before testing.

Rheological measurements

Dynamic rheological properties of the dough were assessed using a Haake dynamic rheometer (MARS 40, Thermo-Haake, Karlsruhe, Germany). This rheometer was equipped with 40 mm parallel plates and a Peltier heating system to maintain a constant temperature. Dough samples were positioned between the plates with a 3 mm gap, and any excess was removed. To prevent moisture loss, the exposed dough surface was coated with a thin layer of vaseline. Before testing, samples rested for 120 s in the measurement position. Measurements were conducted within the dough's linear viscoelastic region, determined by strain sweep tests (0-100 Pa strain, 1 Hz oscillation frequency). Frequency sweep tests were performed at a shear stress of 15 Pa across frequencies from 0.01 to 20 Hz. Temperature sweep tests were run at 4.0 ± 0.1 °C/min, heating from 20 to 100 °C, to identify initial (Ti) and final (Tg) gelatinization temperatures.

Complementing these, creep-recovery tests involved a sudden application of 50 Pa stress for 60 s, followed by a 180 s recovery period to allow the dough to relax. The Burgers model was used for processing the creep and recovery data.

Texture measurements

To assess the pasta dough's texture, 50 g dough spheres were subjected to a double compression down to 50% of their original height. This was performed using a Perten TVT–6700 texturometer (Perten Instruments, Sweden) fitted with a 35 mm cylindrical probe. During the test, a speed of 5.0 mm/s was applied, along with a 20 g trigger force and a 12-second recovery time between compressions. The dough's hardness, springiness, gumminess, and resilience were subsequently recorded.

Statistical analysis

Measurements were performed in at least three replicates. Comparisons of means were conducted via ANOVA, utilizing version 2023 of the XLSTAT program. Principal Component Analysis was performed to evaluate the relationships between the characteristics.

Results and discussion

Characteristics of carrot pomace and wheat flour

Proximate composition of wheat flour and carrot pomace from different carrot varieties was discussed in our previous paper (Luca et al., 2022) an it is shown in Table 1.

Table 1

Proximate composition of wheat flour and carrot pomace from different carrot varieties

(Luca et al., 2022)

Content,%	**Pomace from carrot variety**				**Wheat flour**
	Baltimore	**Belgrado**	**Niagara**	**Sirkana**	
Moisture	4.04 ± 0.02^{c}	3.78 ± 0.01^{c}	5.88 ± 0.46^{b}	5.91 ± 0.15^{b}	11.84 ± 0.04^{a}
Protein	6.87 ± 0.06^{e}	8.01 ± 0.06^{d}	8.84 ± 0.12^{c}	9.14 ± 0.06^{b}	10.95 ± 0.00^{a}
Fat	1.00 ± 0.02^{c}	1.01 ± 0.03^{c}	0.70 ± 0.02^{d}	1.13 ± 0.03^{a}	1.06 ± 0.02^{b}
Fiber	12.25 ± 0.29^{c}	13.44 ± 0.73^{b}	14.23 ± 0.19^{b}	15.23 ± 0.76^{a}	0.56 ± 0.04^{d}
Ash	5.29 ± 0.04^{c}	5.89 ± 0.02^{a}	5.56 ± 0.03^{b}	5.89 ± 0.01^{a}	0.46 ± 0.01^{d}

Content of wet gluten in wheat flour was 26.75%. Wheat flour had gluten deformation index 2.50 mm and falling number 354.50 s.

Dough rheological features

The incorporation of carrot pomace affected the rheological properties of the dough (Figure 1). All the samples tested exhibited a solid-like behavior, as suggested by the G′ values greater than G". In general, increasing the level of carrot pomace addition led to higher viscoelastic moduli. Compared to the control, all the samples enriched with carrot pomace presented higher G’ and G". Samples containing Belgrado carrot pomace showed lower G′ and G" values compared to the other varieties. Similar to our results, Bigne et al. (2016) obtained higher G’ compared to G" for dough with Mesquite flour – a leguminous plant, indicating a prevalence of elastic forces above the viscous ones. Carrot pomace is rich in fibers (Table 1) (Luca et al., 2022) and has significant amounts of polyphenols (Luca et al., 2024). The addition of fiber-rich ingredients to wheat dough determines the increase in water absorption requirements and disrupt the development of the gluten network (Gan et al., 1992).

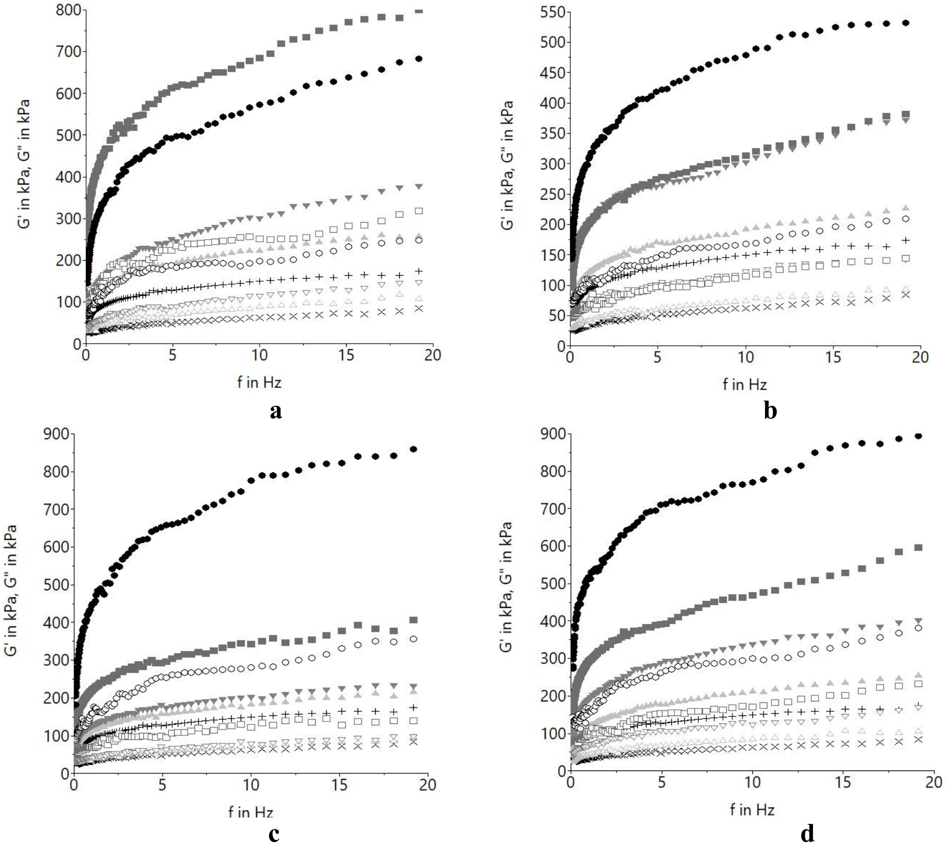

Figure 1. Variation of elastic (G′) and viscous (G") moduli with frequency:
a – Baltimore, b – Belgrado, c – Niagara, d – Sirkana carrot varieties; F2 (0%): + G′, ×G"; 3%: ▲G′, △G"; 6%: ▼G′, ▽G"; 9%: ■G′, □G"; 12%: ●G′, ○G"

These changes in the dough's viscoelastic properties, particularly when enriched with insoluble dietary fibre, can be attributed to modifications in the gluten structure, interactions with starch, and the fiber's water-binding capacity, leading to more plastic than elastic dough matrix (Almoumen et al., 2024). Furthermore, the strengthening of the dough may be attributed to the presence of polyphenols in carrot pomace. Interactions between proteins and phenolic compounds can occur through both covalent and non-covalent bonds, playing a key role in the formation, structure, and development of dough (Ozdal et al., 2013).

Creep and recovery experimental data were successfully fitted to Burgers model ($R^2 > 0.97$). A reduction of instantaneous elasticity – J_{Co}, J_{Ro} and retarded elastic compliance – J_{Cm}, J_{Rm} in both creep and recovery phases was observed as the addition level of carrot pomace was higher, with some exceptions (Table 2). Maximum creep compliance – J_{max} also decreases, as the amount of carrot pomace was higher, suggesting dough strengthening and higher resistance to deformation. Steady-state viscosity – η_o exhibited a general increasing trend as the level of carrot pomace raised. The recovery compliance – J_r was lower as the amount of carrot pomace was higher. The results are in agreements with those reported previously for wheat dough with tomato pomace (Mironeasa and Codina, 2019).

Table 2

Burgers model parameters for creep and recovery tests

Sample	**$J_{Co}\cdot10^5$ (Pa^{-1})**	**$J_{Cm}\cdot10^5$ (Pa^{-1})**	**λ_C (s)**	**$\eta_o\cdot10^{-6}$ ($Pa\cdot s$)**	**$J_{max}\cdot10^5$ (Pa^{-1})**	**$J_{Ro}\cdot10^5$ (Pa^{-1})**	**$J_{Rm}\cdot10^5$ (Pa^{-1})**	**λ_R (s)**	**$J_r\cdot10^5$ (Pa^{-1})**
F2	1.47 ± 0.09^{abAB}	5.31 ± 0.29^{abAB}	33.71 ± 1.99^{aA}	2.98 ± 0.10^{bcA}	5.67 ± 0.46^{abA}	1.29 ± 0.09^{aAB}	2.84 ± 0.18^{abAB}	50.14 ± 7.23^{aAB}	$4.13\pm0.27a^{bAB}$
F2Ba3	1.06 ± 0.10^{aB}	2.80 ± 0.27^{aB}	25.47 ± 1.20^{aA}	5.29 ± 0.47^{cA}	8.05 ± 0.70^{aA}	1.76 ± 0.12^{aB}	3.44 ± 0.28^{aAB}	45.36 ± 3.42^{aA}	5.20 ± 0.40^{aB}
F2Ba6	0.57 ± 0.04^{abB}	3.91 ± 0.15^{bB}	49.66 ± 1.10^{aA}	5.23 ± 0.99^{bcA}	3.58 ± 0.20^{bA}	1.00 ± 0.05^{aB}	1.38 ± 0.10^{bAB}	50.18 ± 0.40^{abA}	2.38 ± 0.15^{abcB}
F2Ba9	0.55 ± 0.04^{bB}	2.40 ± 0.16^{bcB}	26.98 ± 6.17^{aA}	5.64 ± 0.55^{bA}	2.87 ± 0.16^{bcA}	0.87 ± 0.03^{aB}	1.26 ± 0.05^{bAB}	49.58 ± 0.93^{abA}	2.13 ± 0.08^{bcB}
F2Ba12	0.66 ± 0.02^{bB}	2.23 ± 0.14^{cB}	37.87 ± 2.03^{aA}	5.33 ± 0.31^{aA}	2.60 ± 0.03^{cA}	0.43 ± 0.03^{aB}	1.19 ± 0.07^{bAB}	44.68 ± 1.84^{bA}	1.62 ± 0.10^{cB}
F2Be3	3.01 ± 0.14^{aA}	9.95 ± 0.08^{aA}	33.47 ± 1.62^{aA}	1.05 ± 0.07^{cA}	12.64 ± 0.90^{aA}	1.13 ± 0.09^{aA}	7.07 ± 0.18^{aA}	84.43 ± 1.77^{aA}	8.20 ± 0.27^{aA}
F2Be6	1.95 ± 0.04^{abA}	6.41 ± 0.36^{bA}	33.18 ± 2.04^{aA}	3.05 ± 0.19^{bcA}	7.46 ± 0.52^{bA}	3.54 ± 0.20^{aA}	2.94 ± 0.21^{bA}	35.87 ± 0.90^{abA}	6.47 ± 0.40^{abcA}
F2Be9	0.97 ± 0.04^{bA}	3.58 ± 0.15^{bcA}	34.25 ± 0.24^{aA}	4.06 ± 0.17^{bA}	3.98 ± 0.18^{bcA}	1.89 ± 0.07^{aA}	1.34 ± 0.04^{bA}	38.37 ± 2.02^{abA}	3.23 ± 0.11^{bcA}
F2Be12	0.71 ± 0.05^{bA}	2.03 ± 0.12^{cA}	36.80 ± 0.17^{aA}	6.38 ± 0.21^{aA}	2.88 ± 0.27^{cA}	1.69 ± 0.06^{aA}	0.78 ± 0.03^{bA}	12.39 ± 0.45^{bA}	2.46 ± 0.09^{cA}
F2Ni3	0.89 ± 0.06^{aB}	4.96 ± 0.34^{aB}	43.16 ± 1.15^{aA}	2.62 ± 0.16^{cA}	4.67 ± 0.32^{aA}	1.10 ± 0.02^{aAB}	0.89 ± 0.02^{aB}	31.24 ± 0.56^{aA}	1.99 ± 0.04^{aB}
F2Ni6	1.24 ± 0.11^{abB}	3.64 ± 0.39^{bB}	35.86 ± 0.78^{aA}	3.74 ± 0.22^{bcA}	5.82 ± 0.29^{bA}	1.81 ± 0.17^{aAB}	1.13 ± 0.11^{bB}	23.12 ± 0.75^{abA}	2.94 ± 0.28^{abcB}
F2Ni9	0.60 ± 0.04^{bB}	1.61 ± 0.07^{bcB}	35.46 ± 3.49^{aA}	5.99 ± 0.13^{bA}	2.80 ± 0.10^{bcA}	1.60 ± 0.08^{aAB}	0.66 ± 0.01^{bB}	23.82 ± 2.23^{abA}	2.26 ± 0.09^{bcB}
F2Ni12	0.44 ± 0.04^{bB}	1.38 ± 0.09^{cB}	33.78 ± 0.11^{aA}	9.25 ± 0.86^{aA}	1.94 ± 0.14^{cA}	0.84 ± 0.08^{aAB}	0.64 ± 0.03^{bB}	36.64 ± 5.27^{bA}	1.48 ± 0.11^{cB}
F2Si3	1.99 ± 0.18^{aAB}	7.71 ± 0.65^{aAB}	34.13 ± 1.53^{aA}	1.65 ± 0.11^{cA}	8.55 ± 0.67^{aA}	3.43 ± 0.28^{aAB}	2.58 ± 0.09^{aAB}	42.34 ± 1.47^{aA}	6.01 ± 0.36^{aAB}
F2Si6	0.80 ± 0.07^{abAB}	3.10 ± 0.26^{bAB}	33.87 ± 1.09^{aA}	3.93 ± 0.16^{bcA}	3.52 ± 0.39^{bA}	1.04 ± 0.10^{aAB}	1.81 ± 0.11^{bAB}	54.42 ± 4.92^{abA}	2.86 ± 0.21^{abcAB}
F2Si9	1.05 ± 0.08^{bAB}	3.30 ± 0.13^{bcAB}	41.80 ± 2.26^{aA}	4.03 ± 0.09^{bA}	3.88 ± 0.13^{bcA}	0.93 ± 0.03^{aAB}	1.58 ± 0.12^{bAB}	47.75 ± 3.20^{abA}	2.50 ± 0.14^{bcAB}
F2Si12	0.42 ± 0.04^{bAB}	1.90 ± 0.16^{cAB}	42.04 ± 3.73^{aA}	6.78 ± 0.14^{aA}	1.87 ± 0.02^{cA}	0.93 ± 0.03^{aAB}	0.49 ± 0.02^{bAB}	17.49 ± 1.30^{bA}	1.41 ± 0.04^{cAB}

In the same column, a-c – small letters indicate significant differences ($p < 0.05$) among different addition levels, A-B – uppercase letters indicate significant differences ($p < 0.05$) among different carrot varieties, Ba – Baltimore, Be – Belgrado, Ni – Niagara, Si – Sirkana carrot varieties, F2 – common wheat flour; 3, 6, 9, and 12 – levels of incorporated carrot pomace.

Lower elastic compliance during the recovery phase indicates smaller recoverable energy, suggesting a less extensively cross-linked gluten network in samples with high amounts of carrot pomace (Mironeasa and Codina, 2019). The reduction in instantaneous compliance may result from gluten dilution in the dough matrix, as a portion of the wheat flour is replaced by non-gluten components from the carrot pomace (Atudorei et al., 2023).

The diminished recovery behavior further suggests that the incorporation of carrot pomace may have led to partial disruption of elastic bonds within the gluten network. The observed strengthening effect may be attributed to the hydroxyl groups present in phenolic compounds from carrot pomace (Iuga et al., 2019; Luca et al., 2024). The phenolic compounds can directly interact with proteins through both non-covalent and covalent bonds, altering protein structure and functionality, thereby influencing the overall quality and functional properties of food products (Baxter et al., 1997). Furthermore, interactions between the insoluble dietary fiber in carrot pomace and wheat proteins may also contribute to increased dough stiffness, supporting previous findings (Iuga et al., 2019).

Dough behavior during heating indicated a delay in starch gelatinization. Ti values increased as the addition level was higher, with some exceptions for 12% Baltimore, Belgrado and Niagara carrot pomace (Figure 2). Tg values exhibited slight increase with carrot pomace addition level raise. Carrot pomace variety did not show significant impact on Tg and Ti. The presence of polyphenols in carrot pomace may explain these results since it was demonstrated that there is an interaction between phenolics and gluten, leading to delay in starch gelatinization and staling (Xu et al., 2019). There are two primary interactions mechanisms between starch and gluten during thermal processing (Jekle et al., 2016): (1) competitive hydration, where both components compete for available water as their structures undergo transformation, and (2) the formation of a gluten network that encases starch granules, thereby restricting their water uptake and hindering gelatinization. Additionally, hydrophilic phenolic compounds may compete with starch for water and form non-covalent interactions with starch molecules. These interactions can alter the system's pH, subsequently influencing starch granule hydration, gelatinization behavior, and pasting characteristics (Jekle et al., 2016). Another study observed that incorporating certain phenolics from blackcurrant into wheat flour increases starch gelatinization temperatures (Sivam et al., 2012).

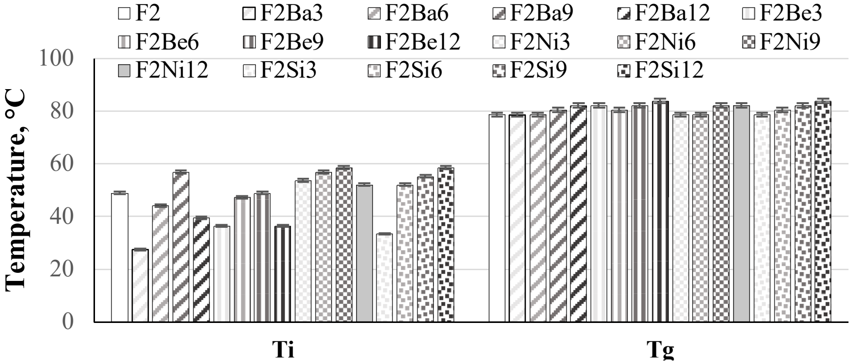

Figure 2. Initial (Ti) and final (Tg) gelatinization temperatures of dough with carrot pomace: Ba – Baltimore, Be – Belgrado, Ni – Niagara, Si – Sirkana carrot varieties, F2 – common wheat flour

Dough texture features

The structure of the gluten network plays a key role in determining dough texture. Incorporation of fiber-rich ingredients like carrot pomace led to significant changes of textural parameters, depending on the addition level (Table 3).

Table 3

Texture features of dough

Sample	Hardness (g)	Springiness (%)	Gumminess (g)	Resilience (adim.)	Cohesiveness (adim.)
F2	1423.99 ± 119.74^{dB}	99.90 ± 0.15^{aA}	657.35 ± 27.08^{cA}	0.91 ± 0.08^{aA}	0.40 ± 0.05^{aA}
F2Ba3	2093.00 ± 83.44^{dA}	99.73 ± 0.18^{aA}	779.05 ± 59.47^{cA}	1.04 ± 0.07^{aA}	0.35 ± 0.02^{abC}
F2Ba6	2863.67 ± 784.31^{cA}	99.61 ± 0.05^{aA}	860.29 ± 79.68^{cA}	0.95 ± 0.04^{aA}	0.32 ± 0.02^{bcC}
F2Ba9	3393.50 ± 286.38^{bA}	99.55 ± 0.21^{aA}	904.20 ± 60.24^{bA}	0.93 ± 0.03^{aA}	0.31 ± 0.02^{bcC}
F2Ba12	3550.00 ± 95.35^{aA}	99.64 ± 0.13^{aA}	1194.02 ± 172.00^{aA}	0.88 ± 0.08^{aA}	0.32 ± 0.01^{cC}
F2Be3	1676.50 ± 75.94^{dAB}	99.80 ± 0.05^{aA}	626.16 ± 53.51^{cA}	0.99 ± 0.03^{aA}	0.39 ± 0.01^{abB}
F2Be6	2221.67 ± 295.17^{cAB}	99.76 ± 0.16^{aA}	639.54 ± 37.79^{cA}	0.98 ± 0.06^{aA}	0.36 ± 0.01^{bcB}
F2Be9	3225.00 ± 219.73^{bAB}	99.72 ± 0.09^{aA}	1085.23 ± 55.51^{bA}	1.01 ± 0.02^{aA}	0.34 ± 0.01^{bcB}
F2Be12	3461.25 ± 350.08^{aAB}	99.66 ± 0.08^{aA}	1100.24 ± 107.77^{aA}	0.97 ± 0.05^{aA}	0.33 ± 0.01^{cB}
F2Ni3	1520.00 ± 75.74^{dAB}	99.71 ± 0.20^{aA}	593.41 ± 70.45^{cA}	0.89 ± 0.03^{aA}	0.36 ± 0.01^{abBC}
F2Ni6	2202.25 ± 50.17^{cAB}	99.73 ± 0.21^{aA}	776.08 ± 74.66^{cA}	1.00 ± 0.07^{aA}	0.33 ± 0.02^{bcBC}
F2Ni9	2395.00 ± 99.98^{bAB}	99.69 ± 0.19^{aA}	848.05 ± 83.60^{bA}	0.95 ± 0.04^{aA}	0.35 ± 0.00^{bcBC}
F2Ni12	4579.25 ± 297.69^{aAB}	99.86 ± 0.05^{aA}	1498.21 ± 134.04^{aA}	1.03 ± 0.07^{aA}	0.32 ± 0.02^{cBC}
F2Si3	1894.00 ± 67.55^{dAB}	99.78 ± 0.24^{aA}	651.89 ± 92.87^{cA}	0.96 ± 0.06^{aA}	0.35 ± 0.02^{abBC}
F2Si6	2281.25 ± 36.68^{cAB}	99.77 ± 0.10^{aA}	847.08 ± 25.7^{cA}	0.97 ± 0.04^{aA}	0.36 ± 0.02^{bcBC}
F2Si9	2748.75 ± 94.75^{bAB}	99.58 ± 0.08^{aA}	951.85 ± 69.95^{bA}	1.01 ± 0.03^{aA}	0.35 ± 0.02^{bcBC}
F2Si12	3500.67 ± 193.71^{aAB}	99.53 ± 0.13^{aA}	1150.39 ± 108.06^{aA}	0.94 ± 0.04^{aA}	0.33 ± 0.01^{cBC}

In the same column, a-d – small letters indicate significant differences ($p < 0.05$) among different addition levels, A-C – uppercase letters indicate significant differences ($p < 0.05$) among different carrot varieties, Ba – Baltimore, Be – Belgrado, Ni – Niagara, Si – Sirkana carrot varieties, F2 – common wheat flour

Dough hardness and gumminess increased with higher levels of carrot pomace, while springiness, resilience, and cohesiveness generally decreased. Carrot pomace level influenced significantly ($p < 0.05$) only cohesiveness. Fibers from carrot pomace could be responsible for hardness increase and reduction of elasticity of pasta dough. For instance, studies have shown that components like water-unextractable solids, found in wheat flour with higher milling extraction rates, could reduce both gluten yield and extensibility, as well as disrupt gluten particle interactions (Wang et al., 2003). Additionally, fiber components may hinder gluten agglomeration (Noort et al., 2010), physically disrupt the gluten matrix, and limit the water available for gluten development (Ktenioudaki et al., 2013; Wang et al., 2003). Dahdah et al. (2024) reported smaller extensibility of dough with freeze-dried olive pomace due to a weaker gluten matrix due to fibre incorporation, leading to stiffer and firmer dough. Glutenin serves as the base of the gluten network and plays a key role in providing elasticity to dough by forming intermolecular disulfide bonds with other glutenin molecules (Dahdah et al., 2024; Shewry et al., 2000). These cross-links result in a stable, elastic structure that contributes to the dough's resistance (Shewry et al., 2000). Therefore, diluting the gluten content with fiber-rich ingredients like carrot pomace leads to a reduction in springiness and dough resilience.

Relationships between variables

Pearson correlations coefficients are presented in Table 4.

Table 4

Pearson correlations between variables

Variables	G'_{10Hz}	G''_{10Hz}	T_i	Hardness	Springiness	Gumminess	Resilience	Cohesiveness	J_{Co}	J_{Cm}	λ_C	η_o	J_{max}	J_{Ro}	J_{Rm}	λ_R	J_r
G'_{10Hz}	**1**																
G''_{10Hz}	**0.99**	**1**															
T_i	0.34	0.36	**1**														
Hardness	**0.89**	**0.87**	0.19	**1**													
Springiness	-0.48	**-0.49**	-0.26	-0.37	**1**												
Gumminess	**0.83**	**0.80**	0.17	**0.95**	-0.23	**1**											
Resilience	-0.04	-0.03	-0.23	0.15	0.24	0.18	**1**										
Cohesiveness	**-0.68**	**-0.66**	-0.15	**-0.79**	**0.65**	**-0.65**	-0.03	**1**									
J_{Co}	**-0.60**	**-0.59**	-0.43	**-0.64**	**0.50**	**-0.64**	0.20	**0.68**	**1**								
J_{Cm}	**-0.64**	**-0.61**	-0.39	**-0.68**	0.44	**-0.71**	0.01	**0.67**	**0.94**	**1**							
λ_C	0.03	0.04	0.26	0.07	-0.37	0.09	-0.36	-0.15	-0.25	-0.05	**1**						
η_o	**0.79**	**0.77**	0.25	**0.85**	-0.29	**0.85**	0.14	**-0.69**	**-0.80**	**-0.87**	0.02	**1**					
J_{max}	**-0.68**	**-0.65**	**-0.57**	**-0.69**	0.47	**-0.70**	0.29	**0.62**	**0.94**	**0.89**	-0.33	**-0.76**	**1**				
J_{Ro}	-0.45	-0.42	-0.31	-0.37	0.32	-0.46	0.26	0.22	**0.50**	0.46	-0.27	-0.42	0.48	**1**			
J_{Rm}	**-0.50**	**-0.49**	**-0.55**	**-0.55**	0.44	**-0.54**	0.26	**0.65**	**0.89**	**0.82**	-0.35	**-0.64**	**0.92**	0.21	**1**		
λ_R	-0.29	-0.29	-0.32	-0.33	0.29	-0.33	0.09	0.44	**0.57**	**0.60**	-0.20	**-0.49**	**0.58**	-0.18	**0.78**	**1**	
J_r	**-0.60**	**-0.58**	**-0.58**	**-0.61**	**0.49**	**-0.64**	0.33	**0.62**	**0.94**	**0.86**	-0.40	**-0.71**	**0.95**	**0.61**	**0.91**	**0.56**	**1**

Ba – Baltimore, Be – Belgrado, Ni – Niagara, Si – Sirkana carrot varieties, F2 – common wheat flour, values with bold are significant at $p < 0.05$.

Strong positive correlation was obtained between G' and G" ($p < 0.05$, r = 0.99), which was in agreement with previous report for grape peels-containing dough (Mironeasa et al., 2019).

G' and G" were positively correlated with hardness, gumminess, η_o ($p < 0.05$, r > 0.79), and negatively with cohesiveness, J_{Co}, J_{Cm}, J_{max}, J_{Rm}, and J_r ($p < 0.05$, r > -0.50). Similar strong positive correlations between G' and G" with hardness and gumminess were obtained by (Ganesan et al., 2023) for cookies with brewer’s spent grain by-product. Negative correlations ($p < 0.05$, r > -0.55) were observed between T_i and J_{max}, J_{Rm}, and J_r. Hardness and gumminess were positively correlated with η_o ($p < 0.05$, r > 0.85), and negatively with J_{Co}, J_{Cm}, J_{max}, J_{Rm}, and J_r ($p < 0.05$, r > -0.54). Springiness was positively correlated with cohesiveness, J_{Co} and J_r ($p < 0.05$, r > 0.49). Significant positive correlations ($p < 0.05$, r > 0.62) were obtained between cohesiveness and J_{Co}, J_{Cm}, J_{max}, J_{Rm}, and J_r, while with η_o the correlation was negative ($p < 0.05$, r = -0.69). J_{Co}, and J_{Cm} were positively correlated with J_{max}, J_{Rm}, λ_R and J_r ($p < 0.05$, r > 0.50), and negatively with η_o ($p < 0.05$, r > -0.80). Negative correlations were observed between η_o and J_{Rm} and λ_R ($p < 0.05$, r > -0.49), while J_{max} was positively correlated with J_{Rm}, λ_R and J_r ($p < 0.05$, r > 0.58). Significant positive correlations were obtained between J_r and J_{max}, J_{Ro}, J_{Rm}, and λ_R ($p < 0.05$, r > 0.61). Previous research regarding wheat flour dough rheological properties revealed significant positive correlations between extensibility and maximum recovery strain (Wang and Sun, 2002).

Principal Component analysis (PCA) revealed the relationships between the rheological and texture parameters of dough (Figure 3).

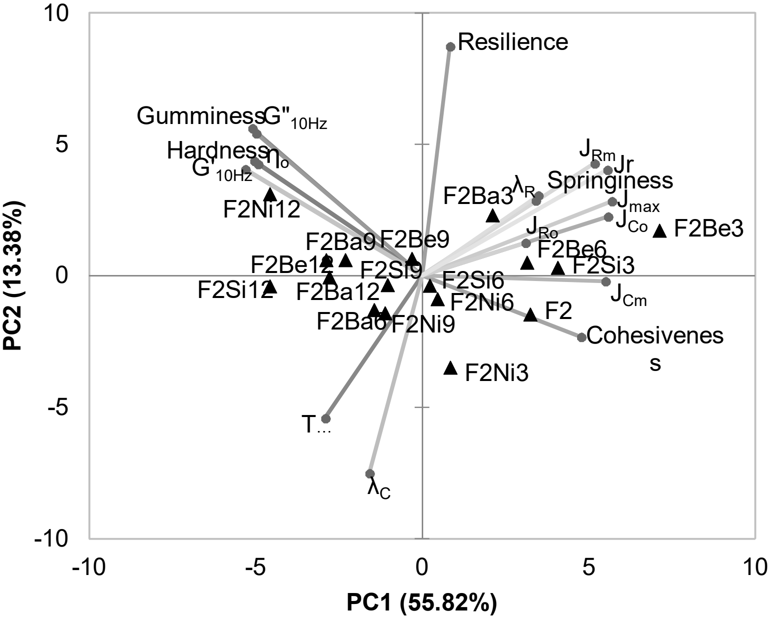

Figure 3. Principal Component Analysis bi-plot:
Ba – Baltimore, Be – Belgrado, Ni – Niagara,
Si – Sirkana carrot varieties, F2 – common wheat flour

A total variance of 69.20% was explained by PC1 (55.82%) and PC2 (13.38%). PC1 was associated with J_{Co}, J_{Cm}, η_o, J_{max}, J_{Ro}, J_{Rm}, λ_R, J_r, springiness, cohesiveness, hardness, gumminess, G' and G", while PC2 was associated with resilience, T_i and λ_C. Almost all the samples with 3 and 6% carrot pomace, along with the control (F2) were positioned in the right part of the graphic, while the samples with higher addition levels (9 and 12%) were located in the left quadrants.

Conclusions

The incorporation of carrot pomace has a significant effect on the rheological and textural properties of the dough. The results demonstrate that increasing carrot pomace levels generally lead to a more solid-like dough behavior, characterized by higher viscoelastic moduli and increased resistance to deformation. Furthermore, the addition of carrot pomace was found to delay starch gelatinization, indicating a modification in the dough's thermal properties. From a texture point of view, the enriched dough exhibited increased hardness and gumminess, while springiness, resilience, and cohesiveness were lower. Correlation analysis reinforced these observations, revealing strong positive relationships between viscoelastic moduli and dough hardness/gumminess, and negative correlations with various creep and recovery parameters. These results underscore the potential of carrot pomace as a functional ingredient for modifying dough properties, although further research is needed to optimize its application for specific product development.

Acknowledgment
Mădălina Ungureanu-Iuga was funded by the Romania National Council for Higher Education Funding, CNFIS, project number CNFIS-FDI-2025-F-0603.

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Cite:

UFJ Style

Luca M.I., Ungureanu-Iuga M., Batariuc A., Mironeasa S. (2025), Effect of carrot pomace addition on rheology and texture of soft wheat pasta dough, *Ukrainian Food Journal*, 14(2), pp. 259–272, https://doi.org/10.24263/2304-974X-2025-14-2-6

APA Style

Luca, M.I., Ungureanu-Iuga, M., Batariuc, A., & Mironeasa, S. (2025). Effect of carrot pomace addition on rheology and texture of soft wheat pasta dough. *Ukrainian Food Journal, 14*(2), 259–272. https://doi.org/10.24263/2304-974X-2025-14-2-6

Variation in enzymatic activity and phenolic content in grapes across maturity stages

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Abstract

Introduction. This study is aimed at investigating the changes in the activity of certain oxidoreductase-class enzymes and content of phenolic compounds depending on the level of grape maturity.

Materials and methods. The white technical grape varieties Bayanshira and Rkatsiteli grown in Azerbaijan, as well as the introduced red technical variety Cabernet Sauvignon were used. The activities of ascorbate oxidase, o-diphenol oxidase, peroxidase, and catalase in grapes were determined at different stages of grape maturation. High-performance liquid chromatography was used for the analysis of phenolic compounds.

Results and discussion. Depending on the degree of grape maturity, oxidoreductase-class enzymes remain continuously active. The enzyme activity in the grape varieties Bayanshira, Rkatsiteli, and Cabernet Sauvignon, grown in the Samukh and Goygol regions, varies throughout the ripening stages, with the most pronounced changes observed in unripe and overripe berries. In the Bayanshira variety, enzyme activity in unripe berries increased by 12.5–22.8% and 15.8–38.4%; in ripe berries — by 3.1–5.6% and 3.5–7.9%; and in overripe berries — by 20.4–42.5% and 19.0–33.3%, respectively, which is consistent with the data for the other studied varieties. The lowest enzyme activity was recorded in ripe grapes.

Using chromatographic–mass spectrometric analysis, it was established that the studied grape varieties are rich in phenolic compounds such as flavonoids, phenolcarboxylic acids, and procyanidins. A total of 18 monomeric phenolic compounds were identified in the grape varieties, along with 21 anthocyanin compounds found in the Cabernet Sauvignon variety (a red technical grape). This indicates a higher content of phenolic compounds in the studied grape varieties.

Conclusions. To obtain high-quality brandy wine material, it is recommended to use fully ripened grapes, as they exhibit the lowest enzymatic activity. To enrich the wine material with phenolic and extractive substances, the grape juice should be fermented in contact with the mash for several days.

Keywords:

Grape
Maturity stages
Enzymatic activity
Phenolic content

Article history:

Received 22.09.2024
Received in revised form 21.02.2025
Accepted 30.06.2025

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DOI: 10.24263/2304-974X-2025-14-2-7

Introduction

One of the most significant groups of enzymes involved in the biochemical transformations of grapes is the oxidoreductase class of enzymes (Nabiev, 2010). These enzymes play a key role in oxidation–reduction reactions, which underlie processes, such as respiration, the increase in antioxidant content, the degradation of phenolic compounds, and other metabolic changes. Oxidoreductase activity is influenced by the degree of grape ripeness.

Phenolic compounds play an important role in winemaking. The quality of various types of wines, as well as brandy wine material, brandy spirit, and the brandy itself, largely depends on phenolic compounds and their transformation products, or the metabolites formed because of their conversion. It is important to note that the distinctive taste and aroma of brandy are closely associated with phenolic compounds (Fataliyev, 2012). Phenolic compounds have a significant impact on the sensory profile of wines, particularly in terms of color intensity and sensory characteristics such as density, astringency, and bitterness. Most of these compounds originate from the grapes themselves. Therefore, their content in wines is determined by the grape variety, its phenolic potential, and reserves (Ghiselli et al., 1998; Lee and Koh, 2001).

Phenolic compounds are widely distributed in plants, including grapes, and possess important technological properties (Cosme, 2018). The concentration of phenolic compounds is higher in grape skins than in the juice and pulp (Bendaali, et al., 2022). One of the key biological and technological features of phenolic compounds is their antioxidant and antimicrobial activity. Grape varieties and wines rich in phenolic compounds are not only more resistant to diseases but are also rich in extractive substances (Nabiev and Moslemzadeh, 2008).

To produce high-quality brandy, it is essential first to prepare high-quality brandy wine material that meets modern requirements in terms of sensory and chemical characteristics, ensuring a rich flavor and product stability. In the production of brandy wine material, special attention must be paid to the grape variety, its degree of ripeness, its richness in chemical components, and other factors. Brandy wine material should be rich in extractive substances (Dhiman et al., 2022).

Despite the large number of studies devoted to brandy production technology, the impact of the maturity level of technical grape varieties on enzymatic activity and the accumulation of phenolic compounds during the production of brandy wine material remains insufficiently studied, especially with regard to specific grape varieties cultivated in Azerbaijan, such as Bayanshira and Rkatsiteli.

To this end, we examined the changes in the activity of certain oxidoreductase-class enzymes depending on the degree of grape maturity, and identified phenolic compounds, such as flavonoids, phenolcarboxylic acids, stilbenes, and procyanidins in grape varieties used for the production of brandy wine material. Studying the relationship between grape maturity, enzymatic activity, and the chemical composition of the wine material is a relevant and scientifically grounded area of research that contributes to improving the quality of the final product — brandy.

The aim of this study was to determine the influence of the maturity stages of technical grape varieties (Bayanshira, Rkatsiteli, and Cabernet Sauvignon) on the activity of oxidoreductase-class enzymes and the content of phenolic compounds, in order to justify the selection of optimal raw materials for the production of brandy wine material.

Scientific novelty: A comparative study was conducted on the enzymatic activity of oxidoreductases and the content of phenolic compounds in technical grape varieties (Bayanshira, Rkatsiteli, and Cabernet Sauvignon) at different stages of maturity.

Characteristic changes in enzyme activity and the profile of phenolic compounds depending on the maturity level were identified. A correlation between berry maturity and biochemical composition was also established, which is important for optimizing grape processing technologies in the production of high-quality brandy wine material.

Materials and methods

Grape varieties widely cultivated in the Samukh and Goygol regions of Azerbaijan — the white technical varieties Bayanshira and Rkatsiteli, as well as the red variety Cabernet Sauvignon were used in the study (Figure 1). Grapes were collected at different stages of ripening: unripe, ripe, and overripe berries.

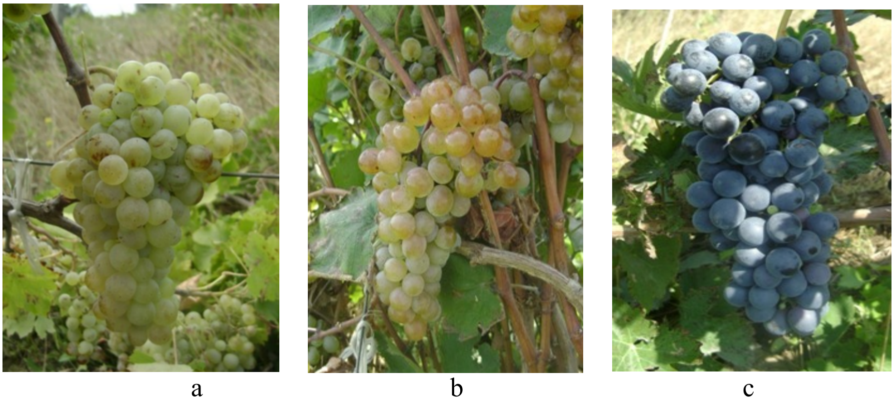

Figure 1. White technical grape variety Bayanshira (a), white technical grape variety Rkatsiteli (b), and red technical grape variety Cabernet Sauvignon (c)

Bayanshira. This white technical grape variety originates from Azerbaijan, where it has become widely cultivated due to its excellent adaptation to local conditions. Bayanshira is also successfully grown in Uzbekistan, Turkmenistan, Kazakhstan, Tajikistan, Ukraine, Moldova, and other countries, where it is widely used in winemaking. The grape is characterized by a light fruity aroma with citrus notes. The sugar content in the berries ranges from 16 to 20%, and the acidity is between 5.5 and 8 g/L. The variety is classified as late-ripening (Fig. 1a).

Rkatsiteli. The homeland of the Rkatsiteli grape variety is Kakheti, Georgia. In Azerbaijan, it is primarily cultivated in the Goygol and Samukh regions. Grapes grown in foothill zones tend to accumulate more sugars compared to those cultivated in lowland areas. Currently, Rkatsiteli is used not only for the production of table and brandy wine materials but also for various types of fortified wines. This variety belongs to the medium-late ripening group. In fully ripened Rkatsiteli berries, the sugar content reaches 18–24%, while the titratable acidity is 5.5–6.5 g/L. The variety is also known for its good resistance to grapevine diseases (Figure 1b).

Cabernet Sauvignon is the most widely cultivated grape variety in the world (Lončarić et al., 2022; Ramos et al., 2018). It is a red technical grape variety that has been cultivated since the 17th century in the Bordeaux region of France. In Azerbaijan, Cabernet Sauvignon began to be cultivated in the late 19th – early 20th century, initially in collections of research institutions as well as in farms of the Karabakh, Shirvan, and Ganja-Gazakh regions. Today, this variety is most widely grown in the winemaking farms of the Goygol and Samukh districts (Figure 1c).

Methods

Determination of enzymatic activity

Enzyme activity in this study was determined using the method described by Nabiyev et al. (2008). Enzyme activity is defined by the amount of substrate in mg, broken down by the enzyme in 30 minutes in 1 gram of sample mass.

The determination of ortho-diphenol oxidase activity is based on the oxidation of dimethyl-p-phenylenediamine. The resulting compound is colored blue-violet (Nabiyev et al., 2008). In the cuvette, 2 mL of dimethyl-p-phenylenediamine solution, 1 mL of buffer (if necessary), and 0.5 mL of enzyme extract were mixed. The mixture was stirred, and the timing was started immediately. The optical density of the solution was measured after 1, 2, 3, and 5 minutes at a wavelength of approximately 540–550 nm. A control sample was prepared without the enzyme (buffer was added instead) to eliminate the influence of auto-oxidation. This method made it possible to quantitatively determine the activity of ortho-diphenol oxidase by the increase in color intensity of the solution caused by the oxidation of dimethyl-p-phenylenediamine.

Oxidative and reductive processes play a crucial role in the activity of the enzyme peroxidase. This enzyme catalyzes the oxidation of various phenols and aromatic amines. The method for determining peroxidase activity is based on measuring the time it takes for the test solution to reach a specific optical density. Benzidine is used as the substrate, and its oxidation results in the formation of a blue-colored compound. Based on the intensity of this color, the enzyme's activity can be preliminarily assessed using a photoelectric colorimeter (Nabiyev et al., 2008).

The reaction mixture containing 2.0 ml of benzidine solution, 0.5 ml of H_2O_2 solution, and 0.5 ml of buffer was mixed thoroughly with 0.2 ml of the enzyme solution (peroxidase extract). Time measurement was started immediately, and the cuvette was simultaneously placed in the photoelectric colorimeter. The optical density of the solution was measured at 590–610 nm every 10 seconds. The time at which a specific optical density was reached was recorded. A control experiment without the enzyme (with buffer added instead) was carried out in parallel to exclude the possibility of spontaneous benzidine oxidation.

Catalase breaks down hydrogen peroxide, which is formed during tissue respiration, into water and molecular oxygen. A titrimetric method for determining catalase activity was based on measuring the remaining hydrogen peroxide using potassium permanganate, was selected by us as a prototype (Nabiyev et al., 2008).

In the experiment, 5 ml of H_2O_2 solution and 1 ml of enzyme solution (catalase) were added to a test tube. The mixture was then incubated at 37 °C for 1–3 minutes. The reaction was stopped by adding 5 ml of 10% H_2SO_4. Immediately afterward, the solution was titrated with 0.01 N $KMnO_4$ until a persistent pink color appeared. A control sample was prepared following the same procedure, but the enzyme was added after the acid to prevent the decomposition of H_2O_2.

Determining of phenolic compounds

For the analysis, 50 g of grape berries were taken and crushed in a porcelain mortar, then transferred into an Erlenmeyer flask. The sample was then rinsed with methanol in a 1:2 ratio. The flask was sealed and stored in a refrigerator at +4°C for 24 hours. Afterward, the resulting extract was filtered through a Büchner funnel. The residue was extracted again with methanol to remove colored substances. The filtrate was combined with the initial extract and evaporated using a rotary evaporator at 40°C.

The obtained aqueous residue was brought to the original volume by adding deionized water and then passed through a C18 cartridge (Waters Sep-Pak 6 cc tC18). Prior to this, the cartridge is preconditioned by passing through it two volumes (2 ml each) of methanol. The remaining methanol was rinsed off with deionized water. Then, the solution containing phenolic compounds was transferred into the cartridge. To remove non-adsorbed substances (e.g., phenolic acids and others), two volumes of deionized water are added to the cartridge. Adsorbed anthocyanins were eluted from the cartridge with 50 ml of deionized water. The resulting solution was then evaporated in a rotary evaporator at 40°C until a dry residue is obtained. The resulting dry residue was dissolved in deionized water, and the sample is ready for analysis.

High-performance liquid chromatography (HPLC) ProStar-MS 500 (Varian, USA) was used for the analysis of phenolic compounds in the grape sample (Flamini and Traldi, 2010).

Statistical Analysis

The changes in enzyme activities depending on the ripeness stage of the studied grape varieties were determined by the triplicate assay method. Results are presented as mean ± standard deviation. Statistical calculations were performed using software and Microsoft Excel.

Results and discussion

The changes in the enzymatic activities in the grape varieties depending on the ripeness stage

The changes in the activities of the enzymes ascorbate oxidase, o-diphenol oxidase, peroxidase, and catalase in the studied grape varieties were investigated depending on their ripeness stage (unripe, ripe, and overripe).

The changes in enzyme activity at different ripeness stages of grape varieties used for brandy wine production are presented in Tables 1–4 and Figures 2 and 3.

As seen (Tables 1 and 2), the enzyme activity of the grape varieties Bayanshira, Rkatsiteli, and Cabernet Sauvignon, grown in the Samukh district, tends to consistently increase depending on their ripeness stage. The study established that, depending on the ripeness of the grapes, more pronounced changes in enzyme activities were most often observed in unripe and overripe grape berries (Table 1).

In the unripe Bayanshira grape variety, the activity of the enzyme ascorbate oxidase increased by 12.5% before processing; in the ripe grapes, this increase was 5.4%, and in the overripe ones — 42.5% (Table 2).

Analysis of the research results revealed that the increase in enzyme activity in unripe Bayanshira grape berries ranged from 12.5% to 22.8%, in ripe berries — from 3.1% to 5.4%, and in overripe berries — from 20.4% to 42.5%. These values are consistent with those observed in the Rkatsiteli and Cabernet Sauvignon grape varieties.

Table 1

Activities of oxidoreductase class enzymes depending on ripeness stage of grape varieties grown in Samukh district, µmol/min

Enzymes	**Unripe**			**Ripe**			**Overripe**		
	Days of activity measurement								
	1	**5**	**10**	**1**	**5**	**10**	**1**	**5**	**10**
•									
Ascorbate oxidase	0.64 ±0.01	0.68 ±0.01	0.72 ±0.01	0.74 ±0.01	0.75 ±0.01	0.78 ±0.02	0.80 ±0.01	0.98 ±0.01	1.14 ±0.02
o-Diphenol oxidase	0.70 ±0.01	0.78 ±0.01	0.86 ±0.01	0.88 ±0.01	0.90 ±0.01	0.92 ±0.01	0.94 ±0.02	1.01 ±0.01	1.28 ±0.02
Peroxidase	2.20 ±0.01	2.42 ±0.02	2.54 ±0.02	2.60 ±0.02	2.62 ±0.01	2.68 ±0.01	2.70 ±0.01	3.02 ±0.02	3.25 ±0.02
Catalase	0.33 ±0.01	0.35 ±0.01	0.39 ±0.01	0.40 ±0.02	0.41 ±0.02	0.42 ±0.01	0.42 ±0.01	0.48 ±0.01	0.54 ±0.02
Rkatsiteli									
Ascorbate oxidase	0.68 ±0.01	0.72 ±0.01	0.78 ±0.01	0.80 ±0.01	0.82 ±0.01	0.86 ±0.02	0.88 ±0.01	1.02 ±0.01	1.20 ±0.02
o-Diphenol oxidase	0.76 ±0.01	0.84 ±0.01	0.92 ±0.02	0.94 ±0.01	0.98 ±0.01	0.01 ±0.01	1.04 ±0.02	1.16 ±0.01	0.38 ±0.01
Peroxidase	2.42 ±0.01	2.54 ±0.01	2.76 ±0.02	2.78 ±0.01	2.82 ±0.01	2.88 ±0.01	2.90 ±0.01	3.10 ±0.01	3.34 ±0.01
Catalase	0.40 ±0.02	0.44 ±0.01	0.48 ±0.01	0.51 ±0.02	0.53 ±0.01	0.54 ±0.01	0.56 ±0.01	0.62 ±0.01	0.71 ±0.01
Cabernet Sauvignon									
Ascorbate oxidase	0.55 ±0.01	0.58 ±0.01	0.62 ±0.01	0.63 ±0.01	0.64 ±0.01	0.66 ±0.01	0.68 ±0.01	0.82 ±0.01	0.98 ±0.01
o-Diphenol oxidase	0.66 ±0.01	0.72 ±0.02	0.78 ±0.01	0.80 ±0.01	0.82 ±0.01	0.84 ±0.01	0.88 ±0.01	0.98 ±0.01	1.20 ±0.01
Peroxidase	1.76 ±0.01	1.98 ±0.01	2.02 ±0.02	2.04 ±0.01	2.08 ±0.01	2.12 ±0.01	2.20 ±0.02	2.42 ±0.01	2.52 ±0.01
Catalase	0.29 ±0.01	0.33 ±0.02	0.35 ±0.01	0.36 ±0.01	0.37 ±0.01	0.39 ±0.01	0.39 ±0.02	0.42 ±0.01	0.48 ±0.01

According to Tables 1 and 2, depending on the ripeness stage of the grapes, the highest activity was exhibited by the enzyme peroxidase, followed by o-diphenol oxidase, then ascorbate oxidase, while the lowest enzymatic activity was observed for catalase.In the Bayanshira grape variety grown in the Samukh district, the activity of ascorbate oxidase in unripe berries increased by 12.5%, in ripe berries by 5.4%, and in overripe berries by 42.5%. This pattern remained consistent up to the stage of grape processing.

Analysis of the literature indicates that the enzyme ascorbate oxidase catalyzes the conversion of ascorbic acid (vitamin C) into D-dehydro-L-ascorbic acid, while the enzyme o-diphenol oxidase catalyzes the oxidation of ortho- and para-diphenols into o-quinones (Nabiev et al., 2008). The change or darkening in the color of grape and other plant juices, as well as certain food products, is associated with increased activity of o-diphenol oxidase (Gonzalez et al., 2023).

It is known that enzyme activity increases with the ripening stage of grapes, contributing to the breakdown of nutritional components in the fruit for use in respiratory processes (García-Martínez et al., 2021). This once again confirms that metabolic processes in the studied grape varieties occur constantly.

Table 2

Activity of oxidoreductase class enzymes depending on ripeness stage of grape varieties, grown in Samukh district, µmol/min

Enzymes	Unripe				Ripe				Overripe			
	In	F	Δ	%	In	F	Δ	%	In	F	Δ	%
Bayanshira												
Ascorbate oxidase	0.64	0.72	0.08	12.5	0.74	0.78	0.04	5.4	0.80	1.14	0.34	42.5
o-Diphenol oxidase	0.70	0.86	0.16	22.8	0.88	0.93	0.05	5.6	0.94	1.28	0.34	36.2
Peroxidase	2.20	2.54	0.34	15.4	2.60	2.68	0.08	3.1	2.70	3.25	0.55	20.4
Catalase	0.33	0.39	0.06	18.2	0.40	0.42	0.02	5.0	0.42	0.54	0.12	28.6
Rkatsiteli												
Ascorbate oxidase	0.68	0.78	0.10	14.7	0.80	0.86	0.06	7.5	0.88	1.20	0.32	36.4
o-Diphenol oxidase	0.76	0.92	0.16	21.0	0.94	1.01	0.07	7.4	1.04	1.38	0.34	32.7
Peroxidase	2.42	2.76	0.34	14.0	2.78	2.88	0.10	3.6	2.90	3.34	0.44	15.2
Catalase	0.40	0.48	0.08	0.20	0.51	0.54	0.03	5.9	0.56	0.71	0.15	26.8
Cabernet Sauvignon												
Ascorbate oxidase	0.55	0.62	0.07	12.7	0.63	0.66	0.03	4.8	0.68	0.98	0.30	44.1
o-Diphenol oxidase	0.66	0.78	0.12	18.2	0.80	0.84	0.04	5.0	0.88	1.20	0.32	36.4
Peroxidase	1.76	2.02	0.26	14.8	2.04	2.12	0.08	3.9	2.20	2.52	0.32	14.5
Catalase	0.29	0.35	0.06	20.7	0.36	0.39	0.03	8.3	0.39	0.46	0.07	17.9

Note: In- initial, F – final, Δ – difference.

The changes in enzyme activity depending on the ripeness stage of the Bayanshira grape variety grown under the conditions of the Samukh district are shown in Figure 2.

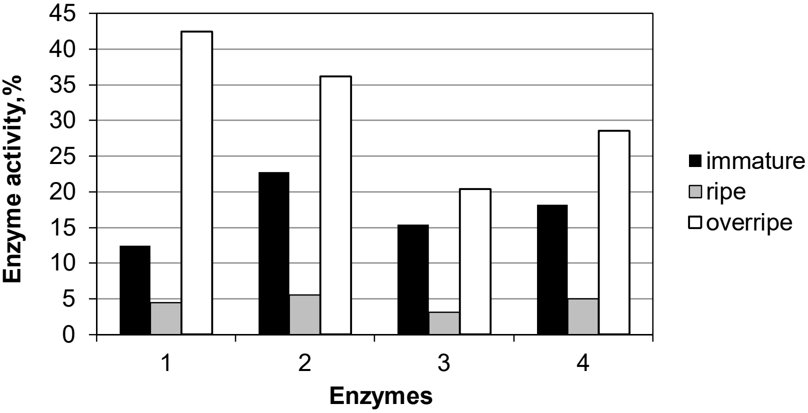

Figure 2. Enzyme activity, depending on the ripeness stage of the Bayanshira grape variety grown under the conditions of the Samukh district:
1 – ascorbate oxidase, 2 – o-diphenol oxidase, 3 – peroxidase, 4 – catalase

As seen in the figure, the activity of the studied oxidoreductases varies depending on the ripeness stage of the Bayanshira grape variety. Enzyme activity was high in both unripe and overripe grape berries. A similar pattern was also observed in the other grape varieties.

Tables 3 and 4 show the changes in the activity of the studied enzymes during grape storage prior to processing, depending on the ripeness stage of the technical grape varieties Bayanshira, Rkatsiteli, and Cabernet Sauvignon, grown in the Goygol district.

It became evident that the activity of these enzymes changes differently depending on the degree of ripening of the grapes. For each grape variety, the dynamics of enzyme activity during ripening were determined through triplicate measurements. For example, in unripe Bayanshira grapes, the activity of ascorbate oxidase was 0.58 on the first day, 0.62 on the fifth day, and 0.68 on the tenth day. As shown in the table, a consistent activation of enzymes was observed across all the studied grape varieties.

Table 3

Activity of oxidoreductase class enzymes depending on the ripeness stage of grape varieties grown in the Goygol district, µmol/min

Enzymes	Unripe			Ripe			Overripe		
	Days of activity measurement								
	1	5	10	1	5	10	1	5	10
Bayanshira									
Ascorbate oxidase	0.58 ±0.01	0.62 ±0.01	0.68 ±0.01	0.70 ±0.01	0.72 ±0.01	0.73 ±0.02	0.75 ±0.01	0.88 ±0.01	1.10 ±0.02
o-Diphenol oxidase	0.68 ±0.01	0.76 ±0.01	0.84 ±0.01	0.85 ±0.01	0.86 ±0.01	0.88 ±0.01	0.90 ±0.02	1.05 ±0.01	1.20 ±0.02
Peroxidase	1.90 ±0.01	2.00 ±0.02	2.20 ±0.02	2.30 ±0.02	2.35 ±0.01	2.40 ±0.01	2.40 ±0.02	2.60 ±0.02	2.95 ±0.01
Catalase	0.26 ±0.01	0.32 ±0.01	0.36 ±0.01	0.38 ±0.02	0.39 ±0.02	0.41 ±0.01	0.42 ±0.01	0.46 ±0.01	0.50 ±0.02
Rkatsiteli									
Ascorbate oxidase	0.64 ±0.01	0.68 ±0.01	0.75 ±0.01	0.76 ±0.01	0.78 ±0.01	0.81 ±0.02	0.82 ±0.01	0.96 ±0.01	1.05 ±0.02
o-Diphenol oxidase	0.72 ±0.01	0.80 ±0.02	0.88 ±0.01	0.90 ±0.02	0.92 ±0.01	0.94 ±0.01	0.94 ±0.01	1.10 ±0.02	1.35 ±0.01
Peroxidase	2.32 ±0.01	2.54 ±0.02	2.74 ±0.01	2.76 ±0.02	2.86 ±0.01	2.90 ±0.02	2.90 ±0.02	3.10 ±0.02	3.26 ±0.01
Catalase	0.36 ±0.01	0.44 ±0.01	0.44 ±0.01	0.46 ±0.01	0.48 ±0.01	0.49 ±0.01	0.50 ±0.02	0.58 ±0.01	0.64 ±0.01
Cabernet Sauvignon									
Ascorbate oxidase	0.51 ±0.01	0.55 ±0.02	0.59 ±0.01	0.60 ±0.03	0.62 ±0.02	0.64 ±0.01	0.65 ±0.01	0.74 ±0.01	0.96 ±0.01
o-Diphenol oxidase	0.62 ±0.01	0.70 ±0.02	0.76 ±0.01	0.78 ±0.01	0.80 ±0.02	0.82 ±0.01	0.84 ±0.01	1.01 ±0.02	1.10 ±0.01
Peroxidase	1.70 ±0.02	1.84 ±0.01	1.96 ±0.01	1.98 ±0.01	2.05 ±0.02	2.10 ±0.02	2.10 ±0.02	2.32 ±0.01	2.48 ±0.01
Catalase	0.28 ±0.02	0.31 ±0.01	0.34 ±0.01	0.36 ±0.01	0.38 ±0.01	0.40 ±0.02	0.40 ±0.02	0.44 ±0.01	0.48 ±0.01

Changes in enzyme activity depending on the ripeness stage of the studied grape varieties are expressed as percentages (Table 4). Among the studied grape varieties, relatively small changes in enzyme activity were recorded in ripe berries compared to the other ripeness stages.

The study of ripe Rkatsiteli grapes prior to processing revealed a 4.4% increase in o-diphenol oxidase activity. In unripe grapes, this increase was 22.2%, while in overripe grapes it reached 43.6%. Among unripe grapes grown in the Goygol district, o-diphenol oxidase activity increased by 23.5% in the Bayanshira variety, 22.2% in Rkatsiteli, and 22.6% in Cabernet Sauvignon. In ripe grapes, the enzyme activity increase was 3.5% for Bayanshira, 4.4% for Rkatsiteli, and 5.1% for Cabernet Sauvignon.

Table 4

Activity of enzymes depending on the ripeness stage of grape grown in the Goygol district

Enzymes	Unripe				Ripe				Overripe			
	In	**F**	**Δ**	**%**	**In**	**F**	**Δ**	**%**	**In**	**F**	**Δ**	**%**
Bayanshira												
Ascorbate oxidase	0.58	0.68	0.10	17.2	0.70	0.73	0.03	4.2	0.75	1.10	0.35	26.2
o-Diphenol oxidase	0.68	0.84	0.16	23.5	0.85	0.88	0.03	3.5	0.90	1.20	0.30	33.3
Peroxidase	1.90	2.20	0.30	15.8	2.30	2.40	0.10	4.3	2.40	2.95	0.55	22.9
Catalase	0.26	0.36	0.10	38.4	0.38	0.41	0.03	7.9	0.42	0.50	0.08	19.0
Rkatsiteli												
Ascorbate oxidase	0.64	0.75	0.11	17.2	0.76	0.81	0.05	6.6	0.82	1.05	0.23	28.1
o-Diphenol oxidase	0.72	0.88	0.16	22.2	0.90	0.94	0.04	4.4	0.94	1.35	0.41	43.6
Peroxidase	2.32	2.74	0.42	18.1	2.76	2.90	0.14	5.1	2.90	3.26	0.36	12.4
Catalase	0.36	0.44	0.08	22.2	0.46	0.49	0.03	6.5	0.50	0.64	0.14	28.0
Cabernet Sauvignon												
Ascorbate oxidase	0.51	0.59	0.08	15.7	0.60	0.65	0.05	8.3	0.65	0.96	0.31	47.7
o-Diphenol oxidase	0.62	0.76	0.14	22.6	0.78	0.82	0.04	5.1	0.84	1.10	0.26	30.9
Peroxidase	1.70	1.96	0.26	15.3	1.98	2.10	0.12	6.1	2.10	2.48	0.38	18.1
Catalase	0.28	0.34	0.06	21.4	0.37	0.40	0.04	7.5	0.40	0.48	0.08	20.0

Note: In – initial, F – final, Δ – difference.

As shown in Table 4, o-diphenol oxidase activity increased by 33.3% in overripe Bayanshira grapes, 43.6% in Rkatsiteli, and 30.9% in Cabernet Sauvignon. The data also indicate that the activity of other enzymes changed only slightly in the ripe grape varieties. These findings are consistent with the results obtained for the other enzymes studied.

During grape processing, as well as throughout fermentation and even during the formation and maturation of wine material, the activity of o-diphenol oxidase must be continuously regulated. Otherwise, increased activity of this enzyme may lead to changes in the color of the final products and the degradation of nutritional components in their composition (Nabiev et al., 2008).Therefore, when producing cognac wine material from various grape varieties, special attention must be paid to the ripeness stage of the grapes.

This is because the production of high-quality cognac wine material largely depends on the ripeness stage of the grapes. A significant increase in enzyme activity creates conditions for the breakdown of the grape's nutritional components for respiration processes. Therefore, in unripe and overripe grape varieties, the lower content of extractive substances makes it impossible to produce high-quality cognac wine material. These grape varieties also exhibit a low content of aromatic compounds.

The dynamics of changes in enzyme activity depending on the ripeness stage of the Cabernet Sauvignon grape variety grown under the conditions of the Goygol district are shown in Figure 4. Enzymatic activity was expressed in percentage as relative activity, i.e. demonstrating how much the enzyme activity increased comparative to the initial value.

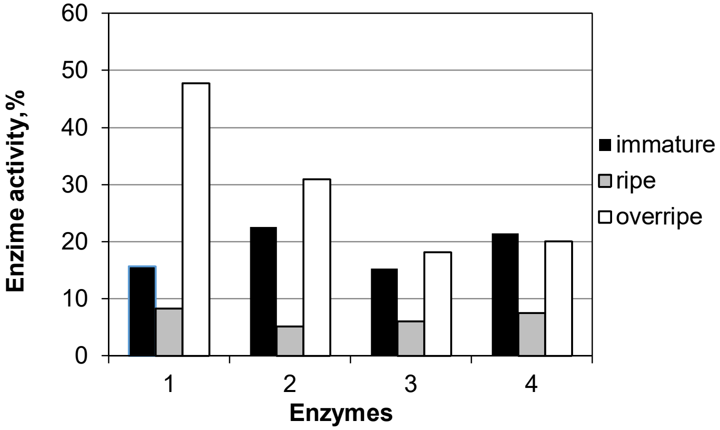

Figure 3. Enzyme activity, depending on the ripening stage of Cabernet Sauvignon grapes grown in the Goygol region:
1 – Ascorbate oxidase, 2 – o-Diphenol oxidase, 3 – Peroxidase, 4 – Catalase

The biochemical processes occurring in grapes at different stages of ripening determine the quality and chemical composition of the berries, which ultimately affects the organoleptic and technological properties of the final product (Fataliyev, 2012). The quality of the raw materials used in processing, as well as the final product, largely depends on enzymatic processes. Therefore, it is essential to regulate enzymatic processes that occur both during the ripening of raw materials, including grapes, and during their processing.

The enzyme peroxidase catalyzes the oxidation of polyphenols and a number of aromatic amines in the presence of hydrogen peroxide. The enzyme catalase, by breaking down hydrogen peroxide, formed during tissue respiration, into water and molecular oxygen, protects the product from toxic effects (Nabiev et al., 2008).

As seen in Figure 2, the activity of all enzymes tends to increase depending on the ripening stage of Cabernet Sauvignon grapes. A relatively slight increase in enzyme activity was observed in fully ripened grape berries (Figure 2). In unripe and overripe grapes, higher activity of the enzyme ascorbate oxidase contributes to a more intensive breakdown of vitamin C, exceeding normal levels. This pattern is also observed in the other enzymes under study. Based on the data in Figures 1 and 2, it becomes evident that the smallest changes in enzyme activity were detected in ripened grape varieties.

Thus, the results of the study showed that enzymes of the oxidoreductase class remain in an active form throughout the entire ripening period of the grapes.

The slightest increase in enzyme activity was observed in fully ripened grape varieties. In these ripened grapes, the minimal changes in enzyme activity reduce the breakdown of nutrients during respiration. Therefore, for the production of high-quality cognac wine material, it is advisable to use ripened technical grape varieties, with careful attention paid to their ripening stage.

The presence of phenolic compounds in grapes and wine contributes to their enrichment with aromatic substances (Dutra et al., 2021). As a result of the hydrolysis of dimeric, oligomeric, and polymeric phenolic compounds present in wine, aromatic substances (vanillin, syringaldehyde, coniferyl, sinapic, ferulic compounds, and their corresponding aldehydes, alcohols, etc.) are formed, which positively influence the organoleptic properties and flavor quality of the wine (Kazimova et al., 2022).

In the course of the study, the content of phenolic compounds in grape varieties intended for the production of cognac wine material was determined using chromate-mass spectrometry.

Table 5 presents selected phenolic compounds identified in the studied grape varieties. Chromatography–mass spectrometry analysis revealed the presence of 18 distinct phenolic compounds.

Table 5

Phenolic compound content in grape varieties

Phenolic compounds	**M.m.**	**Grape varieties**					
		Bayanshira		**Rkatsiteli**		**Cabernet Sauvignon**	
		DT, min	**mg/kg**	**DT, min**	**mg/kg**	**DT, min**	**mg/kg**
(-)epicatechin	289	20.344	253.98	20.130	670.9	20.124	54.76
(+)catechin	289	16.570	164.37	16.376	840.1	16.206	88.58
Chlorogenic acid	352	15.286	324.64	15.375	632.2	14.990	54.53
Caffeic acid	179	17.880	103.8	18.162	474.54	17.806	23.93
Vanillin	151	22.095	3.98	21.908	81.0	22.212	27.95
Trans-p-coumaroylvinyl acid	295	traces	-	traces	-	-	-
ProcyanidinB1,B3,B4,B5	577	traces	-	-	-	-	-
ProcyanidinC1,T2,T3	865	-	-	-	-	-	-
Quercetin-3-o-glucuronide	477	traces	-	traces	-	traces	-
Quercetin-3-o-galactoside	463	-	-	traces	-	-	-
Myricetin-3-o-glucuronide	493	traces	-	-	-	-	-
Trans-caffeoylvinyl acid	311	-	-	traces	-	-	-
Cis-trans-resveratrol-3-o-glucoside	389	-	-	traces	-	-	-
Myricetin-3-o-glucoside	479	-	-	traces	-	-	-
Trimeric procyanidin	865	-	-	traces	-	-	-
Gallic acid	301	traces	-	-	-	traces	
Astilbin	449	-	-	-	-	-	-
Epicatechin-3-o-gallate	441	-	-	-	-	-	-

Note: M. m. – molecular mass; DT – determination time, min.

Data present in Table 5 show that the content of phenolic compounds is higher in red grape varieties compared to white varieties. For example, the red grape variety Cabernet Sauvignon contained 54.76 mg/kg of epicatechin, while the white grape variety Rkatsiteli contained 670.9 mg/kg, and the Bayanshira variety contained 253.98 mg/kg.

A comparison of the varieties shows that vanillin is present in the greatest amount in the Rkatsiteli variety. While Rkatsiteli contained 81.0 mg/kg of vanillin, this value was 3.98 mg/kg in Bayanshira and 27.95 mg/kg in Cabernet Sauvignon. The other phenolic compounds studied in red grape varieties, particularly in Cabernet Sauvignon, are present in higher amounts compared to white grape varieties (Table 5).

The study revealed that the grape variety Bayanshira contains seven different phenolic compounds, among which quercetin-3-o-glucuronide and gallic acid are present in trace amounts. In the Rkatsiteli variety, 14 phenolic compounds were identified, five of which were found in higher concentrations, while nine were present in trace amounts. In the Cabernet Sauvignon variety, seven phenolic compounds were detected (Table 5).

The molecular weights of the phenolic compounds also vary (Jimenez-Lopez et al., 2021). The lowest molecular weight was observed in vanillin, while the highest molecular weight was found in trimeric procyanidin and procyanidins C1, C2, and C3 (molecular weight 865) (Capanoglu et al., 2013). It was established that the studied grape varieties contain a significant amount of phenolic compounds. The results of the analysis showed that the Rkatsiteli grape variety surpasses the other varieties in terms of phenolic content.

Phenolic compounds are considered to be the main indicators of the composition of wines obtained from other fruits, as well as in grapes (Chiusano et al., 2015). Phenolic compounds determine the quality indicators of the product, such as its appearance, taste and aroma. Phenolic compounds play an important role in the maturation of wines, giving them physiological and colloidal properties (Fataliyev et al., 2025).

Another group of phenolic compounds—anthocyanins—are widespread in plants, including grapes (Moldovan, 2020). Anthocyanins are the most common flavonoids found in grapes and wine (Baydar et al., 2010). They are dyes that primarily accumulate in the skins of red and pink grape varieties (Baiano et al., 2009). However, there are also grape varieties, such as Saperavi, in which anthocyanins have been detected in the pulp (Garcha et al., 2003).

Recent studies have shown that plants, including grapes, contain more than 10 anthocyanin aglycones (Kulcan et al., 2015). Newly identified anthocyanins are typically named after the plant in which they are found. In plants, anthocyanin aglycones are usually present in the form of glycosides (Kanakis et al., 2005). Anthocyanins are also referred to as anthocyanidins and anthocyanins (Woźniak et al., 2014). The distribution of anthocyanins in grapes depends on the characteristics of the variety, climatic conditions, and other factors (Yılmaz et al., 2015).

Since anthocyanins possess antioxidant and antimicrobial properties, they play an important role in the processing of table wines and other types of wine (Lorrain et al., 2015). Anthocyanin monoglucosides inhibit the growth of fungi such as *Botrytis cinerea* (Rodriguez-Saona and Wrotstad, 2001). One of the main biological properties of anthocyanins is their ability to reduce the levels of complex fats, including cholesterol, in the body, as well as their beneficial effect on the elimination of radionuclides from the body (Lima, 2014). Therefore, in this study, changes in anthocyanin content in the red grape variety Cabernet Sauvignon were examined.

Quantitative and qualitative changes in anthocyanins in the Cabernet Sauvignon grape variety observed during the study are presented in Table 6.

Table 6

Anthocyanin content in Cabernet Sauvignon grape (% of total anthocyanins)

Anthocyanins	M.m.	Content,%	Determination time, min
delphinidin-3,5-o-diglucoside	627	traces	-
petunidin-3,5-o-diglucoside	641	0.0875	14.299
delphinidin-3-o-monoglucoside	465	2.9745	14.796
malvidin-3,5-o-diglucoside	655	-	-
cyanidin-3-o-monoglucoside	449	1.5789	15.163
petunidin-3-o-monoglucoside	479	5.1786	-
Peonidin-3-o-monoglucoside	463	10.6781	-
malvidin-3-o-monoglucoside	493	45.6857	16.467
delphinidin-3-o-acetylmonoglucoside	507	0.1534	-
petunidin-3-(6-o-p-coumaroyl), 5-o-diglucoside	787	traces	-
petunidin-3-o-acetylmonoglucoside	521	0.7645	-
malvidin-3-(6-o-p-coumaroyl),5-o-diglucoside	801	traces	-
peonidin-3-o-acetylmonoglucoside	505	0.0085	-
malvidin-3-o-acetylmonoglucoside	535	8.7632	19.624
delphinidin-3-(6-o-p-coumaroyl)monoglucoside	611	traces	-
malvidin-3-(6-o caffeoyl)monoglucoside	655	traces	-
cyanidin-3-(6-o-p-coumaroyl)monoglucoside	595	traces	20.705
petunidin-3-(6-o-p-coumaroyl)monoglucoside	625	2.8756	-
peonidin-3-(6-o-p-coumaroyl)monoglucoside	609	traces	21.998
malvidin-3-(6-O-p-coumaroyl)monoglucoside	639	18.0546	21.305
delphinidin-3-o-(6-o-p-coumaroyl)-pyruvic acid-monoglucoside	679	traces	-

As seen in Table 6, under local growing conditions, 21 mono- and diglucosideanthocyanins were identified in the technical grape variety Cabernet Sauvignon. According to a study by the world-renowned winemaker, who used chromatographic methods to identify between 6 and 17 anthocyanins in European grape varieties, including those grown in France, only monoglucosideanthocyaninswere found in red grape varieties cultivated in Europe (Spacil et al., 2008), with diglucosides absent from their composition. It should be noted that the presence of diglucosides in grapes—especially malvidindiglucoside—indicates a hybrid or genetically modified origin (Urpi-Sarda et al., 2009). The Cabernet Sauvignon variety, however, is natural and environmentally pure (Xia et al., 2010). It is known that Cabernet Sauvignon is an introduced grape variety (Dani et al., 2007). The diversity of the chemical characteristics of Cabernet Sauvignon grown in the western region of Azerbaijan is associated with the specific local soil and climatic conditions. As seen in Table 5, the molecular weights of anthocyanins vary. The highest molecular weights were recorded for malvidin-3-(6-o-p-coumaroyl), 5-o-diglucoside (801) and petunidin-3-(6-o-p-coumaroyl), 5-o-diglucoside (787). The lowest molecular weight was found in cyanidin-3-o-monoglucoside (449).

In the Cabernet Sauvignon grape variety, malvidin-3-o-monoglucoside accounted for 45.7% of the total anthocyanin content, malvidin-3-(6-o-p-coumaroyl) monoglucoside for 18.05%, and peonidin-3-o-monoglucoside for 10.7%. Other anthocyanin compounds present in Cabernet Sauvignon were found in smaller amounts. The studied white technical grape varieties (Bayanshira and Rkatsiteli), as well as the red variety Cabernet Sauvignon, are rich in phenolic compounds, including anthocyanins (Spranger et al., 2008). Phenolic compounds in grapes are mainly concentrated in the skin (Cosme et al., 2008). Therefore, in order to enrich the brandy wine material with extractive substances, it is advisable to ferment the grape juice in contact with the mash for several days (Ribeiro, 2018).

The conducted study confirms the feasibility of using the studied grape varieties for the production of cognac wine materials. Let's consider it promising to study in-depth the aspects covered in the work. The conducted study allowed us to establish the influence of the degree of grape maturity on the enzymatic activity and the content of phenolic substances in technical varieties grown in various agroclimatic conditions of Azerbaijan. However, like any scientific study, it has both advantages and certain limitations that should be taken into account when interpreting and practically applying the results obtained. The study of three stages of grape maturity (unripe, ripe, and overripe) allowed us to comprehensively evaluate the biochemical processes occurring in the berries during their ripening. This gives a more complete picture of the dynamics of enzymatic activity and the accumulation of phenolic compounds.

Conclusions

1. During the study, it has been established that enzymatic activity in the grape varieties Bayanshira, Rkatsiteli, and Cabernet Sauvignon, grown in the Samukh and Goygol regions of Azerbaijan, varies depending on the degree of berry ripeness. The most significant fluctuations in enzyme activity were observed in unripe and overripe samples. For example, in the Bayanshira variety, enzyme activity in unripe berries increased by 12.5–22.8% and 15.8–38.4%, in ripe berries by 3.1–5.6% and 3.5–7.9%, and in overripe berries by 20.4–42.5% and 19.0–33.3%, respectively. Similar trends were observed in the other grape varieties studied. The lowest enzymatic activity values were recorded in ripe grapes. In the ripe varieties, the smallest increase in enzyme activity was noted, indicating the completion of metabolic processes, including the breakdown of extractive substances.
2. Chromatography–mass spectrometry analysis revealed that the studied grape varieties are rich in phenolic compounds such as flavonoids, phenolic carboxylic acids, and procyanidins. Across the studied grape varieties, 18 monomeric phenolic compounds were identified, while 21 anthocyanin compounds were detected exclusively in the Cabernet Sauvignon variety, classified as a red technical grape. This indicates a higher content of phenolic substances in the grape varieties under study.
3. Studies have shown that the content of phenolic compounds, including anthocyanins, varies significantly among the grape varieties Cabernet Sauvignon, Rkatsiteli, and Bayanshira. Notably, Cabernet Sauvignon exhibits a high level of mono- and diglucoside anthocyanins, making it particularly suitable for producing high-quality wine materials – especially for brandy, where extractive substances play an important role. A key aspect is the variation in the molecular weights of anthocyanins and other phenolic compounds, which reflects the uniqueness of each variety depending on the region of cultivation.

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Cite:

UFJ Style

Nabiyev A., Kazimova I., Omarova E., Gasimova A. (2025), Variation in enzymatic activity and phenolic content in grapes across maturity stages, *Ukrainian Food Journal*, 14(2), pp. 273–289, https://doi.org/10.24263/2304-974X-2025-14-2-7

APA Style

Nabiyev, A., Kazimova, I., Omarova, E., & Gasimova, A. (2025). Variation in enzymatic activity and phenolic content in grapes across maturity stages. *Ukrainian Food Journal, 14*(2), 273–289. https://doi.org/10.24263/2304-974X-2025-14-2-7

Decolorization of liquid sugar products by carbon sorbents

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Keywords:

Sugar
Syrup
Purification
Decolorization
Sorbent

Article history:

Received 14.12.2024
Received in revised form 17.03.2025
Accepted 30.06.2025

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DOI: 10.24263/2304-974X-2025-14-2-8

Abstract

Introduction. This study aims to justify the use of carbon sorbents for adsorption purification (decolorization) of liquid sugar products and to determine the key patterns of the process.

Materials and methods. A sugar-containing product derived from sweet sorghum was used as the raw material in the study. To enhance the purification process and effectively remove coloring agents from the liquid sugar product, various carbon sorbents were utilized. These included novel carbon sorbent CS produced by modifying regenerated wood-based raw materials, and commercially available NORIT, and coconut shell-based activated carbon C607.

Results and discussion. It was established that the decolorization efficiency of the investigated sugar solutions using carbon sorbents depends on the duration of the process and the sorbent dosage, as described by mathematical relationships. High purification efficiency of sugar solutions from coloring substances is achieved with carbon sorbents at dosages of 0.5–1.5% relative to the product mass and a process duration of 20–30 minutes. An analysis of the decolorization kinetics of sugar solutions using the studied carbon sorbents was conducted. Based on a comparative analysis of the adsorption characteristics of the tested sorbents, it was found that the carbon sorbent (CS), at a dosage of 0.5–1.5%, achieved a decolorization efficiency of up to 80–85% and an adsorption capacity of 28–32 mg/g. This resulted in a reduction of solution color to 220–350 ICUMSA units, outperforming the corresponding values obtained with sorbents C607 and NORIT. The high efficiency of the CS carbon sorbent is attributed to its surface characteristics, particularly its micro- and mesoporous structure, which play a key role in determining the sorption kinetics of coloring substances during the liquid-phase purification of sugar-containing products.

Conclusion. The research results indicate the potential for the industrial implementation of carbon sorbent grade CS and its use for the purification of liquid sugar-containing products.

Introduction

Global trends in the development of the sugar industry are defined by key directions such as product range diversification, high quality of finished products, and environmental sustainability (Chorna et al., 2023). A relevant issue is the production of organic sugar, liquid sugars (sugar or invert syrup), as well as the manufacture of sugar-containing syrups from non-traditional plant raw materials, particularly from sweet sorghum (Abdelsalam et al., 2020; Husiatynska et al., 2023; Li et al., 2017; Thilagavathi et al., 2016). Liquid sugar products are becoming increasingly popular in the food and beverage industry due to their long shelf life and ease of use in the food industry (Khan et al., 1996; Mouelhi et al., 2014).

An important aspect of the quality of both crystalline sugar and liquid sugars is meeting organoleptic standards and color specifications. However, during the production of sugar-containing products, especially at the stage of concentration to syrup, reactions occur under the influence of high temperatures, resulting in the formation of intensely colored compounds (colorants) (Lohinova and Petrusha, 2023). The colorants in sugar production have a rather complex chemical structure that remains insufficiently studied, as continuous transformations occur within them. In general, colorants are divided into compounds formed as a result of the breakdown of hexoses and natural compounds present in the raw material that pass into the juice during the extraction or pressing process. These include plant pigments (chlorophylls, carotenoids, and xanthophylls), phenolic compounds, and flavonoids, which undergo complex chemical transformations during processing (Cabral et al., 2022; Zavaleta and Velasco, 2019). Because of a series of chemical reactions, high-molecular-weight compounds with colors ranging from yellow to dark brown are formed (Eggleston et al., 2009; Vercellotti et al., 2010). The intensity of color compound formation in juices and syrups depends on the quality of the raw materials, the duration and temperature of the technological process, the pH of the medium, the concentration of sugar-containing solutions, and the presence of non-sugar substances, which may act as catalysts or inhibitors of color formation.

The formation of color compounds can be classified into the following groups: melanins, melanoidins, products of alkaline-thermal transformation of hexoses, and caramels (Zhu et al., 2021; Zolfaghari et al., 2020). Their presence in sugar syrups leads to a deterioration in product quality and increased coloration, as well as an increase in the viscosity of intermediate products and a decline in the organoleptic properties of the final product due to the development of a bitter taste and a characteristic specific odor. The removal of color compounds is an extremely important task in the production of crystalline sugar, liquid sugar, invert syrup, and food syrups from sweet sorghum.

Currently, several methods are proposed and used for the purification of sugar-containing juices and syrups, including chemical treatment (Kibret, 2019); membrane filtration methods (including ultrafiltration) (Chibrikov et al., 2022); the use of adsorbents such as activated carbon and ion exchange purification (Xiao et al., 2022), among others. However, some of these methods pose potential health risks. For instance, residual amounts of sulfur dioxide during sulfitation or styrene and divinylbenzene monomers during ion-exchange purification may enter food solutions during processing, thereby potentially compromising food safety. Additionally, phosphate treatment removes only certain color compounds, mainly those with high molecular weight and a significant number of carboxylic groups (Kibret, 2019).

The use of carbon-based adsorbing materials, particularly activated carbon derived from agricultural waste, is becoming increasingly relevant for the decolorization of liquid sugar products. Due to its highly developed porous structure and large specific surface area, activated carbon possesses a high adsorption capacity for most contaminants and is widely used across various industries (Ahmad et al., 2010; Bello et al., 2017; Kosheleva et al., 2018). Its application is technologically simple, energy-efficient, relatively inexpensive, and poses no environmental risks. Owing to its ability to absorb and retain a wide range of pollutants, activated carbon serves as an effective adsorbent for the treatment and purification of drinking water, the removal of harmful substances from industrial wastewater, as well as the elimination of high-molecular-weight and colored compounds during the purification of various liquid food solutions (Ruiz-Velducea et al., 2024). Given the above, the use of a developed carbon sorbent based on renewable organic waste and its integration into the technological process of liquid sugar product manufacturing presents scientific and practical interest.

It should be noted that adsorption decolorization processes using activated carbon are widely applied in starch and syrup production. Both powdered and granular forms of activated carbon are used for this purpose. However, despite the significant advantages of activated carbon as a sorbent, its application is not widespread in sugar and liquid syrup production. This is likely due, largely, to economic factors, as food industries currently face a shortage of high-efficiency domestically produced sorbent materials. This necessitates the use of foreign analogues in technological processes, leading to increased production costs. At the same time, approximately 177.5 million tons of plant waste are generated annually in Ukraine, which could be used as raw material for the production of sorbent materials (Zhuk, 2022).

In the present study on the purification (decolorization) of colored compounds, a sugar-containing syrup derived from the stem juice of sweet sorghum was used. The sorghum stems were first cleaned of leaves and then pressed using a roller press to extract the raw juice, which was then used to produce food syrup.

Specifically, the stem sap has balanced nutritional value, containing easily absorbable sugars, comprising 53-85% sucrose and 15-54% fructose and glucose (Blatke, 2019; Rao et al., 2013). The sap contains bioactive substances: amino and organic acids, polyphenols, proteins, vitamins, minerals, and more (Eggleston et al., 2013). When sweet sorghum syrup is utilized in food product technologies, excellent taste and technological characteristics for new product types can be achieved. It can be considered a natural, safe, and complete substitute for sugar in food products.

This study aims to justify the use of carbon sorbents for the adsorption-based decolorization of liquid sugar products, particularly syrup obtained from sweet sorghum stem juice, and to determine the key patterns governing this process.

Materials and methods

Sugar syrup from sweet sorghum

The production of the syrup was carried out according to Hryhorenko et al. (2020), which involved thermal coagulation of non-sugars, a two-stage enzymatic hydrolysis of starch, separation of the coagulated precipitate by filtration with the addition of filter perlite, and additional ultrafiltration purification of the juice using a polyethylene terephthalate membrane with a pore diameter of 0.08–0.15 µm. The purified juice was then concentrated into syrup with a dry matter content of 70–75%.

The physicochemical characteristics of the syrup from sweet sorghum are presented in Table 1.

Table 1

Physicochemical characteristics of the syrup from sweet sorghum

Characteristics	Unit	Value
Dry matter (DM)	%	73.30 ± 0.03
Total sugars (TS)	% to product weight	65.54 ± 0.15
Reducing sugars (RS)	% to product weight	23.72 ± 0.05
Sucrose	% to product weight	41.82 ±. 0.04
Purity	% TS to DM	89.41 ± 0.11
H+ activity		5.12 ± 0.02

Carbon sorbents

The novel carbon sorbent (CS), and commercial available sorbents NORIT GAC 1240 and C607 18x40, supplied by Chemviron Carbon (USA), were used in the study.

The carbon sorbent (CS) was derived from pine pellets with the following specifications: diameter – 6 mm, length – 8–25 mm, moisture content – 7%, ash content – 0.7%, mechanical strength – 98%, bulk density – 0.6 g/cm³. The sorbent production technology involved carbonization in a quartz reactor followed by steam activation, as described in (Brey et al., 2024). This sorbent, further referred to as carbon sorbent (CS), was used in the purification (decolourisation) process of sugar syrup to compare its efficiency with known sorbents NORIT GAC 1240 and C607 18x40.

NORIT GAC 1240 is a coal-based granular activated carbon, while C607 18×40 is a coconut shell-based granular activated carbon. Both are suitable and commonly used for the decolorization of sugar syrups and other liquid sugar products.

Evaluation of the efficiency of colorant removal by carbon sorbents

To evaluate the potential of CS for the decolorization of syrup from sweet sorghum, a series of adsorption experiments were conducted. Each experiment was performed using an individual conical flask containing 100 NORIT of dye solution, to which CS was added. Adsorption of coloring compounds from the solutions was carried out under standard conditions. Fixed volumes, 100 ml, of the sugar-containing product were treated with carbon sorbent samples at dosages of 0.5%, 1.0%, and 1.5% by product mass. The test solutions were kept in a thermostat at 80 °C under constant stirring for 30 minutes. Product samples were taken at 10, 20, and 30 minutes to determine optical density and dry matter content.

Based on the experimental data, the following parameters were calculated: solution color intensity (ICUMSA units), decolorization efficiency (%), and adsorption capacity (A, mg/g). During the study, the following parameters were varied: type and dosage of the sorbent, and duration of the adsorption process. The study was conducted using traditional control and analysis methods commonly used in the sugar industry and accepted in international practice.

The content of total sugars (TS), reducing substances, and sucrose was determined using the Luff-Schoorl method (Asquieri et al., 2007; Dekker, 1950). The purity index was calculated as the ratio of the total sugars content in the product to the dry matter content multiplied by 100% (Hryhorenko et al., 2021).

Dry matter content (DM, % to the weight of the product) was determined at a temperature of 20 °C by the refractometric method (Asquieri et al., 2007) using refractometer RPL-3.

The activity of H^+ ions was determined by the potentiometric method (Hryhorenko et al., 2021) using universal ionometer EV-74. The quantitative assessment of sugar solution decolorization was performed in accordance with the methodology of the International Commission for Uniform Methods of Sugar Analysis (ICUMSA) (Chai et al., 2016). The ICUMSA color value was then calculated using following formula (1):

$$\mathrm{C\ (ICUMSA)} = \frac{100 \times 1000 \times D}{l \times DM \times \rho} \tag{1}$$

where: C is color intensity, opt. densityICUMSA units; D is optical density of the solution at 560 nm; l is cuvette length; DM is total solids content,%; p is true density of the solution g/ml.

Decolorization effect (%) was calculated using the following formula (2) (Wang et al., 2023):

$$\text{Decolorization effect} = \frac{(C1-C2) \times 100}{C1} \tag{2}$$

where: C1 and C2 are color intensity of the solutions before and after treatment with the carbon sorbent.

Sorption capacity (A, mg/g) was determined by the formula (Sych et al., 2017):

$$A = \frac{k \cdot (C_0 - C_t) \cdot V}{m} \tag{3}$$

where: k is coefficient that accounts for the ratio between the concentration of coloring substances in the solution and its optical density; V is volume of the solution, ml; m is mass of the carbon sorbent, g; C_0 and C_t are concentrations of coloring substances (g/ml) before and after treatment with the carbon sorbent, respectively.

Taking into account the ratio that:

$$k = 1/Kl \text{ and } C = k \cdot D,$$

where K is the conversion coefficient, which equals 1150 for sugar and refined sugar products; l is cuvette length; D is optical density of the solutions; C is concentration of coloring substances, g/ml.

Accordingly, the Sorption capacity was calculated using the following formula:

$$A = \frac{1}{1150} \cdot \frac{l \cdot (D_0 - D_t) \cdot V}{m} \tag{4}$$

where D_0 and D_t are optical density of the solutions before and after treatment with the carbon sorbent, respectively.

Characteristics of the structural-adsorptive and physicochemical properties of the studied carbon sorbents

The **textural characteristics** of the studied carbon sorbents were calculated from nitrogen adsorption/desorption isotherm curves using an automated Quantachrome analyzer (Nova 2200e, Quantachrome, USA) with Nova Win 2.0 software.

The **total surface area of the materials** (S total, m²/g) was calculated using the Brunauer–Emmett–Teller (BET) method (Kotynska et al., 2025).

The **total pore volume** (V total) was calculated from the volume of adsorbed nitrogen converted to liquid at a relative pressure close to $P/P_0 = 1$ (Gregg et al., 1982; Kotynska et al., 2025).

To determine the sorption properties of the adsorbents, an aqueous solution of the dye (methylene blue) was used. The methods involved measuring the change in dye concentration after interaction with the adsorbent. The sorption activity of the studied samples was determined by the methylene blue sorption method following standard procedures using a KFK-3 photoelectric colorimeter (Tarnovsky et al., 2021).

Mathematical and statistical processing of experimental data

To obtain regression equations that adequately describe the adsorption purification process of sugar-containing solutions, methods of mathematical and statistical processing of experimental results were used (Castillo, 2007), along with the Mathcad Professional application software.

The main factors selected were: duration of the purification process (minutes), decolorization of the solutions (%), and carbon sorbents dosage (%). The variation of these factors was conducted within the ranges typical for the purification and decolorization technology of solutions of the sugar-containing product from sweet sorghum.

The research program was incorporated into the experimental design matrix as follows: base level — time: 20 min; sorbent dosage: 1%; variation interval — time: ±10 min; sorbent dosage: ±0.5%; upper level — time: 30 min; sorbent dosage: 1.5%; lower level — time: 10 min; sorbent dosage: 0.5%.

Results and discussion

Adsorption removal of colored compounds from sugar solutions using carbon sorbents

The key parameters investigated during the adsorption process included the type of adsorbent material, adsorbent dosage, contact time, and temperature. In addition, a comparative analysis was conducted to evaluate the relative performance of CS in comparison with other industrially manufactured adsorbents that are commercially available on the market, including NORIT GAC 1240 (further NORIT) and coconut-based charcoal, grade C607 18x40 (further C607), supplied by Chemviron Carbon (USA).

Characteristics of CS were compared with those of industrially manufactured adsorbents that are commercially available on the market, including NORIT and coconut-based charcoal, grade C607 (Table 2).

Table2

Characteristics of the carbon sorbents

Characteristics	**C607**	**NORIT**	**CS**
Bulk density, g/cm³	0.53	0.45	0.34
Adsorption capacity for methylene blue, mg/g	80	155	300
Total specific sorption pore volume, ml/g	0.48	0.55	1.28
Specific surface area, m²/g	1077	1100	1240

It is known (Hryhorenko et al., 2021; Husiatynska et al., 2023), that the purity and chemical composition of sugar-containing syrups depend on the quality of the raw materials, the efficiency of the purification processes, and are determined by the content of non-sugar substances, including colored compounds. Among the factors influencing the adsorption purification process, the following should be highlighted (Dubinin et al., 1987; Qureshi et al., 2008): contact time, sorbent dosage, temperature, concentration of sugar-containing solutions, and the physicochemical properties of the sorbent (such as surface area, and the presence of a significant number of meso- and micropores in the sorbent structure).

According to the characteristics of the carbon sorbent (CS), it can be assumed that the obtained material, due to its sufficiently high surface area and developed micro- and meso-porosity, may serve as an effective sorbent for medium- and large-sized molecules, particularly for high-molecular-weight compounds and colored substances in liquid-phase purification processes of sugar-containing products.

To confirm the effectiveness of using the carbon sorbent (CS), a series of studies was conducted to investigate the removal of coloring compounds from liquid sugar-containing products in comparison with industrial carbon sorbents. The results of the color intensity changes in sugar-containing solutions during a treatment period of 10–30 minutes and with sorbent dosages of 0.5–1.5% are presented in Table 3.

Table 3

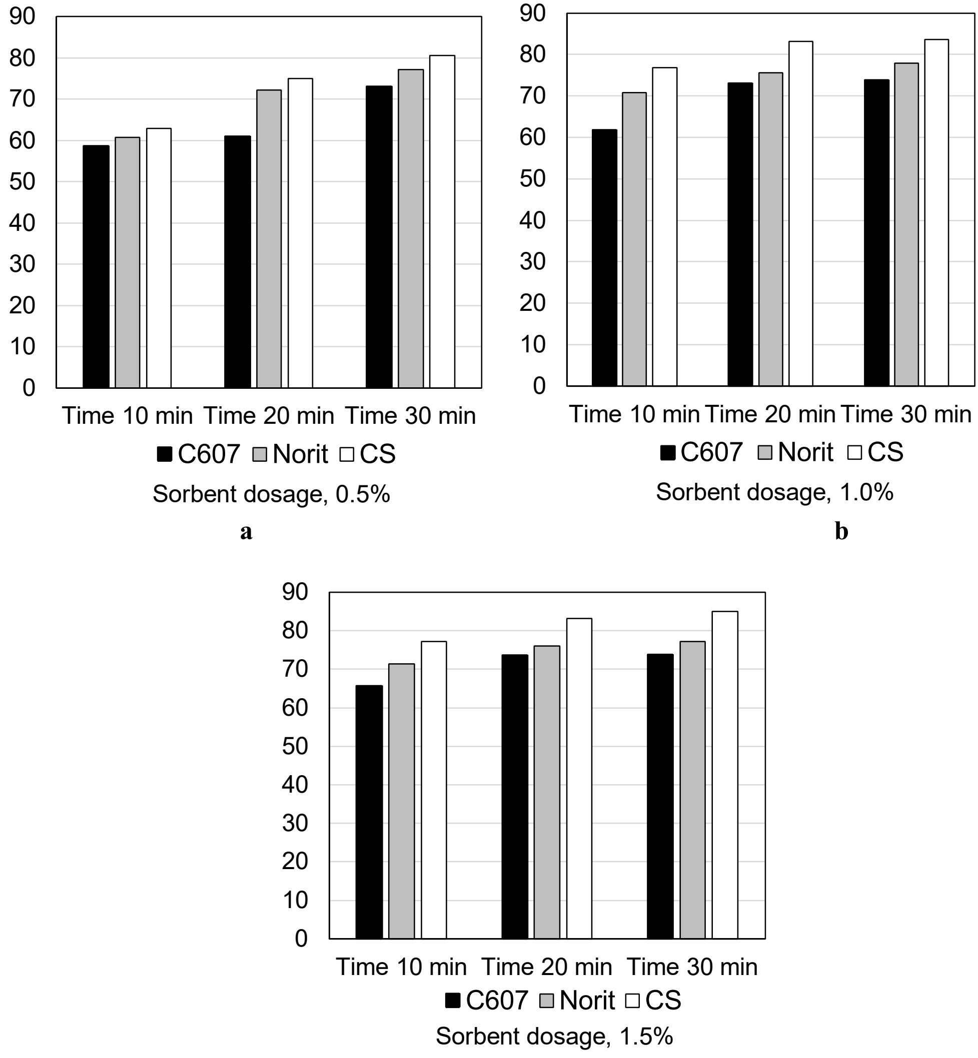
**Change in the color intensity
of sugar solutions treated with carbon sorbents**

Time, min	**C607**	**NORIT**	**CS**
	Color, ICUMA units		
Sorbent dosage, 0.5%			
10	615.67	585.23	552.60
20	580.35	562.43	323.89
30	402.66	341.89	292.27
Sorbent dosage, 1.0%			
10	569.99	433.60	347.99
20	402. 12	366.36	251.72
30	392.15	338.70	228.83
Sorbent dosage, 1.5%			
10	509.91	426.70	340.18
20	394.16	358.67	252.19
30	390.65	329.69	222.81

An increase in the process duration up to 30 minutes and sorbent dosage in the range of 0.5–1.5% (relative to product mass) led to a gradual decrease in colour intensity of the treated sugar-containing liquids for all tested sorbents.

For example, when purifying the initial solution with a color intensity of 1489.06 ICUMSA units using activated carbon, a 30-minute contact time and a sorbent dosage of 1.5% resulted in the following reductions in color intensity: 390.65 ICUMSA units for C607; 329.69 ICUMSA units for NORIT; and 222.81 ICUMSA units for CS.

At the same time, a favourable decolourisation dynamic was also observed at lower treatment durations (20 minutes) and carbon dosages of 1.0%, for all sorbents C607, NORIT, and CS.



Sorbent dosage, 0.5%

a

Sorbent dosage, 1.0%

b

Sorbent dosage, 1.5%

Figure 1. Decolorization effect of syrup at different process durations. Dosage of sorbent, %: a – 0.5; b – 1.0; c – 1.5.

To determine the optimal parameters of adsorption purification, it is advisable to assess the process based on the decolorization effect achieved under varying process parameters. This was done by calculating the percentage reduction in solution color intensity before and after treatment with the tested carbon sorbents using formula (2). The diagrams showing the changes in the decolorization effect of sugar-containing solutions over a process duration of 10–30 minutes and sorbent dosages of 0.5–1.5% are presented in Figures 1 (a, b, c).

Based on the comparison of the adsorption characteristics of the carbon sorbent CS with commercial activated carbon samples (Fig. 1a, b, c), it was established that the decolorization effect achieved using sorbent CS was 80–85% at a dosage of 0.5–1.5%, which exceeds the corresponding performance of sorbents C607 and NORIT.

The data on the decolorization effect of the solutions (Fig. 1 a, b, c) correlate with the previously presented results on the change in color intensity of the solutions during purification with the studied carbon sorbents (Table 3), and are also consistent with findings reported by other researchers (Wang et al., 2023; Zolfaghari et al., 2020).

In particular, the use of an adsorbent synthesized from bagasse with the addition of hydroxyapatite enabled the decolorization of juice and syrup from sugarcane by 92.41% and 93.19%, respectively (Wang et al., 2023). Also, a combined method for the decolorization of the beet sugar syrup using activated carbon granules was proposed, which gave a decolorization performance at the level of 83.68% (Zolfaghari et al., 2020).

Mathematical and statistical analysis of experimental results

An important step in determining the optimal parameters for adsorption purification is obtaining mathematical relationships that allow for the prediction of decolorization efficiency of sugar solutions within the selected range of key process variables. As a result of the mathematical and statistical analysis of the experimental data, regression equations were obtained that adequately describe the influence of the studied factors on the purification and decolorization of solutions of the sugar-containing product from sweet sorghum when using the following substances as adsorbents:

NORIT:

$$\text{Effect decolor} = 37.046 + 1.651 \cdot t + 27.879 \cdot c - 0.534 \cdot t \cdot c - 0.016 \cdot t^2 - 5.608 \cdot c^2$$

C 607:

$$\text{Effect decolor} = 30.734 + 1.718 \cdot t + 26.571 \cdot c - 0.305 \cdot t \cdot c - 0.020 \cdot t^2 - 6.886 \cdot c^2$$

CS:

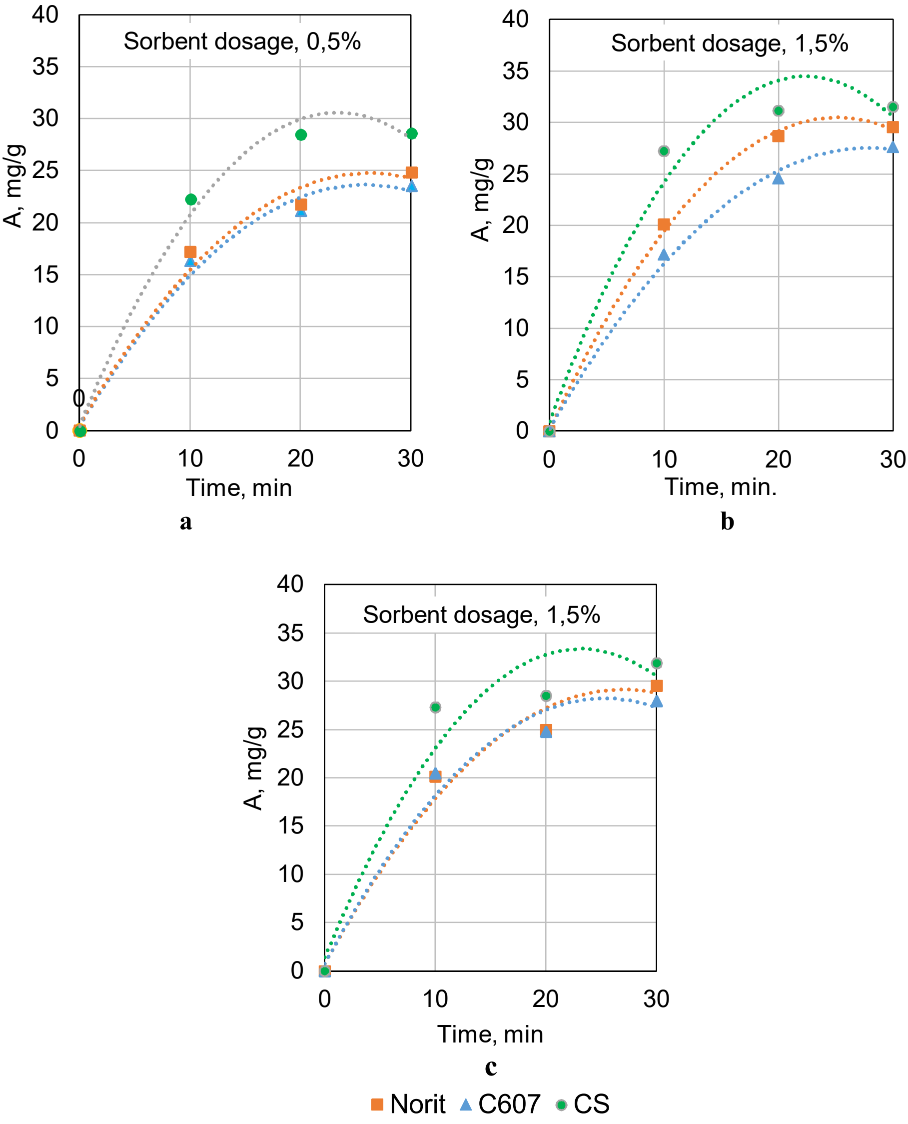
$$\text{Effect decolor} = 26.588 + 2.227 \cdot t + 46.291 \cdot c - 0.540 \cdot t \cdot c - 0.029 \cdot t^2 - 12.983 \cdot c^2$$

t is process duration, minutes;
c is carbon sorbent dosage, %.

The obtained equations adequately describe the dependence of the decolorization effect of sugar-containing solutions at a temperature of 80 °C, within a process duration of 5–35 minutes, and a carbon sorbent dosage range of 0.3–1.7%.

He obtained regression equations can be used to analyze the decolorization process of sugar-containing solutions and to determine the optimal parameters for process duration and sorbent dosage when using the specified carbon sorbents.

The next stage of the mathematical and statistical analysis of the experimental results involved calculating the sorption capacity (A, mg/g) and conducting a comparative analysis of the decolorization kinetics solutions using the studied carbon sorbents. The calculated adsorption values under the investigated process durations, presented in Figures 2 (a, b, c), show that the sorption-kinetic decolorization properties of the studied carbon sorbents gradually increase with higher sorbent dosages and longer sorption times. For instance, at a dosage of 1.5% (relative to the product mass) and a process duration of 30 minutes, the sorption capacity (A, mg/g) was as follows: 28.02 mg/g for C607; 29.55 mg/g for NORIT; and 31.91 mg/g for CS.



a

b

c

Figure 2. Change in colorant absorption (A) from sugar syrup at sorbents dosage of: 0.5% (a), 1.0% (b), and 1.5% (c).

Moreover, the data presented in Figures 2 (a, b, c) clearly illustrate the sorption-kinetic performance of the studied carbon sorbents in removing colorants from sugar-containing solutions. Accordingly, their performance may be ranked in the following order:

CS > NORIT > C607.

Conclusions

The carbon sorbent CS, obtained through the modification of renewable woody raw materials, demonstrated effective performance in purifying sugar-containing solutions from coloring compounds and, in terms of its properties, showed superior results compared to the commercial available C607 and NORIT sorbents.

The CS carbon sorbent possesses sufficient mechanical strength of the granules, indicating its practicality for long-term use and the possibility of reuse after regeneration.

The obtained results indicate the potential for industrial implementation of the CS carbon sorbent and its application in the purification of liquid sugar-containing products. Furthermore, based on its physicochemical characteristics, the sorbent shows promising potential for use in the purification of aqueous solutions in the chemical, food, and pharmaceutical industries, as well as for water treatment and wastewater purification from organic pollutants.

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Cite:

UFJ Style

Husiatynska N., Hryhorenko N. (2025), Decolorization of liquid sugar products by carbon sorbents, *Ukrainian Food Journal*, 14(2), pp. 290–303, https://doi.org/10.24263/2304-974X-2025-14-2-8

APA Style

Husiatynska, N., & Hryhorenko, N. (2025). Decolorization of liquid sugar products by carbon sorbents. *Ukrainian Food Journal, 14*(2), 290–303. https://doi.org/10.24263/2304-974X-2025-14-2-8

Sorbent selection for solid-phase clean-up of nitrofuran metabolites from meat

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Keywords:

Nitrofuran
Metabolite
Meat
Extraction
Sorbents
Chromatography

Article history:

Received 20.10.2024
Received in revised form 25.02.2025
Accepted 30.06.2025

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DOI: 10.24263/2304-974X-2025-14-2-9

Abstract

Introduction. In this study there are presented the results of selecting a sorbent for clean-up of an extract of 4 nitrofuran metabolites extracted from the meat, preparing a solid-phase column with this sorbent, and confirming the advantages of this column using validation of method.

Materials and methods. The extract was clean-up using a solid-phase column, prepared by us with the sorbent Silica gel 60; identification and quantitative determination of nitrofuran metabolites: 3-amino-2-oxazolidinone (AOZ), 3-amino 5-methylmorpholino-2-oxazolidinone (AMOZ), 1-aminohydantoin (AHD), semicarbazide (SEM), and their nitrophenyl derivatives was carried out using a high-performance liquid chromatography with diode array detector.

Results and discussion. Based on the physicochemical properties of the analyzed substances, sorbents and literature data, two sorbents were selected – silica gel 60 and a polymer sorbent – polypropylene, which is most often used in ready-made columns for nitrofuran purification. Columns were made from the selected sorbents and their suitability was determined. The advantage of the manufactured columns using silica gel was experimentally proven. 96-98% extraction of nitrofuran metabolites was achieved with good reproducibility (RSD ≤ 2.0%), since when using a polymer sorbent, the extraction does not exceed 94%.

When using silica gel 60, the limit of quantification for metabolites – AOZ, AMOZ, AHD and SEM is within 0.1–0.12 µg/kg, for their nitrophenyl derivatives (NP) within – 0.11–0.14 µg/kg. The decision limit is within 0.01–0.07 µg/kg. When using a polymer sorbent: the limit of quantification for metabolites – AOZ, AMOZ, AHD and SEM is within 0.21–0.32 µg/kg; for their NPs within 0.25–0.43 µg /kg. The decision limit is within 0.13–0.28 µg/kg.

Conclusions. Silica gel 60 and columns prepared with this sorbent were identified as preferable for the purification and concentration of nitrofuran metabolites isolated from meat. The advantages of the proposed column over disposable, expensive, commercially available columns were demonstrated through method validation.

Abbreviations:

SPE – solid-phase extraction;
HPLC – high-performance liquid chromatography;
DAD – diode array detector;
HPLC/DAD – high-performance liquid chromatography with diode array detector;
HPLC-MS/MS – liquid chromatography-tandem mass spectrometry;
UPLC-MS/MS – ultra-high-performance liquid chromatography-tandem mass spectrometry;
AOZ – 3-amino-2-oxazolidinone;
AMOZ – 3-amino 5-methylmorpholino-2-oxazolidinone;
AHD – 1-aminohydantoin;
SEM – semicarbazide;
NP-derivatives – nitrophenyl derivatives;
NPAMOZ – 5- (morpholinomethyl) – 3 – (2 – nitrobenzylidenamino)-2-oxazolidinone;
NPAOZ – 3-(2-nitrobenzylidenamino)-2-oxazolidinone, NPSEM – 2-nitro-benzaldehyde-Semicarbazone;
NPAHD – 1-(2-nitrobenzylidenamino)-2,4-imidazolidinedione;
MRL – Maximum Residual Limit;
MRPL – Minimum Required Performance Limit;
LC-MS/MS – Liquid chromatography- tandem mass spectrometry;
2-NBA – 2-nitrobenzaldehyde;
RSD – Relative standard deviation;
LOQ – Limit of quantification;
LOD – Limit of detection.

Introduction

Nitrofurans are antibacterial drugs, which can be considered one of the most used drugs for the treatment of microbial infections in farm animals (Torre et al., 2015; Wang et al., 2017). However, the most commonly used nitrofurans, such as furazolidone, furaltadone, nitrofurantoin, and nitrofurazone, have been banned worldwide for farmed animals due to their carcinogenic and teratogenic risks (Commission implementing regulation (EU) 2021/808; García et al., 2017; Yuan et al., 2020; Zuma et al., 2019). However, these drugs continue to be used in some countries because of their low cost, effectiveness against infections, and availability (Antunes et al., 2006; Gotsiridze et al., 2022, 2023; Yu et al., 2019; Zhang et al., 2016; Zhao et al., 2016). Therefore, it is necessary to control their illegal use in food products by establishing effective and reliable methods. As of today, the analytical strategy for the quantification of nitrofurans is based on the determination of four metabolites: AOZ, AMOZ, AHD, and SEM (Alkan et al., 2016; Craken et al., 2001; Tsai al., 2010).

Many analytical methods have been developed to determine nitrofuran metabolites, such as LC-MS (Gong X. et al., 2022), HPLC-MS/MS (Park et al., 2017), UPLC-MS/MS (Gong J. et al., 2020; Noelia et al., 2013), enzyme-linked immunosorbent assay (Cháfer et al., 2010), HPLC/DAD (Kangkang et al., 2020). However, not all of these methods are suitable due to the very low detection limit required for the analyte, with an MRPL of 0.5 µg/kg (Luo et al., 2019); therefore, the HPLC-MS/MS method is mainly used due to its high sensitivity and accuracy (Shi et al., 2016). The use of this analytical method is limited in economically developing countries due to the expensive equipment and high maintenance costs of this equipment (Ong et al., 2013).

The HPLC/DAD method is convenient and relatively less expensive compared to other methods, but it is difficult to achieve the required detection limit using a diode detector (Luo et al., 2019). For the determination of nitrofuran metabolites in food, sample preparation is of particular importance. Most often, for clean-up of samples solid-phase purification is used through various expensive cartridges (Barbosa et al., 2007; Conneely et al., 2002; Kaufman et al., 2015; Tripathi et al., 2023), where polymers are used as sorbents, followed by determination by HPLC-MS/MS. Currently, commercially available ready-made cartridges used for sample cleaning are disposable and expensive. Moreover, for commercial purposes, sorbents are often designed for a wide range of substance groups and are primarily polymer-based, which limits the ability to account for the specific chemical nature of individual target compounds.

Based on the above, the aim of this study was to select an appropriate sorbent and prepare a column for solid-phase sample purification in accordance with MRPL (Minimum Required Performance Limits) requirements. The goal was to achieve specificity and selectivity toward nitrofuran metabolites, thereby enhancing the sensitivity of the HPLC method and enabling the use of a diode array detector instead of a more costly mass spectrometric detector.

Materials and methods

Materials

Reagents and chemicals

Acetonitrile (HPLC grade), potassium dihydrogen orthophosphate, 35% hydrochloric acid, sodium hydroxide, 2-nitrobenzaldehyde (2-NBA), dimethyl sulfoxide anhydrous, ≥99.9%, ethyl acetate, methanol (HPLC grade), were from Sigma Aldrich Chemical Company (Germany) and Silica gel 60 from Roth (Germany). Ultrapure water was filtered through a Milli-Q system Millipore (USA). The metabolites AOZ, AMOZ, AHD, SEM, NP-AOZ, NP-AMOZ, NP- AHD and NP- SEM were supplied by Sigma (Aldrich Chemical Company, Germany). Stock solutions may be stored, refrigerated below -14°C for at least 12 months, and standard solutions should be stored refrigerated from 2°C to 8°C. Intermediate standards should be replaced at least monthly and working solutions at least weekly.

Standard solutions

Individual standard stock solutions of 1 mg/mL were prepared in acetonitrile. Working solutions of 10 ng/mL were diluted by water. All standard stock solutions were stored -20 ºC, and the working solutions were stored in refrigerator.

The concentration and content of mix standard solution were used to spiked samples with AMOZ, AOZ, AHD, NP-AOZ, NP-AMOZ, NP- AHD, NP- SEM and SEM at an 8.0, 9.0, 10.0, 11.0 and 12.0 ng/mL respectively.

Sample preparation end extraction

2.0±0.05 g of homogenized meat was weighed into a 50 mL polypropylene centrifuge tube. A mix of standard spiking solutions 50.0 µL, 5 mL of 0.1 M hydrochloric acid solution and 50 µl of a solution of 2-nitrobenzaldehyde in dimethyl sulfoxide (DMSO) (8 mg±0.6 mg in 5 mL of DMSO) were added. Thoroughly mixed for 1 minute and incubated for 12 hours at 37° C to hydrolyze the protein-bound metabolites and convert the metabolites to their nitrophenyl derivatives. After the sample solution was cooled to room temperature, 500 µL of potassium dihydrogen orthophosphate solution, 300 µL of sodium hydroxide solution were added to adjust the pH to 7.0±0.5, and 5 mL of acetonitrile were added. Thoroughly stirred for 1 minute, centrifuged at 4000 rpm for 10 minutes (Bongers et al., 2021; Śniegocki et al., 2018; Zuma al., 2019) and the acetonitrile layer was transferred to a pre-activated silica gel column, prepared by us, at a flow rate of 5 ml/min. After sample loading, the column was washed with 10 mL of ethyl acetate and eluted with 10 mL of acetonitrile. The column was then evacuated, the eluates were combined and evaporated under a moderate nitrogen flow.The residue was dissolved in 1 ml acetonitrile, 1 ml of the mobile phase was added, mixed for 1 minute, filtered through a Syringe Filter PTFE for HPLC 0.22um and chromatographed.

Selection of sorbent and column preparation for solid phase purification

The choice of sorbent was made based on the physicochemical properties of the analyzed substances, sorbents, literary and experimental data. The columns with the selected sorbent silica gel 60 were prepared by the dry method. For activation, the prepared column was washed with 5 mL of methanol, dried under vacuum, then washed with 10 mL of acetonitrile, and again dried under vacuum.

Quantitative determination of metabolites

Quantitative of AOZ, AMOZ, AHD, SEM, NP-AOZ, NP-AMOZ, NP-AHD and NP-SEM were determined by high-performance liquid chromatography with diode array detector. The LC/DAD system consisted of an Agilent Series 1260 HPLC system (Agilent Technologies, Germany) with DAD detector.

The chromatography was performed on a C18 column 3 µm x 2 mm 150 mm (Phenomenex, Torrance, CA, USA), connected to a C18 precolumn 3 µm x 2 mm x 4 mm (Phenomenex, USA). The mobile phase was Acetonitrile: 0.01 M sodium acetate buffer, pH 6. 0 – 250:750, λ 376 nm, flow rate of 1 mL/min, Injection volume was 50 µL. The column was thermostated at 30^0C. All determinations were carried out under standard conditions: Air temperature – (20±5) ^{0}C, atmospheric pressure – 84.0 – 106.7 kPa (630 – 800 mm Hg), air humidity no more than – 80%, mains voltage 198 – 242 V, frequency AC – 50±1 Hz. Acceptance criteria: Reference points for action (RPA) 0.5 µg/kg for each of the metabolites (Commission implementing regulation (EU) 2023/411).

Validation

The HPLC/DMD method for the determination of nitrofuran metabolites in meat extract purified using our column was validated in accordance with the European Union criteria for the analysis of veterinary drug residues in food (Commission implementing regulation (EU) 2021/808; Commission implementing regulation (EU) 2024/2052).

The validation was carried out according to the following parameters: Specificity/selectivity. The limit of detection (LOD) and the limit of quantification (LOQ), Recovery, Linearity, Trueness and Accuracy, Repeatability and Reproducibility also determined Decision limit (CC_α) and detection capability (CC_β), Stability and Matrix effects,%.

Specificity/selectivity

The specificity of the method was tested by analyzing 20 different blank meat samples. These samples were randomly selected from previously analyzed nitrofuran-free samples (eight pork, seven beef, and five poultry) and 20 matrix samples spiked with nitrofuran metabolite standards (concentration 0.5 µg/kg each). Acceptance criteria: No interference.

Limit of detection (LOD) and limit of quantification (LOQ)

LOD: smallest measured content, from which it is possible to deduce the presence of the analyte with reasonable statistical certainty.

LOQ: Analyte content that can be determined with a certain level of precision.

The calculation of LOD was performed according to equation (1) under the condition of a pseudo-blank sample and equal probabilities (α=ß=0.05) for false positive and false negative decisions.

$$x_{LOD} = 3.3 \times \frac{S_{y,b}}{b},$$

where: X_{LOD} is limit of detection;

$S_{y,b}$ is standard deviation of the blank (pseudo-blank) signals;

b is a slope of the calibration curve;

3.3 is the multiplication factor.

The calculation of LOQ was performed according to equation:

$$X_{LOQ} = 10 \text{ x } \sigma / S,$$

where: σ is SD of the obtained results;

S is a slope of the calibration curve.

Acceptance criteria: LOD is three times the standard deviation of the mean of blank determinations ($n > 20$) and S/N (Signal/Noice) > 2 or 3; LOQ is numerically equal to 10 times standard deviation of the mean of blank determinations ($n > 20$) and S/N > 10 (ISO 11843-2).

Recovery

The recoveries and RSDs were determined from 6 replicates at four concentration levels spiking blank samples over three days.

The absolute recovery was calculated as:

Rec (analyte) = (area matrix-fortified standard) / (area matrix-matched standard)×100.

Acceptance criteria: 70-120%, % RSD of recovery concentration must be < 2;

Decision limit (CC_α) and detection capability (CC_β)

The CC_α and CC_β were calculated with the application of the following formulas:

$$\mathrm{CC}\alpha = t_{(\alpha,IJ-2)}\frac{\sigma}{b}\sqrt{\frac{1}{K}+\frac{1}{IJ}+\frac{\bar{x}^2}{\sum_{i=1}^{I}(x_i-\bar{x})^2}}$$

$$\mathrm{CC}\beta = \delta\frac{\sigma}{b}\sqrt{\frac{1}{K}+\frac{1}{IJ}+\frac{\bar{x}^2}{\sum_{i=1}^{I}(x_i-\bar{x})^2}}$$

where: σ is an estimation of the residual standard deviation of the regression function;

b is an estimation of the slope of the calibration curve obtained through regression calculation;

$\overline{X}$ is the mean of the x_{ij} values;

$t_{(\alpha,I\,J-2)}$ – is the Student's t at risk of α and for degrees of freedom (dof) equal to IJ-2 with I the number of calibrating levels of concentration and J the number of replicates per level of concentration;

K is the number of replicates for the real state;

$\delta_{(I\,J-2;\alpha;\beta)}$ is a statistical function that can be fairly approximated by using $2t_{(\alpha,I\,J-2)}$ (ISO 11843-2; Verdon et al., 2007).

Acceptance criteria: For prohibited substances (Nitrofuran metabolites), for which an Reference points for action is established under Regulation (Commission regulation (EU) 2023/411) CCα shall be lower than or equal to the reference point for action (MRL of nitrofurans -0.5 µg/kg).

Linearity

Calibration curves were constructed on extracts from AOZ, AMOZ, AHD, SEM, NP-AOZ, NP-AMOZ, NP-AHD and NP-SEM spiked matrices with the following analyte concentrations: 0.4, 0.45, 0.5, 0.55 and 0.6 µg/kg for each metabolites.

Acceptance criteria: Range – concentration where data can be reliable detected (MRL – 0.5 µg/kg) 80 – 120%; Linearity -Correlation coefficient – NLT 0.999.

Trueness and accuracy

Trueness and accuracy were validated in the studied matrices using blank samples fortified at three concentration levels: 0.25, 0.5, and 0.75 µg/kg; which correspond to 0.5, 1, and 1.5 times the RPA (0.5 µg/kg) value, respectively. At each concentration level 20 replicate analyses were performed.

Accuracy for each target analyte was determined using the following equation:

$$\text{Accuracy (\%)} = C_{\text{analyte}}/C_{\text{spiked}}\times 100,$$

where C_{analyte} is the found analyte concentration using standard calibration with matrix-fortified standards;

C_{spiked} is the known spiked concentration.

The obtained accuracy values (%) were expressed as the average value±standard deviation ($n \geq 20$).

From these values, trueness (%) was calculated as bias according to the following equation:

$$\text{Trueness (bias,\%)}=100-\text{Accuracy (\%)}.$$

Acceptance criteria: −50% to +20% for levels ≤1 µg/kg, and -30% to +20% for levels >1 µg/kg to 10 µg/kg.

Repeatability

A set of identical blank matrix samples were spiked with the analyte to obtain concentrations equivalent to 0.5, 1.0 and 1.5 times the RPA i.e. for furan metabolites (RPA is 0.5 µg/kg) 0.25, 0.5, and 0.75 µg/kg. At each level, the analysis was performed at least six times. The concentration found in each sample, the mean concentration, the standard deviation and the coefficient of variation (%) of the spiked samples were calculated. These steps were repeated twice. Overall, mean concentrations, standard deviations (by averaging the squares of the standard deviation of individual cases and taking the square root of it) and the coefficients of variation for the spiked samples were calculated. Acceptance criteria: $CV_R \leq 30\%$ for concentration levels <10 µg/kg

Reproducibility

Reproducibility values, expressed as CV (CV_R, %), were obtained in repeated series of analyses (4 series of 5 replicates) of blank samples fortified at the studied concentration levels conducted by different operator applying the following equations:

$$S_R^2 = S_L^2 + S_r^2,$$

where S_R^2 is the variance of reproducibility;

S_L^2 is the variance of the different analytical series;

S_r^2 is the variance of repeatability.

From these values, reproducibility (CV_R) was calculated:

$$CV_R\,(\%) = S_R/C_M \times 100,$$

where S_R is the standard deviation for reproducibility;

C_M is the average found concentration considering all analytical series.

Acceptans critetia: CVr values should be ≤ 2/3 CV_R (i.e., ≤ 20%).

Stability

The stability of the stored analyte in solution and analyte(s) in matrix was tested under four different conditions: storage in light for 8 hours per day at 25°C; in the dark at 25°C; in the dark at 4°C; and in the dark at -20°C (Tsai C, 2010). The storage time was extended to 30 days until degradation phenomena became visible during quantification. The maximum storage time and optimal storage conditions were recorded.

The calculation of the concentration of the analyte(s) in each aliquot was carried out by using the solution of the analyte freshly prepared at the time of analysis as 100%.

$$\text{Analyte Remaining (\%)} = Ci \times 100/C_{fresh},$$

where Ci is a concentration at time point;

C_{fresh} is a concentration of fresh solution.

Acceptance criteria: The mean value of five replicate solutions, which were stored, shall not differ by more than 15% from the mean value of five freshly prepared replicate solutions. The mean value of the five freshly prepared solutions shall be used as the basis for calculating the percentage difference.

Matrix effects, %

The calculation of the matrix effect, %, was carried out at 20 different blanks lots (matrix/species).

The blank matrix was fortified after extraction with the analyte and was analysed together with a pure solution of the analyte.

The matrix effect, % was calculated as:

Matrix effect, % = (peak area of solution standard – peak area of extract of fortified matrix) / peak area of solution standard x 100.

Acceptance criteria: RSD < 20%.

Statistical analysis

The software package SPSS Statistics 2015 was used for the statistical analysis.

Results and discussion

Selection of sorbent and conditions of solid-phase purification of nitrofuran metabolite extract from meat

Based on the physicochemical properties of the analyzed substances, sorbents, literature, and experimental data, silica gel 60 with a particle size of 0.2–0.5 mm was selected for purifying nitrofuran metabolite extracts. Solid-phase extraction (SPE) columns for nitrofuran metabolite purification were prepared using this sorbent, with cartridge dimensions of 20 × 400 mm. Ethyl acetate was chosen as the washing solvent, and acetonitrile as the elution solvent.

Validation

Specificity/Selectivity. According to the results of the analysis for the investigated substances, the specific wavelength is 376, there were no significant peaks with an S/N ratio (signal/noise) of 3 and chromatographic interference during the retention times of the target metabolites of nitrofurans;

The coefficient of variation of the specificity of the obtained results during the working day is within 0.02–0.18, during the working week – within 0.02–0.29. That is satisfactory as required by Commission implementing regulation (EU) 2021/808/EC.

Limit of Detection (LOD) and (Limit of Quantitation). When using silica gel 60, the limit of detection for metabolites is – 0.08, 0.05, 0.01 and 0.03 μg/kg, for their NP- derivatives 0.10, 0.09, 0.11 and 0.10 μg /kg respectively; the limit of quantification for metabolites – is 0.10, 0.09, 0.11 and 0.10 μg /kg, for their NP- derivatives is – 0.13, 0.11, 0.14 and 0.12 μg /kg respectively.

When using a polymer sorbent: the limit of detection for metabolites is – 0.20, 0.17, 0.21 and 0.14 μg/kg, for their NP- derivatives 0.34, 0.18, 0.13 and 0.15 μg/kg respectively; the limit of quantification for metabolites – is 0.32, 0.23, 0.29 and 0.21 μg/kg, for their NP- derivatives is – 0.43, 0.29, 0.25 and 0.36 μg /kg respectively. The mean value of LOD and LOQ (Table 1 and 2).

Recovery. The recovery was observed within 96.27 – 98.79% and%RSD < 2 when used sorbent silica gel, 89.56 – 91.24% and%RSD < 2 when used polymer sorbent.

Despite the fact that the data both when using silica gel 60 and when using a polymer sorbent meet the acceptability criteria of Commission implementing regulation (EU) 2021/808, the advantage of using silica gel 60 is obvious (Table 1 and 2).

Table 1

Mean value of recovery, LOD, LOQ, CCα and CC$_\beta$ of nitrofuran metabolites using silica gel 60

Analytes	Calibration range (µg/kg)	Mean value of recovery (%)*	LOD (µg/kg)	LOQ (µg/kg)	CCα (µg/kg)	CC$_\beta$ (µg/kg)
AOZ	0.1–1.0	96.55±0.97	0.08	0.12	0.04	0.10
AMOZ	0.1–1.0	97.49±1.04	0.05	0.11	0.01	0.09
AHD	0.1–1.0	97.28±1.36	0.01	0.12	0.05	0.07
SEM	0.1–1.0	98.79±1.29	0.03	0.10	0.03	0.09
NP-AOZ	0.1–1.0	96.27±0.37	0.10	0.13	0.06	0.12
NP-AOZ	0.1–1.0	98.81±0.41	0.09	0.11	0.04	0.08
NP-AHD	0.1–1.0	97.54±1.38	0.11	0.14	0.07	0.14
NP-SEM	0.1–1.0	97.20±1.23	0.10	0.12	0.05	0.08

*µg/kg

Table 2

Mean value of recovery, LOD, LOQ, CCα and CC$_\beta$ of nitrofuran metabolites using polymer sorbent

Analytes	Calibration range (µg/kg)	Mean value of recovery (%)*	LOD (µg/kg)	LOQ (µg/kg)	CCα (µg/kg)	CC$_\beta$ (µg/kg)
AOZ	0.1–1.0	90.30±0.63	0.20	0.32	0.28	0.54
AMOZ	0.1–1.0	90.61±3.65	0.17	0.23	0.17	0.36
AHD	0.1–1.0	90.63±2.05	0.21	0.29	0.23	0.43
SEM	0.1–1.0	90.36±2.65	0.14	0.21	0.19	0.27
NP-AOZ	0.1–1.0	90.96±2.17	0.34	0.43	0.26	0.39
NP-AOZ	0.1–1.0	91.24±2.24	0.18	0.29	0.16	0.30
NP-AHD	0.1–1.0	89.56±2.44	0.13	0.25	0.22	0.47
NP-SEM	0.1–1.0	90.24±2.85	0.15	0.36	0.13	0.31

*Standard 1 µg/kg

Decision limit (CC$_\alpha$) and detection capability (CC$_\beta$). In all cases, CCα is below the MRL (0.5 µg/kg). Despite the fact that the data both when using silica gel 60 and when using a polymer sorbent meet the acceptability criteria of Commission implementing regulation (EU) 2021/808, the advantage of using silica gel 60 is obvious (Table 1 and 2).

Linearity. The plots are linear over the reported range,% RSD of peak area of 6 standard samples – 0.98%,% RSD of peak area of 6 standard samples relative to bracketing peak area – 1.08% and acceptable as the correlation coefficient r^2 is above 0.999.

Trueness and Accuracy

The data for determining the Trueness values ranged and Accuracy values ranged when using silica gel 60 and polymer sorbent are presented in Table 3 and 4. As can be seen in Table 3 and 4, the obtained accuracy values: when using silica gel 60, ranged from 95.99 to 98.64% for 0.4 µg/kg; from 95.81 to 99.09% for 0.5 µg/kg and from 95.49 to 100.08% for 0.6 µg /kg. Trueness values ranged from −2 to -5% for 0.4 µg/kg; from −1 to -4 for 0.5 µg/kg and from 0 to -5 for 0.6 µg/kg, meeting in all cases the acceptability criteria of Commission implementing regulation (EU) 2021/808.

Table 3

Mean value of Accuracy and Trueness of nitrofuran metabolites using silica gel 60

Analytes	Accuracy (%)*			Trueness (bias, %)**		
	0.4 µg/kg	0.5 µg/kg	0.6 µg/kg	0.4 µg/kg	0.5 µg/kg	0.6 µg/kg
AOZ	97.24±0.35	96.92±4.44	95.49±2.18	-3	-4	-5
AMOZ	96.62±0.16	97.24±2.31	98.61±6.52	-4	-3	-2
AHD	98.37±0.49	95.81±5.56	97.66±5.14	-2	-5	-3
SEM	97.53±0.52	98.75±6.12	100.08±3.39	-3	-2	0
NP-AOZ	96.01±1.08	96.68±8.29	96.12±4.52	-4	-4	-4
NP-AOZ	98.64±0.67	98.52±1.45	99.28±7.74	-2	-2	-1
NP-AHD	96.82±1.23	99.09±3.57	96.71±9.17	-4	-1	-4
NP-SEM	95.99±0.24	97.22±2.63	98.39±8.81	-5	-3	-2

*Accuracy values (%) obtained in the analysis of fortified samples, expressed as the average value±standard deviation (n ≥ 20);

**Trueness values (%) expressed as bias (100 – Accuracy%).

Table 4

Mean value of Accuracy and Trueness of nitrofuran metabolites using polymer sorbent

Analytes	Accuracy (%)*			Trueness (bias, %)**		
	0.4 µg /kg	0.5 µg /kg	0.6 µg /kg	0.4 µg /kg	0.5 µg /kg	0.6 µg /kg
AOZ	90.62±1.28	89.65±2.41	90.64±1.90	-10	-11	-10
AMOZ	93.44±0.99	91.42±3.85	86.98±1.49	-7	-9	-14
AHD	92.37±1.39	90.86±4.16	88.67±2.72	-8	-10	-10
SEM	90.09±1.42	88.11±2.92	92.88±3.27	-10	-12	-12
NP-AOZ	89.74±0.89	93.23±4.53	89.90±4.14	-11	-7	-7
NP-AOZ	91.25±0.97	89.19±3.17	93.27±3.56	-9	-11	-11
NP-AHD	90.88±1.11	87.04±1.08	90.76±2.68	-10	-13	-13
NP-SEM	87.63±1.48	90.33±1.11	92.77±3.02	-13	-10	-10

*Accuracy values (%) obtained in the analysis of fortified samples, expressed as the average value±standard deviation (n ≥ 20);

**Trueness values (%) expressed as bias (100 – Accuracy%).

When using polymer sorbent, ranged from 87.63 to 93.44% for 0.4 µg /kg; from 87.04 to 91.42% for 0.5 µg/kg and from 86.98 to 92.88% for 0.6 µg/kg. Trueness values ranged from −7 to -13% for 0.4 µg/kg; from −7 to -11 for 0.5 µg/kg and from -7 to -14 for 0.6 µg/kg, meeting in all cases the acceptability criteria of Commission implementing regulation (EU) 2021/808.

Despite the fact that the data both when using silica gel 60 and when using a polymer sorbent meet the acceptability criteria of Commission implementing regulation (EU) 2021/808, the advantage of using silica gel 60 is obvious.

Repeatability and reproducibility

The data for determining the repeatability values ranged and reproducibility values ranged when using silica gel 60 and polymer sorbent are presented in Table 5 and 6.

As shown in the tables, the obtained accuracy values met the acceptability criteria established by Commission Implementing Regulation (EU) 2021/808 in all cases. When using silica gel 60, repeatability ranged from 1.14% to 8.57%, and reproducibility ranged from 1.98% to 8.99%. In comparison, the use of a polymer sorbent resulted in repeatability values ranging from 7.77% to 19.25%, and reproducibility values from 12.55% to 22.11%. Despite the fact that the data both when using silica gel 60 and when using a polymer sorbent meet the acceptability criteria of Commission implementing regulation (EU) 2021/808, the advantage of using silica gel 60 is obvious.

Table 5

Mean value of Accuracy and Trueness of nitrofuran metabolites using silica gel 60

Analytes	Repeatability (CV_r, %)*			Reproducibility (CV_R, %)**		
	0.4 µg/kg	0.5 µg/kg	0.6 µg/kg	0.4 µg/kg	0.5 µg/kg	0.6 µg/kg
AOZ	8.57	2.92	2.16	8.99	3.27	4.29
AMOZ	6.33	7.26	4.65	7.22	7.64	5.23
AHD	5.08	4.83	1.98	6.18	4.93	3.61
SEM	7.64	3.75	5.78	8.27	4.29	6.43
NP-AOZ	2.67	2.94	3.48	3.13	3.65	5.55
NP-AOZ	3.43	1.45	6.23	4.21	1.98	8.63
NP-AHD	1.14	5.86	5.88	2.38	6.81	7.28
NP-SEM	1.19	7.35	2.99	3.45	7.97	5.33

*Repeatability values (CV_r,%) obtained in the replicate analysis of fortified samples (n ≥ 20).
**Reproducibility values (CV_R,%) obtained in the replicate analysis of fortified samples in different analytical series (4 series, n = 5).

Table 6

Mean value of Accuracy and Trueness of nitrofuran metabolites using polymer sorbent

Analytes	Repeatability (CV_r, %)*			Reproducibility (CV_R, %)**		
	0.4 µg/kg	0.5 µg/kg	0.6 µg/kg	0.4 µg/kg	0.5 µg/kg	0.6 µg/kg
AOZ	12.26	17.11	14.56	15.54	21.42	16.31
AMOZ	11.58	9.24	12.87	13.22	16.27	15.72
AHD	15.57	14.45	9.93	18.43	18.32	10.57
SEM	18.71	12.28	11.69	21.61	15.19	13.59
NP-AOZ	9.26	9.99	7.77	15.18	13.76	15.43
NP-AOZ	14.73	13.17	10.44	17.24	16.25	12.55
NP-AHD	16.54	11.32	16.08	19.76	14.39	19.23
NP-SEM	17.08	16.67	19.25	22.11	19.42	20.45

*Repeatability values (CV_r, %) obtained in the replicate analysis of fortified samples (n ≥ 20).
**Reproducibility values (CV_R, %) obtained in the replicate analysis of fortified samples in different analytical series (4 series, n = 5).

Stability

The mean value of five stored duplicate solutions was within 2.66 – 7.89%, i.e. did not differ by more than 15% from the mean value of five freshly prepared duplicate solutions. The mean value of five freshly prepared solutions was used as the basis for calculating the percentage difference.

Matrix effects, %

The calculated ion inhibition of the matrix effect was in the range of 5.98 – 8.47%, which is acceptable for the validated method (N ≤ 20%).

For the solid-phase extraction of nitrofurans, ready-made columns with polymer-based sorbents – typically polypropylene cartridges containing a polystyrene-divinylbenzene copolymer – are most commonly used (Barbosa et al., 2007; Conneely et al., 2002; Kaufman et al., 2015; Leitner et al., 2001; Śniegocki et al., 2018; Tripathi et al., 2023). According to these authors, such sorbents enable strong and selective retention of nitroaromatic derivatives through π–π interactions.

Oasis MAX cartridges contain mixed- mode polymeric sorbent with reversed phase and anion exchange function. During sample loading, NBA may be retained by the ion exchange groups, while the NP derivative of nitrofurans may be retained by the reversed-phase sorbent. Ammonia (2%) can be used to remove the matrix co-extractive from the cartridge while enhancing the bonding of the NBA to the sorbent by the ion exchange mechanism. NP gets held by hydrophobic interaction with reversed phase functionality of the sorbent, which can be eluted with methanol.

In case of dual MAX/HLB SPE, while SPE on Oasis™ MAX cartridges gave good recovery of NP derivative a further clean-up step was needed to remove excess NBA. Although the MAX cartridge had the capacity to remove up to 93% NBA a significant amount of NBA still remained, as typically, NBA is added in1000-fold excess to that of NPAOZ to ensure complete derivatization of any AOZ present (Tripathi, 2023).

When choosing the right SPE sorbent, there are four main considerations to consider the type of sorbent, the sorption mechanism appropriate for the sample and analyte to be tested, the type of matrix and the method of extraction (GL Science, 2025).

As of today, the most widely used nitrofurans in the treatment of cattle are furaltadone, furazolidone, nitrofurazone and nitrofurantoin, all of which are broad-spectrum antibiotics containing a 5-nitrofuran ring structure and contain an azomethine bond ~C=N~. The presence of this bond and the nitro group causes the similarity of their physicochemical properties (Betsy, 2006).

The parent nitrofurans are rapidly metabolized to protein-bound metabolites, making the use of the parent drugs as marker residues ineffective; however, tissue-bound metabolites are stable in the body for several weeks after treatment and thus represent a better choice as marker residues (Gaastra et al., 2024; Ramos et al., 2017; Rixt et al., 2018). Based on this, the analytical strategy for the quantitative assessment of nitrofurans is based on the determination of 4 stable and persistent metabolites that can be released from proteins (Alkan et al., 2016). These stable metabolites are 3-amino-2-oxazolidone AOZ, 3-aminomorpholinomethyl-2-oxazolidinone AMOZ, 1-aminohydantoin AHD and semicarbazide SEM (Craken et al., 2001) (Figure 1).

Nitrofuran metabolites in a living organism meet in protein-bound forms and are released in an acidic environment, forming free compounds with a low molecular weight, are well soluble in polar solvents, and can be easily ionized, which complicates their analysis (Kaufmann et al., 2015; Xiaoing et al., 2022).

The relative molecular weights of AOP, AMOZ, AGD and CEM are 102.1, 201.2, 115.1 and 75.1, respectively, with no characteristic ion spread, making qualitative and quantitative analysis difficult. Derivatization increases the side chain molecular weight and, accordingly, the relative molecular weights of the nitrofuran metabolites to 248.2, 334.3, 235.2 and 208.2, respectively. Due to the covalent bonding of the metabolites to the tissue, solvent extraction of the sample will not recover the residues. Instead, an acid hydrolysis step is required to cleave the covalently bound marker residues from the tissue prior to analysis (Craken et al., 2001).

The metabolites are simultaneously treated with acid hydrolysis and derivatization reagents to increase the relative molecular weights of the target compounds, which, under appropriate conditions, allows for quantitative determination (Leitner et al., 2001). Derivatization of nitrofuran metabolites is performed using 2-nitrobenzaldehyde to yield the NP- derivatives shown in Figure 1, after which the sample is incubated at 37°C for 16 hours. The process using furazolidone as an example is shown in Figure 2 (Verdon et al., 2007).

Nitrofuran metabolites are Hydrogen Bond Acceptor Count and generally polar. This is because they contain functional groups such as amines, hydroxyls, and other polar groups that make them soluble in polar solvents. For example, main metabolites, SEM, AOZ and AMOZ are highly polar (NCBI, 2025).

Nitrofuran metabolites have a different structure than the parent compound, and these structural change significantly are changing affect polarity. The polarity of nitrofuran metabolites is a crucial factor in their analysis. Liquid chromatography (LC), in particular reversed-phase LC, is often used for their separation and detection. However, precisely because of the high polarity of these metabolites, derivatization with 2-nitrobenzaldehyde (2-NBA) is used to improve their retention and separation in reversed-phase columns (Arystanova, 2022; Vass et al., 2008).

Nitrofuran metabolites are generally stable in nature, meaning that they can persist in the environment and food products. Their polarity also affects how they interact with various matrices and how they are extracted and analysed (Moragues et al., 2024). The main goal in selecting the sorbent was to minimize the loss of nitrofuran metabolites directly related to the sample preparation procedure. To effectively isolate nitrofuran metabolites using solid-phase extraction (SPE), an appropriate sorbent must be selected based on the chemical properties of the analytes and the characteristics of the sample matrix.

Generally, sorbents like C18 or polymer material are used for non-polar solutions, while silica gel, diol, aminopropyl, or cyanopropyl are preferred for polar solutions. The specific sorbent should be tailored to the target analyte, considering its solvent solubility and the sample matrix's nature.

Nitrofuran metabolites have different solubility in different solvents and therefore it is necessary to select a sorbent that will ensure effective binding of analytes while minimizing the retention of interfering compounds (NCBI, 2025). Since nitrofuran metabolites are polar, a sorbent with polar characteristics (e.g. silica gel, diol) is recommended. Silica gel is a polar sorbent because its surface is covered with polar silanol (Si-OH) groups. These silanols act as hydrogen bond donors and acceptors, allowing silica gel to interact strongly with polar compounds. If the solvent polarity is weak, the adsorbent will exhibit a strong adsorption capacity for the solute (Shimadzu, 2025).

The polar nature of the Si-O bonds in the bulk structure of silica gel makes the entire material polar. Because of this polarity, silica gel can interact with other polar substances. While the bulk structure of silica gel is polar, what makes silica gel exceptionally versatile is its surface properties. Silica gel has a vast surface area due to its porous nature, and this surface can be modified in various ways to tailor its polarity for specific applications (Shimadzu, 2025).

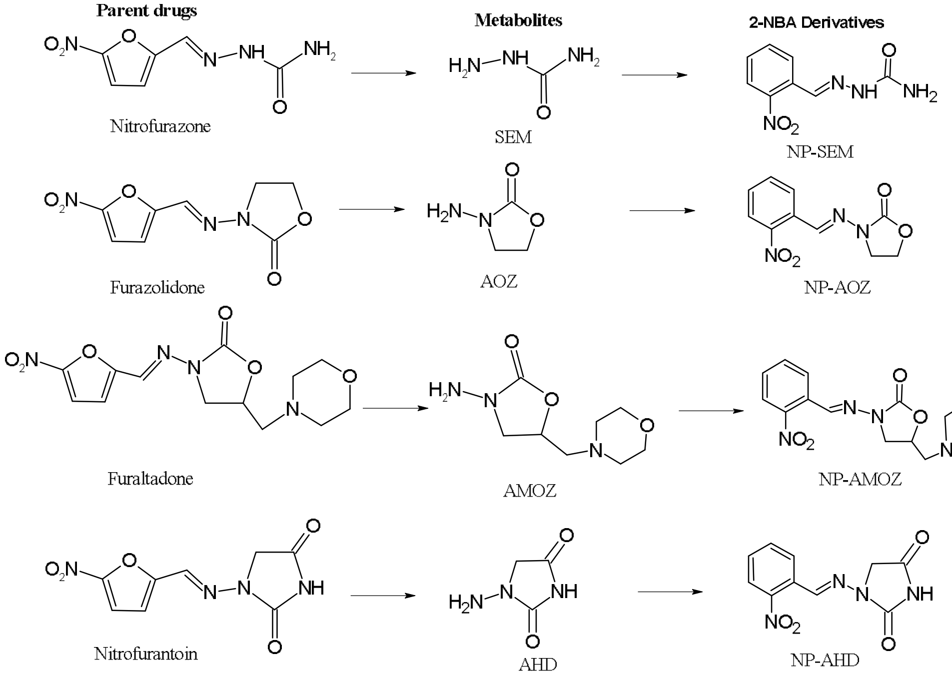

Figure 1. 2-NBA derivative products

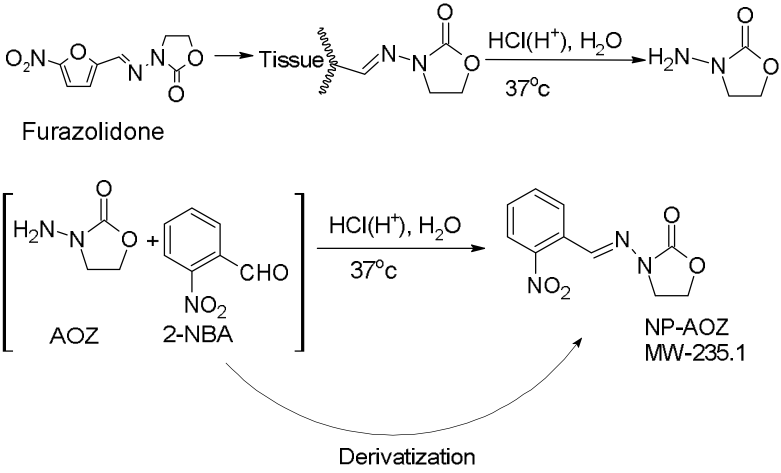

Figure 2. Derivatization of Furazolidone

In desiccation, silica gel's polar nature, combined with its high surface area, makes it highly effective at selectively adsorbing polar substances. The polar nature of silica gel allows it to interact strongly with polar compounds, causing them to be retained on the column's surface. Meanwhile, non-polar substances are less attracted to the silica gel and tend to pass through the column more quickly. This differential interaction helps separate complex mixtures into their individual components, a fundamental process in many analytical and preparative chemistry procedures.

Silica gel 60 is a widely used silicon dioxide (SiO_2) based sorbent with high adsorption capacity and stability. It is characterized by a small pore size (60 angstroms), which allows it to adsorb molecules of various sizes. Silica gel 60 also has a high specific surface area (about 500 m^2 g^{-1}) and pore volume (0.75 mL g^{-1}), which makes it effective for various applications. The characteristics of the adsorbent ensure high resolution and flow coefficient, maximum reproducibility of analytical results, as well as scalability of the method without the need for adjustments.

Based on the above, the choice of Silica gel 60 as a sorbent, rather than polymers, is justified (Besbes et al., 2009). The size of the molecules of the substance purified by the SFE method should be smaller than the pore size of the sorbent. The mass of the target component retained by the sorbent from the solution subjected to SFE, including retained impurities, is close in chemical nature to this component (approximately 5% of the sorbent mass). Thus, a 100 mg cartridge can retain approximately 5 mg of dissolved substances, which made it possible to calculate the required sorbent mass. The free volume of the sorbent is the sum of the volume of space between the particles and the pore volume of the sorbent. The free volume of the sorbent determines how much solvent is needed for SPE. To create optimal conditions for dilution, purification and elution, it is required that the volume of solvent exceeds the free volume of the sorbent by 4-8 times. Otherwise, the risk of incomplete solvation and low analyte recovery increases. Silica-based SPE products usually have a free volume to sorbent mass ratio of approximately 150 µL per 100 mg. Polymer sorbents require a larger volume of solvent. It is recommended to use 250 µL per 100 mg of sorbent.

Silica gel is a polar sorbent that can be used to extract nitrofuran metabolites from polar solutions. Selecting appropriate wash and eluent solvents for solid phase extraction (SPE) of nitrofuran metabolites requires consideration of both the analyte and sample matrix properties. Wash solvents are used to remove interfering compounds while eluents selectively extract the target nitrofuran metabolites.

Polar solvents have high permittivity, which allows for efficient separation of ionic charges and dissolution of polar molecules. Polar solvent molecules have partial charges, which makes them capable of interacting with polar solutes (Thermofisher, 2025).

The purpose of wash solvents is to remove co-extracted compounds that are not of interest while preserving the target analytes on the SPE cartridge. Connelly et al. (2002) used 2% acetic acid in 50% methanol, which served to disrupt the interaction of acidic analytes such as NBA, and used 2% ammonia in the wash solution for the dual effect of firstly cleaning the sample and secondly improving the binding of NBA to the sorbent for more selective elution of NPAOZ. Hexane along with water removes excess water trapped in the polymer phase of the cartridge before eluting the compounds (Khong et al., 2004).

Wash solvents should be less polar than the eluent to remove interferences without eluting the target analytes. Less polar solvents are usually non-polar. They include alkanes (such as pentane, hexane, and heptane) and aromatic compounds (such as benzene, toluene, and xylene). Other such solvents include diethyl ether, ethyl acetate, methylene chloride, chloroform, and others. Among these solvents, ethyl acetate is a moderately polar substance. The polar carbonyl group and polar oxygen atom create a dipole moment in the molecule, making it polar. At the same time, the ethyl group, which is non-polar, partially compensates for the polarity of other groups, so ethyl acetate is a moderately polar compound (Thermofisher, 2025)

Weak organic solvents, such as lower concentrations of methanol or acetonitrile in water, may also selectively remove certain types of interferences. The volume of wash solvents should be sufficient to remove the interferences, typically 2–5 times the void volume of the SPE cartridge.

The purpose of the eluent is to selectively and efficiently elute nitrofuran metabolites from the SPE cartridge into the final extract. To effectively wash nitrofuran metabolites from silica gel in solid-phase extraction (SPE), a solvent with a good balance of polarity and ability to dissolve the target compounds is required. Solvents with moderate to high polarity, such as methanol or acetonitrile, are usually more effective in dissolving them.

The solvent chosen should be compatible with subsequent analytical methods (e.g., LC) to ensure accurate and reliable quantification. It is necessary to elute with a smaller volume of solvent to obtain a more concentrated extract (Thermofisher, 2025).

To sum it up, we can say that, methanol can be a versatile solvent for washing polar substances, especially when used in combination with other polar solvents or water, acetonitrile is another good choice for washing polar substances due to its ability to dissolve polar compounds and its relatively low boiling point.

The polarity of the sample matrix can influence the choice of solvents. If the sample contains particles, filtration or centrifugation may be required prior to SPE to avoid clogging of the cartridge. The meat matrix exhibits both polar and non-polar characteristics due to the presence of both hydrophilic and hydrophobic components. This is because meat proteins, like other proteins, contain amino acid side chains that can be polar or non-polar. In addition, the fats and lipids in meat also contribute to the non-polar aspects of the matrix (García et al., 2023).

Meat contains water, which is a polar solvent. In addition, some of the amino acid side chains in meat proteins are polar, which contributes to the overall polarity of the matrix. Meat also contains fats and lipids, which are non-polar and hydrophobic. These contribute to the overall non-polar nature of the meat matrix. Meat proteins, like many other proteins, are amphipathic, meaning they have both polar and non-polar regions. This allows them to interact with both water and fat. Meat is therefore not simply polar or non-polar, but rather a complex matrix with hydrophilic and hydrophobic properties (Decker et al., 2022).

By carefully considering these factors, suitable solvents were selected for efficient and accurate washing and elution of nitrofuran metabolites from the solid-phase column. In particular, acetonitrile was used for washing and methanol was used for elution.

In conclusion, it should be repeated that the selected sorbent and solid-phase purification mode are optimal, which has been confirmed experimentally, in particular, 89–96% extraction of nitrofuran metabolites with good reproducibility (RSD ≤ 2.0%) was achieved.

The SPE purification column we propose is specifically designed for nitrofuran metabolites, offering high selectivity and specificity. It enhances the quantitative detection limit of the HPLC method, enabling the use of a diode array detector instead of more expensive cartridges and mass spectrometers. This approach achieves the determination of nitrofuran metabolite levels below the established MRL of 0.5 µg/kg, while maintaining relatively low financial costs.

The proposed conditions for determining nitrofuran metabolites are relatively accessible, enabling small farmers and producers in developing countries and regions to implement the method, thereby ensuring the safe use of meat and meat products.

Conclusions

The following conclusions can be drawn from the conducted studies:

(1) a sorbent – silica gel 60, particle size 0.2 – 0.5 mm, – was selected for purification of the extract of nitrofuran metabolites and confirming the advantages of using this column using validation;

(2) columns for solid-phase purification of nitrofuran metabolites were prepared from the selected sorbent, cartridge size – (20 x 400 mm);

(3) selected solvents for washing nitrofuran metabolites from the solid-phase column – ethyl acetate and for elution – acetonitrile;

(4) the advantage of the columns manufactured using silica gel compared to the polymer sorbent – polypropylene was experimentally proven. 96-98% extraction of nitrofuran metabolites was achieved with good reproducibility (RSD ≤ 2.0%), since when using a polymer sorbent, the extraction does not exceed 94%;

(5) the advantage of the proposed column over disposable, expensive, ready-made columns for purification and concentration of four nitrofuran metabolites isolated from meat is its significantly lower cost combined with high specificity and selectivity. This enhances the sensitivity of the HPLC method, enabling the use of a diode array detector instead of a costly mass spectrometer to detect nitrofuran metabolites below the EU's Minimum Required Performance Limit of 0.5 µg/kg in the complex meat matrix. As a result, the method becomes financially acceptable for small farmers, developing countries and regions, which in turn will ensure the safe use of meat and meat products in these regions.

Evaluation of results

The method has been validated in accordance with the requirements of European Commission Decision 2021/808/EC. The effectiveness of the proposed column and the use of a diode detector for HPLC determination of nitrofuran metabolites was confirmed by the results of professional testing by the Globaltest testing laboratory accredited according to ISO 17025 by the Accreditation Agency of Georgia.

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Cite:

UFJ Style

Gotsiridze D., Chikviladze T., Baramidze K. (2025), Sorbent selection for solid-phase clean-up of nitrofuran metabolites from meat, *Ukrainian Food Journal*, 14(2), pp. 304–324, https://doi.org/10.24263/2304-974X-2025-14-2-9

APA Style

Gotsiridze, D., Chikviladze, T., & Baramidze, K. (2025). Sorbent selection for solid-phase clean-up of nitrofuran metabolites from meat. *Ukrainian Food Journal, 14*(2), 304–324. https://doi.org/10.24263/2304-974X-2025-14-2-9

Effect of stabilizers on the colligative properties of whey-based ice cream

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Keywords:

Ice cream
β-glucan
Cryoscopic temperature
Ice crystals
Texture

Article history:

Received 20.11.2024
Received in revised form 21.03.2025
Accepted 30.06.2025

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DOI:
10.24263/2304-974X-2025-14-2-10

Abstract

Introduction. The effect of β-glucans derived from oats and yeast on the physicochemical properties, cryoscopic temperature, frozen water content, and ice crystal growth in whey ice cream during storage was investigated.

Materials and methods. The cryoscopic temperature was measured using a cryoscope Marcel osm 3000. Based on the data obtained on the freezing point of the ice cream mixes, the frozen water content under variable low-temperature processing conditions and thermodynamic parameters were calculated. The growth of ice crystals in the ice cream was studied by microscopy and modeled using power-law equations derived from the experimental measurements.

Results and discussion. β-glucans from oats or yeast in the amount of 0.5% significantly affect the physicochemical and structural properties of ice cream. The β-glucan from oats reduces the cryoscopic temperature of the mixture the most (–6.040°C), compared to the sample without the stabilizer (–4.222°C) and the mixture with Cremodan SI 320 (–4.688°C). Along with its high cryoprotective ability, β-glucan from oats provides the maximum content of frozen water in ice cream 97.27% at –30°C, which is probably due to the formation of low-energy weak bonds between water molecules and β-glucan macromolecules.

At the same time, β-glucan from yeast with a lower effect on the cryoscopic temperature (–3.846°C) more significantly counteracted the growth of ice crystals, the average diameter of which did not exceed 11.08 μm on the 30th day of ice cream storage, compared to the control sample (27.5 μm). This fact indicates a different mechanism of the structuring effect of β-glucan from yeast, associated with the formation of a spatial gel matrix in the aqueous phase, which limits crystal growth and stabilizes the air phase of ice cream. The osmotic pressure in the samples with yeast β-glucan was slightly higher (up to 8.84 kPa) compared to the sample with oat β-glucan, which reflects the peculiarities of its colloidal structure and enhances the stabilizing effect. The heat of fusion decreased in the samples with oat β-glucan, indicating a decrease in the stability of the crystalline phase and an easier phase transition. On the contrary, an increase was observed in mixtures with yeast β-glucan, indicating an increase in the stability of the ice structure. The developed stepwise model of ice crystal growth based on experimental data makes it possible to predict and prevent changes in ice cream texture during long-term storage.

Conclusion. Addition of oat β-glucan to whey ice cream exhibited cryoprotective properties, while addition of yeast β-glucan contributed to structural gel stabilization, inhibiting the growth of ice crystals during low-temperature storage of ice cream.

Introduction

Colligative properties of food systems such as freezing point depression, osmotic pressure, and others are closely linked to the physicochemical transformations occurring during freezing. In ice cream production, the cryoscopic temperature serves as a critical parameter, as it governs the degree of free water removal through freezing and consequently determines the product's texture, structural stability, and ice crystal formation within the ice cream matrix (Harfoush et al., 2024). Simultaneously, osmotic pressure provides insight into the water-holding capacity of ingredients and their influence on the state of water in the system (Liu et al., 2021).

Whey is widely used as a raw material in the production of dairy desserts (Kuzmyk and Bohdanova, 2022; Mykhalevych et al., 2024a; Tomczyńska-Mleko et al., 2024). Unlike conventional ice cream, whey-based ice cream is formulated using fresh or dried milk whey, sometimes supplemented with milk, cream, and flavoring agents (Meneses et al., 2020). This product type is typically characterized by a reduced content of total solids and proteins, resulting in a higher proportion of free water, lowered osmotic pressure, and a relatively modest impact on the cryoscopic temperature (Shevchenko et al., 2022). These factors collectively promote intensive freezing of unbound water, which may lead to structural destabilization of ice crystals during low-temperature processing and storage. Accordingly, a more detailed investigation into the processes of water crystallization, the binding mechanisms of free water, and the morphological characteristics of the ice phase is essential to ensure the desired textural properties and stability of ice cream formulated with novel ingredients.

At the preliminary stage of the study, the formulation of whey-based ice cream was scientifically developed using a liquid hydrolyzed concentrate of demineralized whey, supplemented with whey protein isolate and stabilizing agents, specifically β-glucans derived from oats or yeast (Mykhalevych et al., 2024a, 2024b). This formulation has been shown to improve the rheological properties of ice cream mixes. However, the cryoprotective properties of β-glucans used as a natural alternative to traditional commercial stabilizing systems have not yet been fully studied, especially under low-temperature processing and long-term storage conditions.

Stabilizers traditionally incorporated into ice cream play a dual role, contributing both to structure formation and cryoprotection (Kamińska-Dwórznicka et al., 2022). These compounds influence the concentration of dissolved solids, viscosity, and water-binding interactions, thereby affecting the cryoscopic temperature, osmotic pressure, and phase transitions of water (Goff et al., 2025; Kot et al., 2023). In light of evolving consumer demands for clean-label formulations and enhanced product functionality, replacing synthetic stabilizing systems with natural, functionally active ingredients has become increasingly relevant.

In this context, β-glucans, especially those sourced from oats and yeast, have attracted significant attention. Their hydrophilic nature, gel-forming capacity, antioxidant activity, and ability to increase viscosity in food matrices position them as promising ingredients for ice cream applications (Ahmad et al., 2024; Mykhalevych et al., 2022; Shevchenko et al., 2023; Stabnikova et al., 2023; Tomczyńska-Mleko et al., 2024; Zhao et al., 2023). Nevertheless, the widespread adoption of β-glucans necessitates validation of their cryoprotective efficacy, particularly in terms of their ability to reduce the extent of free water crystallization and to enhance the structural stability of the final product.

A critical component in evaluating the functional-technological potential of β-glucans involves assessing the dynamics of free water removal through freezing within the ice cream matrix. This is of fundamental importance, as free water participates directly in phase transitions and induces structural rearrangements in the product as temperature decreases (Tan et al., 2021). The rate of free water freezing determines the size, morphology, and fractional distribution of the resulting ice crystals (Petzold et al., 2009). Excessively large crystals can disrupt the protein–polysaccharide matrix, compromising textural integrity and diminishing the sensory quality of the product (Kamińska-Dwórznicka et al., 2015; 2020). Hence, the kinetics of ice crystal growth not only represent a physical parameter of the system but also serve as a critical indicator of structural stability and the efficiency of matrix formation. It is therefore imperative to investigate the influence of β-glucans on the cryoscopic temperature, the rate of free water crystallization during technological processing, and the subsequent ice crystal development during storage.

Accordingly, this study aims to conduct a comprehensive examination of the colligative properties of whey-based ice cream systems formulated with natural stabilizing agents. Such an investigation will advance the mechanistic understanding of structure formation and stabilization processes during hardening and storage of frozen dairy desserts.

Materials and methods

Raw materials and sample preparation

To produce the liquid hydrolyzed concentrate of demineralized whey (containing 40% total solids), the following ingredients were employed: spray-dried demineralized whey with 90% demineralization (PJSC "Dairy Alliance," Ukraine), the β-D-galactosidase enzyme (5000 NLU/g, Danisco, Denmark), the starter culture *Lactobacillus acidophilus* LYO 50 DCU-S (Danisco, Denmark), and potable water. Ice cream formulations were prepared with the addition of a commercial stabilizing system (Cremodan SI320), whey protein isolate with a protein content of 90% (Spomlek, Poland), white sugar, vanillin, and water.

The study employed two types of β-glucans:

- Oat β-glucan (Grupa Feniks 2050, Poland) is a 1→3, 1→4 β-D-glucan, a non-cellulosic polysaccharide extracted from Avena sativa L. grains or their derivatives (bran, oat fiber) using a proprietary method. According to the manufacturer's specifications, this highly purified product contains 72% oat β-glucan, with a moisture content not exceeding 6% and an ash content not exceeding 5%. The remaining composition consists primarily of starch, protein, insoluble fiber, and water. Its high purity is evidenced by complete solubility and the absence of odor, color, and taste. Even at low concentrations, it readily forms stable hydrogels upon interaction with water.
- Yeast β-glucan (GOLDCELL, Brazil) derived from the cell walls of baker's yeast (*Saccharomyces cerevisiae*), presented in spray-dried powder form ranging in color from light to dark bronze. The product contains no less than 70% (1→3),(1→6)-linked β-glucan, with moisture content not exceeding 8% and ash content between 5% and 8%.

The starter culture was activated in skim milk at 38–42 °C until reaching a pH of 5.4–5.2. The dry whey was reconstituted at 40–45 °C, followed by pasteurization (85–88 °C, 3–5 min), cooling to 40–43 °C, and enzymatic hydrolysis for 8 hours to achieve at least 95% lactose degradation, as previously described (Mykhalevych et al., 2024c).

Dry ingredients of the ice cream mix were blended with the liquid whey concentrate and water, filtered through a 1 mm sieve, pasteurized (80–82 °C, 1–2 min), homogenized at 12.0±2.5 MPa, cooled, inoculated with the activated starter, and fermented until pH 5.10–5.25 was reached. The fermented mixture was subsequently cooled and subjected to a two-stage freezing process: preliminary cooling to −1 °C (shear rate 4.5 s^{-1}, 120 s), followed by dynamic freezing to −5.0±0.5 °C (shear rate 9 s^{-1}, 180 s). Ice cream samples were hardened and stored at −18±1 °C for 30 days. Each formulation was produced in triplicate to ensure data reproducibility.

The following sample groups were prepared:

- C – control sample without stabilizing ingredients;
- 0.6%SS – sample with 0.5% Cremodan SI320 stabilizing system;
- 0.25%OBG – sample with 0.25% oat β-glucan;
- 0.5%OBG – sample with 0.5% oat β-glucan;
- 0.25%YBG – sample with 0.25% yeast β-glucan;
- 0.5%YBG – sample with 0.5% yeast β-glucan.

The formulation of the ice cream mix was as follows (in% w/w): liquid hydrolyzed demineralized whey concentrate – 75.0%; sucrose – 9.0%; whey protein isolate – 3.0%; activated starter culture – 3.0%; vanillin – 0.01%; water – 9.39–9.74%; stabilizer – 0.25–0.6%.

Methods

The total solids content in the ice cream samples was determined using the gravimetric method by drying at 105 °C to constant weight, in accordance with standard arbitration procedures.

The mass fractions of carbohydrates, namely sucrose, lactose, glucose, and galactose, were quantified by high-performance liquid chromatography (HPLC) using a Shimadzu LC-6A chromatographic system (Kyoto, Japan) equipped with a refractive index detector. Separation was performed on an SCR-101-N column (250 × 4.7 mm), with degassed deionized water as the mobile phase. The flow rate was maintained at 0.5 mL/min (Mykhalevych et al., 2025).

The freezing point (cryoscopic temperature) was calculated theoretically based on the sucrose equivalence method, following the approach proposed by Marshall et al. (2003):

$$ES = (TS_w \times 0{,}765) + S,$$

where ES is the sucrose equivalence; TS_w is total solids of dried demineralized whey,%; S is the sugar content,%.

For samples containing stabilizing ingredients, the formula was modified to take into account their water-binding capacity:

$$ES = (WB \times MF) + (TS_w \times 0{,}765) + S,$$

where WB is the amount of bound water (g water per g stabilizing agent); MF is the mass fraction of the stabilizing ingredient in the mixture. Weakly bound water was not taken into account in the calculation, so coefficients of 0.66 for mixtures with a commercial stabilization system, 0.80 for mixtures with oat β-glucan, and 0.50 for mixtures with yeast β-glucan were introduced.

The cryoscopic temperature was also measured using a Marcel osm 3000 osmometer (Marcel, Poland) with an accuracy of 0.002 °C (Kamińska-Dwórznicka and Kot, 2024).

The difference between the calculated (Tf_t) and experimentally (Tf_e) measured cryoscopic temperature was determined by the formula:

$$\Delta Tf = Tf_e - Tf_t.$$

The content of frozen water at different stages of low-temperature processing of model ice cream samples was calculated using the Zhadan formula (Dibirasulaev et al., 2016):

$$\omega = 1 + \frac{1{,}12 - 0{,}05 \times t}{t} ,$$

where ω is the mass fraction of frozen water,%; 1.12 is the empirical coefficient; t is the cryoscopic temperature and the processing temperature without the "–" sign.

The molarity of ice cream mixtures was calculated using the formula (Atkins and de Paula, 2010):

$$b = \frac{\frac{n}{M}}{\text{m water}} ,$$

where b is the molarity, mol/kg of solvent (water); m is the mass of each osmotically active substance (glucose, galactose, lactose, etc.), g; M is the molecular weight of the substance, g/mol; m_{water} is the mass of water in the system, kg.

The osmotic pressure was calculated according to the Van Hoff equation:

$$\pi = i \times C \times R \times T$$

where π is the osmotic pressure, Pa; i is the isotonic coefficient (≈1); C is the molar concentration, mol/L; R is the gas constant (8.314 J/mol-K); T is the temperature, K.

The phase transition energy was determined by a modified thermodynamic formula (Atkins and de Paula, 2010):

$$\Delta Hf = \frac{R \times Tf2 \times b}{\Delta Tf},$$

where ΔHf is the heat of phase transition (J/mol), T_f is the freezing point of the pure solvent (273.15 K), b is the molarity, ΔT_f is the decrease in freezing point, K, calculated as a module of the experimental T_f; R=8.314 J/(mol-K) is the universal gas constant.

The molar Gibbs free energy was determined by the formula (Atkins and de Paula, 2010):

$$\Delta G = -R \times T \times \ln b,$$

where lnb is the natural logarithm of the molarity.

The size of ice crystals in ice cream was studied using an Olympus BX53 microscope with a Linkam LTS420 cooling system (measuring temperature range from -196 °C to -420 °C) and an Olympus SC50 digital camera. For each sample, 300 to 500 crystals were labeled and the area, equivalent diameter, and standard deviation were calculated using NIS Elements D Imaging software (version 5.30.00, Nikon).

To model the kinetics of ice crystal growth during whey ice cream storage, the authors developed an empirical power-law model that allows describing the change in the average diameter of ice crystals depending on storage time:

$$d(t) = a \times t^b ,$$

where d(t) is the average diameter of ice crystals at time t, μm; t is the storage time, days (1, 7, 30); a is the empirical growth coefficient characterizing the initial growth rate of crystals; b is the exponent (reflects the nature of changes: slowing or accelerating growth).

To determine the parameters a and b, the least-squares approximation in logarithmic form was used:

$$\ln(d) = \ln(a) + b \cdot \ln(t).$$

After linearizing the dependence and plotting ln(d) versus ln(t), the model parameters were determined from the straight line equation. For each sample, a regression analysis was performed in Microsoft Excel, based on which the coefficients a and b, as well as the coefficient of determination R^2 were determined (Montgomery and Runger, 2018).

Data were expressed as means with standard deviations in triplicate. Statistical analysis was performed using Statistika 10 software. Differences were considered significant at a validity of $\alpha = 0.95$.

Results and discussion

Chemical composition

The chemical composition of the main ingredients that affect the colligative properties of ice cream, in particular, the lowering of the freezing point, is given in Table 1. The appearance of ice cream samples with β-glucans is shown in Figure 1.

The total solids content in the investigated ice cream formulations ranged from 42.05% to 42.61%, which is consistent with full-fat premium ice cream standards (fat content 14–18%) (Goff and Hartel, 2013). The mass fractions of key saccharides, namely glucose (16.02–16.51%), galactose (15.79–16.35%), lactose (0.72–0.78%), and sucrose (8.97–9.05%), play a pivotal role in determining the cryoscopic behavior of the system, as their cumulative colligative activity significantly influences the freezing point of the food matrix.

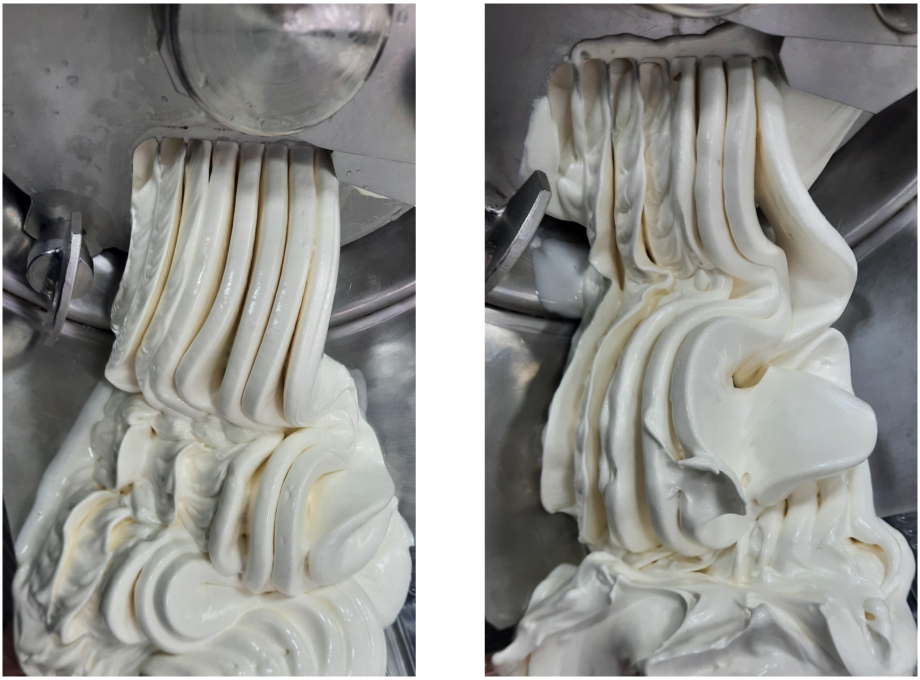
The sucrose content in the samples was notably lower than the standard levels typically observed in conventional ice cream formulations. According to Goff and Hartel (2013), the total sweetener content in classical ice cream usually ranges from 14% to 22%. Similarly, Meneses et al. (2020) reported a sugar content of 17.44% in whey- and buttermilk-based ice creams. Meanwhile, Goff (2018) notes that sugar content in both commercial and artisanal ice creams can vary widely, from 9% to 28%, depending on the formulation and market segment.

Table 1

Chemical composition of the test samples

Ice cream	Solids,%	Lactose,%	Glucose,%	Galactose,%	Sucrose,%
C	42.05^{a}±0.91	0.73^{a}±0.05	16.45^{a}±0.02	15.90^{a}±0.02	9.02^{a}±0.03
0.6%CC	42.61^{a}±1.02	0.75^{a}±0.02	16.51^{a}±0.14	15.79^{a}±0.07	8.98^{a}±0.04
0.25%OBG	42.28^{a}±0.67	0.72^{a}±0.05	16.32^{ab}±0.08	16.07^{a}±0.52	8.97^{a}±0.02
0.5%OBG	42.50^{a}±0.95	0.75^{a}±0.03	16.02^{ab}±0.10	16.35^{a}±0.15	8.99^{a}±0.05
0.25%YBG	42.33^{a}±1.12	0.78^{b}±0.01	16.48^{ab}±0.17	15.88^{a}±0.12	9.05^{a}±0.02
0.5%YBG	42.24^{a}±1.04	0.76^{a}±0.06	16.39^{b}±0.23	15.94^{a}±0.05	9.01^{a}±0.04

Notes:
C – ice cream without stabilizing ingredients;
0.6%SS – ice cream with 0.5% of the Cremodan SI 320 stabilizing system;
0.25OBG – ice cream with 0.25% oat β-glucan;
0.5%OBG – ice cream with 0.5% oat β-glucan;
0.25%YBG – ice cream with 0.25% yeast β-glucan;
0.5%YBG – ice cream with 0.5% yeast β-glucan.
$^{a\text{-}b}$-mean values denoted in columns by different letters differ statistically significantly at $p \le 0.05$.



a b

Figure 1. Appearance of soft whey ice cream at the exit of the freezer:
a – with oat β-glucan, b – with yeast β-glucan.

In the present study, the reduced sucrose concentration is compensated by a high content of monosaccharides, which results from the hydrolysis of lactose in demineralized whey, which serves as the main raw material. This compositional feature directly affects water mobility, crystallization kinetics, and ice crystal growth, thereby influencing the microstructure and textural stability of the resulting frozen food system.

Freezing point and frozen water content

The freezing point characterizes the ability of a mixture to freeze, which directly affects the texture quality of ice cream. The theoretical and experimental values of the freezing point (Tf) of the ice cream samples, as well as the difference between them (ΔTf), are presented in Table 2.

Table 2

Freezing point of ice cream mixtures

Sample	Tf_t, °C	Tf_e, °C	ΔT_f, °C
C	–4.432	$–4.222^b±0.14$	–0.210
0.6%SS	–4.600	$–4.688^b±0.03$	+0.088
0.25%OBG	–5.111	$–5.108^c±0.25$	–0.003
0.5%OBG	–5.177	$–6.040^d±0.18$	+0.863
0.25%YBG	–3.926	$–3.888^a±0.07$	–0.038
0.5%YBG	–3.924	$–3.846^a±0.02$	–0.078

Notes:
C – ice cream without stabilizing ingredients;
0.6%SS – ice cream with 0.5% of the Cremodan SI 320 stabilizing system;
0.25OBG – ice cream with 0.25% oat β-glucan;
0.5%OBG – ice cream with 0.5% oat β-glucan;
0.25%YBG – ice cream with 0.25% yeast β-glucan;
0.5%YBG – ice cream with 0.5% yeast β-glucan.
a-d-mean values denoted in columns by different letters differ statistically significantly at $p \leq 0.05$.

For the control sample, the theoretical freezing point (Tf) was –4.432 °C, while the experimental value was –4.222 °C. The difference of 0.210 °C is within an acceptable deviation range, indicating accurate measurement procedures. The inclusion of 0.6% Cremodan SI 320 stabilizing system led to a decrease in the theoretical Tf to –4.600 °C, whereas the experimental Tf was –4.688 °C, yielding a positive deviation (ΔTf = +0.088 °C). This small difference suggests that Cremodan SI 320 contributes to a slight additional depression of the freezing point, likely due to its capacity to form stable colloidal structures that bind water and influence phase transitions.

The incorporation of β-glucans into the ice cream mixtures also affected their freezing points. The sample with 0.25% oat β-glucan exhibited nearly perfect agreement between theoretical (–5.111 °C) and experimental (–5.108 °C) values, with ΔTf = –0.003 °C, indicating a predictable system behavior at this low concentration. However, increasing the oat β-glucan concentration to 0.5% resulted in a significant decrease in experimental Tf to –6.040 °C, compared to a theoretical value of –5.177 °C. The corresponding deviation (ΔTf = +0.863 °C) demonstrates a marked impact of the polysaccharide on water–phase interactions.

This enhanced cryoscopic effect is likely attributable to the formation of more structured hydrogels capable of more effectively binding water. β-glucans are reported to form viscous gels and pseudoplastic solutions due to the complex conformational arrangement of their polymer chains (Wang et al., 2017; Vaikousi et al., 2004). As such, β-glucan solutions may function as stabilizers or thickeners in food formulations (Kaur et al., 2020).

Samples containing 0.25% and 0.5% yeast-derived β-glucan showed only minor differences between theoretical and experimental Tf values (–0.038 °C and –0.078 °C, respectively), indicating a moderate influence of this additive on the freezing behavior of the mixture.

Thus, the stabilizing system Cremodan SI 320 and the β-glucans exert differential effects on the freezing point of ice cream. Cremodan SI 320 contributes to a moderate depression of Tf through colloidal structuring, whereas elevated concentrations of oat β-glucan significantly intensify this effect. These findings are critical for optimizing ice cream formulations with respect to desired textural attributes and product stability.

A general trend was observed wherein the proportion of frozen water increased with decreasing temperature, in accordance with the fundamental freezing principle: the lower the temperature, the greater the amount of liquid transitioning into the solid phase (Table 2). For the control sample (C), this proportion ranged from 81.82% at –5 °C to 97.73% at –40 °C.

Table 3

Content of frozen water in ice cream at low temperature processing, %

t, °C	C	0.6%SS	0.25%OBG	0.5% OBG	0.25%YBG	0.5% YBG
–5	81.82	82.29	82.71	83.64	81.49	81.45
–10	90.91	91.14	91.35	91.82	90.74	90.72
–15	93.94	94.10	94.24	94.55	93.83	93.82
–20	95.46	95.57	95.68	95.91	95.37	95.36
–25	96.36	96.46	96.54	96.73	96.30	96.29
–30	96.97	97.05	97.12	97.27	96.91	96.91
–35	97.40	97.47	97.53	97.66	97.36	97.35
–40	97.73	97.79	97.84	97.96	97.69	97.68

Notes:
C – ice cream without stabilizing ingredients;
0.6%SS – ice cream with 0.5% of the Cremodan SI 320 stabilizing system;
0.25OBG – ice cream with 0.25% oat β-glucan;
0.5%OBG – ice cream with 0.5% oat β-glucan;
0.25%YBG – ice cream with 0.25% yeast β-glucan;
0.5%YBG – ice cream with 0.5% yeast β-glucan.

The impact of added stabilizing agents on the frozen water content was manifested in a slight increase in this parameter compared to the control sample across the studied temperature range. Specifically, the sample containing 0.6% Cremodan SI 320 exhibited a marginally higher frozen water content than the control, indicating a slight enhancement of the crystallization process due to the stabilizing system.

Oat β-glucan added at concentrations of 0.25% and 0.5% demonstrated a more pronounced effect: the frozen water content in these samples consistently exceeded that in the control and the sample containing the commercial stabilizer under all temperature conditions. This suggests that the polysaccharide intensifies water freezing, which can be attributed to its ability to form hydrogel structures that modulate water molecule mobility within the mixture (Kaur et al., 2020).

Samples enriched with yeast-derived β-glucan showed frozen water contents comparable to the control and Cremodan-containing samples and were less pronounced in this regard than those with oat β-glucan. This may reflect structural differences and varying hydrophilicity between β-glucans, which in turn affect the freezing behavior of the aqueous phase. Lazaridou and Biliaderis (2004) found that β-glucans derived from cereals exhibit enhanced gelation at subzero temperatures, improving the functional properties of gel-forming materials. Regarding yeast β-glucan, Xu et al. (2009), based on small-amplitude oscillatory rheological analyses, and identified its unique capacity to actively participate in gelation processes. Yeast β-glucan possesses significant potential for energy-based bonding, with its effectiveness notably increasing in systems with a low κ-carrageenan content, where it acts not only as a structural component but also as an active element of the gel matrix. This suggests that while yeast β-glucan may not form structures as firm as those formed by oat β-glucan, it contributes strength through a high number of molecular interactions. Nevertheless, further investigation is required to elucidate the cryoprotective properties of yeast β-glucan. It is worth noting that the difference in the effect between 0.25% and 0.5% doses of yeast β-glucan was minimal, indicating a potential saturation effect at low inclusion levels.

Thus, the findings indicate that the presence of β-glucans, especially oat-derived, leads to an increase in the fraction of frozen water during low-temperature treatment of ice cream. This phenomenon may potentially enhance the product’s textural attributes and structural stability (Hagiwara and Hartel, 1996). In ice cream systems, it is primarily the free (unbound) water that undergoes freezing. β-glucans were expected to bind more free water and thus reduce the amount of frozen water. However, the data in Table 3 indicate the opposite, an increase in frozen water content, which may be explained by the formation of low-energy, weak interactions between water molecules and β-glucan macromolecules. Therefore, a promising direction for future research lies in the comparative analysis of the binding energy between water and various glucans versus classical stabilizers. Different glucans may form a varying number of hydrogen bonds, which could influence the extent of water freezing during the dynamic freezing stage. In contrast, traditional stabilizers may bind water more strongly, with some of this bound water freezing only during the hardening phase.

Thermodynamic properties of ice cream mixtures

The total number of moles (Σ moles) and molality vary slightly between samples, indicating similar concentrations of solutes in the systems. The molality varies between 3.59-3.63 mol/kg (Table 4).

The osmotic pressure of the control sample was determined to be 8.73 kPa. The addition of 0.6% of the Cremodan SI320 stabilizing system led to a slight increase in osmotic pressure to 8.82 kPa, which may reflect modifications in the colloidal network and an increase in the total activity of dissolved solutes. The influence of β-glucans of different origins varied. Samples containing 0.25% and 0.5% oat β-glucan exhibited osmotic pressures close to that of the control, ranging from 8.73 to 8.76 kPa. In contrast, the corresponding yeast β-glucan samples showed slightly elevated osmotic pressures of 8.84 kPa and 8.78 kPa, respectively. These differences likely stem from variations in molecular architecture and solubility: yeast-derived β-glucans possess higher molecular weights and a tightly packed triple-helix conformation, rendering them largely water-insoluble (Wang et al., 2025), whereas oat β-glucans contain a higher proportion of water-soluble fractions (Yu et al., 2021).

Table 4

Thermodynamic and colloidal parameters of ice cream mixtures with different stabilizing ingredients

Sample	Σ moles	Mole content, mol/kg	Osmotic pressure, kPa	ΔHf, J/mol	ΔG, J/mol
C	0.2080	3.59	8.73	527 473.88	−3124
0.6%CC	0.2076	3.62	8.82	478 902.30	−3143
0.25%OBG	0.2072	3.59	8.73	437 817.36	−3124
0.5%OBG	0.2071	3.60	8.76	371 739.38	−3131
0.25%YBG	0.2092	3.63	8.84	576 367.85	−3151
0.5%YBG	0.2085	3.61	8.78	580 799.70	−3137

Notes:
C – ice cream without stabilizing ingredients;
0.6%SS – ice cream with 0
.5% of the Cremodan SI 320 stabilizing system;
0.25OBG – ice cream with 0.25% oat β-glucan;
0.5%OBG – ice cream with 0.5% oat β-glucan;
0.25%YBG – ice cream with 0.25% yeast β-glucan;
0.5%YBG – ice cream with 0.5% yeast β-glucan.

The phase transition enthalpy (ΔHf), representing the heat of fusion, decreased with increasing concentrations of oat β-glucan, from 527 473.88 J/mol in the control sample to 371 739.38 J/mol at 0.5% inclusion. This suggests a facilitation of the solid-to-liquid phase transition, likely due to the structural influence of polymeric networks on ice crystal stability. In contrast, an opposite trend was observed in samples with yeast β-glucan: ΔHf increased to 580,799 J/mol at 0.5% concentration, indicating enhanced crystalline phase stability and a higher enthalpic requirement for melting.

The molar Gibbs free energy (ΔG) values were negative across all samples, consistent with the thermodynamic favorability of the freezing process (Atkins and de Paula, 2014). ΔG values ranged from –3124 J/mol (control) to –3151 J/mol (0.25% yeast β-glucan), suggesting small but notable differences in system stability.

These thermodynamic observations underscore the distinct impacts of β-glucan types on the thermal behavior of ice cream matrices: oat β-glucan promotes phase transition by reducing fusion enthalpy, while yeast β-glucan enhances melting energy requirements, potentially contributing to greater structural integrity during storage. These results are in line with previous findings concerning the textural modifications induced by β-glucans, specifically, a softening effect from yeast β-glucan and an elasticity-promoting effect from oat β-glucan incorporation (Mykhalevych et al., 2024b).

Ice crystal growth

Table 5 presents the temporal evolution of the average diameter of ice crystals in the ice cream samples over storage periods of 1, 7, and 30 days. Both experimental measurements and predicted values derived from a power-law growth model are provided. The high coefficients of determination (R^2 ranging from 0.91 to 0.98) confirm the strong predictive capability and reliability of the applied mathematical model.

Table 5

Measured and modelled ice crystal sizes in ice cream samples over storage

Sample	Storage time, days	D_A, µm, measured	D_A, µm, modelled	Power-law model	R^2
C	1	$18.50^{a}\pm1.21$	18.90	y = 0.1186x + 2.9389	0.9569
	7	$25.01^{b}\pm1.06$	23.80		
	30	$27.50^{c}\pm0.78$	28.28		
0.6%CC	1	$15.80^{a}\pm0.67$	15.16	y = 0.2048x + 2.7186	0.9454
	7	$20.50^{b}\pm0.77$	22.58		
	30	$32.15^{c}\pm1.18$	30.42		
0.25%OBG	1	$18.74^{a}\pm0.04$	18.69	y = 0.019x + 2.9282	0.9774
	7	$19.29^{b}\pm0.50$	19.40		
	30	$20.01^{b}\pm0.72$	19.94		
0.5%OBG	1	$11.38^{a}\pm0.17$	11.08	y = 0.1031x + 2.4048	0.9114
	7	$12.71^{b}\pm0.16$	13.54		
	30	$16.31^{c}\pm0.15$	15.73		
0.25%YBG	1	$8.49^{a}\pm0.37$	8.54	y = 0.0343x + 2.1449	0.9579
	7	$9.26^{b}\pm0.12$	9.13		
	30	$9.52^{b}\pm0.16$	9.60		
0.5%YBG	1	$10.24^{a}\pm0.02$	10.19	y = 0.0227x + 2.3212	0.9322
	7	$10.52^{a}\pm0.49$	10.65		
	30	$11.08^{b}\pm0.20$	11.01		

Notes:
C – ice cream without stabilizing ingredients;
0.6%SS – ice cream with 0.5% of the Cremodan SI 320 stabilizing system;
0.25OBG – ice cream with 0.25% oat β-glucan;
0.5%OBG – ice cream with 0.5% oat β-glucan;
0.25%YBG – ice cream with 0.25% yeast β-glucan;
0.5%YBG – ice cream with 0.5% yeast β-glucan.
a-c-mean values denoted (according to storage time within the group) in the columns by different letters are statistically significantly different at $p \leq 0.05$.

The practical application of this model lies in its capability to predict changes in the textural properties of the product during extended storage, which is a critical factor for ensuring the stability, quality, and consumer attributes of ice cream. Additionally, the model can be utilized in further scientific research and in optimizing ice cream manufacturing technologies, taking into account the effects of various functional additives.

The analysis demonstrated that additives, particularly 0.5% oat and yeast β-glucans, effectively retard the growth of ice crystals, maintaining a fine-crystalline structure of the ice cream. This positively influences texture and quality throughout the entire storage period (Table 5).

Thus, the ice crystal growth model, developed based on empirical measurements, represents a reliable tool for evaluating and controlling ice cream stability and can be valuable for both researchers and food industry practitioners.

Figure 2 illustrates the linear dependencies of the changes of average ice crystal diameter during the storage of the studied samples. Both the actual experimental data (measured values) and the lines modeled using the power-law model are visualized, allowing for comparison between observed and predicted crystal growth over time. The high coefficients of determination (R^2) indicate a strong agreement between the model and the empirical observations.

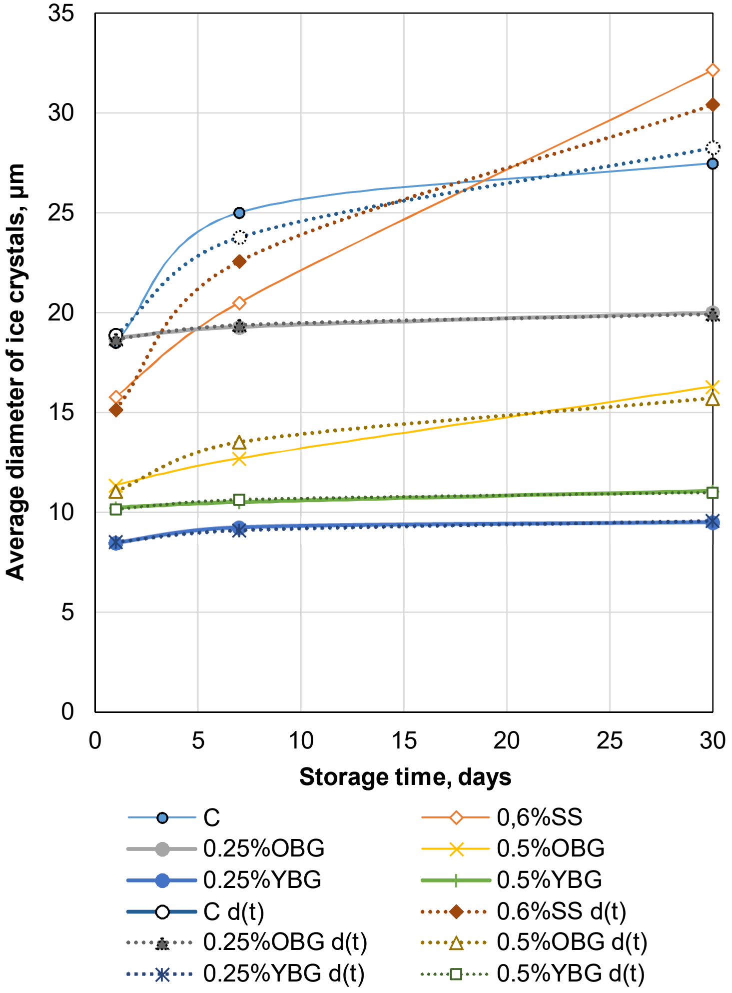


Figure 2. Ice crystal growth in ice cream during storage

Notes.

0.6%SS – ice cream with 0.5% of the Cremodan SI 320 stabilizing system;
0.25OBG – ice cream with 0.25% oat β-glucan;
0.5%OBG – ice cream with 0.5% oat β-glucan;
0.25%YBG – ice cream with 0.25% yeast β-glucan;
0.5%YBG – ice cream with 0.5% yeast β-glucan.
—— Solid lines – growth dynamics based on measured values,
······ Dashed line – modeled growth dynamics.

It is worth noting the specific behavior of yeast β-glucan, which results from a different mechanism of influence on the ice cream structure compared to oat β-glucan. Despite the increased cryoscopic temperature observed in samples with yeast β-glucan, this type of polysaccharide promotes the formation of the smallest ice crystals throughout the entire storage period. This indicates that yeast β-glucan exerts a structural rather than a colligative effect. It is likely that within the aqueous phase, it forms a spatial gel matrix that inhibits ice crystal growth and modulates their morphology (Zhang et al., 2019). Such behavior is characteristic of hydrocolloids and polymeric stabilizers and holds promise for industrial applications aimed at improving texture and reducing undesirable structural changes in ice cream during storage.

Conclusions

1. Oat β-glucan at a concentration of 0.5% in ice cream acts as an effective cryoprotector, lowering the cryoscopic temperature to –6.04 °C, which indicates enhanced water binding and the formation of a structured gel matrix. In contrast, samples with yeast β-glucan increase the cryoscopic temperature but provide better control over ice morphology. The addition of β-glucans leads to an increase in the fraction of frozen water during low-temperature processing, which can be explained by low-energy weak bonds between water molecules and β-glucan macromolecules.
2. Yeast β-glucan exhibits a different structuring mechanism in ice cream by forming a spatial gel matrix in the aqueous phase that inhibits ice crystal growth and modulates their size and shape. This is confirmed by the stabilization of the smallest ice crystal sizes (8.49–11.08 µm) in ice cream over 30 days of storage, despite a smaller decrease in cryoscopic temperature. Conversely, oat β-glucan acts through enhancing the cryoscopic effect by lowering the freezing temperature and influencing water phase transitions.
3. Analysis of melting enthalpy (ΔHf) showed that oat β-glucan reduces the heat of phase transition, facilitating ice crystal melting, whereas yeast β-glucan increases this energy, enhancing the stability of the ice phase.
4. The power-law model of ice crystal growth, developed based on experimental data, demonstrates high accuracy with a coefficient of determination $R^2 > 0.91$ and allows forecasting changes in the textural characteristics of ice cream during storage. This makes it an effective practical tool for optimizing production technologies and quality control of ice cream.
5. A promising direction for further research is a comparative analysis of the binding energies of different types of glucans and classical stabilizers with water to substantiate the mechanism of action of glucans under subzero temperature conditions.

Acknowledgments. The work was carried out within the framework of state-funded scientific research projects of the Problem Scientific and Research Laboratory of the National University of Food Technologies: "Development of technology for the reuse of secondary dairy resources for the production of new products and reduction of food waste generation" (state registration number – 0124U000965) and "Scientific justification and development of resource-efficient technologies for targeted food products as an imperative of Ukraine's food security" (registration number 0123U102060).

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Cite:

UFJ Style

Mykhalevych A., Polishchuk G., Buniowska-Olejnik M. (2025), Effect of stabilizers on the colligative properties of whey-based ice cream, *Ukrainian Food Journal*, 14(2), pp. 325–341, https://doi.org/10.24263/2304-974X-2025-14-2-10

APA Style

Mykhalevych, A., Polishchuk, G., & Buniowska-Olejnik, M. (2025). Effect of stabilizers on the colligative properties of whey-based ice cream. *Ukrainian Food Journal*, *14*(2), 325–341, https://doi.org/10.24263/2304-974X-2025-14-2-10

Discrete-pulse energy input for preretreatment of corn stalk in bioethanol production

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Keywords:

Bioethanol
Corn
Stalk
Lignin
Cellulose
Hemicellulose
Rotor-pulsating unit

Article history:

Received 20.11.2024
Received in revised form 14.04.2025
Accepted 30.06.2025

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DOI: 10.24263/2304-974X-2025-14-2-11

Abstract

Introduction. The aim of the work is to determine the effect of discrete-pulse energy input on the reactivity of lignocellulosic biomass, in particular corn stalks, during their preliminary processing for the production of bioethanol fuel.

Materials and methods. The corn stalks were crushed in a disintegrator to particles no larger than 500 micrometers, poured with a 1% NaOH solution in a ratio of 1:10 and sent for processing in a rotory-pulsating unit. The treatment in the rotary-pulsating unit was carried out in recirculation mode for 1–70 minutes at a temperature of 50 °C.

Results and discussion. To increase the reactivity of lignocellulosic biomass during enzymatic conversion, preliminary treatment is necessary, the main task of which is to remove lignin and disrupt the structure of cellulose fibers. With a processing time of 70 minutes and a flow shear rate from $20 \cdot 10^3$ to $60 \cdot 10^3$ s-1 at different pulsation frequencies, the reactivity (degree of conversion) of corn stalks increases to 61%. The main increase in reactivity from 10 to 61% occurs within 50 minutes at a shear rate of $60 \cdot 10^3$ s^{-1}. Further increase in treatment duration is ineffective. The amount of lignin in corn stalks without pretreatment was 15% of the dry matter content. During the 50-minute treatment at a pulsation frequency of 6 kHz and a flow shear rate of $60 \cdot 10^3$ s^{-1}, the content of lignin decreased from 15% to 1.3%. Increase in treatment time to 70 minutes at pulsation frequencies of 2 kHz, 4 kHz, and 6 kHz, the content of lignin decreased slightly and reached 1%. Therefore, increasing the treatment time from 50 to 70 minutes is impractical and leads to an increase in energy consumption by 10–15%. The results obtained are explained by the fact that with an increase in the rotor speed, there is an increase in the frequency of flow pulsations. When opening and closing the holes of the stator and rotor, sharp pressure fluctuations are observed, resulting in microcavitation, which leads to explosive autohydrolysis, while the average particle size of lignocellulosic biomass decreases and the total contact area of the phase increases, ensuring high reactivity.

Conclusion. The use of discrete-pulse energy supply and the proposed modes of preliminary processing of corn stalk biomass allows increasing its reactivity during enzymatic conversion to 61% compared to the known method of explosive autohydrolysis, which provides an increase in conversion in the presence of H_2SO_4 by 6–21%, and in the presence of $Ca(OH)_2$ by 25%.

Introduction

In recent years, there has been growing interest in the use of alternative fuels derived from plant biomass against the decline in crude oil production (Stabnikova et al., 2023a; Tran et al., 2019). Biofuels are considered a renewable energy source because they are produced from biomass, which can be replenished. One of the most common types of biofuel is bioethanol, which is produced from sugar- and starch-containing raw materials. A significant increase in bioethanol production, a reduction in its cost price and an increase in competitiveness are possible with the use of non-food raw materials, in particular lignocellulosic biomass, such as corn stalks (Shukla et al., 2023). The main components of lignocellulosic biomass are cellulose (23–50%), hemicellulose (12–29%), and lignin (13–31%) (Abolore et al., 2024; Tran et al., 2019).

Cellulose is a polysaccharide having a linear structure composed of d-glucose monosaccharides. The macromolecules of cellulose form fibrils. Hemicellulose has the form of a linear and branched heterogeneous polymer consisting of five saccharides – xylose, arabinose, mannose, galactose, and glucose. Lignin consists of phenylpropanoid alcohols, such as coniferyl, sinapyl, and coumaryl and contains different hydroxyl, methoxy, and carbonyl functional groups. The presence of lignin, due to its structure, significantly complicates the treatment of lignocellulosic biomass (Sheehan, 2001).

Lignin connects cellulose and hemicellulose, making the structure of lignocellulosic raw materials stronger and harder (Peciulyte et al., 2016). Lignocellulosic biomass is poorly suited for enzymatic degradation due to its low reactivity (Houfani et al., 2020). Cellulose is resistant to the action of enzymes due to its insolubility and strong structure, which is caused by its high crystallinity (Nitsos et al., 2019). Hemicelluloses in the form of lignocellulosic complexes have low reactivity (Karimi and Taherzadeh, 2016). Overall, the factors that contribute to the resistance of lignocellulosic biomass to enzyme action include a limited surface area available for enzyme interaction; a high degree of screening of cellulose by lignin; a high level of 'wrapping' of cellulose by hemicelluloses.

To increase the reactivity during the enzymatic conversion, pretreatment of lignocellulosic biomass is necessary to remove lignin and the breakdown of the structure of cellulose fibers. This is achieved by reducing the particle size of the raw material, increasing its porosity, and enhancing the surface area accessible to enzymatic action (Sun et al., 2014). Schematic of the breakage of the lignocellulosic material is shown in Figure 1 (adapted from Medeiros et al., 2023).

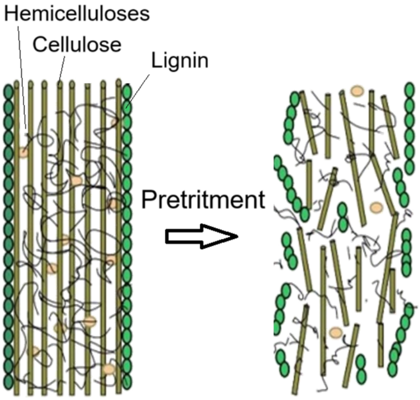

Figure 1. Schematic of the structure of lignocellulosic material and its breakage during pretreatment
(adapted from Medeiros et al., 2023)

Corn is the most widely produced grain crop in Ukraine, and its production in 2023/2024 amounted to 31 million metric tons (Statista, 2024). It was estimated that approximately one ton of corn stover is generated for every ton of corn grain (Momayez et al., 2019), meanwhile stalks make up the largest part (from 32.9 to 39.4% of dry biomass) of corn stover (Berchem et al., 2017).

The cellulose content in corn stalks is within 40–50%, hemicellulose 20–30% and lignin 10–15% (Liang et al., 2023).The issue of recycling agricultural waste and turning it into useful products is receiving increased attention in Ukraine (Stabnikova et al., 2023b), and corn stalks can be an excellent raw material for producing biofuels, in particular bioethanol.

There are several methods for the pretreatment of lignocellulosic biomass: grinding (Hideno et al., 2009; Paudel et al., 2017); radiation methods (Kapoor et al., 2017; Yang et al., 2008); ultrasound (Bak et al., 2009); microwave radiation (Velmurugan and Muthukumar, 2011); acids (Mishra and Ghosh, 2019; Stabnikova et al., 2010); organosolv (Ostovareh et al., 2015); alkalis (Bali et al., 2015); ionic liquids (Karimi and Taherzadeh, 2016); explosive autohydrolysis (Alvira et al., 2014; Gogate et al., 2011; Putra, 2018); hydrothermal treatment (Putra, 2018); carbon dioxide explosive decompression (Kim and Hong, 2001; Gu et al., 2013), and biological treatment using enzymes (Dai et al., 2017; Rabemanolontsoa and Saka, 2016).

Methods for obtaining pure components from lignocellulosic biomass by its thermal treatment without the use of chemical reagents are effective and environmentally attractive. The aim of the present study is to determine the influence of discrete-pulse energy input on the reactivity of lignocellulosic biomass, specifically corn stalks, during their pretreatment for bioethanol fuel production.

Materials and methods

Materials

Corn stalks were used in the research. Lignin content and reactivity were determined in the original corn stalks and pre-treated ones. In both cases, the corn stalks were originally ground in a disintegrator to particles no larger than 500 μm.

The reactivity was determined by hydrolysis of lignocellulosic biomass with a mixture of cellulase produced by *Penicillium verruculosum* B151 and cellobiase (β-glucosidase) produced by *P. verruculosum* F10 at 50 °C, pH 5.0 for 48 hours. The degree of conversion of lignocellulosic biomass into reducing saccharides because of enzymatic hydrolysis was taken as a criterion of reactivity.

Methods

Pretreatment of corn stalks

Corn stalks ground in the disintegrator were mixed with 0.5% or 1% NaOH solution in a ratio of 1:10, and then processed in a rotary-pulsation apparatus. The processing was carried out in the recirculation mode for 10 to 70 min at a temperature of 50 °C. During the processing, the flow pulsation frequency was changed from 2 to 6 kHz and the flow displacement rate was changed within the range from $20\cdot10^3$ to $60\cdot10^3$ s^{-1}. The flow pulsation frequency and the flow displacement rate were regulated by the rotation speed of the rotor of the rotary-pulsation apparatus and the size of the gap between the stator and the rotors.

Determination of lignin content. Two samples of liquid from the mixture, 50 ml each, were taken before the start of treatment and every 10 minutes of mixture treatment. Sulfuric acid was added to one sample until pH 2 was reached to precipitate lignin, separate it from other components and stabilize the system after alkaline hydrolysis. Then the resulting suspension was filtered through pre-weighed filter paper. The filter with the lignin sediment was dried and weighed on an Axis AN200 analytical balance with an accuracy of 0.0001 g. The mass of lignin was determined as the difference between the mass of the dried filter with sediment and the mass of the original filter.

Another sample was used to determine the total dry matter content of the solution according to the generally accepted method (drying the samples to a stable mass at a temperature of 103±2 °C (ISO 24196, 2022). The lignin content was determined in% as the ratio of the mass of the obtained lignin to the mass of dry matter in each of the studied samples.

The Laff-Schurl method was used to determine reducing sugars (Ibrahima et al., 2020).

Pretreatment of lignocellulosic raw materials. The pretreatment of lignocellulosic raw material for hydrolysis was conducted at a pilot plant shown in Figure 2, which operates on the principle of discrete-pulse energy input.

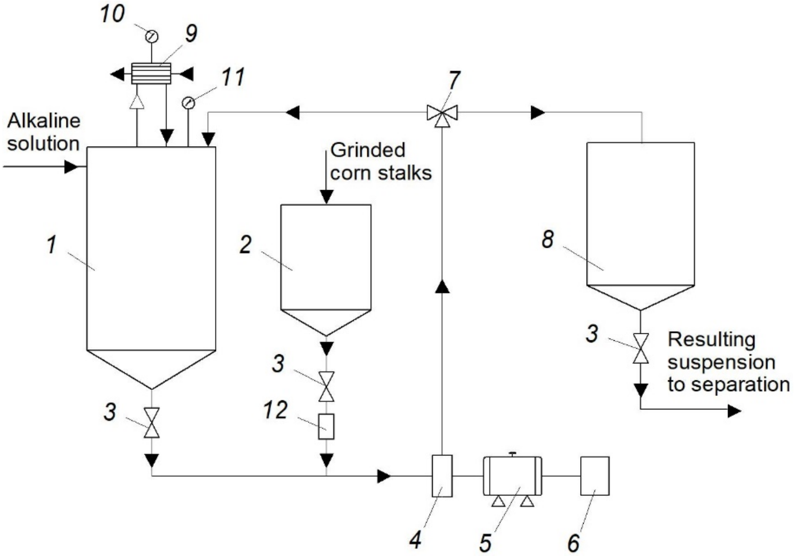

Figure 2. Schematic of pilot plant of discrete-pulse energy input:
1 – receiving tank; 2 – tank for raw materials; 3 – two-way valve; 4 – rotary-pulsation apparatus; 5 – electric motor; 6 – control unit; 7 – three-way valve; 8 – tank for pre-prepared raw materials; 9 – dephlegmator; 10 – pressure gauge; 11 – safety valve; 12 – vibratory batcher

The pilot plant (Figure 2) consists of a receiving tank (1); a tank for raw materials (corn stalks) (2); a rotary pulsation apparatus (4); an electric motor (5); a container for pre-prepared raw materials (8); water supply and power supply systems, shut-off and control valves (3, 7); power supply systems and control devices (10).

The receiving tank with a useful volume of 200 l is designed to store plant biomass during processing. The tank is equipped with a heat exchange jacket with a volume of 27 l to maintain the required process temperature during processing. The tank lid is equipped with a pressure gauge and a safety valve. The jacket is equipped with a 5-liter flow-through water heater, an open-loop circulation pump with a capacity of 8 l/min with a controller for adjusting the temperature of the coolant heating from 0 to 95 ºC. The heating element power is 2 kW.

The pilot plant operates as follows. The receiving tank (1) and rotary-pulsation device (4) are filled with alkaline solution. Corn stalks after mechanical grinding on the disintegrator are loaded into the container for raw materials (2), and the motor of the pump unit of the flow-through water heater is turned on.

After reaching the set temperature of the solution, the two-way valve (3) on the inlet pipe is opened and the vibratory batcher (12) is started. The alkaline suspension of grounded corn stalk biomass is circulated for the set processing time. When processing the lignocellulose complex, lignin and hemicellulose pass into solution. The resulting suspension is fed to a device for separating the solid phase (cellulose) from the liquid (lignin and hemicellulose) one. Phase separation can occur through filtration, ultrafiltration, membrane technologies, and separation.

Results and discussion

Compared to explosive autohydrolysis in the presence of acids and alkalis, the proposed method offers two key advantages: (a) it increases the reactivity without a significant rise in temperature (remaining below 50 °C), and (b) it intensifies the pretreatment process of corn stalks by generating uniformly distributed vapor–gas bubbles throughout the dispersed medium. Upon collapse, these bubbles convert stationary thermal, mechanical, or other energy forms within the system into powerful, discrete energy pulses in both time and space. The shock waves accompanying these phenomena, interphase turbulence, microcavitation, vortices, and penetrating cumulative microjets cause instabilities of the Rayleigh-Taylor or Kelvin-Helmholtz type at interphase surfaces, leading to intense fragmentation of disperse inclusions, significant increase in the total contact surface area of the phases, and enhancement of mass and heat transfer processes (Dolynskyi and Ivanytskyi, 2008; Dolinsky et al., 2012).

Results (Figures 3–6) were obtained in a pilot plant that enabled the production of a dispersed phase with an average particle size of lignocellulosic biomass ranging from 50 to 100 micrometers. The total surface area of contact between the phases of the dispersed system and the intensity of biomass conversion of corn stalks depend on the size of these particles. Pretreatment, after the disintegrator, of the original corn stalks using the method of discreet-pulse energy input through a rotor-pulsation apparatus for 50 minutes does not significantly reduce the size of the disperse phase particles. As a result, the reactivity does not increase, and the lignin content does not decrease.

Effect of treatment regimes on lignin content in corn stalks

At the first stage, the lignin content in corn stalks was determined depending on the operating parameters of their processing in a rotary-pulsation apparatus. The lignin content was determined depending on the duration of processing of corn stalks in a rotary pulsation apparatus of a heat and mass transfer unit at a pulsation frequency of 2 to 6 kHz for 0 to 70 min (Figure 3). The content of lignin in the original corn stalks was 15% of the dry matter (d.m.). With an increase in treatment duration to 70 minutes and a pulsation frequency ranging from 2 to 6 kHz, the lignin content decreased from 15% to 1.0%. During the treatment from 0 to 50 min. at a flow pulsation frequency of 6 kHz, the lignin concentration decreased from 15 to 1.3%. With an increase in the treatment duration to 70 min., the lignin concentration remained virtually unchanged and reached 1%. Finishing from 50 to 70 min. reduces the lignin content by only 0.3%, but leads to an increase in energy consumption by 10-15%.

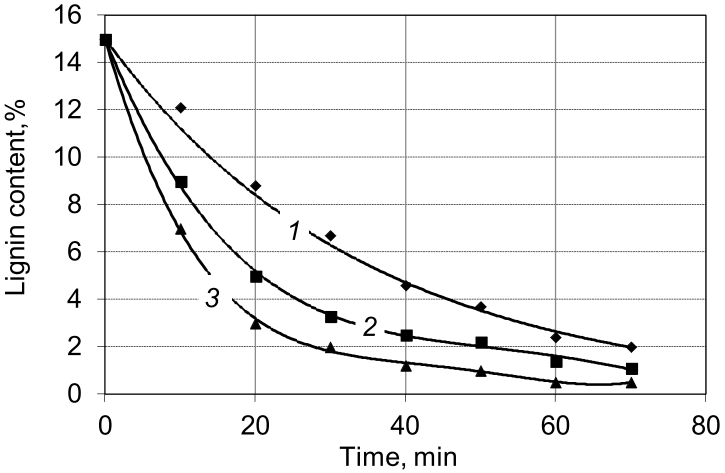

Figure 3. Effect of treatment duration and flow pulsation frequency on lignin content in corn stalks. Pulsation frequency:
1 – 2 kHz, 2 – 4 kHz, 3 – 6 kHz

Dependence of the lignin content in corn stalks from the duration of treatment and the flow rate in the rotary pulsation apparatus are shown in Figure 4.

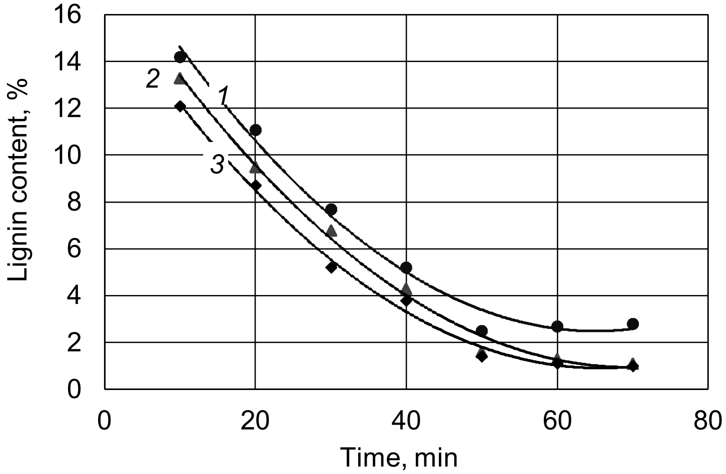

Figure 4. Effect of treatment duration and shear rate on lignin content in corn stalks. Shear rate: 1 – 20·10^3 s^{-1}; 2 – 40·10^3 s^{-1}; 60·10^3 s^{-1}

Increasing the treatment duration from 0 to 70 minutes and raising the shear rate from 20·10^3 to 60·10^3 s^{-1} leads to a reduction in lignin content from 15.0% to 1.0% from dry matter. The delignification process occurs most intensively over 50 minutes at a shear rate of 60·10^3 s^{-1}, with a reduction of 1.3%. Extending the process duration from 50 to 70 minutes allows for a further decrease in lignin content by 0.3%, but results in a 10-15% increase in energy consumption.

Effect of treatment regimes on reactivity (degree of conversion) of corn stalks

At the second stage, studies were conducted to determine the reactivity of corn stalks depending on the process parameters of their processing in a rotary pulsation apparatus (Figure 5).

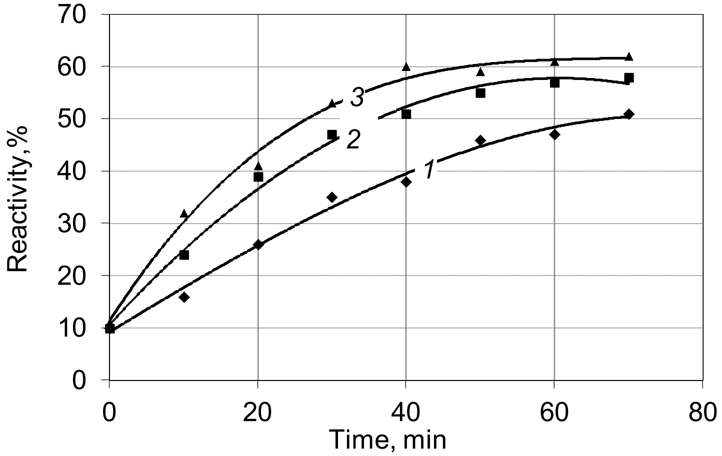

Figure 5. Effect of treatment duration and flow pulsation frequency on reactivity (degree of conversion) of corn stalks.
Pulsation frequency: 1 – 2 kHz, 2 – 4 kHz, 3 – 6 kHz

The highest reactivity of 63% is achieved with a pulsation frequency in the rotary pulsation apparatus of 6 kHz and a processing duration of 70 min. The main increase in reactivity from 10% to 61% is observed in the time interval from 0 to 50 min. A further increase in the processing time from 50 to 70 min has little effect on the reactivity of corn stalks. Under these conditions, the lignin content decreased from 15% to 1% of the dry matter content in corn stalks. At the same time, with a processing time of 50 min, the lignin content decreased to 1.3%. Therefore, processing of corn stalk biomass in the range from 50 to 70 min reduces the lignin content by only 0.3%, while the energy consumption for the process increases by 10-15%.

Dependence of corn stalk reactivity on the duration of processing and the shear rate of the flow in the rotary-pulsation apparatus is shown in Figure 6.

With an increase in the duration of treatment from 0 to 70 minutes and an increase in the shear rate from $20 \cdot 10^3$ to $60 \cdot 10^3$ s^{-1}, the reactivity reaches 61%. Among the investigated parameters, the most effective treatment of corn stalks in the rotor-pulsating apparatus is carried out for 50 minutes at a shear rate of $60 \cdot 10^3$ s^{-1}. Under these treatment conditions, the reactivity increases to 61%. An increase in the treatment duration from 50 to 70 minutes results in a 10 – 15% increase in energy costs, while the reactivity only increases by 2%, which is economically unfeasible.

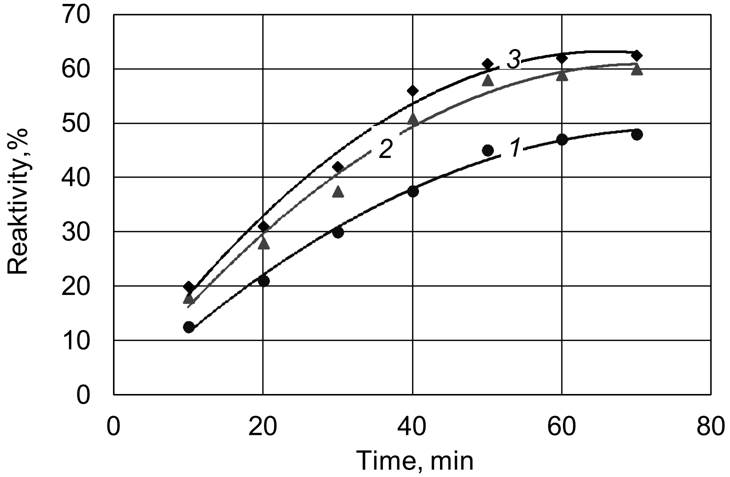

Figure 6. Effect of treatment duration and flow shear rate on the reactivity (degree of conversion) of corn stalks.
Shear rate: 1 – 20·10^3 s^{-1}; 2 – 40·10^3 s^{-1}; 60·10^3 s^{-1}

Dependence of the reactivity (degree of conversion) of corn stalks on the duration of processing in a rotary pulsation apparatus in the presence of NaOH solution (0.5% or 1.0%) at a pulsation frequency of 6 kHz is shown in Figure 7.

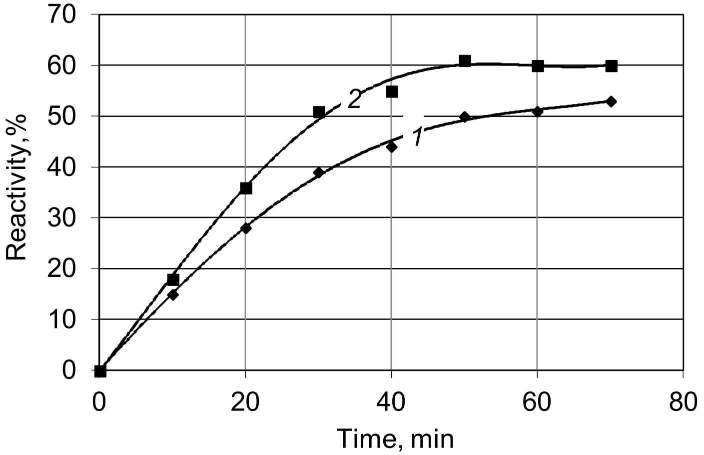

Figure 7. Effect of the duration of treatment and concentration of NaOH on the reactivity (conversion rate) of corn stalks.
1 – 0.5% NaOH; 2 – 1.0% NaOH.

Increasing the NaOH concentration to 1.0% with a treatment duration of 50 minutes leads to an increase in reactivity to 61%. Further increase in treatment duration does not lead in an increase in reactivity.

Comparison of reactivity (degree of conversion) of original corn stalks pretreated by various methods

The results of the researches on pretreated corn stalks using the method of discrete-pulse energy input in the presence of a 1% NaOH solution were compared with the results (according to literary sources) obtained from treatment through explosive autohydrolysis. The reactivity, i.e., the conversion degree of the original corn stalks after disintegration, is 10%, while those pretreated through explosive autohydrolysis in the presence of H_2SO_4 range from 40% to 55%, and in the presence of $Ca(OH)_2$ reach 36% (Chen et al., 2023; Shukla et al., 2023).

Comparative characteristics of the reactivity (conversion degree) of original corn stalks and those pretreated through various methods for 50 minutes at a temperature of 50 °C, at a pulsation frequency of 6 kHz and a shear rate of $60 \cdot 10^3$ s^{-1} are presented in Table 1.

Table 1

Reactivity (degree of conversion) of original corn stalks pretreated by various methods

Indicator	Original corn stalks ground in disintegrator	Pretreatment method		
		Explosive autohydrolysis in presence of H_2SO_4	Explosive autohydrolysis in presence of $Ca(OH)_2$	Discrete-pulse energy input method in presence of NaOH
Reactivity,%	10	40–55	36	61

The pretreatment of corn stalk biomass using the discrete-pulse energy input method in a rotor-pulsation apparatus under the specified conditions resulted in the highest reactivity, reaching 61%. This is 6–21% higher than that achieved through explosive autohydrolysis in the presence of H_2SO_4, and 25% higher than when using $Ca(OH)_2$. This is explained by the fact that as the rotor speed increases, the frequency of opening and closing of the stator and rotor holes increases, i.e., the frequency of flow pulsations, which is defined as the product of the angular speed of the rotor's rotation and the number of stator holes. During the opening and closing of the stator and rotor holes, sharp pressure drops occur, which means microcavitation, resulting in explosive autohydrolysis. The frequency of explosive autohydrolysis occurring in the rotor-pulsation apparatus essentially corresponds to the pulsation frequency (Dolinsky et al., 2012; Myronchuk et al., 2019).

Conclusions

1. The conversion of lignocellulosic biomass, particularly corn stalks, is not very productive without pretreatment, i.e., separation into cellulose, hemicellulose, and lignin. The most effective method currently available for the conversion of lignocellulosic biomass in bioethanol production technology is explosive autohydrolysis in the presence of acids or bases, which allows for a maximum increase in the reactivity of corn stalks from 10 to 55%.

2. The use of discrete-pulse energy input method in a rotor-pulsation apparatus at a temperature of 50 ºC and a treatment duration of 50 minutes enables an increase in reactivity to 61%. For this purpose, pre-ground biomass of corn stalks should be treated to a size of no more than 500 microns in a rotor-pulsation apparatus at a pulsation frequency of 6 kHz and a flow shear rate of $60 \cdot 10^3$ s^{-1}. Meanwhile, the lignin content in the corn stalk biomass is reduced from 15% (original) to 1.3%.
3. The developed equipment for discrete-pulse energy input and the proposed technological modes of treatment enable an increase in the reactivity (conversion degree) of corn stalk biomass compared to explosive autohydrolysis in the presence of H_2SO_4 by 6 to 21% and in the presence of $Ca(OH)_2$ by 25%.

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Cite:

UFJ Style

Myronchuk V., Obodovych O., Stepanova O., Gavva O., Volodin S., Tselen B. (2025), Discrete-pulse energy input method for preretreatment of corn stalk in bioethanol production, *Ukrainian Food Journal*, 14(2), pp. 342–353, https://doi.org/10.24263/2304-974X-2025-14-2-11

APA Style

Myronchuk, V., Obodovych, O., Stepanova, O., Gavva, O., Volodin, S., & Tselen, B. (2025). Discrete-pulse energy input method for preretreatment of corn stalk in bioethanol production. *Ukrainian Food Journal, 14*(2), 342–353. https://doi.org/10.24263/2304-974X-2025-14-2-11

Marketing communications by Ukrainian food enterprises to improve consumer attitudes towards food additives

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Abstract

Introduction. The aim of the study was to investigate the marketing communication activities of food market enterprises in respect to enhancing consumer attitudes towards food additives.

Materials and methods. The online survey was conducted using single-stage cluster sampling, covering 621 respondents. The questionnaire was developed based on validated scientific surveys and consisted of 33 questions.

Results and discussion. Technological education has a significant impact on the consumer perception of food additives. 67.5% of consumers with such education trust labels versus 33.3% without such education, which indicates a significant impact of technological education on consumer behavior and perceptions regarding food additives, emphasizing its role in increasing awareness and frequency of label reading.

Technological education has a significant impact on consumer perception of food additives. 67.5% of consumers with technological education trust labels, versus 33.3% of consumers without such education. Thus, having a technological education plays a significant role in increasing consumer awareness and the frequency with which they read product labels. A statistically significant relationship was shown between technological education of consumers and the frequency of reading labels, as well as trust in the information on them ($p<0.001$). Almost half of respondents (48.3%) believe that there is insufficient information about food additives on labels, with the main reasons being difficulties in understanding the nature of food additives (46.5%) and insufficient labeling (40.1%). Most consumers want to find more detailed and understandable information about food additives on labels: they expect to see the amount of food additives in the product (68.6%), the purpose of additive application (69.1%), recommendations for consumption (66.7%), and possible harmful effects (63.3%). To improve consumer attitudes towards food additives and enhance their trust in products, the study proposes an omnichannel approach to marketing communications, which includes the following key activities: (a) expanding the use of social networks and digital channels; (b) developing educational programs in partnership with specialized educational institutions; (c) improving information directly at points of sale by placing information stands in the entrance areas of supermarkets with brief explanations of the most common food additives; (d) introducing labels or QR codes on shelves near products with food additives; (e) adapting information for different consumer groups by using simple, and (f) understandable explanations for a general audience.

Conclusions. Increasing consumer knowledge of food additives will help to develop informed choices among them and strengthen trust in food producers.

Keywords:

Food additive
Marketing
Consumers
Attitude

Article history:

Received
20.12.2024
Received in revised form
17.03.2025
Accepted
30.06.2025

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DOI:
10.24263/2304-974X-2025-14-2-12

Introduction

The modern food market is undergoing significant transformations under the influence of changes in consumer preferences, increased public awareness and demands for the quality and safety of food products. One of the most controversial topics in the food industry is the use of food additives, which are often perceived by consumers as dangerous or undesirable components.

Given the rapid development of the food additives market, consumers are concerned about the long-term and short-term risks of their consumption (Bearth et al., 2014). Much of the negative consumer attitude towards food additives is due to low levels of knowledge about them (Kostynets et al., 2023; Mnerie et al., 2015), lack of reliable information and the spread of myths in the media (Lanytsia, 2011). Consumers often rely on unofficial sources, social networks and rumors, which create mistrust of food manufacturers.

At the same time, global experience shows that competent communication campaigns based on scientifically sound data, transparency and open dialogue with consumers can significantly improve the perception of food additives (Mnerie et al., 2016; Shim et al., 2011), make consumer choices more conscious and increase brand loyalty.

Growing competition in the food market requires enterprises not only to ensure high quality products, but also to effectively manage their reputation and consumer trust. Marketing communications play a crucial role in this process by informing consumers about product composition, explaining the functions of food additives, and emphasizing their safety and compliance with international standards. This issue is especially relevant in the context of the spread of the concept of healthy eating (Asioli et al., 2017) and environmental awareness of the population (Kumar et al., 2021), which forces companies to find optimal ways to communicate with the audience.

Thus, the study of consumer attitudes towards food additives is extremely relevant. It allows not only to understand the effectiveness of various methods of forming a positive image of products using effective marketing communication strategies, but also to develop recommendations for enterprises on transparent and trusting communication with consumers. These strategies will contribute not only to sales growth, but also to raising public awareness of food additives, which is important for the development of innovations in the food industry.

Studies conducted in the USA, Korea, UAE, China, the UK, and other countries have revealed generally low levels of consumer awareness regarding food additives (Kayışoğlu et al., 2016; Koyratty et al., 2014). Research has shown that awareness and attitudes toward food additives are closely linked to educational level (Grujić et al., 2013; Miao et al., 2016), that the percentage of consumers who read food labels remains low (Koyratty et al., 2014), and that there is a clear need for simple and accessible information on food additives (Al-Ghamdi et al., 2021). Additionally, some studies have highlighted consumer awareness of the potential relationship between food additives and obesity (Ingram, 2020). However, research on these issues in Ukraine is very limited.

The aim of the present study was to substantiate the marketing communication activities of enterprises in the Ukrainian food market to improve consumer attitudes towards food additives, taking into account their level of knowledge and attitude towards the use of food additives in products.

Materials and methods

An online survey using a probability sampling method (cluster single-stage sampling) was conducted in October-November 2023. Data were collected and processed from 621 completed respondent questionnaires. The demographic profile of the sample is presented in Table 1.

Table 1

Demographic profile

Characteristics	Absolute value	Percentage, %
Gender		
Men	171	27.5
Women	450	72.5
Age (years)		
14-24	399	64.3
25-44	174	28.0
45-60	45	7.2
> 60	3	0.5
Education		
Technological	351	57.0
Other	270	43.0

The general methodology and questions of the questionnaire were formed using the experience of surveys described in the scientific literature: a survey on consumer awareness and perception of the safety of food additives (Shim et al., 2011), consumer perception of food additives and the most important variables associated with consumer acceptance of food additives (Bearth et al., 2014), level of consumer knowledge about food additives (Kayışoğlu et al., 2016), consumer knowledge and attitudes towards the use and safety of food additives (Osaili et al., 2023). The questionnaire consisted of 33 questions, which were divided into 6 conditional blocks:

- demographic information of respondents (age, gender, education);
- sources of information about food additives and trust in food labels;
- assessment of consumer knowledge about food additives;
- assessment of consumer attitudes towards food additives;
- assessment of consumer behavior towards food additives
- needs and suggestions of consumers regarding food additive labeling.

The questionnaire was formed using Google-Forms, the survey results were processed using IBM SPSS Statistics software.

Results and discussion

Sources of information about food additives

The main sources of information for respondents were the Internet/social media (82.9%), information on labels (73.7%), and food specialists (41.7%). The least frequently used sources of information were newspapers, magazines (8%), and government agencies or official information (13.7%) (Figure 1).

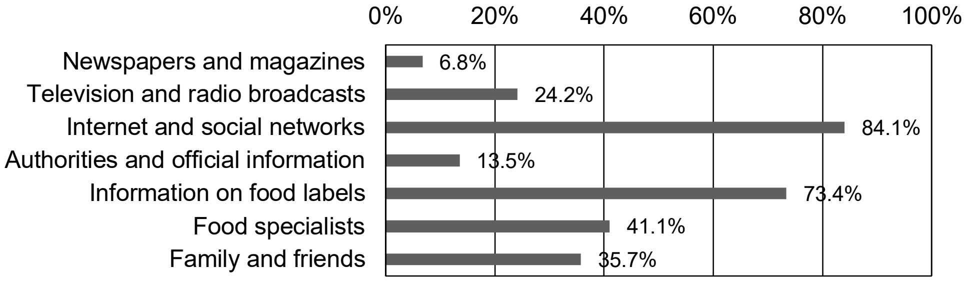

Figure 1. Sources of information about food additives (multiple answer, %)

Similar results regarding the source of information on food additives were reported in other studies (Osaili et al., 2023) – Internet/social media (69.3%), information on labels (63.4%). A study in the UK (Ismail et al., 2017) showed that the majority of respondents (56%) received information from radio and television programs, followed by the Internet (42%) and government information (36%).

As for trust in the information provided on the label, half of the respondents surveyed by the authors (52.7%) trust it, 11.6% of respondents do not trust this information, and another 35.7% of respondents are undecided. The presence of a significant proportion of respondents who do not have a clear position indicates the need to improve the transparency, accessibility and understandability of information on labels.

If this issue is considered in terms of respondent education, 67.5% of people with education trust the information on labels and only 33.3% without technological education trust such information. In the study of Osaili et al. (2023), the majority of respondents (46.2%) took a neutral position on trust in food labels, and only 34.9% of respondents trusted them sufficiently.

Media coverage of food additives, locations of food production, and local food regulatory inspection information all influence consumer trust in food labels (Coveney, 2008).

Assessing consumer knowledge and attitudes towards food additives

According to the knowledge of the respondents, the majority of them are aware of food additives such as malt (95.7%), rowan berries (95.2%), preservatives (91.8%), flavorings (91.3%). The least known food additives for consumers are anti-caking agents (29.0%), sodium benzoate (37.7%), SO_2 and sulfites (Figure 2).

Regarding consumer awareness of the use of individual food additives, the majority (71%) of respondents correctly answered that monosodium glutamate (MSG) is used to enhance the taste of food. This is higher than in a study conducted in Mauritius, where 44.4% of respondents knew that MSG is a flavor enhancer (Koyratty et al., 2014) and in a study (Osaili et al., 2023), where 33.8% knew this. 73.9% of respondents knew that gelatin gives food a gel-like consistency. In the study (Osaili et al., 2023), this percentage was higher – 80.5%.

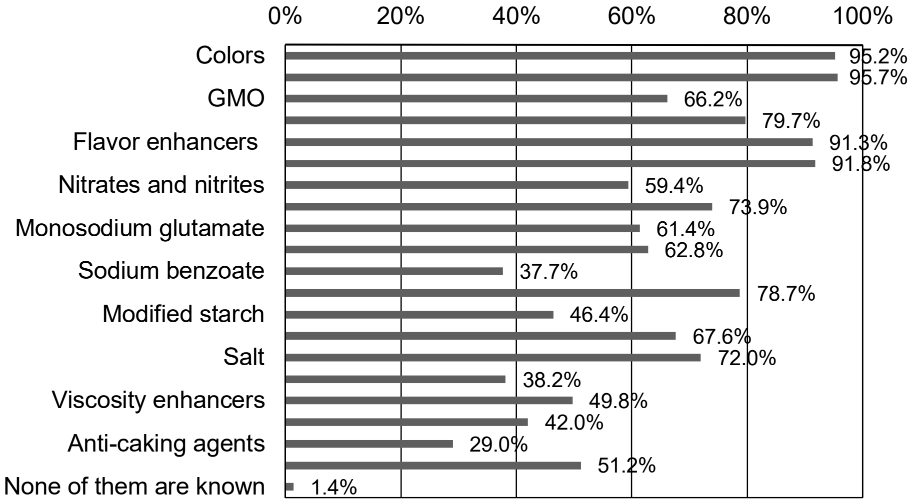

Figure 2. Respondent awareness of the presented types of food additives (multiple answer, %)

48.3% of respondents in the author's study knew of the effect of nitrates on food color. "In a comparable study by Osaili et al. (2023), only 25.7% of respondents were aware that nitrates are used as colorants in food products. The study also found that more than half of consumers knew that flour/semolina/oatmeal (90.3%), pickles (55.6%), nut butter (81.2%) and natural cheeses (77.8%) contained food additives (Figure 3).

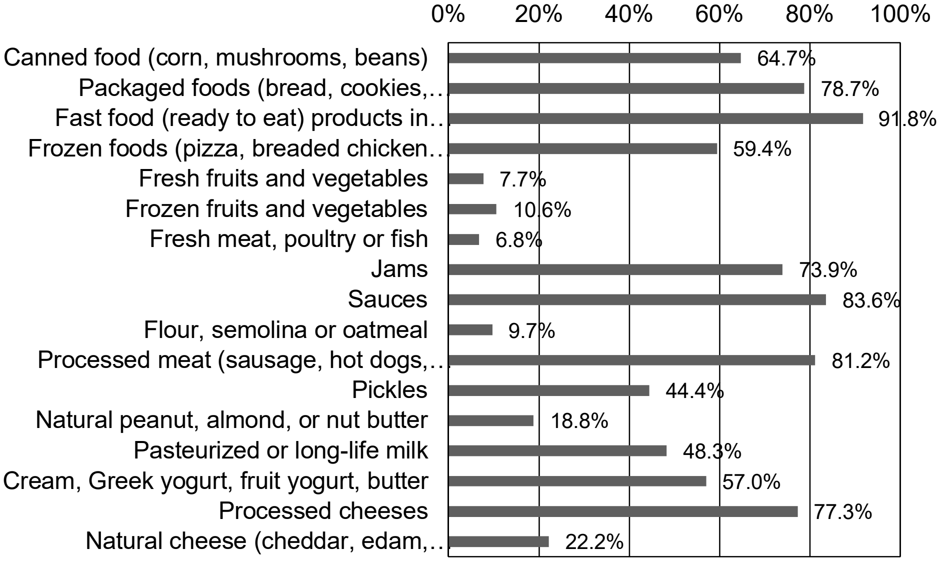

Figure 3. Consumer awareness of the content of food additives in certain food products (multiple answer, %)

However, a significant percentage of respondents knew that fresh meat/poultry/fish (93.2%), frozen fruits and vegetables (89.4%), and fresh fruits and vegetables (92.3%) did not contain food additives

According to the survey results, the majority of respondents (74.4%) agree with the statement that consuming processed foods containing preservatives is safe if it occurs within the permissible limits. This indicates a relatively high level of trust in the scientific and regulatory standards that determine the permissible amount of preservatives in food. At the same time, this trust is higher among respondents who have a technological education, in particular, 80.3% of such respondents agree with this statement, in contrast to 66.7% of respondents who do not have any other education. At the same time, 16.6% of respondents disagreed with this statement, which may be due to a skeptical attitude towards food additives in general, concerns about possible long-term health consequences, or the influence of negative information in the media. This indicates the existence of a group of consumers who avoid processed foods and prefer natural products. This indicates the existence of a group of consumers who avoid processed foods and prefer natural products. It is noteworthy that 9.7% of respondents selected the “don’t know” response. These respondents do not hold a clear opinion and can therefore be effectively targeted with marketing communications aimed at informing them about the safety of food additives.

Overall, the data obtained indicate a predominantly positive or neutral perception of preservatives in products, although there remain consumers who avoid them. This indicates the importance of marketing communication work by food market enterprises to explain the role of preservatives, their safety, and compliance with regulatory requirements.

An analysis of respondents' perceptions of the harmfulness of food additives shows that consumers have varying levels of concern about individual groups of food additives (Figure 4).

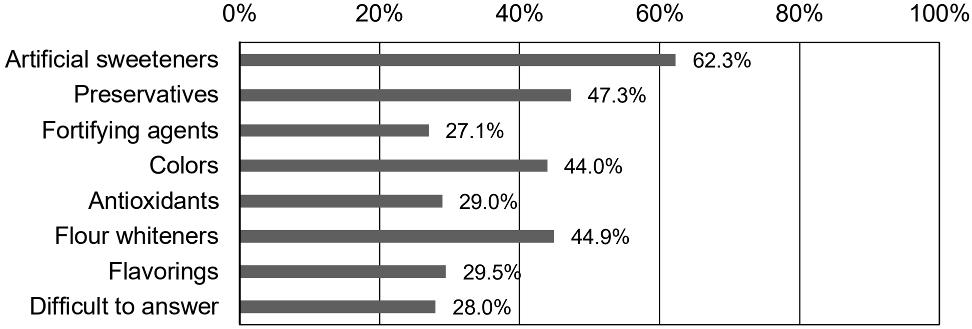

Figure 4. Respondents' opinion on the most harmful food additives for human health (multiple answer,%)

Artificial sweeteners caused the most negative perception (62.3%), which may be due to numerous discussions about their possible health effects, in particular regarding the increased risk of metabolic disorders, negative effects on intestinal microflora, and the possible carcinogenic effect of some substances.

However, it is worth noting that many studies do not confirm serious risks when artificial sweeteners are used within established norms. Preservatives were the second most concerned food additives (47.3%). This may be due to common stereotypes about their "chemical nature" and possible negative effects on the body, including allergic reactions and digestive disorders. Consumers are particularly concerned about synthetic preservatives used to extend the shelf life of products. Flour bleaches are at a slightly lower level (44.9%), which is an interesting result, as this group of additives is less discussed. Perhaps consumers associate them with over-processing of products and the loss of beneficial properties of natural ingredients. It is known that some artificial food colors have been banned in various countries, which may have affected consumer perceptions of this category of food additives. So, artificial colors are associated with possible allergic reactions, especially in children (44%). Meanwhile, flavors (29.5%) are of moderate concern. This may be because natural flavors are often perceived as harmless, and the negative attitude is mostly directed at synthetic analogues, which can mask the low quality of raw materials. To a lesser extent, respondents are concerned about firming agents (27.1%) and antioxidants (29%). This may reflect a lack of consumer awareness of their role in food products. Antioxidants are commonly associated with positive health effects, which may explain their lower position in the harmfulness rankings.

The survey results show that consumers are most concerned about food additives that are widely discussed in the media and associated with potential health risks. At the same time, the level of mistrust towards certain additives may be due to a lack of reliable information or the spread of myths. This highlights the need for increased public awareness and transparency from manufacturers on the composition and functions of food additives in products.

A survey conducted in Germany found that respondents believed that flavour enhancers (84%), sweeteners (69%), and colors (64%) were unhealthy (Southey, 2021). Another 60% of respondents expressed concerns about preservatives, and 43% about emulsifiers. Survey questionnaire, which was provided among 430 Seoul residents, Korea, also showed consumer concerns about safety of preservatives, colors, and artificial sweeteners in foods (Shim et al., 2011). Based on the surveys conducted, it was found that respondents agreed that food additives are vital for the food industry because they provide better appearance and pleasant taste (Grujić et al., 2013), are used for food preservation (Ismail et al., 2017), and extend the shelf life of processed foods
(Al-Ghamdi et al., 2021). At the same time, respondents were concerned about their negative impact on health (Eiser et al., 2002). Regarding the use of food additives, the nature of origin and the risk of disease, only 20.8% of respondents indicated that they have sufficient knowledge on the use of food additives.

Finding out the knowledge of the respondents of this study on the purpose of using food additives showed that most of them believed that the purpose of adding food additives is to extend shelf life (93.7%), improve the taste and aroma of food (97.1%), and increase nutritional value (40.1%), improved consistency and texture (84.1%) and appearance and color (91.3%) (Figure 5).

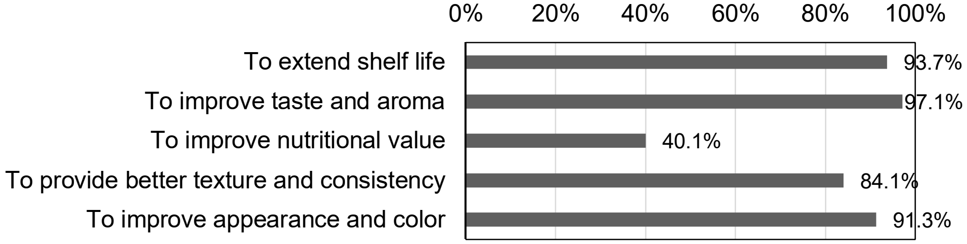

Figure 5. Respondents' opinion on the purpose of using food additives in food products (multiple answer, %)

An analysis of respondents' opinion on the purpose of using food additives shows that consumers have a clear understanding of the main functions of food additives in products. However, the level of acceptance of their use varies depending on the type of additive. Regarding improving taste and aroma, the largest part of respondents (97.1%) believe that food additives are used to improve the taste characteristics of products. This is quite an expected result, since taste and aroma are key factors influencing consumer choice. This includes flavor enhancers, flavors, and sweeteners, which are widely used in the manufacturing of beverages, confectionery, meat, and dairy products. The high level of agreement indicates that consumers have a good understanding of this function, although attitudes toward it can be both positive and negative.

The second most important function, according to respondents' opinion, is the function of extending shelf life (93.7%). This indicates that consumers understand that preservatives and antioxidants help prevent food spoilage, the development of microorganisms and oxidation. However, this aspect can also be a concern for consumers who have a negative perception of artificial preservatives, even if they are approved by regulatory authorities.

A large proportion of respondents (91.4%) recognized that additives are employed to improve the color and visual appeal of food products. Colors are widely used in the confectionery, meat and dairy industries, as well as in the production of beverages. At the same time, it is colors that are often perceived negatively due to their potential health effects, especially synthetic ones.

A slightly smaller, but still significant, number of respondents (84.1%) understand that food additives are also used to form the correct structure of products. Stabilizers, thickeners and emulsifiers play an important role in maintaining the uniformity and attractive appearance of many food products, including sauces, dairy products, ice cream and confectionery.

The smallest part of respondents (41.1%) know that the purpose of adding food additives is to increase the nutritional value of products. Such a low percentage of knowledge is because increasing the nutritional value of products is not the main purpose of using food additives.

Overall, the results of the survey shown in Figure 5, demonstrate that consumers are well aware of the functional role of food additives, especially in terms of improving taste, preservation of products, and their structure.

However, the relatively low understanding of the role of food additives in enhancing nutritional value suggests that additional information provided by manufacturers is needed. This could help change the perception of certain additives from negative to neutral or even positive, thereby increasing trust in food products.

In the study by Kayisoglu et al. (2016), 93.3% of respondents were informed that additives were added to increase shelf life, to improve taste and aroma (69%), and for appearance and color (63.7%). Similar results are shown in the study (Osaili et al., 2023), according to which 92.1% of respondents were reported that the purpose of adding food additives was to extend shelf life, 75.0% of respondents – to improve the taste and aroma of food, 23.5% – to increase nutritional value, 56.6% – to improve consistency and texture, 69.4% – to improve appearance and color. At the same time, it was shown that only 31.3% of respondents knew that food additives could be used to extend the shelf life of products. This indicates that knowledge about food additives varies among respondents from different countries, highlighting the need to inform the public about their role to support informed decision-making.

Most respondents (90.3%) believe that food-processing enterprises use various types of food additives during food production. Only 31.9% of respondents in this survey agreed that food processing enterprises follow the food safety standards set by the government. At the same time, the percentage of respondents with technological education is higher than that of respondents with other education (38.5% versus 23.3%).

In the survey presented, less than half (31.9%) of respondents were confident that legally permitted food additives are safe. Almost the same percentage of respondents (31.4%) considered legally permitted food additives unsafe. At the same time, respondents with technological education are more likely to consider additives dangerous (37.6%) compared to those without such education (23.3%), which indicates both greater awareness of potential risks and a more critical attitude towards additives permitted by law. It should also be noted that the percentage of respondents who found it difficult to answer this question was excessively high (36.7%), which also indicates a lack of consumer awareness.

The survey of 300 people in the Tekirdag province, Turkey, showed that 74% of respondents consider food additives unsafe (Kayışoğlu et al., 2016). Interestingly, the results of another study showed that there was a significant difference in consumer responses before and after a campaign on food additive safety (Shim et al., 2011). Before this awareness campaign, only 14% considered food additives safe within acceptable limits; however, after the campaign, this percentage increased to 74%. This demonstrates the need to inform consumers about the safety of food additives in order to increase consumer trust in the food industry.

The analysis of consumers' perceptions of the safety of food additives in the present study showed that only 9.7% of respondents believe that all food additives are harmful to health. This indicates that the vast majority of consumers are aware of the fact that not all food additives are dangerous and that their effect depends on the type, dosage, and purpose of use.

However, a significant part of respondents has some concerns about the potential health effects of food additives:

- allergic reactions (40.5%). The possible development of allergies due to the consumption of products with food additives is the greatest concern. This can be explained by the increasing prevalence of allergic diseases, especially among children, as well as reports of a possible connection between some colors, flavor enhancers and allergic reactions;
- cancer. About a third of respondents (31.5%) believe that food additives can cause cancer. This opinion was probably formed under the influence of media publications, where certain synthetic additives (for example, nitrites in meat products) are associated with an increased risk of developing cancer. Although scientific studies confirm that the permissible levels of such substances are safe, the public still tends to be afraid of their consumption;
- skin rashes and swelling (31.3%). The same percentage of respondents believe that food additives can cause skin problems. This is probably due to the fact that certain colors, preservatives and flavor enhancers can be triggers of allergic reactions such as hives or dermatitis;
- respiratory problems. The smallest percentage of respondents (7.6%) expressed concern about the possible impact of food additives on the respiratory system. This may be due to exposure to sulfites, which in rare cases can cause asthma exacerbations or other reactions in sensitive individuals.
- According to the German Federal Institute for Risk Assessment, surveyed respondents most often associate the harmfulness of food additives with problems such as intolerance (27%), allergic reactions (26%), poisoning (23%), diabetes (17%), and cardiovascular diseases (10%) (Southey, 2021).

- In addition to possible health risks, consumers also noted the main reasons that increase their distrust in the safety of food additives:
- lack of trust in food manufacturers.The most important factor is the low level of trust in manufacturers. About 59.5% of respondents doubt that companies honestly declare the composition of products and comply with all regulatory requirements. This may be due to a lack of transparent communication or insufficient openness of brands regarding the quality and origin of ingredients;
- lack of knowledge on the topic. 40.5% of respondents admitted that they lacked knowledge about food additives. This indicates the need for communication campaigns that would help consumers better understand which additives are safe, which have clear regulatory standards, and what role they play in food products;
- negative media reports. A quarter of respondents (26.8%) noted that their concerns about food additives are largely influenced by negative media reports. The media often focus on sensational headlines, which creates panic among consumers, even if real scientific evidence does not confirm such risks.

The results obtained indicate that the absolute majority of consumers do not consider all food additives harmful, but there are a number of serious concerns related to their possible health effects. The biggest concerns are allergic reactions, cancer risks and skin problems. In addition, low trust in manufacturers, lack of information and the influence of negative media reports are key factors contributing to mistrust of food additives even within safe limits. These points to the need for transparent communication by food industry companies, active consumer awareness and dissemination of scientifically based information on the functions and safety of food additives.

It should be noted that earlier studies of consumer attitudes have shown that negative messages in the media and social networks are an important factor in the formation of mistrust of food additives by consumers (Shim et al., 2011).

Evaluation of consumer preferences regarding food additives

In our study, 37.2% of respondents consumed food containing additives daily, and 39.1% consumed such products 2-4 times a week. In the study provided in Saudi Arabia, 28.7% of the 868 participants purchased processed foods two to three times a week (Al-Ghamdi et al., 2021). It was shown that among food products containing food additives, the highest frequency of consumption was found for table salt (77.3% per day), followed by dairy products (38.2% per day), and the lowest consumption was for processed meat (0.6% per day) (Osaili et al., 2023).

A study (Aoki et al., 2010) showed that taste-related information has a greater influence on consumer choice and determines the purchase of a product, even if there is information about the presence of food additives in it. At the same time, a study (Southey, 2021) showed that among 1,015 respondents surveyed, 55% try to avoid potentially harmful food additives. In contrast, 40% indicated that the presence of additives does not affect their decision when purchasing food.

Among the factors influencing the purchase of products with food additives, the main ones were identified as the composition of ingredients on the label (42%), the price of the product (24.6%), and the brand (manufacturer) (19.8%).

According to the survey, about 64% of respondents plan to check information on food labels in the future. More than half (73.9% of consumers) said they would use information on food additives if the store provided it. It is worth noting that the obtained data are consistent with a study conducted in Korea, where 73.7% of respondents would be grateful for information about food additives if it was provided by the seller (Shim et al., 2011). This indicates that consumers are willing to expand their knowledge about food additives and correct their misconceptions.

At the same time, less than half of the respondents (46.4%) reported that they read labels, 38.6% of respondents took a neutral position, and 15.0% of respondents do not read labels on food products. In particular, respondents with a technological education are significantly more likely to read labels (59.0%) compared to those without such education (30.0%). There is a statistically significant relationship between technology education and frequency of reading and trust in information on labels (p<0.001) (Table 2).

Table 2

Relationship between socio-demographic variables and trust in information on labels and their reading frequency (n = 207)

Characteristics	Level of consumer trust in information on food labels (1-5)			Level of reading information on labels (1-5)		
	Mean	**Standard deviation**	**P-value**	**Mean**	**Standard deviation**	**P-value**
Gender						
Men	3.74	0.892	<0.001	3.30	1.158	0.08
Women	3.39	0.923		3.54	0.958	
Age (years)						
14–24	3.56	0.871	<0.001	3.25	1.008	<0.001
25–44	3.38	0.982		3.91	0.936	
45–60	3.07	1.009		3.67	0.798	
> 60	5.0	-		5.0	-	
Education						
Technological	3.79	0.687	<0.001	3.76	0.985	<0.001
Other	3.08	1.037		3.10	0.945	

Age groups also show differences in behavior (p<0.001), in particular, the highest frequency of reading labels is observed in the 25–44 year old group, while respondents aged 14–24 years read labels the least. Regarding gender, the difference in label reading frequency between men and women is not statistically significant, although women show slightly higher scores. While standard deviations reflect moderate variability in scores, the lower values observed in the technology education groups indicate greater response consistency within these categories. This indicates that technological education and specialized knowledge of food additives significantly increase awareness and interest in the content of food labels. This finding is also consistent with the results of a study (Shim et al., 2011), which found that knowledge and understanding of food additive labels influences Korean consumers' label reading habits.

Therefore, an important aspect in consumer confidence in food products is the availability, clarity and completeness of the information provided on the labels. To determine the level of consumer satisfaction with this information, a question was asked whether they had enough information on food additives on the product labels.

The results of the study showed that almost half of respondents (48.3%) believe that there is insufficient information about food additives on labels.

The main reasons for this are:

- difficulty in understanding the nature of food additives (46.5%) – a large part of consumers do not have sufficient awareness of what certain additives mean, what functions they perform and how safe they are. This may be due to the use of complex terminology, scientific names or code designations (for example, “E-numbers”), which are not always understood by the general public;
- insufficient labeling, as 40.1% of respondents noted that labels lack detailed explanations of the functions of food additives, their origin and possible impact on health. Sometimes manufacturers provide only general designations without additional explanations, which can contribute to suspicion and mistrust on the part of consumers;
- indifference to the topic – some respondents (16.2%) noted that the issue of food additives does not interest them. This may indicate that for a certain segment of consumers, information campaigns on the composition of products are not a priority.

The results obtained are consistent with the research of other scientists (Osaili et al., 2023), in which 60.1% of respondents noted insufficient information on food labels. The main reasons for this were inadequate knowledge about food additives (46.5%) and difficulties in understanding the nature of food additives (41.6%). Another study conducted in Turkey found that only 13.3% of participants could easily understand information about food additives (Kayişoğlu et al., 2016). 66.3% of respondents reported that they had difficulty in understanding this information, and 20.3% of respondents could not understand food labels at all. These points to a global problem of complexity in the presentation of information about the composition of products.

In Korea, however, the results were even more striking: 76.8% of respondents admitted that they had difficulty in understanding the topic of food additives (Shim et al., 2011). This was due to both the labelling features and the insufficient level of educational work on food safety.

An analysis of sources of information about food additives showed that consumers receive information from different sources, but the most popular are social media (67.1%) (Figure 6)

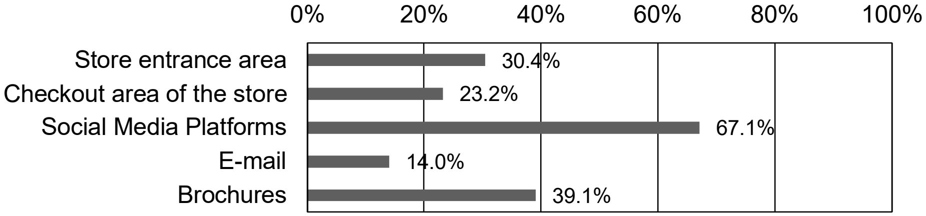

Figure 6. Priority methods for obtaining information about the existing food additives (multiple answer,%)

Similar results were obtained in the study of Osaili et al. (2023) where social media were recognized as the most preferred platforms for consumers to receive information about food additives (41.1%). This indicates the growing role of digital channels in disseminating knowledge about food products, in particular about their composition and safety.

In addition to social media, respondents also noted the following sources of information:

- brochures. Printed materials such as booklets and leaflets remain an important way of delivering information for 39.1% of respondents, especially for those who prefer traditional formats;
- information stands in the entrance area of stores – this channel is convenient for attracting the attention of 30.4% of buyers even before the start of the process of choosing goods;
- checkout area of stores – short information messages or advertising materials near the checkouts serve as an effective method of conveying key facts to 23.2% of consumers while waiting in line;
- emails – although this format is less popular compared to others, it remains relevant for those 14.9% of respondents who subscribe to newsletters from manufacturers or retail chains.

Consumer needs and suggestions regarding food additive labeling

Consumer suggestions regarding food additives include (a) clearer labeling (66.7%); (b) improvements in regulations and standards (54.6%); (c) more information and awareness (73.9%); and (d) decreased of their use (51.2%) (Figure 7).

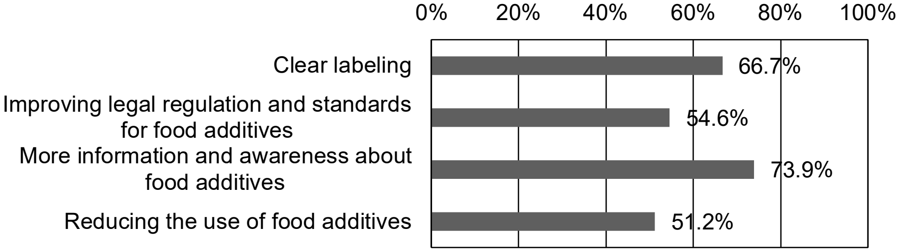

Figure 7. Consumer needs and suggestions regarding food additive labeling (multiple answer, %)

An analysis of consumer expectations for changes in the use of food additives and food labelling showed that consumers express specific desires for better information and regulation of the use of food additives in food products. The main aspects they would like to see changed include:

- clearer labeling (66.7%). More than two thirds of respondents believe that product labels should be designed in a more accessible and understandable form. This indicates the need to simplify terminology, explain the functions of food additives and improve the visual design of labeling;
- improvement of legal norms and standards (54.6%). More than half of the respondents say that it is necessary to strengthen control over the use of food additives through updating the legal framework, which indicates the importance of state regulation in this area;
- providing access to information about food additives (73.9%). Almost three quarters of respondents want to receive more reliable data about the composition of products and the influence of food additives on health, which indicates a significant demand for educational campaigns and initiatives from brands and regulatory bodies;
- reducing the use of food additives (51.2%). More than half of survey participants believe that manufacturers should limit the use of food additives in food products. This indicates a growing demand for natural and minimally processed products.

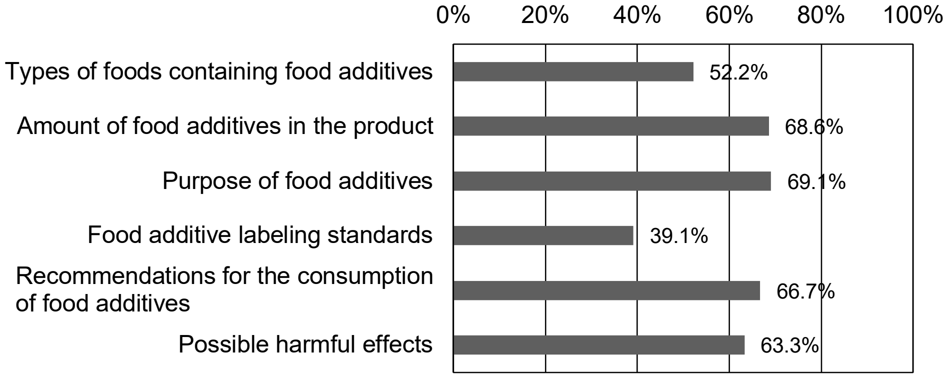

Figure 8. Consumer expectations regarding the information they would like to see on food labels (multiple answer, %)

The results of a survey on consumer suggestions for improving food-labeling show that the majority of consumers would like to receive more detailed and understandable information about food additives on product labels (Figure 8). Thus, respondents noted the following issues that, in their opinion, should be shown on the label (packaging):

- the amount of food additives in the product. 68.6% of respondents want to know the amount of added components, which will help them assess the level of processing of the product and its compliance with individual food preferences;
- the purpose of food additives. 69.1% of respondents want to understand the role of each additive plays in the product: preservative, flavor enhancer, coloring or thickener;
- recommendations for the consumption of food additives. 66.7% of respondents noted the need to provide information on acceptable consumption rates and conditions under which additives can be safe for health;
- possible harmful effects of using food additives. 63.3% of respondents want to see warnings about the potential risks of excessive consumption of certain additives, especially for vulnerable categories of the population (children, pregnant women, people with chronic diseases).

The survey showed that caution when purchasing food products with additives is caused by the following main factors:

1. Spreading myths and misinformation. The media, the Internet, and social media can be sources of myths and misinformation about food additives. The media and social media often spread information about the harms of food additives without scientific justification. Rumors and sensational news about "dangerous" E-additives can cause panic among consumers, even if these substances are actually permitted and safe in the prescribed doses. Various surveys have shown that people are concerned about the dangers of food additives and often find the "E" numbers mysterious (Ismail et al., 2017). The dishonest practices of some food manufacturers and the dissemination of false information have also led to consumers forming an erroneous opinion about food additives and their designation with the letter "E". When seeing such a mark on the packaging, some consumers refused to buy the product, associating it with information

about harm to health (Lanytsia, 2011). Therefore, such unreliable, incomplete information can lead to incorrect beliefs and decisions of consumers. 2.

2. Mistrust of food additive safety. Many consumers believe that food additives can have a negative impact on health. This is due to the spread of myths and lack of awareness about the functions and safety of various substances. Some additives (e.g. preservatives, colorants) are associated with allergic reactions, chronic diseases or carcinogenic effects, although their use is strictly regulated by international and national standards.
3. Confusing food labeling for consumers. Many consumers do not understand the meaning of complex chemical names or codes on labels of products containing food additives. This makes it difficult to choose healthy and safe products.
4. Lack of knowledge about food additives. Schools and other educational institutions do not provide enough information about food additives, their role in products, and their possible effects on health.
5. Effects of advertising and other marketing communications. Advertising may provide false information about the benefits of certain products containing food additives. This may lead to underestimation of the real benefits or possible risks.
6. Difficulty in understanding of product labeling and composition. Not all consumers possess sufficient knowledge to accurately interpret information on product labels. Designation of additives with codes (for example, "E621" instead of "monosodium glutamate") raises suspicions, although many such substances are safe and of natural origin. The lack of a detailed explanation on the packaging can increase the level of mistrust.
7. Naturalness and eco-friendliness in nutrition. The modern trend of healthy lifestyle and eco-friendly consumption makes people choose products with minimal processing and without artificial ingredients. Consumers tend to consider natural products more useful, even if some food additives (for example, antioxidants or probiotics) can have a positive effect on the body.
8. Consumer experience and personal beliefs. If a person or someone they know has had a negative experience with food additives (e.g. allergic reactions, digestive problems), they are more likely to avoid such products in the future. Cultural and religious beliefs also play an important role, as they may limit the use of certain additives.
9. Lack of transparent information from manufacturers. Trust in a product largely depends on the openness of the brand. If a company does not provide clear explanations for the use of certain additives in its products, the consumer may perceive this as concealment of important information and refuse to buy.

Thus, caution when purchasing products with food additives is a multifactorial phenomenon that combines emotional, informational and social aspects. To reduce bias, it is necessary to raise consumer awareness using effective marketing communication mix tools, ensure transparency in communication on the part of manufacturers and promote a scientifically based approach to assessing food additives.

Conclusions

1. A significant part of consumers lack accessible and understandable information about food additives. The main problems are complex terminology, insufficient detail of labelling and low awareness. In this regard, food manufacturers and relevant regulatory authorities are recommended to: (a) introduce more understandable labeling by including not only code designations (e.g., E-numbers) but also brief explanations of

the functions of each additive; (b) increase consumer awareness through educational campaigns and the publication of accessible explanatory materials; (c) ensure transparent communication by providing detailed information on the origin of food additives, their safety, and compliance with international standards. The implementation of these measures may help increase consumer confidence in food products and reduce their concerns about the use of food additives.

2. Based on the obtained results, the main areas of marketing communications are proposed to improve consumer awareness of food additives:
   - expanding the use of social networks and digital channels (creating short videos, infographics and interactive content to explain the functions of food additives; using popular platforms (Instagram, TikTok, Facebook, YouTube) to promote reliable information from food safety experts;
   - ollaboration with authoritative experts, including doctors, dietitians and nutritionists. Conducting open lectures, podcasts, webinars and participating in conferences helps increase consumer confidence in the information provided. Inviting experts to television and radio broadcasts allows to convey scientifically based knowledge to a wide audience;
   - development of educational programs in partnership with specialized educational institutions. Inclusion of the topic of food additives in courses on the basics of healthy eating or integration of information into educational programs helps to increase consumer awareness;
   - regular publication of articles on the official websites of companies specializing in the production or sale of food additives allows consumers to be systematically acquainted with scientific research, recommendations and new products;
   - participation in industry forums and discussion platforms. Interaction with the professional community and active presence in specialized online communities contributes to the dissemination of verified information among interested parties;
   - optimization of printed materials (preparation of brochures and booklets with simple, accessible and visually attractive information about food additives; adding QR codes to printed materials to redirect consumers to more detailed digital sources);
   - improving information directly at points of sale by placing information stands in the entrance areas of supermarkets with brief explanations regarding the most common food additives; introducing labels or QR codes on shelves near products with food additives that would provide instant access to information about their composition and safety;
   - adaptation of information to different consumer groups by using simple and understandable explanations for a general audience; preparing more detailed materials for those who want to delve deeper into the topic; providing information in multiple languages to reach a wider range of consumers;
   - integrating information campaigns into loyalty programs motivates consumers to take an interest in healthy eating. For example, providing bonus points or discounts for completing online educational courses or viewing expert materials helps to increase awareness of food additives;
   - collaborations with well-known healthy food brands. Joint advertising campaigns, integration of products into "healthy diet" packages and joint promotions help increase trust in food supplements among health-conscious consumers;

- using gamification, which can increase the level of audience interest. Developing mobile applications or interactive web platforms where users can take knowledge tests on food additives, receive personalized recommendations and bonuses for activity motivates people to learn more about this topic;
- cooperation with fitness centers, yoga studios and wellness communities. Partnership programs, seminars and placement of information materials in such establishments allow to convey useful information directly to people who are already interested in a healthy lifestyle;
- development of the "transparent brand" concept. Manufacturers can talk about the processes of development and testing of food additives, share quality certificates, conduct open production tours and interactive Q&A sessions with experts. This will form the openness of the brand and increase consumer trust;
- participation in charitable initiatives and social projects. Sponsorship of educational programs at specialized universities, support of public events on the topic of healthy nutrition and cooperation with health funds help to disseminate reliable information among a wide audience.

3. Marketing communications on food additives should be based not only on advertising activities, but also on deep interaction with the audience through educational initiatives, partnership projects and creation of a transparent brand image. A multi-channel approach to marketing communications, covering both personal interaction with consumers and digital and educational projects, is the key to effective information distribution on food additives and building trust in this product category.

   The study confirms that consumers receive information about food additives from a variety of sources, with digital channels playing an increasingly important role. However, traditional printed materials and point-of-sale information remain relevant. To increase public awareness and build trust in food products, it is necessary to implement an omnichannel approach to marketing communications that combines social media, printed materials, in-store information boards, digital technologies and other marketing communications tools. This will provide consumers with accessible, reliable and understandable information, which in turn will contribute to more informed food choices.
4. The findings indicate that consumers are seeking greater transparency in the use of food additives, improved product labeling, stricter regulation, and greater information about the health effects of food additives. At the same time, a significant proportion of respondents want to reduce the use of such ingredients in food products, indicating a growing trend towards natural and organic choices.

   Given consumer expectations, food brands, governments and other stakeholders can take the following steps to increase trust in food products:
   - raising consumer awareness (conducting educational campaigns on food additive safety through social media, webinars, printed materials and events in supermarkets; creating independent online platforms where consumers can get expert advice on food additives);
   - strengthening control over food additives (implementing stricter safety standards and regularly updating them in accordance with new scientific research; increasing transparency in production by mandating disclosure of information on the origin and functions of each additive).

   Changing approaches to labeling, regulation and communication on food additives can significantly affect the level of consumer trust in food brands. The food industry must adapt to modern society's demands, ensuring maximum transparency, accessibility of information and the transition to more natural ingredients in food production.

5. The results of syrvey indicate that the current level of food labeling does not meet consumer needs for accessible and comprehensive information about food additives. People want to know not only that a product contains certain additives, but also to understand why they are used, how much of them are in the composition, what are the safe consumption rates, and whether they can have a negative impact on health.
   This confirms the general trend towards conscious consumption, where consumers seek to make choices based on complete and reliable information. Food brands that adapt to these changes and provide more transparent data on labels can gain a competitive advantage and increase trust in their products. Therefore, in order to improve labeling and increase consumer awareness, it is recommended that when forming a communication mix:
   - use detailed labeling (introducing information about the quantity and purpose of each food additive in the product in a simple and understandable form; using infographics, color markings or symbols for quick perception of information);
   - inform about possible risks and provide recommendations for consumption (applying warnings on packaging about potential health risks (e.g. for people with allergies or certain diseases); providing recommendations for consumption for different ages);
   - use digital solutions for expanded access to information (adding QR codes on packaging that lead to pages with detailed information about food additives; creating official online platforms with verified data on food additives and their impact on health);
   - adapt information to a wide range of consumers (avoid complex chemical terms and technical language that make understanding difficult; use clear explanations about the functions of additives and their proven safety).

   Updating food labels to meet consumer needs will not only increase consumer awareness, but also strengthen trust in producers, promote more informed food choices, and reduce concerns about food additives. Transparency in this area is key to deserve the consumer loyalty and increasing the competitiveness of brands in the food sector.

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Cite:

UFJ Style

Boiko I., Bilyk O., Skryhun N., Kapinus L., Bilokhatniuk V. (2025), Marketing communications of enterprises of Ukrainian food market to improve consumer attitudes towards food additives, *Ukrainian Food Journal*, 14(2), pp. 354–374, https://doi.org/10.24263/2304-974X-2025-14-2-12

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1 автор	(Arych, 2019)
2 автора	(Kuievda and Bront, 2020)
3 і більше авторів	(Bazopol et al., 2022)

Приклад тексту із цитуванням: It is known (Arych, 2019; Bazopol et al., 2022), the product yield depends on temperature, but, there are some exceptions (Kuievda and Bront, 2020).
У цитуваннях необхідно вказувати джерело, звідки взято інформацію.
Список літератури сортується за алфавітом, літературні джерела не нумеруються.

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В Ukrainian Food Journal взято за основу загальноприйняте в світі спрощене оформлення списку літератури згідно стандарту Garvard. Всі елементи посилання розділяються **лише комами**.

1. Посилання на статтю:

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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, https://doi.org/5533.935-3.

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Deegan C. (2000), *Financial Accounting Theory*, McGraw-Hill Book Company, Sydney.

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Приклад:
Kochubei-Lytvynenko O., Kuzmyk U., Yushchenko N. (2022), Spices for dairy products, In: O. Paredes-López, O. Shevchenko, V. Stabnikov, V. Ivanov (Eds.), *Bioenhancement and Fortification of Foods for a Healthy Diet*, pp. 157–178, CRC Press, Boca Raton, https://doi.org/10.1201/9781003225287-11

4. Тези доповідей конференції:

Arych M. (2018), Insurance's impact on food safety and food security, *Resource and Energy Saving Technologies of Production and Packing of Food Products as the Main Fundamentals of Their Competitiveness: Proceedings of the 7th International Specialized Scientific and Practical Conference, September 13, 2018,* NUFT, Kyiv, pp. 52–57, https://doi.org/5533.935-3.

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Приклад:

Cheung T. (2011), *World's 50 most delicious drinks*, Available at: http://travel.cnn.com/explorations/drink/worlds–50-most-delicious-drinks–883542

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Наукове видання

Ukrainian Food Journal

Volume 14, Issue 2
2025

Том 14, № 2
2025

Підп. до друку 30.06.2025 р. Формат 70х100/16.
Обл.-вид. арк. 15.42. Ум. друк. арк. 15.54.
Гарнітура Times New Roman. Друк офсетний.
Наклад 100 прим. Вид. № 33н/24.

НУХТ. 01601 Київ–33, вул. Володимирська, 68

Свідоцтво про державну реєстрацію
друкованого засобу масової інформації
КВ 18964–7754Р
видане 26 березня 2012 року.