ISSN 2409–4951(Online)
ISSN 2310–1008 (Print)

# Ukrainian Journal of Food Science

***Volume 12, Issue 1***
***2024***

**Kyiv** 2024

**Ukrainian Journal of Food Science** publishes original research articles, short communications, review papers, news, and literature reviews.

**Topics coverage:**

| | |
|---|---|
| Food engineering | Food nanotechnologies |
| Food chemistry | Food processes |
| Biotechnology, microbiology | Economics and management |
| Physical property of food | Automation of food processes |
| Food quality and safety | Food packaging |

**Publication frequency – 2 issues per year** (June, December).

The research should be novel, have a clear connection to food science and be of general interest to the international scientific community.

Editors make every effort to ensure prompt and fair review, resulting in the timely publication of accepted manuscripts.

**Ukrainian Journal of Food Science is abstracted and indexed in the following databases:**

Directory of Open Access Journals (DOAJ) (2023)
EBSCO (2013)
Google Scholar (2013)
Index Copernicus (2014)
Directory of Open Access scholarly Resources (ROAD) (2014)
CAS Source Index (CASSI) (2016)
FSTA (Food Science and Technology Abstracts) (2018)

**Reviewing a Manuscript for Publication.** All scientific articles submitted for publication in "Ukrainian Journal of Food Science" are double-blind peer-reviewed by at least two reviewers appointed by the Editorial Board: one from the Editorial Board and one reviewer who is not affiliated to the Board and/or the Publisher.

**Copyright.** Authors submitting articles for publication are expected to provide an electronic statement confirming that their work is not an infringement of any existing copyright and will not indemnify the publisher against any breach of legislation and/or international standards in academic publishing. For ease of dissemination and to ensure proper policing of use, papers and contributions become the legal copyright of the publisher unless otherwise agreed.

**For a Complete Guide for Authors please visit our website:**
**http://ukrfoodscience.nuft.edu.ua**

**Editorial office address:**
National University of Food Technologies
Volodymyrska str., 68
Kyiv 01601
**Ukraine**

**E-mail**:
**Ukrfoodscience@meta.ua**

## Contents

# Influence of whey concentrates and carbohydrate ingredients on rheological and physicochemical parameters of reduced fat sour cream

**Galyna Polishchuk, Olena Mandiuk, Andriy Marynin, Oksana Kochubey-Lytvynenko, Roman Svyatnenko**

*National University of Food Technologies, Kyiv, Ukraine*

**Keywords:**

Sour cream
Whey concentrates
Rheology
Syneresis

**Article history:**

Received 18.02.2024
Received in revised form 21.05.2024
Accepted 28.06.2024

**Corresponding author:**

Galyna Polishchuk
E-mail: milknuft@i.ua

**DOI:**
10.24263/2310-1008-2024-12-1-3

## Abstract

**Introduction.** The objective of this research is to study the patterns of the rheological and physicochemical characteristics of reduced fat sour cream enriched with whey protein concentrates in the presence of mono- and polysaccharides.

**Materials and methods.** The study examined samples of 10% fat sour cream enriched with whey protein concentrate obtained by ultrafiltration, and hydrolyzed concentrate of demineralized whey in the presence of mono- and polysaccharides. Viscosity characteristics were determined using a rotational rheometer "Kinexus Pro+"; water activity was determined using an analyzer "HygroLab2".

**Results and discussion.** Whey protein concentrate and hydrolyzed concentrate of demineralized whey are technologically active ingredients that enhance the rheological characteristics of sour cream with a fat content of 10% and its capacity to retain moisture. Prolonged heat treatment of cream before fermentation at 95–98 °C for 3 h has a significant impact on the physicochemical and viscosity characteristics of 10% fat sour cream due to the thermal denaturation of whey proteins. The incorporation of monosaccharides in the hydrolyzed concentrate of demineralized whey, coupled with the introduction of a glucose-fructose syrup into reduced fat sour cream, has been observed to diminish its structuring and thixotropic potential, along with its water activity. The addition of 0.5% guar gum resulted in a notable enhancement in the rheological parameters, reaching their maximum values. The effective viscosity was observed to be 127.209 Pa·s at a shear rate gradient of 0.1 $s^{-1}$, while the degree of structure recovery during the reverse sweep reached 23.23%. The compatibility of oat β-glucan at a concentration of 0.5% in the presence of 1% whey protein concentrate in reduced fat sour cream was found to be incompatible, as evidenced by an abnormal deterioration of its rheological and physicochemical characteristics. This phenomenon may be attributed to the inhibition of the cream fermentation process at the selected concentration of the polysaccharide or the partial destruction of β-glucan during the homogenization of cream prior to fermentation, which results in the formation of a mobile layer of a colloidal solution of this polysaccharide between protein macromolecules.

**Conclusions.** The present study has established the regularities of changes in the structural-mechanical and physicochemical parameters of reduced fat sour cream enriched with whey proteins in the presence of monosaccharides and polysaccharides of different origin.

## Introduction

Sour cream is produced through the fermentation of cream with varying fat content. The physicochemical and rheological characteristics of sour cream differ significantly depending on the fat content, with notable changes in consistency. In particular, the consistency transitions from viscous and homogeneous to structured with a near complete loss of fluidity (Meunier-Goddik, 2012). A specific feature of the rheological characteristics of sour cream is that it exhibits a viscosity anomaly that depends on the velocity and temperature gradient (Seo, 2022). Reduced fat sour cream with a fat content of 10% differs from full-fat types in terms of its liquid consistency, high acidity, and satisfactory moisture retention (Odnorog et al., 2018). To enhance the physicochemical attributes of reduced fat sour cream, moisture-binding structuring ingredients derived from non-dairy sources, such as carrageenan, guar gum, modified starches, and cellulose derivatives, are frequently employed (Meunier-Goddik, 2012). Danylenko et al. (2020) proposed the addition of 0.01–0.03% dry banana puree to the sour cream composition as a mean of enhancing its rheological characteristics. However, the most appropriate solution is the incorporation of dairy ingredients, specifically whey protein concentrates, into natural reduced fat sour cream. The addition of protein concentrates can enhance the consistency and moisture retention capacity of fermented dairy products, while also increasing their biological value (Luck et al., 2013; Seo et al., 2023).

The role of protein in the formation of the structure of low-fat sour cream with a fat content of 10 to 15% is of primary importance. As reported by Narvhus et al. (2019), an acidic protein gel, comprising casein and denatured whey proteins, forms a structured matrix that incorporates fat globules. It was observed that as the fat content exceeds 20%, acid gels contain a reduced proportion of coagulated free protein. In fermented cream with a fat content of 35%, the structural network is comprised almost entirely of aggregated fat globules covered with protein. It can be reasonably deduced that milk protein in the concentrates will have a significant impact on the physicochemical and rheological characteristics of reduced fat sour cream.

Appliction of whey proteins in different food technologies is well known (Ivanov et al., 2021; Kochubei-Lytvynenko et al., 2022, 2023; Wang et al., 2024). The results of previous studies have demonstrated the feasibility of utilizing whey protein concentrate (WPC) and hydrolyzed concentrate of demineralized whey (HCDW) in sour cream with a fat content of 10% (Mykhalevych et al., 2022). Furthermore, no research has been conducted into the impact of different heat treatment methods applied to cream prior to fermentation. Additionally, the role of whey protein concentrates, including their potential combined use with mono- and polysaccharides, in influencing the formation of structural, mechanical, and physicochemical characteristics in reduced fat sour cream remains undetermined.

The aim of the study was to identify the patterns of formation of rheological and physicochemical characteristics of 10% fat sour cream enriched with whey protein concentrates in the presence of mono- and polysaccharides.

## Materials and methods

### Raw materials

Samples of sour cream with a fat content of 10% of the following composition were studied:

- control (sour cream without additives);
- sample 1 (baked sour cream);
- sample 2 - sour cream with hydrolyzed concentrate of demineralized whey (HCDW);
- sample 3 - sour cream with whey protein concentrate (WPC);
- sample 4 - sour cream with whey protein concentrate (WPC) and β-glucan;
- sample 5 - sour cream with whey protein concentrate (WPC) and glucose-fructose syrup (GFS);
- sample 6 - sour cream with whey protein concentrate (WPC) and guar gum.

The following is a description of the reasons to select the samples under study.

Sample 1 is of scientific interest with regard to the investigation of the impact of prolonged high-temperature cream processing on the viscosity characteristics of baked sour cream, which is distinguished by a creamy color and a pleasant flavor resulting from the Maillard reaction (Lohinova and Petrusha, 2024; Xiang et al., 2021).

Samples 2 and 3 were selected based on the findings of previous studies (Mykhalevych et al., 2022), which identified the most promising milk-protein concentrates (WPC and HCDW) for the enrichment of reduced fat sour cream. These concentrates were found to enhance the biological value of the product and improve its consumer characteristics. However, further in-depth study is required to fully understand these effects.

Samples 4–6 were created because protein-enriched reduced fat sour cream is also intended to be used in the production of structured dessert sour cream products. Therefore, it is advisable to study the combined effect of milk protein concentrates (for example, WPC), monosaccharides (glucose-fructose syrup) and polysaccharides (β-glucan and guar gum) on the structural, mechanical, and physicochemical properties of the samples.

**Samples preparation**

A control sample of sour cream with a fat content of 10% was obtained from normalized cream, which was prehomogenized at 10 MPa and a temperature of 60–65 °C using a laboratory homogenizer-disperser model 15M–8TA "Lab Homogenizer & Sub-Micron Disperser" (GAULIN CORPORATION, Massachusetts, USA), pasteurized at 85–90 °C for 5 min and cooled to an inoculation temperature of 28–32 °C. An activated starter preparation (TM "Vivo", Ukraine) containing *Lactococcus lactis, Lactococcus cremoris, Lactococcus diacetylactis,* and *Streptococcus thermophilus* was used for cream incubation.

The duration of fermentation was 7–8 hours to pH 4.6. The fermented samples were cooled to 4±2 °C, matured for 24 hours, and stored at the same temperature for 5 days.

To prepare the experimental sour cream, the protein concentrates and carbohydrate-containing ingredients were pre-hydrated or dissolved in cream at a ratio of 1:10 in cream heated to 40 °C and then added to the remaining cream with subsequent stirring. The resulting mixes were subjected to mechanical and heat treatment as described for the control sample.

In order to obtain baked sour cream, the cream was preliminarily subjected to a prolonged heat treatment (180 min at 96±1 °C) to produce a creamy brown color, a characteristic nutty taste and a pronounced aroma in the sample (Mandiuk et al., 2024).

The following milk protein ingredients were selected:

– whey protein concentrate obtained by ultrafiltration (WPC) (composition: solids - 94%, protein - 80%, carbohydrates - 7%, fat - 7%);
– hydrolyzed concentrate of demineralized whey (HCDW) with a solids content of 40% (degree of lactose hydrolysis not less than 85%). It was obtained through the reconstitution in water of demineralized whey powder with a demineralization level of 90% (ash - no more than 2.5%, lactose - no less than 79%, and protein content - no less

than 10.7% in terms of solids), in accordance with the developed technology (Osmak et al., 2021).

For the hydrolysis of lactose in the 40% whey concentrate, the following preparations were employed:

- a liquid preparation of β-D-galactosidase-hydrolase "GODO-YNL2" (Danisco, Denmark) with a recommended dosage of 100 g of the preparation per 100 liters;
- the single-strain lyophilized starter "*L. acidophilus* LYO 50 DCU-S" (Danisco, Denmark) at a recommended dosage of 5 g of the preparation per 100 liters of milk.

At a specified degree of lactose hydrolysis (at least 85%), the demineralized whey concentrate contains approximately 26.2% monosaccharides, 4.6% lactose, and 10.7% protein. Therefore, together with 30% of the concentrate, 7.86% of the monosaccharides, 1.38% of the lactose and 3.21% of the protein are added to the sour cream.

The contents of WPC and hydrolyzed whey concentrate in the samples were 1.0% and 30.0%, respectively, according to the recommendations of Mykhalevych et al. (2022), resulting in a total protein content of 3.38% and 3.22% (the control sample of sour cream had a protein content of 2.6%).

The following natural monosaccharides and polysaccharides were utilized: β-glucan extracted from oats (Shanxi, China,) and guar gum (Neelkanth Polymers, India) in the amount of 0.5%, as well as glucose-fructose syrup GFS-42 (Intercorn Corn Processing Industry, Ukraine) in the amount of 12.1%. The selected con tent of GFS-42 (at least 65% monosaccharides) contributes at least 7.8% of monosaccharides to the sour cream, which is comparable to the amount of monosaccharides in the hydrolyzed whey concentrate added to the sour cream.

Polysaccharide concentration was chosen according to generally accepted recommendations (Seo, 2022).

**Experimental procedure**

The effective viscosity and water activity were determined in the prepared samples of sour cream of different chemical composition after aging for 24 h at 4±2 °C. The degree of syneresis was determined within 5 days.

**Research method**

The acidity of the fermented sour cream samples was determined by the potentiometric method using a laboratory analyzer pH/MV/ISE/Temp ADWA AD1200 ATC.

Water activity (Aw) in sour cream samples was determined using a water activity analyzer "HygroLab2" (Rotronic, Switzerland) at 20 °C. The instrument was pre-calibrated with a special humidity standard (95% HR). Water activity, with a measurement accuracy of ± 0.001 Aw units, was expressed as values from 0.00 to 1.00 Aw (0–100% rh).

The viscosity of sour cream samples was measured using a rotational rheometer Kinexus Pro+ (Malvern Instruments Ltd, United Kingdom). The upper geometry C25 DIN L0142 SS (cylinder) and the lower geometry PC25 DIN C0350 AL were chosen for the study. Before measuring the rheological properties, the sour cream samples were heated to the temperature of product consumption (10 °C) and gently mixed for 30 s. The samples were placed in a cylinder, the upper geometry was lowered, they were kept for 5 minutes and viscosity curves were determined for forward and reverse sweep at changes in shear rate from 0.1 to 400 $s^{-1}$ and from 400 to 0.1 $s^{-1}$. The degree of recovery of the structure of the sour cream samples was calculated as a percentage of the effective viscosity at the end of the measurement at a shear rate gradient of $\gamma = 0.1$ $s^{-1}$ (reverse sweep), taking the effective

viscosity of the practically intact structure at the beginning of the measurement ($\gamma = 0.1\ s^{-1}$) as 100%.

The syneresis of sour cream clots was determined by centrifuging calibrated tubes containing mixed samples of 25 $cm^3$ for 20 min at 1000 rpm and 20 °C using a laboratory centrifuge Sigma 2-6E (Germany). The volume of whey separated was expressed in $cm^3$ per 100 g of product (Polischuk et al., 2020). Samples were analyzed after ripening and on 1st, 3rd and 5th days of storage.

### Statistical processing

All results with 3–5 times replication were statistically processed using the software Statistika 10. Diagrams were created in Microsoft Excel 2016.

## Results and discussion

### Study of the viscosity characteristics of sour cream

The effective viscosity of sour cream samples was measured in the range of changes in the shear rate gradient during the forward stroke, from the minimum value ($\gamma = 0.1\ s^{-1}$) to the maximum value ($\gamma = 400\ s^{-1}$). The samples were held under these conditions until the steady-state viscosity of the maximally destroyed structure was reached. Subsequently, the viscosity of the samples was measured during the reverse stroke, wherein the shear rate gradient was reduced from maximum to minimum values.

The effective viscosity of the samples in the most dynamic range of changes in the shear rate gradient ($\gamma = 0.1–10\ s^{-1}$), as well as at the maximum ($\gamma = 400\ s^{-1}$) and minimum shear rates ($\gamma = 0.1\ s^{-1}$) at the end of the measurement are presented in Table 1.

**Table 1**

**Effective viscosity of sour cream samples at a variable shear rate gradient (Pa·s)**

| Samples | Shear rate gradient, $\gamma$, $s^{-1}$ | | | | |
|---|---|---|---|---|---|
| | **0.1 (forward sweep)** | **1 (forward sweep)** | **10 (forward sweep)** | **400** | **0.1 (reverse sweep)** |
| **Control** | 89.981 ±2.924 | 5.609 ±0.133 | 2.402 ±0.102 | 0.117 ±0.001 | 16.940 ±1.503 |
| **Sample 1** | 99.121 ±3.031 | 13.420 ±0.429 | 3.461 ±0.141 | 0.119 ±0.001 | 13.332 ±1.111 |
| **Sample 2** | 115.882 ±3.791 | 11.053 ±0.371 | 3.496 ±0.152 | 0.122 ±0.001 | 23.202 ±1.395 |
| **Sample 3** | 119.990 ±4.015 | 15.372 ±0.411 | 3.639 ±0.182 | 0.151 ±0.001 | 25.905 ±1.582 |
| **Sample 4** | 87.680 ±2.684 | 9.501 ±0.350 | 3.845 ±0.148 | 0.176 ±0.001 | 7.201 ±0.214 |
| **Sample 5** | 105.050 ±3.721 | 8.703 ±0.262 | 1.907 ±0.100 | 0.076 ±0.001 | 13.600 ±0.831 |
| **Sample 6** | 127.209 ±3.932 | 20.336 ±0.81 | 4.033 ±0.182 | 0.183 ±0.005 | 29.548 ±1.460 |

The data indicate that all samples of sour cream exhibited the most significant structural destruction within the range of shear rate changes from 0.1 to 1 $s^{-1}$, with moderate destruction observed within the range from 1 to 10 $s^{-1}$. The highest viscosity was observed for sample 6, which contained a combination of whey protein concentrate (WPC) and guar gum. As anticipated, the milk protein concentrates in sour cream (samples 2 and 3) demonstrated comparable structuring capabilities due to the enhanced coagulation activity of fermented mixtures enriched with whey proteins, reaching a total protein content of 3.22% (sample 2) and 3.38% (sample 3), respectively. This observed effect correlates with the data presented by Sturaro et al. (2014).

The prolonged high-temperature treatment of cream (sample 1) due to increased thermal denaturation of whey proteins resulted in an increase in the viscosity of the sour cream compared to the control sample. This phenomenon can be attributed to the covalent interaction of denatured whey proteins with κ-casein on the surface of protein casein micelles. Denatured whey proteins serve as a binder between casein micelles, thereby facilitating the more efficient formation of an acidic protein gel (Lucey, 2016). It is established that fresh fermented milk products made from milk thermally processed at high temperatures are more viscous and have a lower tendency to release whey (Timothy, 2021). These characteristics were confirmed in the present study. However, the experimental confirmation of the latter trait remains to be conducted.

The distinctive characteristics of the joint structuring effect of whey protein concentrate and mono- and polysaccharides in sour cream warrant a separate discussion. To conduct a comparative analysis of the efficacy of their combined use, the dynamics of changes in sour cream viscosity during rheometric measurement at a variable shear rate were investigated using the example of control and test samples 3–6, as illustrated in Figure 1.

Monosaccharides and polysaccharides exert a markedly disparate influence on the viscosity characteristics of sour cream. Therefore, monosaccharides present in the composition of glucose-fructose syrup exert a slight reduction in the effective viscosity of an almost intact acidic milk-protein gel at a shear rate gradient of 0.1 $s^{-1}$. An increase in the destructive force of the shear rate to 1 and 10 $s^{-1}$ demonstrates a notable weakening of intermolecular forces, with the degree of destruction increasing by 1.17 and 1.9 times, respectively. The effective viscosity of the practically destroyed structure at a shear rate of 400 $s^{-1}$ is 0.151 (Pa·s) for the sample with WPC, and only 0.076 (Pa·s) for the sample with whey protein concentrate and glucose-fructose syrup. The observed effect can be explained by the presence of a layer of mobile monosaccharide molecules between the protein micelles, which probably reduces the strength of intermolecular bonds in the protein clot. A comparable effect was observed when comparing sample 1, which contains monosaccharides in hydrolyzed whey concentrate, with sample 2, which contains whey protein concentrate (WPC). However, the effect was less pronounced in the latter sample. The detrimental impact of monosaccharides is mitigated by the incorporation of whey proteins in the hydrolyzed whey concentrate into the sour cream sample.

In contrast to monosaccharides, the presence of polysaccharides, in particular guar gum, has a notable impact on the viscosity characteristics of sour cream. In this instance, guar gum, a high-molecular-weight compound, serves to reinforce the total intermolecular bonds within the protein gel, thereby forming a more stable structure over time at a variable shear rate. The structuring effect of guar gum has also been observed in sour cream (Mudgil et al., 2014), yogurt (Lee et al., 2016), and ice cream (Javidi et al., 2016).

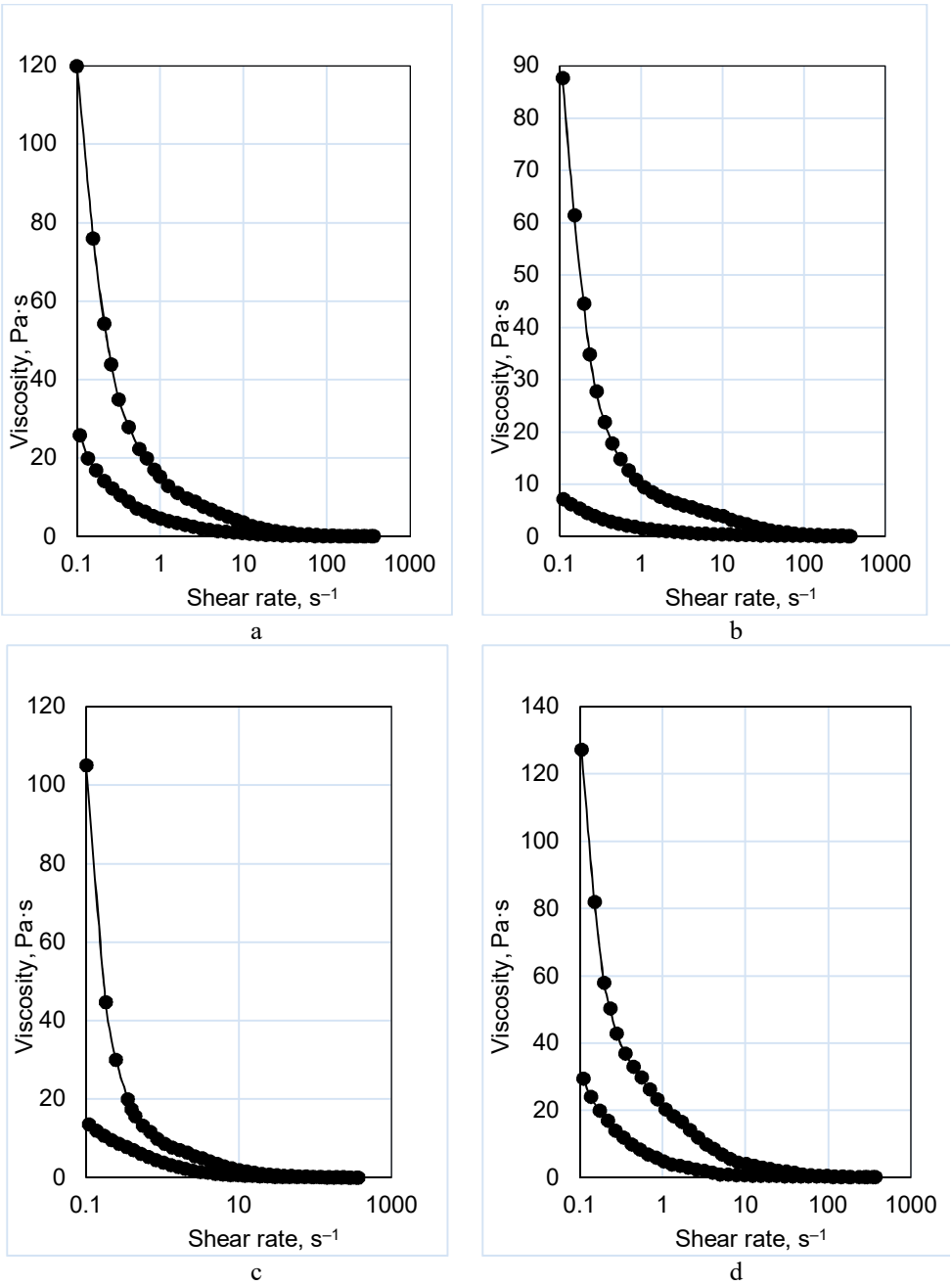


**Figure 1. Changes in the effective viscosity of sour cream samples during rheometric measurement:**

a – sample 3 (sour cream with whey protein concentrate;
b – sample 4 (sour cream with whey protein concentrate and β-glucan);
c – sample 5 (sour cream with whey protein concentrate and glucose-fructose syrup;
d – sample 6 (sour cream with whey protein concentrate and guar gum)

Despite the well-known functional and technological properties of β-glucans, in particular, their ability to form and stabilize the foamy structure of ice cream, prevent the separation of free moisture in the production of cheese, and mimic the presence of fat in low-fat products (Mykhalevych et al., 2022), this polysaccharide was found to be incompatible with sour cream as a structuring ingredient. The addition of β-glucan to sour cream, even in the presence of whey protein concentrate, resulted in the lowest effective viscosity values among all samples. This anomalous effect can be attributed to the distinctive physicochemical characteristics of this polysaccharide, the inhibition of the cream fermentation process, and the potential disruption of its macromolecules during the homogenization of cream mixtures prior to fermentation. A comparable outcome was documented by Zagorska et al. (2013) in the context of chitooligosaccharide. Further studies are required to investigate this phenomenon in greater depth.

Table 1 presents the degree of structure recovery observed in the process of reducing the destructive force during the reverse measurement when the shear rate was changed from 400 $s^{-1}$ to 0.1 $s^{-1}$ for all sour cream samples. The calculated results are illustrated in Figure 2.

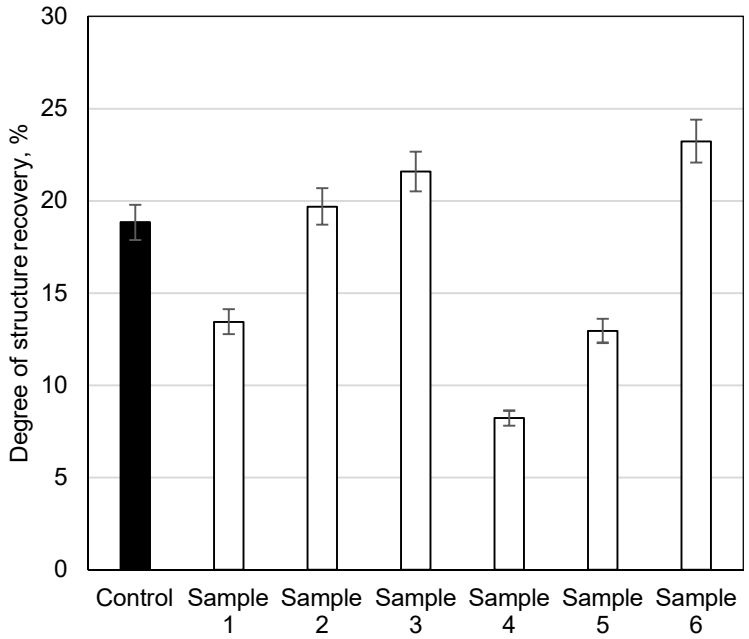


**Figure 2. Degree of structure recovery of sour cream samples with 10% fat content and different chemical composition**

Control (sour cream without additives);
Sample 1 (baked sour cream);
Sample 2 - sour cream with hydrolyzed concentrate of demineralized whey (hcdw);
Sample 3 - sour cream with whey protein concentrate (wpc);
Sample 4 - sour cream with whey protein concentrate (wpc) and β-glucan;
Sample 5 - sour cream with whey protein concentrate (WPC) and glucose-fructose syrup (GFS);
Sample 6 - sour cream with whey protein concentrate (WPC) and guar gum.

As illustrated in Figure 2, the examined food systems exhibit thixotropic behavior, with a considerable range of variation. It is noteworthy that the control sample exhibits a relatively high capacity for spontaneous structural restoration, with values exceeding those observed for samples 2, 3, and 6. This is attributed to the increased protein content, including the combination with guar gum. Concurrently, the presence of monosaccharides in sample 5 and the excessive denaturation of whey proteins during the prolonged heat treatment of cream in sample 1 (Polishchuk et al., 2023) to some extent impede the effective restoration of the sour cream structure. The addition of β-glucan to sour cream, as seen in sample 4, may have an adverse effect on the thixotropic ability of the resulting product. This is due to the probable loss of its structuring ability during homogenization, as previously discussed, as well as the obstruction of contact between protein macromolecules.

**Water activity and the degree of syneresis in sour cream samples with different protein and carbohydrate composition**

The subsequent phase of the study focused on examining the water activity and the capacity of protein-fat clots in sour cream samples with varying chemical compositions to retain moisture. These indicators are of significant importance for the formation of quality indicators in sour cream, and can be correlated with its chemical composition and rheological characteristics.

The water activity of the sour cream samples is illustrated in Figure 3.

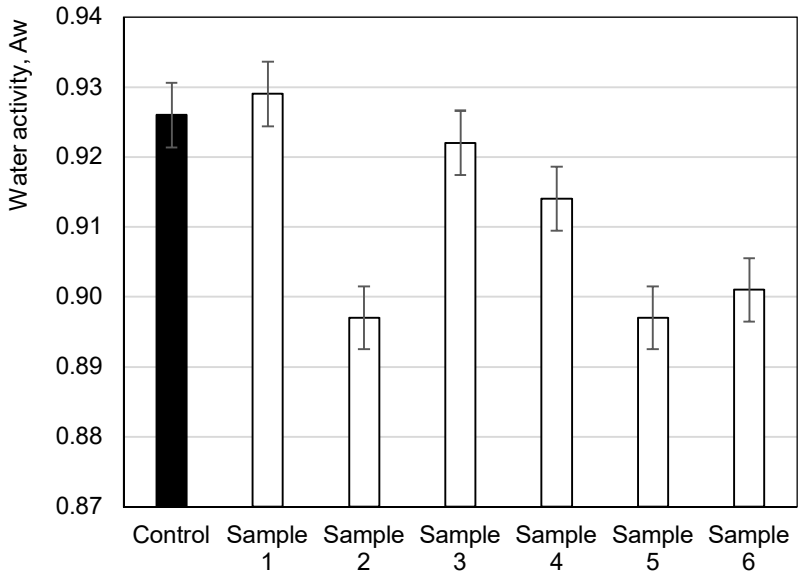


**Figure 3. Water activity in the test samples of sour cream**

Control (sour cream without additives);
Sample 1 (baked sour cream);
Sample 2 - sour cream with hydrolyzed concentrate of demineralized whey (hcdw);
Sample 3 - sour cream with whey protein concentrate (wpc);
Sample 4 - sour cream with whey protein concentrate (wpc) and β-glucan;
Sample 5 - sour cream with whey protein concentrate (WPC) and glucose-fructose syrup (GFS);
Sample 6 - sour cream with whey protein concentrate (WPC) and guar gum.

The water activity in the sour cream samples is notably elevated, ranging from 0.897 (samples 2 and 5 containing monosaccharides) to 0.929 (sample 2, baked sour cream). With regard to the sample of baked sour cream exhibiting the highest water activity, it can be posited that the prolonged and high-temperature processing of cream may result in the excessive coagulation of whey proteins, thereby reducing the moisture retention capacity of sour cream. This hypothesis will be tested in the subsequent series of experiments. Therefore, all sour cream samples can be classified as high-moisture products that are not capable of long-term storage (Plotnikova et al., 2021). It is therefore necessary to develop further technological measures to reduce the water activity in sour cream in order to control the intensity of various physicochemical, biochemical, and microbiological processes during the storage of protein-enriched sour cream.

Another crucial attribute of sour cream is its capacity to retain moisture within the protein-fat gel. The results of determining the syneretic ability of sour cream samples of different chemical composition after ripening and during storage for up to five days are presented in Figure 4.

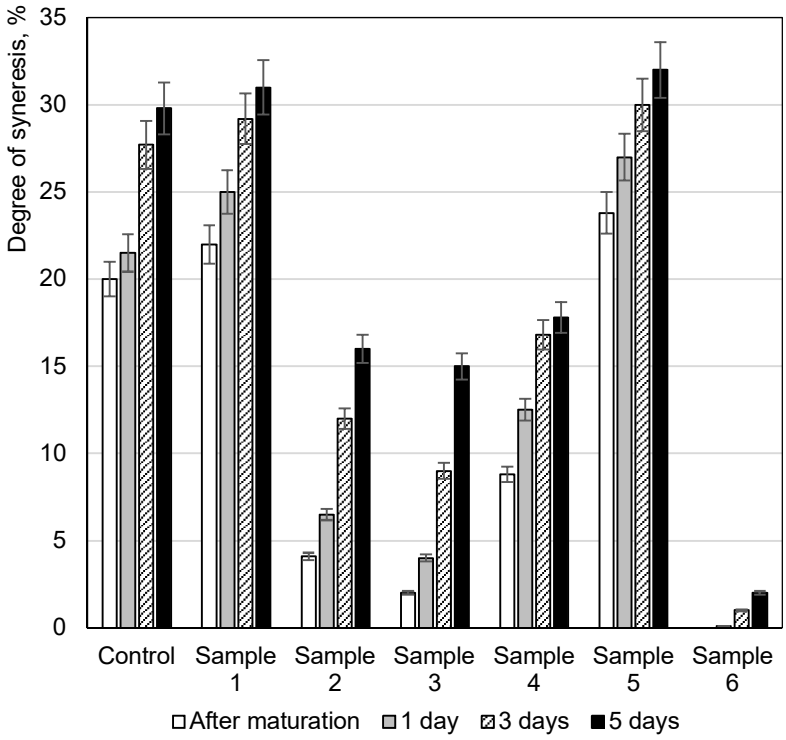


**Figure 4. Degree of syneresis of sour cream samples of different chemical composition during storage**

Control (sour cream without additives);
Sample 1 (baked sour cream);
Sample 2 - sour cream with hydrolyzed concentrate of demineralized whey (hcdw);
Sample 3 - sour cream with whey protein concentrate (wpc);
Sample 4 - sour cream with whey protein concentrate (wpc) and β-glucan;
Sample 5 - sour cream with whey protein concentrate (WPC) and glucose-fructose syrup (GFS);
Sample 6 - sour cream with whey protein concentrate (WPC) and guar gum.

The sample containing whey protein concentrate and guar gum (sample 6) demonstrated the higest moisture retention capacity (Figure 4). The ability of galactomannans, which include guar gum, to form hydrogen bonds with water molecules results in an increase in the moisture retention capacity of food systems (Mudgil et al., 2014). This effect corroborates the hypothesis that polysaccharide macromolecules can be integrated into the protein matrix to form a compact spatial network capable of retaining moisture over an extended period. This assertion is also supported by the findings of Seo (2022).

In the control sample and sample 1 (baked sour cream), pronounced syneresis is observed, which can be explained by the increased content of free water. This is confirmed by the high value of water activity and the substantial thermal denaturation of proteins in sample 1, resulting in a partial loss of their moisture-binding capacity during cream simmering. The results obtained for sample 5, which contained β-glucan, demonstrated that this polysaccharide was incompatible with fermented cream, as evidenced by the degree of syneresis. Therefore, the efficacy of utilizing milk protein concentrates in sour cream with a fat content of 10%, including in conjunction with galactomannan (guar gum), has been substantiated.

Further research should be conducted to elucidate the mechanism of β-glucan structuring and moisture-binding action in sour cream.

## Conclusions

1. The use of whey protein concentrate, obtained by ultrafiltration, and hydrolyzed concentrate of demineralized whey concentrate, in amounts providing a protein content of 3.38% and 3.22%, respectively, in sour cream with a fat content of 10%, results in the formation of technologically active ingredients that serve to improve the rheological characteristics, as well as the moisture retention capacity, of the finished product.
2. Prolonged high-temperature treatment of cream before fermentation (95–98 °C, 3 h) due to thermal denaturation of whey proteins has been observed to significantly reduce the physicochemical and viscosity characteristics of sour cream with a fat content of 10%.
3. The presence of monosaccharides in sour cream has been observed to exert a slight reduction in the functional and technological properties of protein concentrates, with the greatest impact observed with regard to water activity.
4. The addition of polysaccharides at a concentration of 0.5% has been observed to exert a varying degree of influence on the technological properties of whey protein concentrate at a concentration of 1% in sour cream. The incorporation of guar gum has been demonstrated to enhance the rheological characteristics of sour cream, including its thixotropic ability, and to effectively prevent syneresis during storage. Conversely, the use of β-glucan derived from oats has been found to exhibit limited compatibility with sour cream, as evidenced by an anomalous deterioration in the rheological and physicochemical characteristics of the product, necessitating further investigation.

## References

Danylenko S.G., Bodnarchuk O.V., Ryzhkova T.M., Diukareva G.I., Malafaiev M.T., Verbytsky S.B. (2020), The effects of thickeners upon the viscous properties of sour

cream with a low fat content, *Acta Scientiarum Polonorum Technologia Alimentaria*, 19(3), pp. 359–368, https://doi.org/10.17306/J.AFS.0836

Guinee T.P. (2021), Effect of high-temperature treatment of milk and whey protein denaturation on the properties of rennet-curd cheese: A review, *International Dairy Journal*, 121, 105095, https://doi.org/10.1016/j.idairyj.2021.105095

Ivanov V., Shevchenko O., Marynin A., Stabnikov V., Gubenia O., Stabnikova O., Shevchenko A., Gavva O., Saliuk A. (2021), Trends and expected benefits of the breaking edge food technologies in 2021–2030, *Ukrainian Food Journal*, 10(1), pp. 7-36, https://doi.org/10.24263/2304-974X-2021-10-1-3

Javidi F., Razavi S.M.A., Behrouzian F., Alghooneh A. (2016), The influence of basil seed gum, guar gum and their blend on the rheological, physical and sensory properties of low fat ice cream, *Food Hydrocolloids*, 52, pp. 625–633, https://doi.org/10.1016/j.foodhyd.2015.08.006

Kochubei-Lytvynenko O., Bilyk O., Bondarenko Y., Stabnikov V. (2022),Whey proteins in bakery products, In: O. Paredes-López, O. Shevchenko, V. Stabnikov, V. Ivanov, (Eds.), *Bioenhancement and Fortification of Foods for a Healthy Diet*, CRC Press, Boca Raton, London, pp. 67-88, https://doi.org/1201/9781003225287-5

Kochubei-Lytvynenko O., Bilyk O., Stabnikov V., Dubivko A. (2023), Milk whey enriched with magnesium and manganese for food production, In: O. Stabnikova, O. Shevchenko, V. Stabnikov, O. Paredes-López (Eds.), *Bioconversion of Waste to Value-Added Products*, CRC Press, Boca Raton, London, pp. 113-128, https://doi.org/10.1201/9781003329671-4

Lee Y., Chang Y. (2016), Influence of guar gum addition on physicochemical, microbial, rheological and sensory properties of stirred yoghurt, *International Journal of Dairy Technology*, 69, https://doi.org/10.1111/1471-0307.12263

Lisbeth M.G. (2012), Sour cream and crème fraîche, In *Handbook of Animal-Based Fermented Food and Beverage Technology* (pp. 235–246), CRC Press: Boca Raton, USA, https://doi.org/10.1201/b12084-16

Lohinova A., Petrusha O. (2024), Maillard reaction in food technologies, *Ukrainian Journal of Food Science*, 11(2), pp. 81 – 109, https://doi.org/10.24263/2310-1008-2023-11-2-4

Lucey, J.A. (2016). Acid coagulation of milk. In: P. McSweeney, J. O'Mahony (Eds.), *Advanced dairy chemistry*, Springer, New York, NY, https://doi.org/10.1007/978-1-4939-2800-2 12

Luck P.J., Vardhanabhuti B., Yong Y.H., Laundon T., Barbano D.M., Foegeding E.A. (2013), Comparison of functional properties of 34% and 80% whey protein and milk serum protein concentrates, *Journal of Dairy Science*, 96(9), pp. 5522–5531, https://doi.org/10.3168/jds.2013-6617

Mandiuk O., Lohinova A., Arsenieva L., Petrusha O., Polishchuk G. (2023), Effects of protein and carbohydrate ingredients on colour of baked milk products, *Ukrainian Food Journal*, 13(1), pp. 7–19, https://doi.org/10.24263/2304-974X-2024-13-1-3

Mudgil D., Barak S., Khatkar B.S. (2014), Guar gum: Processing, properties and food applications-A review, *Journal of Food Science and Technology*, 51(3), pp. 409–418, https://doi.org/10.1007/s13197-011-0522-x

Mykhalevych A., Kostenko O., Polishchuk G., Bandura U. (2022), Application of milk protein concentrates in preparation of reduced fat sour cream, *Ukrainian Food Journal*, 11(3), pp. 429–447, https://doi.org/10.24263/2304-974X-2022-11-3-8

Mykhalevych A., Polishchuk G., Nassar K., Osmak T., Buniowska-Olejnik M. (2022), β-Glucan as a techno-functional ingredient in dairy and milk-based products—A review, *Molecules*, 27(19), 6313, https://doi.org/10.3390/molecules27196313

Narvhus J.A., Østby N., Abrahamsen R.K. (2019), Science and technology of cultured cream products: A review, *International Dairy Journal*, 93, pp. 57–71, https://doi.org/10.1016/j.idairyj.2019.01.011

Odnorog M., Polishchuk G. (2018), Application of whey protein concentrate for stabilizing sour cream structure, *Food Industry*, 23, pp. 6–12 (in Ukrainian), https://doi.org/10.24263/2225-2916-2018-23-3

Osmak T., Mleko S., Bass O., Mykhalevych A., Kuzmyk U. (2021), Enzymatic hydrolysis of lactose in concentrates of reconstituted demineralized whey, intended for ice cream production, *Ukrainian Food Journal*, 10(2), pp. 277–288, https://doi.org/10.24263/2304-974X-2021-10-2-6

Pintor A., Totosaus A. (2012), Ice cream properties affected by lambda-carrageenan or iota-carrageenan interactions with locust bean gum/carboxymethylcellulose mixtures, *International Food Research Journal*, 19, pp. 1409–1414.

Plotnikova I.V., Zharkova I.M., Magomedov G.O., Magomedov M.G. (2021), Forecasting and quality control of confectionery products with the use of "water activity" indicator, *IOP Conference Series Earth and Environmental Science*, 640(6), 062003, https://doi.org/10.1088/1755-1315/640/6/062003

Polischuk G., Breus N., Kochubey-Litvinenko O., Osmak T., Semko T., Borova M. (2020), Study of the influence of micellar casein and spelt flour on yogurt quality indicator, *EUREKA: Life Sciences*, 4, pp. 44–52, https://doi.org/10.21303/2504-5695.2020.001378

Polishchuk G., Sharakhmatova T., Shevchenko I., Mandiuk O., Mykhalevych A., Pukhlyak A. (2023), Scientific substantiation of cream heating duration in the technology of sour cream, enriched with protein, *Food Science and Technology*, 17(3), pp. 75–83, https://doi.org/10.15673/fst.v17i3.2657

Seo C.W. (2022), Effect of galactomannan addition on rheological, physicochemical, and microbial properties of cultured sour cream, *Food Science and Biotechnology*, 31(5), pp. 571–577, https://doi.org/10.1007/s10068-022-01066-3

Seo C.W., Oh N.S. (2023), Rheological, physicochemical, microbiological, and aroma characteristics of sour creams supplemented with milk protein concentrate, *Food Science of Animal Resources*, 43(3), pp. 540–551, https://doi.org/10.5851/kosfa.2023.e16

Sturaro A., Penasa M., Cassandro M., Varotto A., De Marchi M. (2014), Effect of microparticulated whey proteins on milk coagulation properties, *Journal of Dairy Science*, 97(11), pp. 6729–6736, https://doi.org/10.3168/jds.2014-8157

Wang Q., Ouyang J., Wang L., Wu Y., Xu C. (2024), Impact of whey protein isolate and xanthan gum on the functionality and in vitro digestibility of raw and cooked chestnut flours, *Plant Foods for Human Nutrition,* 79, pp. 189–193, https://doi.org/10.1007/s11130-024-01150-5

Xiang J., Liu F., Wang B., Chen L., Liu W., Tan S. (2021), A literature review on maillard reaction based on milk proteins and carbohydrates in food and pharmaceutical products: Advantages, disadvantages, and avoidance strategies, *Foods*, 10(9), p. 1998, https://doi.org/10.3390/foods10091998

Zagorska J., Pelnik A., Kerch G. (2013), Effect of the addition of chitosans with different molecular structure on fermentation process and viscosity changes during sour cream storage, *Biochemistry and Biophysics (BAB)*, 1(2), pp. 13–21.

# Advantages of cyclic rectification for ethanol production

**Yurii Bulii[1], Andrii Forsiuk[1],**
**Mykola Bondar[1], Olexandr Obodovych[2]**

*1 – National University of Food Technologies, Kyiv, Ukraine*
*2 – Institute of Technical Thermophysics of the National Academy of Sciences of Ukraine, Kyiv, Ukraine*

**Keywords:**

Alcohol
Column
Cyclic
Distillation
Ethanol
Impurity

**Article history:**

Received 20.03.2024
Received in revised form 16.04.2024
Accepted 28.06.2024

**Corresponding author:**

Yurii Bulii
E-mail: yvbuliy@gmail.com

**DOI:**
10.24263/2310-1008-2024-12-1-4

## Abstract

**Introduction.** The efficiency of the ethyl alcohol rectification process depends on the degree of purification of the final product from organic impurities and the heating vapour flow rate in the distillation unit.

**Materials and Methods.** The object of the study was a cyclic column for concentration of impurities. The concentration of volatile impurities of alcohol was determined by chromatographic method, their extraction and concentration ratio – by calculation method. Water flow rate was controlled by means of Rate-Master® flow meters, vapor velocity in the free section of the column – by vortex flowmeter, in the holes of the plates – by calculation method.

**Results and discussion.** Studies of the efficiency of the technology of cyclic rectification of ethyl alcohol, providing for the implementation of controlled cycles of liquid delay on the column plates and its overflow without interruption of heating vapour supply have been carried out. The design of the rectification column for its realization is developed. Hydrodynamic modes of operation of sieve and flake plates in cyclic mode are established: vapour velocity in the free section of the column and plate openings for mass exchange between liquid and vapour and liquid preflow. The research results obtained in production conditions proved the advantages and feasibility of using the innovative technology: in the process of separation of alcohol-containing fractions in full measure head impurities of alcohol are allocated, the degree of extraction of the higher alcohols of sivush oil increases by 38%, methanol – by 15,6%, the multiplicity of concentration of head impurities increases by 25%, higher alcohols – by 40%, methanol – by 34%, acrolein – by 36%, isopropyl alcohol – by 42%. At the same time vapour consumption in the impurity concentration column is reduced by 40% in comparison with typical units operating in the stationary mode and does not exceed 12 kg/dal of absolute alcohol (a.a.) introduced with feed.

**Conclusion.** Increasing the contact time of vapor and liquid on each plate up to 30–40 s allows increasing the degree of purification of rectified alcohol from volatile impurities and reducing energy costs by at least 40%.

## Introduction

The energy dependency of industrialized nations requires for alternative sources of energy, one of which is ethyl alcohol (bioethanol). To ensure the competitiveness of bioethanol over carbohydrate energy sources, the development and implementation of innovative resource- and energy-saving technologies, and the enhancement of operational and technological characteristics of the rectification plants are a prerequisite. A promising direction to solve this problem is to use a non-stationary (cyclic) mode of operation of the plants, based on the alternating change of the periods of the steaming periods and liquid overflow (Botshekan et al., 2022; Nagy et al., 2015).

Investigation of physico-chemical conditions of separation of multicomponent systems in the mode of controlled rectification cycles, development on the basis of the laws of thermodynamics of rational methods of calculation and construction of rectification columns of cyclic action, work in computer modeling of delay cycles and liquid overflow began in the 60's and 70's of the XX-th century (Andersen et al., 2018; Bastian et al., 2012; Kiss, 2014; McWhirter and Cannon, 1961). The authors managed to achieve an increase in steam load by 48% with constant pressure drop without making structural changes in the column, double the capacity of the installation with cap plates compared to the typical at the same degree of separation, to develop a hydrodynamic model of rectification with alternate phase motion, the theory of intermittent cyclic distillation – a new method of periodic control in which the motion of the liquid depended on the ripple of the steam flow, and to propose a new type of plate was a traditional mesh plate with a special inclined section to slow down the liquid overflow (Nielsen et al., 2017; Rasmussen et al., 2020; Toftegard et al., 2016).

Due to rising energy prices, interest in the study of cyclic distillation has increased in Ukraine in recent years. At the National University of Food Technology, special valve contact devices have been proposed to provide controlled cycles of delay and liquid overflow (patent UA60566. Mass exchange contact device). Pilot tests of the devices proved the possibility of reducing the specific consumption of heating **vapor** in columned installations of cyclic action by 30% compared to typical ones. The disadvantages of their operation include the dependence of the operation of the overflow devices on the vapor pressure, the limitation of the rectification column in height, the need to install intermediate plates, the appearance of the impulse to delay the opening and closing of the floating valves (Maleta et al., 2011).

Despite the positive results of experimental studies, justified by the methods of mathematical modeling of the benefits of cyclic rectification, the above methods and models are not widely used. Absence of mass-exchange in the vapour period, fluctuations of vapour pressure in the collector, the bottom section and the deflegmator of the rectification column adversely affect the quality of the finished product and the work of other columns. And the complexity of the proposed constructive solutions to ensure a cyclic mode reduce the reliability of the rectification equipment.

In order to improve the efficiency of mass-exchange by eliminating the above disadvantages, a method of controlled rectification, which provides for periodic (cyclic) motion of the liquid after its delay at the contact stages with continuous supply of heating vapour in the bottom section of the column was proposed. For the cyclic motion of the liquid on the plates of the column, the vapour flow is cut off at each plate by means of valves located on the bypass conduits (Krivosheev and Anufriev, 2015). According to the authors, this technical solution allows to ensure the periodic motion (weeping) of the liquid on the plates during the period of opening of the respective valves and the direction of vapour through the

respective bypass conduits and to exclude its mixing with the portions of the liquid located on the lower adjacent plates.

However, the method was not practically implemented due to the fact that, at the moment of opening of the valves, the vapour flow occurred both through the bypass conduits and through the slits of the upper plates, thus complicating the liquid overflow on the lower plates.

To solve the current problem, the staff of the Department of Biotechnology of Fermentation Products and Winemaking of the National University of Food Technology and LLC "TISER" developed an innovative technology of cyclic rectification, which provided for the delay of liquid on the plates and its cyclic overflow without interrupting the supply of heating vapour, as well as the design of rectification column for its implementation (Buliy et al., 2016; Ukrainets et al., 2018). The efficiency of technical solutions was determined in the process of ethyl alcohol extraction from alcohol-containing fractions enriched with organic impurities. For the purpose of the study, the experimental impurity concentration column was equipped with movable valves connected to actuators whose action was according to the controller program, and flake plates with valve openings.

The aim of the present research was to improve the technology of cyclic rectification, to develop the construction of a distillation column to ensure the cyclic motion of the liquid with continuous vapour supply, to study the effectiveness of innovative technology in the process of rectification of alcohol-containing fractions, to determine the optimal technological modes of operation of the impurity concentration column, specific vapour consumption, degree of extraction and multiplicity of concentration of volatile impurities of alcohol.

To achieve this aim and conduct production tests, the primary task was to determine the hydrodynamic mode of operation of the perforated plates (mesh and cale-shaped) to ensure their cyclic action:

1. Establishment of the working range of vapour speed in the free section of the impurity concentration column and in the slots (openings) of the plates — the lower boundary, beyond which the weeping of the plate stops from the upper plate to the lower plate, and the upper boundary, at which liquid entrainment occurs to the upper plates.
2. Determination of the vapor velocity in the slots (openings) of the plates, at which there is an intense weeping of the liquid from the upper plates to the lower ones.

## Materials and Methods

### Research objects

#### 1. Experimental cyclic column for concentrating alcohol impurities (ICC).

The impurity concentration column is made of stainless steel AISI 304, equipped with flake plates. Technical characteristics: diameter – 426 mm; number of plates – 30; distance between the plates – 300 mm; the cross-sectional area of the holes – 19.42 $mm^2$; thickness of the plate blade – 2 mm; free section of the plate: 5.5% – during the stay of liquid on the plates – 5.5%; during the overflow of liquid – 51.7% (Figure 1).

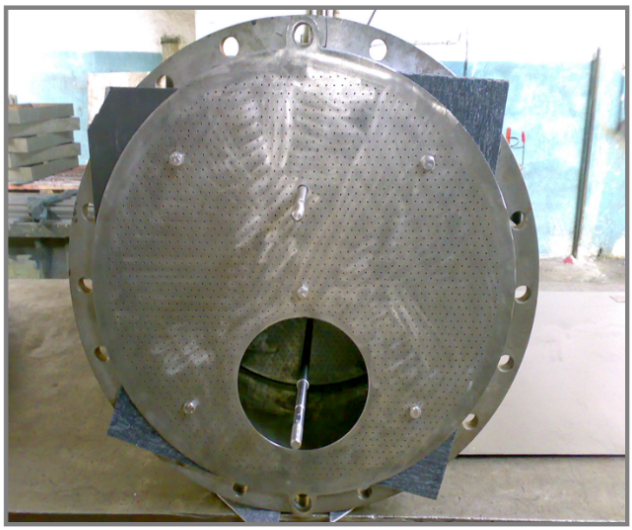

**Figure 1. Cyclic plate with variable live section**

A fragment of the impurity concentration column with movable rods and valves is shown in Figure 2 (patent UA139228. Column mass transfer apparatus of cyclic action).

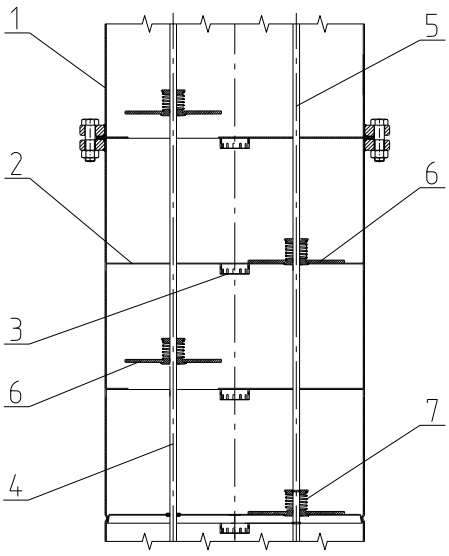


**Figure 2. Fragment of a prototype LC with movable valves:**
1 – body; 2 – plates; 3 – contact elements; 4,5 – rods;
6 – valves; 7 – springs.

The column contained a body 1, plates 2 with contact elements 3, movable rods 4 and 5, on which valves 6 and springs 7 were fixed. The rods moved up and down periodically under the action of drive mechanisms (double-acting pneumatic cylinders of DNT type of

FESTO Company). In this case, valves 6 alternately closed and opened holes for liquid overflow. Pneumatic cylinders were controlled according to the program of controller M340 of Schneider Electric Company.

The operation of the column provided for conducting adjustable in time cycles of liquid residence on the plates and its synchronous overflow from plate to plate over the entire height of the column in two successive stages, repeating periodically in time, alternately, according to the specified algorithm without interrupting the liquid and steam supplyin the column (patent UA89874. Method of liquid overflow on plates of column apparatus in the process of mass-exchange between vapour and liquid). The time interval of liquid retention was determined experimentally depending on the degree of extraction of volatile alcohol impurities and the multiplicity of their concentrationi and was 30-40 s.

The technical solution allowed for one-stage (full) and two-stage (fractional) overflow of liquid from the upper plate on the lower plate. The single-stage method ensured that all liquid overflowed. According to the two-stage method (patent UA 141245. Method of overflow of liquid on plates of mass-exchange column apparatus) at the first stage a part of liquid (30-70% of its volume) was overflowed from the upper plate to the lower one, and after the set time of liquid delay its rests were overflowed.

The experimental impurity concentration column was incorporated into the circuit of an operating distillation unit.

## 2. Installation for extraction of ethyl alcohol from alcohol-containing fractions

The technological scheme of inclusion of the experimental impurity concentration column in the scheme of the distillation unit is shown in Figure 3.

The scheme provided for the introduction on the feed plate of the impurity concentration column of the head fraction of ethyl alcohol, distillate cuts from the condensers of the distillation column and $CO_2$ separator condenser, fusel alcohol and rinse water for fusel oil. The installation included impurity concentration column 6, the plates of which are connected to the pneumatic cylinders bilateral action 7, and their upper and lower parts with vacuum breakers 4, evaporator 5, condenser 9, alcohol catcher 10, softened water container 1 for hydroselection, intermediate collections of distillation residue 17 and alcohol-containing fractions 20, flowmeters 3, 12, 13, 14 and 15, centrifugal pumps 2, 18, 19 and decanter 11.

The consumption of vapour, water for hydroselection, fusel ad ester-aldehyde concentrate and distillation residue was determined using flowmeters.

### Research methods

To evaluate the obtained research results, analytical, chemical, physicochemical, and computational methods were used with the use of devices and research methods used in the production of rectified ethyl alcohol.

**Liquid consumption**. The consumption of alcohol-containing fractions, water for hydroselection, distillation residue and rectified alcohol was monitored using RM flowmeters (Polulyah and Topolov, 2002).

The principle of their operation is based on the perception of the dynamic head of the controlled medium, which depends on the flow rate, by a sensing element (float) placed in the flow. As a result of the flow, the sensing element moves along the height of the flowmeter, and the amount of movement serves as a measure of flow. The readings were taken on the scale of the flowmeter, graduated by water in $dm^3/h$.

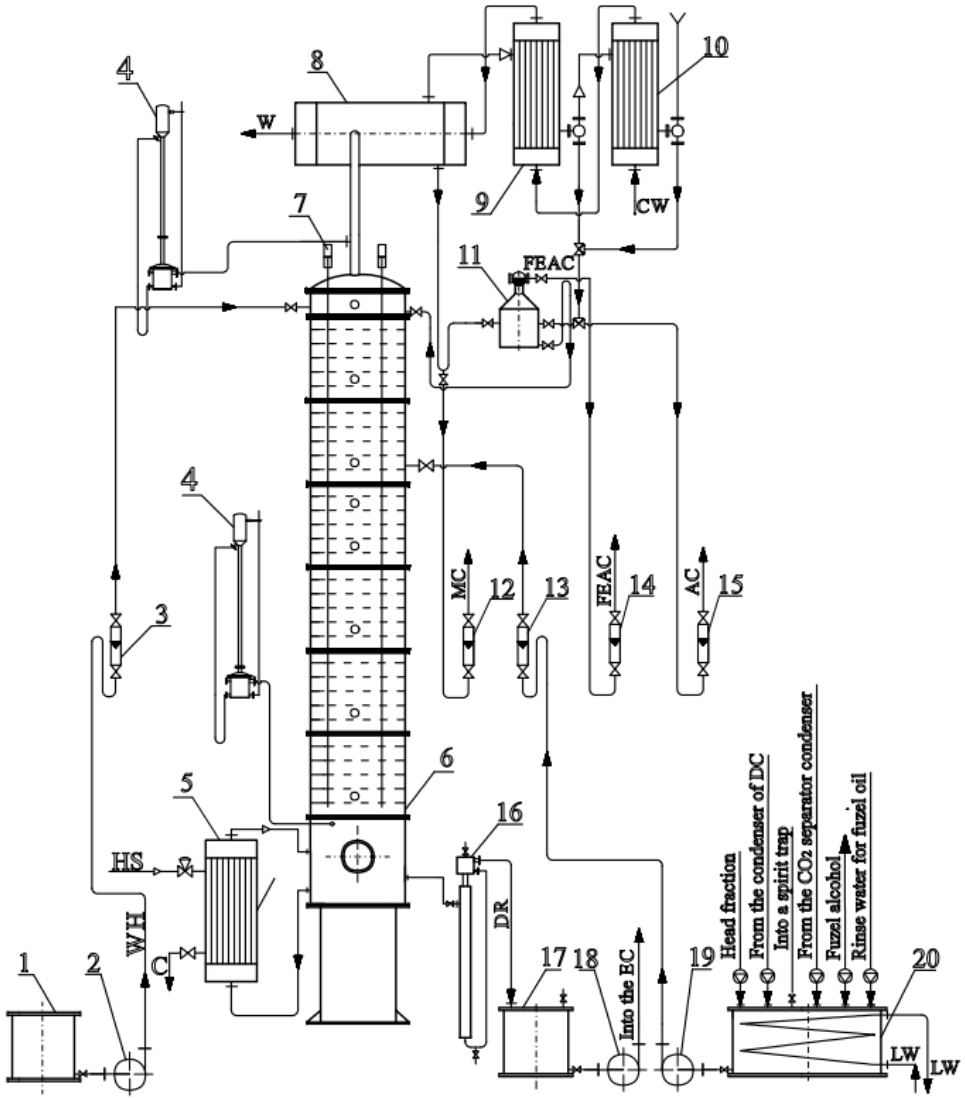


**Fig. 3. Hardware and process flow diagram of the installation for extracting ethyl alcohol from alcohol-containing fractions:**

1 – water container; 2, 18, 19 – centrifugal pumps; 3, 12, 13, 14, 15 – flowmeters; 4 – vacuum breakers; 5 – evaporator; 6 – impurity concentration column (ICC); 7 – pneumatic cylinders; 8 – dephlegmator; 9 – condenser; 10 – alcohol-collecting vessel (spirit trap); 11 – decanter; 16 –hydraulic shutter; 17 – distillation residue tank; 20 – alcohol-containing fraction tank

*Notation conventions:*

CW – cooling water; WH – water for hydroselection; HS – heating steam; DC – distillation column; EC – ether column; C – condensate; DR – distillation residue; FEAC – fusel and ester-aldehyde concentrate; AC – aldehyde concentrate; MC – methanol concentrate; LW – luther water.

**Vapour velocity.** The vapour velocity in the free section of the column was determined using a vortex flowmeter (VFM), which has an ejection rod located on the path of the steam flow from which the vortices are ejected. The frequency of rejection varies linearly with the speed of the steam. Placing a piezoelectric sensor below the flow and calculating the peak frequency of its signal allows you to fix the speed of steam and its flow rate **(**Dattarajan, 2019).

**Concentration of ethyl alcohol in water-alcohol solutions.** The concentration of ethyl alcohol in the liquid distillation residue of the aldehyde concentrate was determined by areometric method). The test solution was poured into a 250 $cm^3$ glass cylinder, the temperature was measured with a thermometer with a division price of 0.1 °C, and then the ASP-1 alcoholmeter was immersed. The actual concentration of ethyl alcohol at a temperature of 20 °C was determined from the readings of the alcoholmeter and using special tables to make appropriate corrections for temperature.

**Concentration of volatile alcohol impurities.** Concentration of aldehydes was determined by reaction with fuchsin-sulfur reagent I; fusel oil – by the method based on the reaction of higher alcohols with salicylic aldehyde solution in the presence of sulfuric acid; free acids – by the amount of sodium hydroxide solution used for titration; complex esters – by titrometric method after their saponification with sodium hydroxide solution; volume fraction of methyl alcohol – by method based on the reaction of methanol oxidation with potassium permanganate and sulfuric acid to form formaldehyde, which forms a color with fuchsin-sulfur reagent II **(**Arslan, 2021).

The concentration of head volatile impurities (aldehydes, higher alcohols of fusel oil, acrolein, isopropyl alcohol) in alcohol-containing fractions, distillation residue of impurity concentration column, concentrate of impurities and rectified ethyl alcohol was determined on a gas chromatograph with an HP FFAP 50 m × 0.32 m column (Dewulf, 2002; Plutowska et al., 2008; Steven et al., 2002). The analysis of the experimental samples was carried out three times. The average values were chosen as determinative.

**Grade of extraction and concentration ratio of volatile alcohol impurities.**

The degree of extraction *(α)* and multiplicity of concentration *(β)* of key organic impurities of alcohol were calculated by the formulas:

$$\alpha = \frac{X_l}{X_{cub}};\ \beta = \frac{X_{seac}}{X_l};$$

where $X_l$, $X_{seac}$, $X_{cub}$ – accordingly, the concentration of volatile alcohol impurities on the feed plate, sivush and ester-aldehyde concentrate and cube liquid, mg/$dm^3$ in terms of a.a. (Linek, 2005; Shiyan et al., 2009).

**Studied modes**

*In the first stage,* the hydrodynamic regimes of failure plates were investigated to ensure the cyclic mode of operation of the experimental impurity concentration column. The efficiency of the perforated plates in a cyclic mode depends on the adopted hydrodynamic modes, which determine the boundaries of stable operation of the column (Kiss et al., 2009; Kiss and Bildea, 2011; Kiss, 2012). There are no general methods for calculating the boundaries of hydrodynamic modes for bubble plates. Therefore, when designing the plant-

like devices, the vapor velocity corresponding to the lower and upper bounds of the plate was determined by calculation, and then the working speed vapor velocity was determined in the free section of the column and in the slits of the plates. The lower limit corresponded to the vapor velocity at which the weeping of the liquid from the plate stopped. The upper limit corresponded to the vapor velocity at which the liquid entrainment onto the upper plate was observed. In this case, the contact surface of the phases sharply decreased. Such plates are called versatile or variable cross-sectional plates. Many studies have been devoted to determining the conditions under which bubbling occurs on the plates (the fluid is retained on the plate) and the overflow of the liquid through the overflow openings and its weeping through the slits of the contact devices (Lita, 2014; Pătruţ et al., 2014; Premkumar and Rangaiah, 2009). In order to eliminate the possibility of the liquid being transferred to the top plate, the vapor velocity in the free section of the column equipped with mesh plates should not exceed 0.7 m/s. The vapour velocity in the holes at which the liquid is retained on the plate is 4-5.5 m/s. When using scale-shaped plates with an optimal live cross-section of 10%, the vapor velocity in the free column cross-section may reach 1.2 m/s or more, and in the openings of the scales must exceed the first critical velocity of 6.5-7.5 m/s. The liquid weeping occurs at a vapor velocity in the openings of 1.5-1 m/s (Dejanovic et al., 2010; Flodman and Timm, 2012).

*At the second stage,* the effectiveness of the innovative technology in the process of distillation and separation of alcohol-containing fractions was investigated: determined the optimal technological mode of operation of the impurity concentration column, the specific vapour consumption, the degree of extraction and multiplicity of concentration of key organic impurities of alcohol.

Alcohol-containing fractions of 135 $dm^3/h$ were calculated on the feed plate of the impurity concentration column in terms of absolute alcohol (a.a.). Of these, the head fraction of ethyl alcohol is 8.5%, the distillate cuts from the condenser of the distillation column – 9.4%, the $CO_2$ separator condenser – 3.0%, fusel alcohol – 1.5%, rinse water for fusel oil – 1.5% of the a .a. of mash. The delay time of the liquid on the plates was 30 s, the overflow time – 2 s. The pressure in the bottom part of the DC was 17.5 kPa, in the upper part – 2–5 kPa. The temperature in the bottom of the column was equal to 102–103 °C, in the upper part – 90-91 °C, water for cooling at the condenser inlet – 15 °C, at the outlet after the deflegmator – 65 °C.

Water for hydroselection in the amount of 2400 $dm^3/h$ was supplied from water container 1 through a flowmeter 3 to the top plate of the column by pump 2. The concentration of ethyl alcohol on the plates of the concentration part was 7% vol., and in the bottom part – 5–6% vol. The alcohol-containing fractions were warmed by the heat of luther water (residue water) in the container 20, from which the feed plate was fed by the pump 18. Released from the head and part of the intermediate volatile impurities distillation residue was pumped into the upper zone of the concentration part of the ether column by centrifugal pump 18. Vapour containing volatile impurities from the upper part of column 6 was sequentially fed into the deflegmator 8 and the condenser 9. In the dephlegmator mainly water, higher alcohols, partially esters and ethanol condensed. There was the condensation of low-boiling aldehydes and methyl alcohol in condenser, which vapours did not condense in the deflegmator. Reflux from the dephlegmator 8 and most of the condensate of the head and final impurities from the condenser 9 was sent to the decanter 11. In the decanter, the heterogeneous mixture was separated, forming the upper layer – FEAC containing esters, higher alcohols and part of aldehydes, and the lower layer – water released from the head, part of the intermediate and final impurities. The FEAC was removed from the installation through the flowmeter 13 and the aqueous-alcoholic liquid from the lower part of the

decanter, which in the form of phlegma flowed on the top plate of the impurity concentration column for irrigation. To reduce the concentration of methyl alcohol in the distillation residue part of the phlegma was removed from the installation in the form of methanol concentrate (MC) through flowmeter 11. From the condenser 9 aldehyde concentrate (AC) was selected through flowmeter 14.

A mnemonic diagram of the impurity concentration column inclusion in operation with controlled cycles of mass-exchange and liquid overflow is shown in Figure 4.

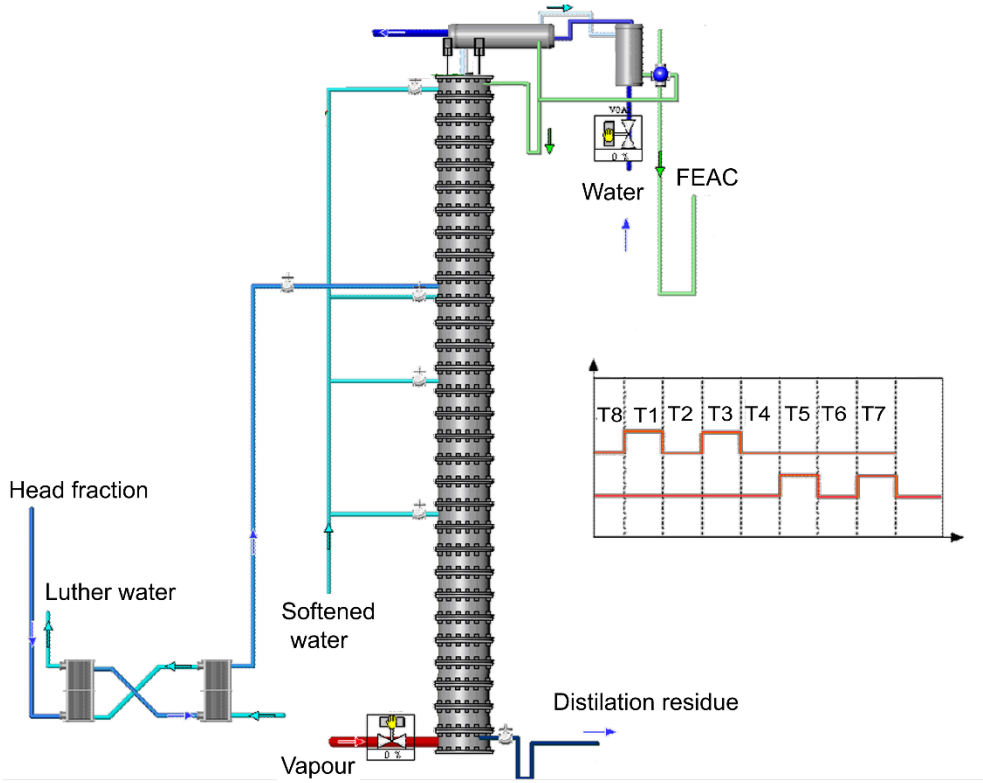


**Figure 4. Mnemonic diagram of the operation of an column concentrating impurities in the mode of controlled cycles of liquid retention and overflow**

For calculation degree of extraction *(α)* and multiplicity of concentration *(β)* of key organic impurities of alcohol during the research period, samples of alcohol-containing fractions entering the column were taken, distillation residue, reflux, fusel and ester-aldehyde concentrate and and samples of liquid from the 2nd, 6th, 11th, 17th and 26th plates of the column were taken and their chromatographic analysis was performed. The studies were carried out in triplicate. The average values were chosen as the determining ones.

**Stages of research**

At the first stage of research it was established that the vapour velocity in the free section of the column equipped with mesh plates should not exceed 0.7 m/s. At such steam velocity the possibility of liquid entrainment to the upper plate is excluded. The vapour velocity in the holes, at which the liquid is retained on the plate, is 4–5.5 m/s; the optimum velocity for mass-exchange is 7.5-8.0 m/s. When using flake plates with an optimum live section of 10%, the vapour velocity in the free section of the column can reach 1,2 m/s and more. In the holes of scales the vapour velocity should exceed the critical velocity of 6.5–7.5 m/s, the optimal velocity is 12–14 m/s (Buliy et al., 2019). Intensive overflow of liquid occurs at vapour velocity in holes 1.5–1 m/s (patent UA 123917. Method of mass-exchange between liquid and vapor in a column apparatus).

At the second stage, it was investigated that to obtain high-quality alcohol, the extraction of FEAC should be 0.23–0.27% of the amount of ethyl alcohol in the fermentation. Of these, AC should be selected in the ratio of 1:2–1:2.5 to FEAC. And MC should be sampled if the concentration of methanol in distillation residue exceeds 0.001% vol.

It's been practically proven that the organization of differentiated selection of alcohol impurities from places of their maximum accumulation, autonomous regulation of their ratio, depending on the qualitative composition of feed, allowed to increase the efficiency of rectification ( patent 69511. Rectification unit for alcohol extraction from fractions enriched with organic impurities). Direction of phlegma of dephlegmator and condensate from the condenser to the decanter to separate the heterogeneous mixture and the additional selection of water-alcohol mixture from the lower part of the decanter helped to reduce the content of the end liquid, parts of the head, intermediate and atypical types of impurities, isopropyl alcohols, crotonaldehyde, acrolein) and esters (methyl acetate, ethyl acetate), due to which the quality of rectified alcohol improved.

The results of the chromatographic analysis of the test samples are given in Table 1. The analysis of the experimental data Table 1 has shown that in the process of rectification of alcohol-containing intermediates and rectification products in the mode of controlled rectification cycles together with the head ones, the intermediate and final impurities of alcohol are effectively removed. Under the given conditions, aldehydes (acetaldehyde), esters (methyl acetate, ethyl acetate, isobutyl acetate, isoamyl acetate), atypical impurities (acrolein, crotonaldehyde), n-pentanol and isopropyl alcohol are completely removed. It is known from practical experience that isopropanol is one of the volatile impurities that is most difficult to separate from ethanol. Therefore, the content of it in the final product evaluates the efficiency of the rectification plant. The concentration of isopropanol in commercial alcohol should not exceed 1.5 mg/dm$^3$ (Xiao-Na Pang et al., 2017).

Among the esters, ethyl acetate had the highest concentration, and isobutyl acetate has the lowest. Of the higher fusel alcohols, isobutanol was most effectively concentrated, n-propanol had the least recovery rate and concentration. During rectification, the concentration of ethyl alcohol on the plates of the column did not exceed 13% vol.

The calculated values of *(α)* and *(β)* for aldehydes, esters, higher fusel alcohols, methyl alcohol and atypical alcohol (acrolein) impurities under typical and cyclic distillation are given in Table 2.

**Table 1**

**Distribution of volatile organic impurities of alcohol along height of the impurity concentration column**

| Name of the impurity | Contertation, mg/dm$^3$ (in terms of a. a.) | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | Distillate cuts | Distillate residue | The plate number | | | | | Reflux | EEAC |
| | | | 2 | 6 | 11 | 17 | 26 | | |
| Acetaldehyde | 605.1 | *traces* | *traces* | 2.6 | 3.3 | 5.3 | 16.7 | 12304.2 | 13591.2 |
| Methyl acetate | 32.3 | *traces* | *traces* | *traces* | *traces* | *traces* | *traces* | 756.1 | 770.8 |
| Ethyl acetate | 509.4 | *traces* | *traces* | 4.7 | 8.3 | 18.6 | *traces* | 14448.9 | 15344.4 |
| Isobutyl acetate | 14.6 | *traces* | *traces* | *traces* | *traces* | 9.1 | 47.5 | 122.9 | 137.2 |
| Isoamyl acetate | 81.4 | *traces* | *traces* | *traces* | *traces* | *traces* | *traces* | 1573.2 | 1652.9 |
| Isopropropanol | 2.0 | *traces* | *traces* | *traces* | 1.0 | 4.4 | 12.5 | 47.7 | 42.2 |
| n-propanol | 10063.8 | 1080.3 | 1419.3 | 2331.1 | 4894.4 | 8035.3 | 6254.5 | 49045.5 | 53570.4 |
| Isobutanol | 6851.2 | 6.5 | 7.6 | 47.9 | 347.5 | 1749.3 | 6718.3 | 218161 | 204757 |
| n-butanol | 36.1 | *traces* | 5.4 | 8.6 | 23.4 | 24.1 | 28.7 | 422.4 | 379.9 |
| Isoamylol | 10354.8 | 7.1 | 8.0 | 156.7 | 731.9 | 1672.4 | 8531.7 | 211033 | 203311 |
| n-pentanol | *traces* | *traces* | *traces* | *traces* | *traces* | *traces* | *traces* | 12.3 | 17.4 |
| Acrolein | 25.9 | *traces* | *traces* | *traces* | *traces* | *traces* | *traces* | 446.9 | 447.6 |
| Crotonaldehyde | *traces* | *traces* | *traces* | *traces* | *traces* | *traces* | *traces* | 121.8 | 136.7 |
| *Aldehydes* | 605.1 | *traces* | *traces* | 2.6 | 3.3 | 5.3 | 16.7 | 12304.2 | 13591.2 |
| *Esters* | 637.7 | *traces* | *traces* | 4.7 | 8.3 | 27.7 | 47.5 | 16901.1 | 17905.2 |
| *Fusel oil* | 27307.9 | 1093.9 | 1440.2 | 2544.4 | 5998.2 | 11485 | 21545 | 478712 | 462078 |
| *Methanol, % vol.* | 0.026 | 0.001 | 0.001 | 0.002 | 0.004 | 0.003 | 0.005 | 2.9 | 2.1 |
| *Ethanol, % vol.* | 82.0 | 5.4 | 10.0 | 13.0 | 13.0 | 12.4 | 7.0 | 65.0 | 68.0 |
| *Atypical* | 25.9 | *traces* | *traces* | *traces* | *traces* | *traces* | *traces* | 446.9 | 447.6 |

**Table 2**

**Degree of extraction *(α)* and concentration multiplicity *(β)* of impurities of alcohol during typical and cyclic rectification**

| Name of the impurity | Conventional distillation | | Cyclic distillation | |
|---|---|---|---|---|
| | α | β | α | β |
| Aldehydes | 86.4 | 16.9 | max | 22.5 |
| Esters | 79.7 | 21.1 | max | 28.1 |
| Fusel oil | 21.1 | 10.0 | 25.0 | 16.9 |
| Methanol | 16.2 | 67.4 | 26.0 | 102.3 |
| Acrolein | 64.7 | 11.2 | max | 17.4 |

Concentration multiplicity *(β)* of key volatile alcohol impurities (aldehydes and higher alcohols of sivush oil) are presented in the diagram (Fig. 5).

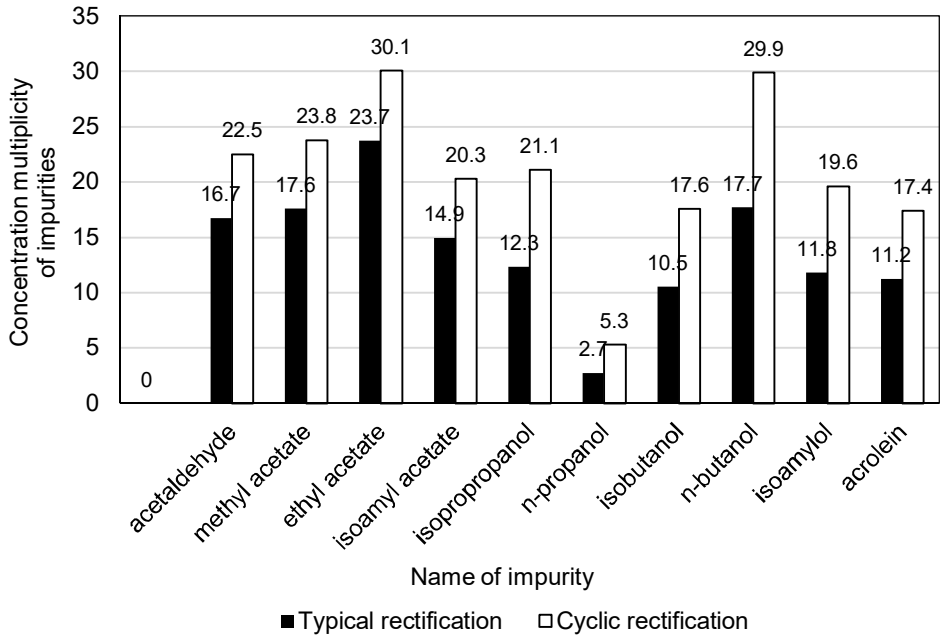


**Figure 5. Multiplicity of concentration of (β) of key alcohol impurities (aldehydes and higher alcohols of fusel oil)**

The data analysis in Table 2 and the diagrams show the advantages of cyclic rectification over typical rectification. It is experimentally proved that in the process of distillation of alcohol-containing fractions aldehydes, esters (head impurities) and acrolein are removed in full measure, and the degree of extraction *(α)* of higher alcohols of sivush oil (upper intermediate impurities) increased by 38 %, methanol – by 15.6 %. The multiplicity of concentration *(β)* of key impurities was increased by 25%, higher alcohols by 40%, methanol by 34%, acrolein by 36%, and isopropyl alcohol by 42%.

When included in the scheme of rectification plant of the impurity concentration column output of rectified ethyl alcohol increased by 3.5–3.7%, its indicators corresponded to the of regulatory documentation.

The costs of heating vapour for the process of rectification were determined from the heat balance by the cost of water for cooling and its temperature at the inlet of the condenser and the outlet of the dephlegmator of column. It was found that, in the conditions of cyclic distillation, heating vapour consumption was reduced by 30% compared to typical plants and did not exceed 12 kg/dal a.a. supplied with feed.

It is proved that for effective separation of the mixture in the mode of controlled cycles of delay and liquid overflow in the impurity concentration column, it is sufficient to install the 30 contact devices mentioned above.

From the experience of operating of the impurity concentration column, it is known that distillation residue is usually returned for a re-cycle of rectification, namely, on the top plate of the ether column (Shiyan et al., 2009). This is due to the fact that the rectification of impurities under the conditions of typical distillation does not always ensure the complete release of the residue from the key organic impurities, which are further concentrated in the process of rectification and impair the quality of the rectified ethyl alcohol. Therefore, rectification plants equipped with typical impurity concentration column require an increased consumption of heating vapour to produce a high quality final product.

The use of innovative cyclic rectification technology allows to obtain the distillation residue, as free from impurities of alcohol. When increasing the contact time of vapour and liquid on the column plates up to 30–40 s in the selected hydrodynamic mode of its operation, the total amount of impurities in the in the marketable product decreases by 94–96% of their quantity in the feedstock. It is expedient to use such distillation residue to conduct hydroselection of impurities in the ether column. The technical solution allows to reduce the consumption of hot softened water for hydroselection, the consumption of heating vapour and prevents the concentration of organic impurities concentration in the distillation unit (Buliy et al., 2019).

## Conclusions

1. Hydrodynamic modes of operation of mesh and flake plates in cyclic mode are established: vapour velocity in the free section of the column equipped with mesh plates should not exceed 0.7 m/s, flake plates – 1.2 m/s; optimum vapour velocity in the holes of the mesh plate is 7.5-8.0 m/s, flake plate – 12–14 m/s; for intensive overflow of liquid the vapour velocity in the holes should not exceed 1.5-1.0 m/s.
2. The results of production studies proved the advantages of cyclic rectification for the production of high quality alcohol: in the process of separation of alcohol-containing fractions in full measure head impurities of alcohol are allocated, the degree of extraction of the higher alcohols of sivush oil increases by 38%, methanol – by 15.6%, the multiplicity of concentration of head impurities increases by 25%, higher alcohols – by 40%, methanol – by 34%, acrolein – by 36%, isopropyl alcohol – by 42%.
3. Vapour consumption in the impurity concentration column is reduced by 40% in comparison with typical units operating in the stationary mode and does not exceed 12 kg/dal a.a. introduced with feed.

## References


Andersen B.B., Nielsen R.F., Udugama I.A., Papadakis E., Gernaey K.V., Huusom J.K., Mansouri S.S., Abildskov J. (2018), Integrated process design and control of cyclic distillation columns, *IFAC-PapersOnLine,* 51(18), pp. 542–547, https://doi.org/10.1016/j.ifacol.2018.09.368

Arslan M., Tahir H., Zareef M., Shi J., Allah A., Bilal M., Xiaowei H., Zhihua Li., Xiaobo Z. (2021), Recent trends in quality control, discrimination and authentication of alcoholic beverages using nondestructive instrumental techniques, *Trends in Food Science & Technology,* 107, pp. 80–113, https://doi.org/10.1016/j.tifs.2020.11.021

Botshekan M., Moheb A., Vatankhah F., Karimi K., Shafiei M. (2022), Energy saving alternatives for renewable ethanol production with the focus on separation/purification units: A techno-economic analysis, *Energy,* 239(PE), pp. 1–12, https://doi.org/10.1016/j.energy.2021.122363

Buliy Y., Shiyan P., Kuts A. (2016), Distillation of alcoholic distillate in controlled cycles mode, *Food Science for Well-being (CEFood 2016): 8th Central European Congress on Food 2016,* pp. 230.

Buliy Y., Kuts A., Shiyan P. (2019), Increasing of operating characteristic of cycle column mass-axchange apparatus, *Scientific Works of National University of Food Technologies,* 25(5), pp. 48–54.

Dattarajan S., Pali S., Fernandes N., Gadwal N., Hulwan D. (2019), Measurement of steam quality using a vortex flowmeter, *Flow Measurement and Instrumentation,* 65, pp. 227–232, https://doi.org/10.1016/j.flowmeasinst.2018.12.004

Dejanovic I., Matijasevic Lj., Olujic Z. (2010), Dividing wall columna breakthrough towards sustainable distilling, *Chemical Engineering and Processing: Process Intensification,* 49(6), pp. 559–580, https://doi.org/10.1016/j.cep.2010.04.001

Dewulf J. (2002), Analysis of volatile organic compounds using gas chromatography, *Trac-nrends in Analitical Chemistry,* 21(9–10), pp. 637–646, http://hdl.handle.net/1854/LU-158121

Flodman H., Timm D. (2012), Batch distillation employing cyclic rectification and stripping operations, *ISA Transactions,* 51(3), pp. 454–460, https://doi.org/10.1016/j.isatra.2011.12.003

Kiss A.A. (2014), Distillation technology – still young and full of breakthrough opportunities, *Journal of Chemical Technology and Biotechnology,* 89, pp. 479–498, https://doi.org/10.1002/jctb.4262

Kiss A., Pragt J., Strien C. (2009), Reactive dividing-wall columns – how to get more with less resources? *Chemical Engineering Communications,* 196, pp. 1366–1374, https://doi.org/10.1080/00986440902935507

Kiss A., Bildea C. (2011). A control perspective on process intensification in dividing-wall columns, *Chemical Engineering and Processing: Process Intensification,* 50(3), pp. 281–292, https://doi.org/10.1016/j.cep.2011.01.011

Kiss A., Landaeta S., Zondervan C. (2012), Cyclic distillation — towards energy efficient binary distillation, *Chemical Engineering and Chemistry,* 30, pp. 697–701, https://doi.org/10.1016/B978-0-444-59519-5.50140-4

Krivosheev V., Anufriev A. (2015), Fundamentals and efficiency of cyclic modes of rectification process. Basic Research, *Scientific Journal of Basic Research,* 11(2), pp. 267–271.

Linek V., Moucha T., Prokopova E., Rejl J.F. (2005), Simultaneous determination of vapor and liquid-side volumetric mass transfer coefficients in a distillation column,

*Chemical Engineering Research and Design,* 83(8), pp. 979–986, https://doi.org/10.1205/cherd.03311

Lita I., Bildea C., Kiss A. (2014), Catalytic cyclic distillation — A novel process intensification approach in reactive separations, *Chemical Engineering and Processing,* 81, pp. 1–12, https://doi.org/10.1016/j.cep.2014.04.006

Maleta V., Kiss A., Taran V., Maleta B. (2011), Understanding process intensification in cyclic distillation systems, *Chemical Engineering and Processing: Process Intensification*, 50, pp. 1–10, https://doi.org/10.1016/j.cep.2011.04.002

McWhirter J.R., Cannon M.R., (1961), Controlled cycling distillation in a packed-plate column, *Industrial and Engineering Chemistry*, 53, pp. 632–634, https://doi.org/10.1021/ie50620a023

Nagy E., Mizsey P., Hancsok J., Boldyryev S., Varbanov P. (2015), Analysis of energy saving by combination of distillation and pervaporation for biofuel production, *Chemical Engineering and Processing: Process Intensification,* 98, pp. 86–94, https://doi.org/10.1016/j.cep.2015.10.010

Nielsen R., Huusom J., Abildskov J., Based D. (2017), Driving force based design of cyclic distillation, *Industrial & Engineering Chemistry Research,* 56(38), pp. 10833–10844, https://doi.org/10.1016/j.cep.2015.10.010 10.1021/acs.iecr.7b01116

Pătruţ C., Bîldea C., Liţă I., Kiss A. (2014), Cyclic distillation — Design, control and applications. *Separation and Purification Technology,* 125, pp. 326–336, https://doi.org/10.1016/j.seppur.2014.02.006

Plutowska B., Wardenski W. (2008), Application of gas hromatography-olfactometry (GC-O) in analysis and quality assessment of alcoholic bewerages – a review, *Food Chemistry,* 107, pp. 449–463, https://doi.org/10.1016/j.foodchem.2007.08.058

Polulyah K., Topolov I. (2002), Determination of the optimum parameters of the autogenerator flowmeter, *Ukrainian Metrological Journal,* 4, pp. 48–50.

Premkumar R., Rangaiah G. (2009), Retrofitting conventional column systems to dividing-wall columns, *Chemical Engineering Research and Design,* 87(1), pp. 47–60, https://doi.org/10.1016/j.cherd.2008.06.013

Rasmussen J., Mansouri S., Zhang X., Abildskov J., Huusom J. (2020), A mass and energy balance stage model for cyclic distillation, *AIChE Journal*, 66(8), 1002, https://doi.org/10.1002/aic.16259

Shiyan P., Sosnytskyi V., Oliinichuk S. (2009), *Innovative technologies of the alcohol indus. Theory and practice: Monograph,* Askania Publishing House, Kyiv.

Lehotay S.J., Hajšlová J. (2002), Application of gas chromatography in food analysis, *Trac-nrends in Analitical Chemistry*, 21(9–10), pp. 686–697, https://doi.org/10.1016/S0165-9936(02)00805-1

Toftegard B., Clausen C.H., Jorgensen S.B., Abildskov J. (2016), New realization of periodic cycled separation, *Industrial & Engineering Chemistry Research,* 55(6), pp. 1720–1730, https://doi.org/10.1021/acs.iecr.5b03911

Ukrainets A., Shiyan P., Buliy Y., Kuts A. (2018), Increasing the efficiency of the distillation unit, *Scientific Works of National University of Food Technologies,* 24(6), pp. 160–166.

Pang X.N., Li Z.J., Chen J.Y., Gao L.J., Han B.Z. (2017), A comprehensive review of spirit drink safety standards and regulations from an international perspective, *Food Protection,* 3(1), pp. 431–442, https://doi.org/10.4315/0362-028X.JFP-16-319

# Cryoprotective properties of functional mixtures in cooked sausage products

**Iryna Shevchenko, Olena Tunik**

*National University of Food Technologies, Kyiv, Ukraine*

**Keywords:**

Cryoprotectant
Sausage
Cryostabilizing
Freezing

**Article history:**

Received 29.03.2024
Received in revised form 02.05.2024
Accepted 28.06.2024

**Corresponding author:**

Olena Tunik
E-mail:
tunikov@nuft.edu.ua

**DOI:**
10.24263/2310-1008-2024-12-1-5

## Abstract

**Introduction.** It is studied the effect of the cryostabilizing mixture in the composition of cooked sausages on the formation and stabilization of their functional, technological, structural, mechanical, and sensory characteristics under conditions of long-term storage in a frozen state.

**Materials and Methods.** The sausage was prepared using a cryostabilizing mixture with a content of 2.0, 2.5, 3.0 and 3.5 %. The control sausage does not contain a cryostabilizing mixture. The cryostabilizing mixture comprised animal protein, bamboo fiber, wheat fiber and sodium alginate in a ratio of 1:0.5:0.5:0.5. The sausages were frozen at –18 °C for 30 days.

**Results and discussion.** The addition of the cryostabilizing mixture to minced meat systems significantly improves their moisture retention (by 9.7–17.3%) and fat retention (by 9.4–9.7%), and increases the stability of the systems by 15.7–16.5%. The use of a mixture of 2.5–3% provided better sensory characteristics, including greater juiciness and structure density. Weight loss during defrosting and heat treatment decreased by 10.24–14.67%, and water activity decreased by 0.048, which contributed to the shelf life of sausage products. The cryostabilizing mixture also reduces the cryoscopic temperature of meat systems by 2.82–4.52°C and improves the structural and mechanical properties of products after defrosting.

The most significant changes in sensory quality indicators: insufficient juiciness, fragility of the structure, lower yield, and higher losses during heat treatment by 6.18–7.25% were observed in control samples after freezing, storage for 30 days, and thawing.

The best structural and mechanical properties (penetration stress 27.37–27.63 Pa) were obtained for thawed samples of sausage products with the addition of a cryostabilizing mixture in the amount of 2.5%–3.5%. The consistency and density of such products almost did not differ from the consistency of chilled products, and single stratifications of the structure were visible in their section, but in a very small amount.

**Conclusions.** The study proved that the use of 2.5–3% cryostabilizing mixture improves the sensory and structural and mechanical properties of cooked sausages.

## Introduction

One of the promising areas for minimizing negative changes in meat products during freezing is the use of cryoprotective food ingredients that prevent the formation of large ice crystals and tissue dehydration (Simakhina et al., 2024; Sun et al., 2022). It has been shown that a composite cryoprotective mixture in the amount of 3% reduces the cryoscopic temperature of minced meat systems by 2.09–2.81°C, reduces the mass fraction of frozen moisture by 1.7%, and reduces the water activity index by 0.031–0.067 (Shevchenko et al., 2020).

The mechanism of action of these ingredients is to reduce the number of crystallization centers, which is especially important for sausage products stored for a long time at low temperatures (minus 18 °C and below) (Tian et al., 2021). The expediency of using hydrocolloids (carrageenans, xanthan gum, guar gum, sodium alginate, etc.) in meat product technologies has been established to improve their functional and technological properties and sensory characteristics (Dromenko et al., 2021; Shevchenko et al., 2020). Such ingredients provide an increased yield of meat products, improve their consistency, and extend shelf life (Ramadhan et al., 2012; Stabnikova et al., 2022).

Cryoprotectants also help to reduce water mobility in minced meat systems, which affects the process of ice formation, increasing the plasticity of products and ensuring the stabilization of their quality characteristics after freezing (Keniyz, 2014; Lee et al., 2002). During the storage of frozen meat products, proteins are denatured and aggregated, which leads to the loss of their functional properties. This process is exacerbated by the formation of intracellular ice and changes in salt concentration during water freezing (Castro-Giraldez et al., 2014; Yancheva et al., 2014). The use of cryostabilizing ingredients allows to protect protein macromolecules from denaturation, maintaining their functional and technological properties, in particular, emulsifying and moisture-binding capacity (Sharpe et al., 2009; Tuan Pham, 2014).

Studies have also confirmed the effectiveness of dietary fiber (wheat, plantain, etc.) in protecting minced meat systems from cellular degradation during freezing due to their hydrophilic properties (Castro-Giraldez et al., 2014; Tomaniak et al., 1998). In addition, polysaccharides, such as sodium alginate, due to their gel-forming properties, reduce the rate of water crystallization, which reduces osmotic drops and protects cells from destruction. The use of these components in sausage stuffing systems is a prerequisite for maintaining their quality and extending shelf life (Keniyz, 2014; Sapiga et al., 2021).

The effect of freezing on the quality of sausages is not well understood, and this requires additional research. It is necessary to substantiate the method of minimizing the negative effects of freezing on cooked sausages, to investigate the cryostabilizing ability of the mixture in model samples, and to assess its effect on the quality of sausages during long-term frozen storage.

The aim of research is to study the cryostabilizing ability of the mixture in the composition of cooked sausages for the purposeful formation and stabilization of their functional, technological, structural, mechanical and sensory characteristics under conditions of long-term storage in a frozen state.

## Materials and methods

### Cryostabilizing mixture

Composition of protein-polysaccharide cryostabilizing mixture: animal protein "ScanPro™ A-95" (Essentia) – a highly functional cold-brewed animal protein made from natural food pork raw materials by mechanical and heat treatment, dietary fiber: bamboo and wheat (Shandong Jianyuan Foods Co, Ltd.), sodium alginate (E401) (Shandong Jiejing Group Corporation) in a ratio of 1:0.5:0.5:0.5. The degree of hydration of the protein-polysaccharide composition was 1:6.

### Recipe of the tested samples of cooked sausages

The sausages were made on the basis of pork (20%), chicken fillet (20%), minced chicken (20%), beef vein emulsion (20%) and lard (20%). In the production of minced meat for the cooked sausage test samples, the protein-polysaccharide mixture was added at 2.0, 2.5, 3.0 and 3.5% to replace the same amount of chicken fillet.

### Preparation of sausage samples

The boiled sausage mince samples were ground with ice in a cutter to a temperature of 12 °C for 15 min, formed into a 55 mm diameter protein sausage casing, and heat-treated in a thermal chamber according to the heat treatment programme for boiled sausages in a natural protein casing. The finished sausage products were cooled to 12 °C for 8 h and frozen at minus 18 °C. The shelf life of the sausage samples at minus 10 °C was 30 days. The heat treatment of the experimental sausage samples, until the temperature in the centre reached 70–72 °C, was carried out before freezing and after defrosting. Defrosting was carried out at a temperature of 20±2 °C for 1.5–2 hours.

### Methods for studying properties of cryostabilizing mixture and cooked sausages and optimizing their composition

The sensory parameters were determined in all samples before and after heat treatment: appearance, cut appearance, smell, taste, color, juiciness, and consistency (Savinok et al., 2017). Physicochemical (pH, moisture, protein, ash, fat content), functional and technological (emulsion stability, moisture binding capacity, emulsifying capacity, fat retention capacity, finished product yield), structural and mechanical (penetration stress), and water activity ($a_w$ ) parameters were also studied.

Sausage products were manufactured and tested in accordance with raw material restrictions that regulate the use of quality meat raw materials, permissible additives, moisture, fat, protein levels, and technological processes to ensure the safety and quality of sausage products.

**Determination of the mass fraction of moisture.** The mass fraction of moisture was determined according to the ISO 1442:1997 method used for meat and meat products. This method involves drying a sample of meat or meat product at a specific temperature to a constant weight. The weight loss of the sample during drying is considered the amount of moisture.

**Determination of the mass fraction of fat.** The mass fraction of fat was determined according to the ISO 1443:1973 method used for meat and meat products. The method involves the extraction of fat from meat or meat product using an organic solvent. After extraction, the fat is determined by measuring its mass.

**Determination of the mass fraction of protein.** The mass fraction of protein was determined by the Kjeldahl method "Agricultural food products. General guidelines for the determination of nitrogen content by the Kjeldahl method (ISO 1871:1975, IDT)".

**Determination of the mass fraction of minerals.** The mass fraction of minerals is determined by the gravimetric method after burning organic matter in a muffle furnace at a temperature of 500–700°C for 5–6 hours to a constant mass.

**Determination of sensory characteristics of sausages.** Sensory characteristics of sausages: the method is to assess the quality of sausage products according to five criteria: appearance, consistency, color in the cut, smell and taste. Each of these indicators is rated on a five-point scale, where 5 is excellent and 1 is unsatisfactory. The assessment is carried out by a group of experts who analyze the product according to the established parameters to determine its compliance with quality standards.

**Determination of Water-Binding Capacity (WBC).** The water-binding capacity of the research objects was determined using the Grau-Hamm press method, modified by Volovynska and Kelman. The method is based on the extraction of water from a 300 mg sample during a 10-minute pressing with a weight of 1 kg. The determination is carried out by measuring the size of the spot left on filter paper after the absorbed moisture is released. The outline of the spot from the pressed meat is drawn with a pencil. The size of the wet spot (outer) is calculated as the difference between the total area of the spot and the area formed by the meat (product). The WBC content is calculated using the formula:

$$WBC = \frac{(A - 8.4B) \times 100}{A}$$

where: WBC – the water-binding capacity, % of total moisture;
A – total moisture content in the sample, mg;
B – wet spot area, ($cm^2$).

**Determination of emulsion stability.** The emulsion stability (ES) of the coarse raw material was determined by heating at 80°C for 30×60s and cooling with water for 15×60s. Then, four 50 $cm^3$ calibrated centrifuge tubes were filled with the emulsion and centrifuged at a rotational speed of 500 $s^{-1}$ for 5×60s. The volume of the emulsified layer was then determined. The stability of the emulsion was calculated by the formula:

$$ES = \frac{V_1}{V_2} \times 100$$

where:
$V_1$ – the volume of emulsified oil, $cm^3$;
$V_2$ – total volume of the emulsion, $cm^3$.

**Determination of Emulsifying capacity.** The emulsifying capacity (EC) was determined after centrifuging a mixture of oil, water, and emulsion at a rotation speed of 500 $s^{-1}$ for 10×60 s. The volume of the emulsified oil was then measured, and the emulsifying capacity was calculated using the following formula:

$$EC = \frac{V_1}{V_2} \times 100$$

where: $V_1$ – volume of emulsified oil, $cm^3$;
$V_2$ – total volume of oil, $cm^3$.

**Determination of the penetration stress**. Penetration stress of the sausages was determined using a Brookfield DV1 digital viscometer by the depth of the indenter immersion in the test sample at 20°C. Three measurements were made on the open surface of the sample at a distance of at least 10 mm from the edge of the product and at the maximum distance from the points of other measurements so that the deformed part of the surface did not enter the measurement area, after which the penetration value was converted to the penetration stress value.

**Determination of water activity.** The water activity ($a_w$) of the model minced meat systems and sausages was determined using a rotronic Hygro Palm-23 analyzer. The cryoscopic temperature of the model minced systems and sausages was measured using the method of thermal analysis based on the construction of temperature change curves over time.

## Results and discussion

### Optimisation of the degree of replacement of meat raw materials with a cryostabilising mixture of animal proteins, polysaccharides and dietary fibers

In order to offset the negative effects of freezing on the functional and technological properties of low-functional meat raw materials and cooked sausages, as well as to prevent significant crystal formation and slow down the freezing process, the cryoprotective properties of a protein-polysaccharide mixture based on animal proteins, polysaccharides and plant fiber were investigated (Yancheva et al., 2014). Protein and vegetable fibers, such as bamboo and wheat, were chosen as cryostabilising components. Protein products, as high molecular weight substances, are able to reduce the growth rate of crystals and protect muscle cells from osmotic and temperature changes (Shevchenko et al., 2018). In addition, they can stabilise meat systems during storage.

Adding vegetable fiber to products subject to freezing and thawing can improve their texture, retain moisture and ensure consistent quality of the final product (Petracci et al., 2013). The sodium salt of alginic acid was used as a polysaccharide component. It is known that sodium alginate and bamboo and wheat fibres improve the consistency of food products, adsorb water, reduce weight loss, and enrich them with ballast substances (Yancheva et al., 2014). The principle of action of these substances is based on the formation of an amorphous structure in the food system, a decrease in the number of crystallisation centres and a decrease in the water activity index.

### Study of chemical composition and functional and technological properties

In order to develop recommendations for the use of the mixture as a cryostabilising ingredient, the chemical composition and functional and technological properties (FTP) of model minced meat systems with different levels of its use were investigated. The results of the study are presented in Table 1.

**Table 1**

**Chemical composition and functional and technological properties of model minced meat systems using the mixture (n=3)**

| Indicators | Samples | | | | |
|---|---|---|---|---|---|
| | Control | Prototypes with the mixture, % | | | |
| | | 2.0 | 2.5 | 3.0 | 3.5 |
| Mass fraction of moisture, % | 72.03±3.14 | 71.47±3.35 | 71.50±3.40 | 71.67±3.39 | 71.56±3.41 |
| Mass fraction of moisture protein, % | 10.04±0.23 | 10.21±0.41 | 10.26±0.28 | 10.30±0.31 | 10.31±0.19 |
| Mass fraction of fat, % | 14.16±0.81 | 14.64±0.77 | 14.62±0.71 | 14.43±0.79 | 14.54 ±0.78 |
| Mass fraction of carbohydrates, % | 3.01±0.01 | 2.91±0.01 | 2.85±0.01 | 2.82±0.01 | 2.81±0.01 |
| Mass fraction of ash, % | 0.76±0.02 | 0.77±0.02 | 0.77±0.01 | 0.78±0.02 | 0.78±0.02 |
| pH | 6.13±0.14 | 6.15±0.21 | 6.16±0.17 | 6.17±0.19 | 6.17±0.07 |
| Water-binding capacity, % | 78.05±3.62 | 79.11±3.14 | 79.85±3.6 | 80.10±3.7 | 81.14±3.5 |
| Water retention capacity, % | 70.90±3.05 | 74.35 ±3.16 | 75.17±3.1. | 76.01±3.0 | 76.79±3.0 |
| Fat retention capacity, % | 68.78±3.28 | 76.40±3.2 | 76.52±3.1 | 77.63±3.4 | 77.57±3.4 |
| Emulsifying capacity,% | 69.80±3.24 | 79.90±3.3 | 79.86±3.1 | 79.91±3.3 | 79.88±3.4 |
| Emulsion stability, % | 87.30±3.15 | 89.40±3.3 | 92.95±3.1 | 96.40±3.2 | 96.06±3.5 |
| Penetration stress, Pa | 117.25±0.75 | 24.87±0.95 | 27.37±1.06 | 27.54±1.08 | 27.63±1.07 |
| Yield of the finished product, % | 117.58±3.1 | 118.16±3.1 | 119.30±3.3 | 120.78±2.1 | 121.08±2.3 |

The introduction of the mixture into minced meat systems as a cryostabilising ingredient has a positive effect on increasing the moisture retention (by 3.45–5.89 %) and fat retention (by 7.62–8.79 %) capacities of model meat systems, which improves the structure of model samples of cooked sausages. When the gelling temperature of the selected polysaccharides approaches the denaturation temperature of meat proteins, water separates from the proteins and is absorbed by the protein-polysaccharide complexes. This creates stable protein-polysaccharide complexes.

An important indicator that determines the quality characteristics of cooked sausages is the stability of minced systems. This indicator characterises the content of bound water and fat in the meat system (Sharpe et al., 2009). The results of the study indicate an increase in

the stability of the meat minced systems of the experimental sausage samples by 2.1–8.76 % compared to the control sample.

When determining the losses after heat treatment of cooked sausage samples, it was found that a higher yield (121.08 %) was characteristic of samples with a mixture of 3.5 %.

The most significant changes in sensory quality indicators (insufficient juiciness, fragility of the structure, lower yield, higher losses during heat treatment by 14.66–14.75 %) were observed in control samples after freezing, storage for 30 days and defrosting.

**Study of sensory properties**

According to the results of the sensory study of model samples of cooked sausages, it was found that the use of the developed cryostabilising mixture does not lead to noticeable changes in the sensory properties of meat systems. The best score, in comparison with the control sample, was given to sausage samples with a mixture content of 2.5–3.0 %, which were characterised by greater juiciness and structural strength, while samples with a mixture content of 3.5 % had too dense a consistency, were not juicy enough and had a worse taste (Figure 1).

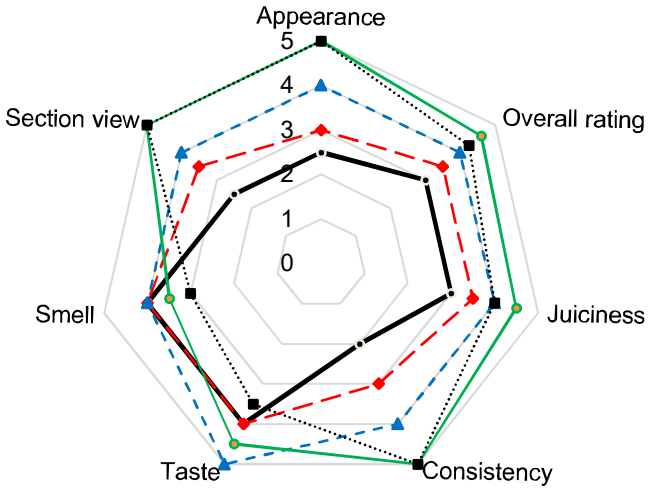


**Figure 1. Quality profiles of cooked sausage samples by sensory characteristics and recipe composition**

**Study of mass loss after heat treatment**

When determining the mass loss of heat-treated model samples of cooked sausages, it was found that the use of a cryostabilising mixture in the amount of 2.0 to 3.5 % in their composition can reduce mass loss regardless of the type of processing. Thus, the weight loss

of the experimental samples of cooked sausages after defrosting and heat treatment was reduced by 10.24–14.67 %, respectively, compared to the control sample (Table 2).

**Table 2**

**Weight loss of heat-treated model minced sausage systems with cryostabilising mixture (n=3)**

| Type processing | Weight loss, %, | | | | |
|---|---|---|---|---|---|
| | Control | Samples with mixture content, % | | | |
| | | 2.0 | 2.5 | 3.0 | 3.5 |
| Heat treatment before freezing | 29.33 ±1.12 | 20.25 ± 0.93 | 17.50 ±0.78 | 16.23 ±0.78 | 16.42 ±0.79 |
| Freezing | 2.64 ±0.12 | 1.96 ±0.09 | 1.76 ±0.07 | 1.65 ±0.08 | 1.66 ±0.08 |
| Heat treatment after defrosting | 33.68 ±1.39 | 23.44 ±1.14 | 20.49 ±0.98 | 18.93 ±0.92 | 19.02 ±0.90 |

Thus, it can be concluded that the use of a cryostabilising mixture in an amount of 2.5–3 % can reduce the amount of frozen moisture to a minimum during the storage period stipulated by the technology at a temperature of minus 18 °C.

**Determination of water activity and cryoscopic temperature**

The preservative effect of the freezing process is aimed at reducing the water activity $a_w$ , which contributes to the shelf life of meat systems (Sharpe et al., 2009). The use of the cryostabilising mixture as a cryostabilising ingredient in the model minced boiled sausage systems also reduces the water activity index $a_w$ by 0.048 compared to the control sample. The dynamics of changes in the water activity index of meat minced systems and the cryoscopic temperature of heat-treated cooked sausage samples after 30 days of storage are shown in Figure 2.

A decrease in the temperature of the onset of moisture crystallisation in minced meat systems and, accordingly, a change in the nature of the process of water crystallisation in the cellular structure of muscle tissue at an increased content of the cryostabilising mixture in the experimental samples also causes a decrease in the water activity index $a_w$ (Keniyz, 2014; Tuan Pham, 2014).

The obtained results confirm the expediency of using a cryostabilising mixture in the amount of 2.5–3 % as a composition of ingredients whose actions are aimed at cryoprotecting minced meat systems from the effects of low temperatures and reducing the water activity index $a_w$.

The protein-polysaccharide mixture also helps to reduce the cryoscopic temperature of minced meat systems by 3.82–4.52 °C and increases the moisture retention capacity by 3.45–5.89 % compared to the control sample, which has a positive effect on the quality of finished products.

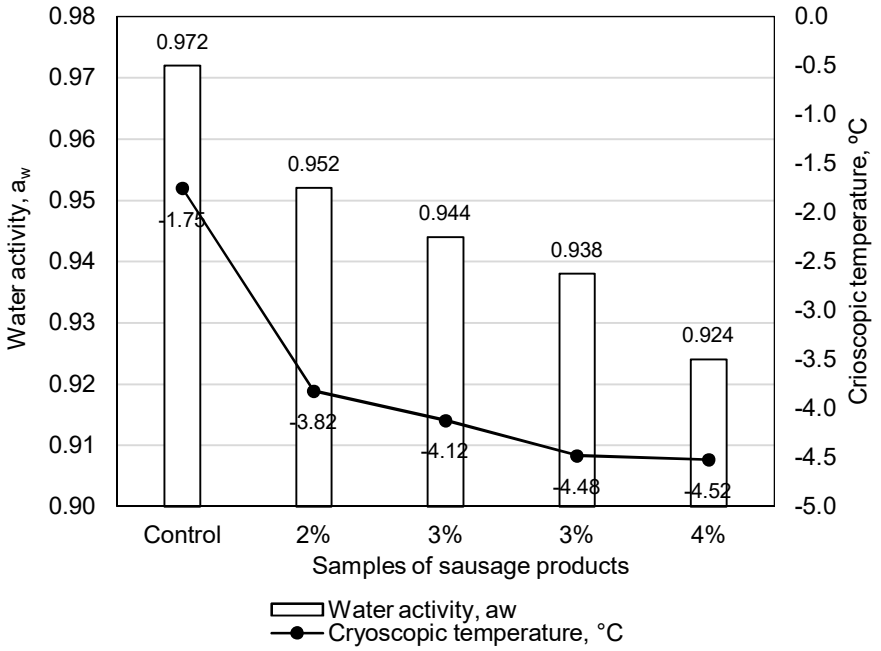


**Figure 2. Dynamics of changes in the water activity index in minced meat systems and cryoscopic temperature of heat-treated samples of cooked sausages after 30 days of storage**

**Impact of cryostabilising mixtures on the quality of sausage products**

According to the results of optimizing the composition of cooked sausages (Table 1), the range of the content of the cryostabilizing mixture in minced meat systems as a cryostabilizing ingredient is in the range of 2.5 to 3 %, which increases the moisture retention (by 3.45–5.89 %) and fat retention (by 7.62–8.79 %) capacities of model meat systems and improves the structure of model samples of cooked sausages. The crystallization process helps to retain moisture in meat products during heat treatment. This allows the product to retain its juiciness and texture, as the water that is usually released when proteins are heated does not escape from the product but is absorbed by the polysaccharides. The results of the study characterize the ability of meat systems to absorb and retain moisture during heat treatment (Tuan Pham, 2014). The determination of losses during heat treatment of model minced meat systems showed that a higher yield is typical for samples with the use of a cryostabilizing mixture in the range of 2.5 to 3 %. This effect can be explained by better retention of immobilized moisture during the heating process due to the capillary effect characteristic of dietary fiber and the high processing properties of thermostable proteins (Keniyz, 2014; Yancheva et al., 2018). The most significant changes in sensory quality indicators (insufficient juiciness, fragility of the structure, lower yield, higher losses during heat treatment by 14.66–14.75 %) were observed in control samples after freezing, storage for 30 days and thawing.

When water freezes, the concentration of soluble compounds in aqueous solutions of meat systems changes. This, in turn, affects changes in pH and the strength of ionic interactions in the layer close to the protein molecule. This phenomenon is a consequence

not only of dehydration and aggregation, but also of the breakdown of glycogen remaining in the meat before freezing and the formation of lactic acid (Petracci et al., 2013; Yancheva et al., 2018).

**Role of cryostabilising mixture in the process of cryopreservation**

The use of cryostabilising mixtures in minced meat systems before the freezing process, due to its hydrophilic properties, can reduce the negative impact of physical and chemical factors and protect meat systems from cell destruction during cryopreservation. In addition, cryopreservation can slow down oxidative processes, prevent denaturation of proteins, molecular complexes and pigments, and deflavour the raw materials.

The research proves the functional, technological and cryostabilising properties of the cryostabilising mixture based on the selected ratio between highly functional animal protein, sodium alginate, bamboo and wheat fibre, which increases the moisture retention, fat retention and emulsifying capacity and forms the stable properties of model minced meat systems and cooked sausages.

Among all the experimental samples of chilled and heat-treated sausage products, the greatest changes in sensory quality indicators (after freezing, storage for 30 days and defrosting) were observed in the control samples after defrosting and heat treatment, which were characterised by insufficient juiciness fragility of the structure, deterioration of consistency due to the formation of large moisture crystals, which destroyed the consistency of the product during freezing, contributed to the stratification and deterioration of the product structure during defrosting and affected the decrease in yield and higher losses by 2.9– 3.1 % higher losses during heat treatment. However, the experimental control samples in the chilled state, before freezing, had better taste properties compared to the taste properties of the samples with the addition of the cryostabilising mixture in the chilled and thawed state. This confirms that the addition of sodium alginate can worsen the taste properties of sausage products, so its use should be well justified and optimally selected in terms of dosage.

**Reasonability of use**

The best structural and mechanical properties were observed in the thawed samples of sausage products with the addition of a cryostabilising mixture in the amount of 2.5 % and 3 %. The consistency and density of such products were almost the same as those of products that were not subjected to freezing, and single stratifications of the structure were visible in their section, but in a very small amount.

Thus, the results obtained confirm the feasibility of using a cryostabilising mixture in the manufacture of sausage products as a substance whose action is aimed at stabilising sausage minced systems and their cryoprotection against low temperatures during storage in the frozen state of such products at low sub-zero temperatures.

Thus, there are two promising areas for the use of the cryostabilising mixture in the production of cooked sausages to be stored frozen for a long time:

- stabilisation of functional and technological properties of meat raw materials and minced systems;
- cryostabilisation of minced meat systems when using cooked sausages for long-term storage in the thawed state.

## Conclusions

1. The use of a cryostabilising mixture with a selected ratio between highly functional animal protein, sodium alginate, dietary fibers of bamboo and wheat fibre can stabilise the sensory and structural and mechanical properties of cooked sausages, help reduce weight loss during heat treatment and increase their yield.
2. The protein-polysaccharide mixture reduces the cryoscopic temperature of minced meat systems by 3.82–4.52 °C and increases the moisture retention capacity by 3.45–5.89% compared to the control sample, which has a positive effect on the quality of finished products.
3. Due to the cryoprotective effect of the ingredients of the cryostabilising mixture in the composition of minced meat systems in the amount of 2.5–3%, the water activity index $a_w$ decreases by 0.048, which helps to extend the shelf life of sausage products of meat systems.
4. Due to their hydrophilic properties, animal protein, sodium alginate, dietary fibres of bamboo and wheat fibre can reduce the harmful effects of physicochemical factors and protect the minced systems of cooked sausages from cell destruction during cryopreservation, so they can be used in the production of cryostabilising mixtures for meat products.

**Acknowledgments.** The work was carried out within the framework of the state research work "Improvement of resource-saving technologies for meat and meat-containing products using targeted refrigeration" (PH 0120U103106)

## References

Castro-Giraldez M., Balaguer N., Hinarejos E., Fito P.J. (2014), Thermodynamic approach of meat freezing process, *Innovative Food Science & Emerging Technologies*, 23, pp. 138–145, https://doi.org/10.1016/j.ifset.2014.03.007

Dromenko O., Potapov V., Yancheva M., Onishchenko V., Bolshakova V., & Inzhyyants A. (2021), Research into the hermophysical characteristics of muscle and adipose tissues in the freezing–thawing process, *Food Science and Technology*, 15(3), https://doi.org/10.15673/fst.v15i3.2120

Keniyz N.V. (2014), *Technology of frozen semi-finished products using cryoprotectants*, Palmarium Academic Publishing, Saarbrücken,

Lee Ch. M., Lian P. (2002), Cryostabilization of unwashed fish mince, *Fisheries Science*, 68, pp. 1355–1358, https://doi.org/10.2331/fishsci.68.sup2_

Petracci M., Bianchi M., Mudalal S., Cavani C. (2013), Functional ingredients for poultry meat products, *Trends in Food Science & Technology*, 33(1), pp. 27–39, https://doi.org/10.1016/j.tifs.2013.06.004

Ramadhan K., Huda N., Ahmad R. (2012), Freeze-thaw stability of duck surimi-like materials with different cryoprotectants added, *Poultry Science,* 91(7), pp. 1703–1708, https://doi.org/10.3382/ps.2011-01926

Savinok O., Novgorodska N., Ovsienko S. (2023), Development of technology of cooked sausages with a changed fatty acid composition for military personnel in the armed forces of Ukraine, *Eastern-European Journal of Enterprise Technologies*, 6(11(126)), pp. 24–32, https://doi.org/10.15587/1729-4061.2023.292777

Savinok O., Pastyukov S., Kuzelov A., Shepelenko D. (2017), Sensory and functional parameters of sausages with reduced sodium nitrite content, *Scientific Works of University of Food Technologies*, 64(1), pp. 32–40.

Simakhina G., Naumenko N., Kaminska S. (2024), Changes in vitamin content and sensory characteristics of frozen wild berries during storage, *Ukrainian Food Journal*, 13(1), pp. 60–75, https://doi.org/10.24263/2304-974X-2024-13-1-5

Sapiga V., Polischuk G., Buniowska M., Shevchenko I., OsmakT. (2021), Polyfunctional properties of oat β-glucan in the composition of milk-vegetable ice cream, *Ukrainian Food Journal*, 10(4), pp. 691–706, https://doi.org/10.24263/2304-974X-2021-10-4-5

Stabnikova O., Danylenko S., Kryzhska T., Shang F., Duan Z. (2022), Effects of different phosphate content on the quality of wheat bran chicken sausage, *Ukrainian Food Journal*, 11(4), pp. 588–600, https://doi.org/10.24263/2304-974X-2022-11-4-8

Sharpe A.A., Azarov N.G., Yankova E.D., Bliznyuk A.A. (2009), The effect of freezing on the functional and technological properties of meat systems, *Food Science and Technology*, 2(7), pp. 12–14.

Shevchenko I., Skochko A. (2018), Advantages of using proteins in the production of truncated semi-finished products, *Food and Environment Safety*, 12(3), pp. 272–277.

Shevchenko I., Polishchuk G., Kotliar Y., Osmak T., Skochko A. (2020), Prospects of using the crystobilizing protein-polysaccharide composition to manufacture semi-finished chopped meat products, *Food Science and Technology*, 14(1), pp. 134–141, https://doi.org/10.15673/fst.v14i1.1642

Skochko O., Druhoveiko V., Shevchenko I., Maslikov M. (2018), The study of cryoprotective properties of protein-polysaccharide mixtures in the composition of chopped semi-finished products, *NAUKh Scientific Papers*, 24(5), pp. 203–207.

Tomaniak A., Tyszkiewicz I., Komosa J. (1998), Cryoprotectants for frozen red meats, *Meat Science*, 50(3), pp. 365–371, https://doi.org/10.1016/S0309-1740(98)00043-6.

Tian J., Walayat N., Ding Y., Liu, J. (2022), The role of trifunctional cryoprotectants in the frozen storage of aquatic foods: Recent developments and future recommendations, *Comprehensive Reviews in Food Science and Food Safety*, 21, pp. 321–339, https://doi.org/10.1111/1541-4337.12865

Tuan P.Q. (2014), Freezing time formulas for foods with low moisture content, low freezing point and for cryogenic freezing, *Journal of Food Engineering*, 127, pp. 85–92, https://doi.org/10.1016/j.jfoodeng.2013.12.007

Sun X., Wu Y., Song Z., Chen X., (2022), A review of natural polysaccharides for food cryoprotection: Ice crystals inhibition and cryo-stabilization, *Bioactive Carbohydrates and Dietary Fibre*, 27(100291), https://doi.org/10.1016/j.bcdf.2021.100291

Xiong Y.L., Mikel W.B. (2001), Meat and meat products, *Meat Science and Applications*, pp. 351–369, https://doi.org/10.1201/9780203908082.pt4

Yancheva M., Zheleva T., Pogozhych N., Hrynchenko A. (2014), Cryoscopy research of solutions of food ingredients of polysaccharide nature, *Eastern-European Journal of Enterprise Technologies*, 2(12), pp. 84. https://doi.org/10.15587/1729-4061.2014.23513

Yancheva M., Dromenko E., Potapov V., Hrynchenko O. (2018), Study of influence of freezing-defrosting on thermophysical properties of meat systems, *EUREKA: Life Sciences*, 1, pp. 32–38, https://doi.org/10.21303/2504- 5695.2018.005370.

# Effect of compression modes on tablet strength and friability

**Oleksandr Zomenko, Oleksii Gubenia**

*National University of Food Technologies, Kyiv, Ukraine*

**Keywords:**

Tablet
Compression
Quality
Strength
Friability

**Article history:**

Received 11.02.2024
Received in revised form 28.05.2024
Accepted 28.06.2024

**Corresponding author:**

Oleksandr Zomenko
E-mail: zemenkoaleksandr@gmail.com

**DOI:**
10.24263/2310-1008-2024-12-1-6

## Abstract

**Introduction.** The aim of study was to determine the effect of the number of compression cycles and rotary table speed on the strength and friability of tablets.

**Materials and methods.** Two tablet samples manufactured on an industrial rotary pressing machine with a rotary table speed of 30 to 80 rpm were studied. Tablet strength was assessed by the breaking stress in the diametrical direction, and abrasion was measured by the percentage of mass loss during processing in a rotating drum.

**Results and discussion.** With an increase in the number of pressing cycles from 28,000 to 280,000, which corresponds to approximately 8 to 80 hours of machine operation, the tablet strength decreases to 10% of the initial value of 1.02–1.52 MPa. The decrease in strength occurs according to a parabolic dependence - during the first 100,000–150,000 cycles (30–35 hours of press operation) it is insignificant, and then accelerates. Under the conditions of this study, the process should be stopped after 200,000–250,000 pressing cycles, that is, after approximately 60–70 hours of press operation.

An increase in the friability value was observed with increasing the compressing cycles: at the beginning of the experiment, the percentage of friability was 0.18–0.30%, at the end of the experiment it increased to 1.19–1.24%. When the rotation speed of the rotary table increases from 35 to 80 rpm, the percentage of friability of tablets increases from 0.15 to 1.24%, and the strength was decreases from initial 2.3–3.5 to 10% with linear dependence. The value of tablets friability was higher than pharmacopoeia recommendation (1%). The decrease in tablet quality is explained by product sticking to the press tool surface, increased surface roughness, friction coefficient, and adhesive strength. As a result, the outer layer of the tablet had lower strength properties and was subject to rapid destruction. The reduction in strength at a higher rotation speed of the rotary table is explained by a decrease in the holding time in the die and a higher speed of pushing out of the tablet, which reduces the strength of its outer layers. Increased friability of the tablet affects subsequent technological operations: dust accumulates in the tablet feed channels for blister packaging, making it difficult to move them, and the tablets lose weight when applying a coating on drum machines. A decrease in the strength of the tablet leads to its fragmentation during various technological operations and storage.

**Conclusions.** For the tablets used in the study, the optimum table rotor speed was 50 rpm. Increased rotation speed and productivity can be achieved by more frequent polishing of the die and punch surfaces.

## Introduction

When pressing on rotary machines, deviations in the strength and friability of tablets may be observed, which leads to a violation of their technological parameters (Siiriä et al., 2011), namely, in their integrity during subsequent coating, packaging, transportation and storage operations (Herasymenko et al., 2024; Natoli et al., 2017), can affect product safety (Gordiienko et al., 2015), and affect the profitability of production.

It is obvious that the strength and friability of tablets are primarily influenced by the technology and degree (effort) of tablet pressing. However, these indicators can also be affected by the operating parameters of the pressing equipment, in particular, the rotation frequency of the rotary table of the pressing machine, and the number of pressing cycles, i.e. the number of tablets produced in one press pair "die-punch" (Herasimenko et al., 2024). The influence of these factors is not sufficiently described in the scientific literature, so the issue requires additional research.

There is disagreement about the terminology characterizing the strength properties of a tablet. The terms "hardness", "strength", "tensile strength", "breaking stress" are used. Most researchers consider the term "strength" to be correct, characterizing the stress at which a tablet breaks under lateral (diametrical) compression. Note that "strength" cannot be called "hardness": from a mechanical point of view, these are different quantities measured by different methods.

Usually, at determining the strength, the shape of the tablet is not paid attention to, however, it has a scientific interest the determination of stresses in tablets of different shapes – from the usual flat (cylindrical) to more complex ones: convex, capsule-shaped, oval, and others. These problems are solved by mathematical modelling, and more recently by using software packages based on the finite element method (Pitt et al., 2016; Siiriä et al, 2011).

To characterize such a phenomenon as the separation of small dust-like particles from a tablet, the terms "scraping", "friability", "abrasion", “wearability” are found, and the incorrect terms "fragility" and “brittleness” is occurred. Brittleness is the property of a product to fracture with little elastic deformation and without significant plastic deformation (Goots et al., 2016; Rösler et al., 2007), this term is relevant for ceramic products, glass, concrete, etc., but it cannot characterize the loss of tablet mass due to the separation of its small particles. The term "friability" is commonly used and corrected.

The value of tablet strength is usually not regulated by external regulatory documents and the Pharmacopoeias (Pharmaeducation, 2024), and is set by the drug manufacturer (CDER, 2015). However, the deviation from the accepted strength should not exceed 5%. Usually, the lateral force at which the tablet breaks is between 40 and 100 N, and the strength derived from it depends on the size of the tablet (Pharmaeducation, 2024).

According to the harmonized European Pharmacopoeia, the value of friability should not exceed 1% (European pharmacopoeia, 2024), however, some researchers consider this value to be overestimated and recommend a friability value of 0.3–0.5% (Podczeck, 2007).

The strength and friability of tablets are influenced by the technology, degree of pressing, type and modes of operation of the equipment, the geometrical parameters and state of the pressing tool surface. Taking into account a significant number of random factors, the strength and friability of tablets are supported technologically, and instrumentally – by setting the pressing tool and the operating modes of the equipment.

Most researchers pay attention to the dependence of strength and friability only on the technology of tablets, compression force or degree of pressing. For example, with an increase in compressing force (and, accordingly, the degree of pressing) from 8 to 30 kN, the strength of the tablet increases from 0.2 to 1.4 kPa; with high compressing force, the strength of the tablet changes slightly (Pitt et al., 2013). However, the experience of engineers and adjusters of compressing equipment indicates that strength, friability and other quality indicators are affected by the kinematic parameters of the press and its operation time. During several workdays of the compression, the tablet strength may decrease by more than 10%, and the friability may exceed 1% (Herasymenko et al., 2024). Similar indicators of failure occur at high speeds of the rotary table. Therefore, the issue of regulating the structural-mechanical and quality indicators of tablets by changing the operating parameters of the equipment and the condition of the surface of the pressing tool requires additional research.

The aim of the study was to determine the effect of the number of compressing cycles and the rotational speed of rotary table of the press on the strength and friability of tablets.

## Materials and methods

The strength and friability of tablets were studied. This study is not medical one, does not make any medical claims, and is intended solely to determine the effect of pressing process parameters on tablet strength and friability.

### Materials

Strength of two well-known medicines in the form of tablets were studied:

- Tablets 1 were made on the basis of: citric acid monohydrate, potato starch, povidone, cocoa, calcium stearate, and API: acetylsalicylic acid, paracetamol, and caffeine.
- Tablets 2 were made on the basis of: potato starch, talc, calcium stearate, and AFI sodium metamizole.

### Equipment

The tablets were produced under industrial conditions on a Korsch XL 400 tablet compression machine. The machine provides the possibility of working with a value of preliminary pressing of 20 kN and main pressing of 100 kN, a maximum productivity of 338,000 tablets per hour at a maximum rotary table speed of 120 rpm.

The schematic diagram of the tablet compression machine is shown in Figure 1. The main operations – filling the die with granulate, preliminary and main compression of the tablet, pushing out the tablet.

During the study of tablets for strength, the tablets were taken every eight hours of continuous operation of the press (or every 28.2 thousand finished tablets per one die). For each study, 10 tablets were selected.

During the study of tablets for friability, the rotary table speed was 35, 50, 65, and 80 rpm. 12 tablets were used for each friability test.

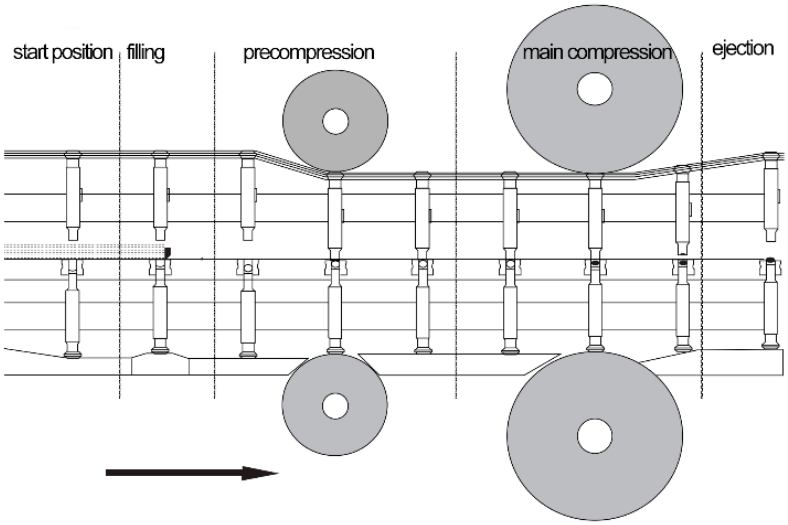


**Figure 1. Schematic diagram of Korsch XL 400 tablet compression machine**

**Determination of tablet strength**

The strength (or ultimate strength) (European pharmacopoeia, 2024) of a tablet was determined by a method generally accepted in pharmaceutical activity: the tablet was compressed in the diametrical direction, the destructive force P at which the tablet was destroyed was determined, and according to the destructive force, the ultimate strength $\sigma_p$ was determined.

The strength (ultimate strength) of the tablet $\sigma_p$ is the ratio of the destructive force to the cross-sectional area of the tablet:

$$\sigma_p = \frac{P}{d \cdot h}, \text{ Pa} \quad (1)$$

where $P$ is the destructive force, H; $d$ – tablet diameter, m; $h$ – tablet height, m.

Destructive force P of a tablet is a force applied in the diametrical direction at which the tablet is destroyed.

In this study, the destructive force of the tablet was determined on the device for strength determining the PTB-M 300 N Pharma Test. The working element of the device is two sponges directed towards each other, which move in the horizontal plane and compress the tablet in the diametrical direction. The accuracy of the device is 1 N. Each test was repeated 10 times.

**Determination of tablet friability**

Tablets were tested for friability using the "ERWEKA TAR II" device, where working element is a rotating drum with one blade (European pharmacopoeia, 2024).

The drum was made from transparent polymer material. The inner diameter of the drum is 283–291 mm. The rotational speed was 25±1 rpm. A blade of a certain shape was installed in the drum, which creates the lifting and lowering of the tablet. During the rotation of the drum, the tablets fall from a height of 156±2 mm or 6 inches (European pharmacopoeia, 2024).

10 tablets are dedusted, weighed and placed in the drum, close the lid and turn on the device for 4 minutes. After that, the procedure of weighing them is repeated.

The friability index P, %, was determined by the formula:

$$F = 100 - \frac{P_b - P_f}{P_b} \cdot 100 \quad (2)$$

where $P_b$, $P_f$ – the mass of tablets before and after experiment.

**Assumptions**

- API does not have a significant effect on strength and friability indicators;
- The change in strength and friability of tablets, like other quality indicators, depends on the number of compressing cycles – that is, how many tablets were produced by one press couple (die-punch). The indicator "Number of compressing cycles" is convenient to use when the results are analysing and comparing, for example, if the experiment was carried out on other compression machine with a different rotary table speed, or on static (crank) presses.
- The strength and friability indicators are depended not the rotary table rotational speed, but by the speed of movement of the punches caused by it, and, accordingly, by the speed of compressing and pushing out the tablet.

## Results and discussion

### Effect of number of compressing cycles on tablet strength

For two tablets of tablets, it was observed a decrease in their strength up to 10% during three hundred thousand cycles of compressing cycles, which is 80 hours of the tablet press operation.

With an increase in the number of compressing cycles from 28 to 280 thousand, which is approximately from 8 to 80 hours of press operation, the strength of tablets 1 decreases from 1.02 to 0.91 MPa, and of tablets 2 – from 1.52 to 1.40 MPa (Figure 2). The decrease in the strength of tablets occurs according to a parabolic dependence – the first 100–150 thousand cycles (30–35 hours of press operation) it is insignificant, and then it accelerates. Over time, the strength of the tablets decreases by more than 5%, therefore, in order to guarantee constant product quality, the process should be stopped and measures taken to restore the initial strength. In this study conditions, the process should be stopped at 200–250 thousand compressing cycles, that is, approximately 60–70 hours of press operation.

The manufacturer must guarantee the stable strength of the tablets, but it decreases during long-term operation of the tablet press. One of the methods known in practice to maintain stable strength is monitoring the condition of the surface of dies and punches, in particular, to carry out periodic polishing (Qiu et al., 2020).

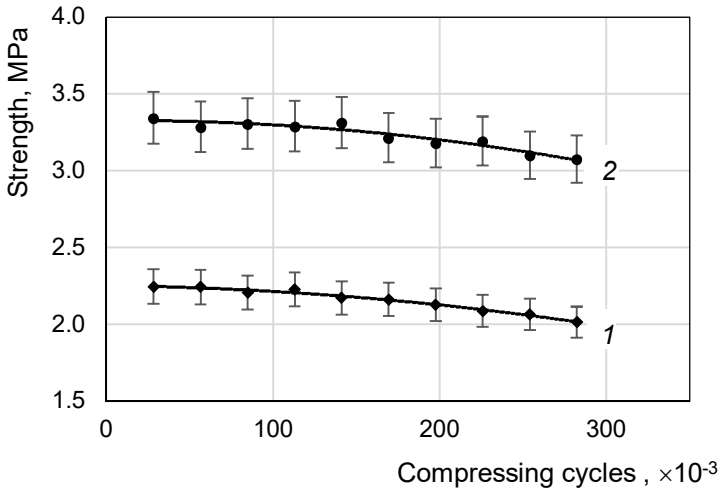


**Figure 2. Effect of number of compressing cycles on the strength of tablet:**
**1 – Tablets 1; 2 – Tablets 2**

Preliminary observations in this experiment showed that after polishing the working surfaces of the punches and dies, the strength of the tablet returns to the initial values within 7–10 times, but then other disturbing factors appear that lead to a decrease in the initial strength of the tablets. This issue requires additional research.

In the case of this study, it is suggested to polish the pressing tool after 48 hours of working operation.

The decrease in tablet strength during long-term press operation can be explained by the fact that a sticky layer is formed on the surface of the press tool, and, accordingly, the force (stress) of friction and pushing out the tablet in the die increases (Figure 3). As a result, the surface layers of the tablet are less strong, and the distribution of the tablet density along the height and width is inconsistent.

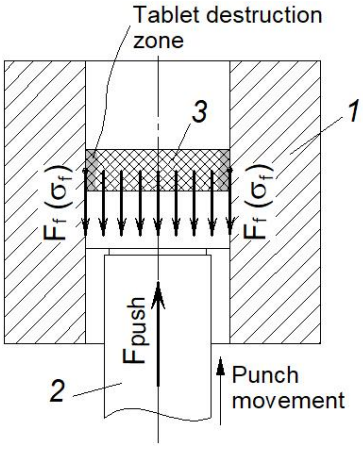


**Figure 3. Forces acting on tablet lateral surface during pushing out from die:**
**1 – die; 2 – punch; 3 – tablet.**
**$F_f$ – friction force, H;**
**σ – friction stress, Pa.**

The gray region is zone of intensive destruction of the tablet lateral surface.

**Effect of rotary table speed on tablet friability**

Information obtained from specialists who maintain tableting equipment and personal observations have shown that tablets made at high rotary speeds have lower strength and higher friability rates. The rational speed of the rotary table is usually determined based on production experience and validation protocols of the drug production process.

When the rotation speed of the rotary table increases from 35 to 80 rpm, the percentage of friability of tablets 1 increases from 0.15 to 1.24% (Figure 4).

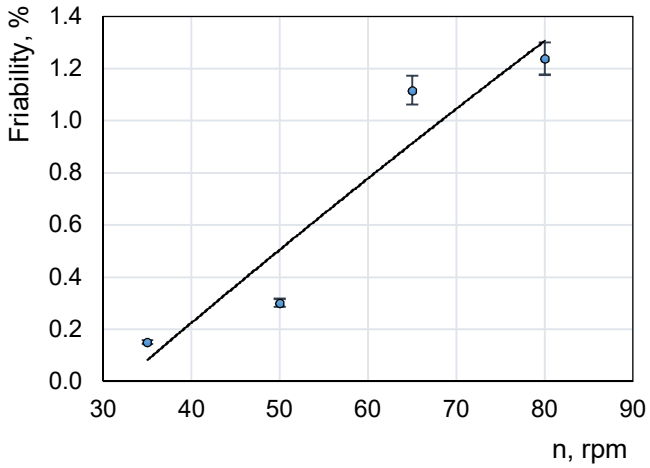


**Figure 4. Effect of rotary table speed (n) on tablet friability**

According to the above assumption, friability is affected not by the rotation speed of the rotary table, but by its derivative – the speed of movement of the punches in the die, and, accordingly, the speed of pushing of the tablet from the die. It is explained by the fact that at high speeds of pushing the tablet out of the die, the outer layer of the tablet is destroyed due to high frictional forces (Fig. 3). This layer is excessively worn away in the following technological and finishing operations. This type of defect is negatively manifested during coating process in drum and fluidized bed machines (Salawi, 2021), transportation between equipment and during the packaging of tablets in blister.

The effect of sliding speed on the friction coefficient or stress has been reported in other studies. For example, for semi-finished products and food products with high adhesion strength indicators (bread, meat products, cheeses), the friction (or adhesion) stress increases significantly at high sliding speeds, and this affects their machining by cutting. For products with low moisture content, the effect of sliding speed on friction (adhesion) stress is insignificant (Gubenia et al., 2010).

Additional research is needed for other factors affecting on a friability, in particular, vibration in the press tool, surface roughness of the die and punch, methods and depth of periodic polishing.

**Effect of number of compressing cycles on tablet friability**

An increase in the friability value was observed with increasing the compressing cycles: at the beginning of the experiment, the percentage of friability was 0.18–0.30%, at the end of the experiment it was 1.19–1.24% (Figure 9).

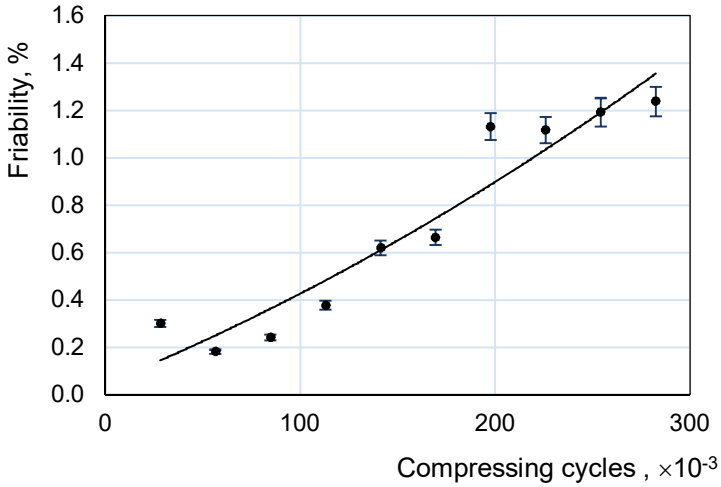


**Figure 5. Effect of number of compressing cycles on friability of tablets**

The consequence of tablet friability during packaging – dusty channels for feeding the tablet into the cell of the blister pack (Trudko et al., 2024) (Figure 6). In the event of dusting, the tablets get stuck in the streams and do not enter into the blister cells – empty cells appear. It takes time to clean equipment elements from dust, and it is undesirable in production.

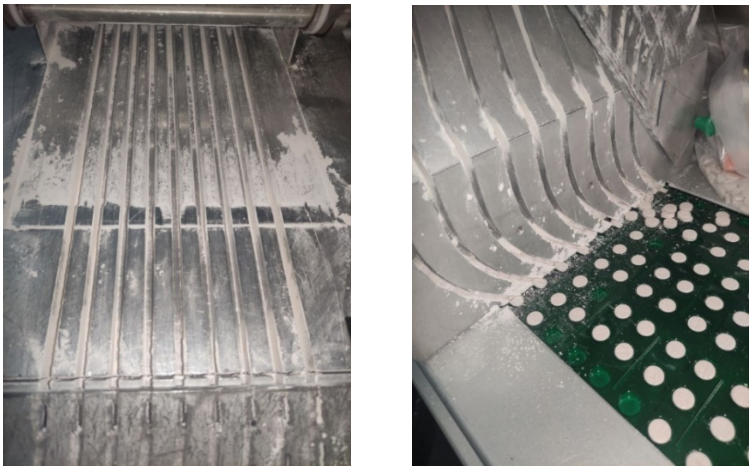

**Figure 6. Dusty cells of feeder in the blister machine**

**Effect of rotary table speed on the strength of the tablets**

In industry conditions, a rational compressing speed is usually set for each type of tablet, which depends on the rotary table speed (Tye et al., 2005). At high compression speeds, tablet strength decreases (Swarbrick, 2007).

During the experiment, an almost linear dependence of tablet strength was observed within the range of rotary table speed from 35 to 80 rpm (Figure 7).

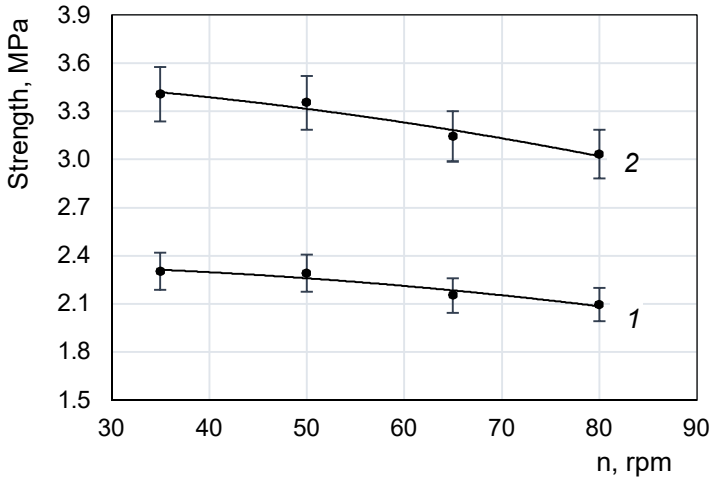


**Figure 7. Effect of rotary table speed (n) on tablet strength:**
1 – Tablets 1; 2 – Tablets 2

It is explained by the fact that at high compression speeds the tablet structure does not have time to form, and high pushing out speeds lead to increased frictional forces and destruction of the outer layers of the tablets.

Within the examined range of rotary table speed, the strength of tablets decreases to 20%. Taking into account the need to ensure stable indicators of the quality of tablets, it is not advisable to exceed the rotary table speed more than 50 rpm.

Tablet strength is probably also affected by the holding time when the upper and lower punches are stationary. The holding time is depended on the size of the flat surface of the punch head flat, which is in contact with the pressure roller (Swarbrick, 2007). It should be noted that the head radius and angle, which are responsible for the movement of the punch, decreases, and, accordingly, the speed and acceleration of the punch will increase.

## Conclusions

1. The number of compressing cycles in the press couple “Die-punch” affects the strength of the tablet: after 300,000 cycles, the tablet loses up to 8% strength on average. This is explained by the sticking of the product on the surface of the die and the punch, an increase in the roughness of their surface and, accordingly, an

increase in the coefficient of friction and adhesion strength. As a result, the outer layer of the tablet has lower strength indicators and is intensively destroyed.

2. Within the working range of the rotary table speed from 30 to 80 rpm, the strength of the tablets can decrease by 10%. This is explained by a change in the compressing speed and a decrease in the holding time of the tablet in the die.
3. With an increase in the rotary table speed from 30 to 80 rpm, the tablet friability increases from 0.2 to 1.2%, it is more than the regulated pharmacopoeial (1%) and recommended (0.5) norms. It is due to a decrease of the holding time of tablet in the die and a higher pushing out speed, which reduces the strength of the outer layers of the tablet. High friability leads to a worse quality of applying a protective coating to the tablet, an increase in the number of scraps at the coating stage, and dustiness of the feeder of the blister machine.
4. Increasing strength and friability indicators imposes restrictions on the rotary table speed. For the tablets used in this study, it is 50 rpm. An increase of speed, and, accordingly, productivity, is possible due to more frequent polishing of the surface of dies and punches.

## References


Adeleye O. (2019), Relationship between compression pressure, mechanical strength and release properties of tablets, *Polymers in Medicine*, 49(1), pp. 27–33, https://doi.org/10.17219/pim/111888

Hoag S.W., Dave V.S., Moolchandani V. (2008), Compression and compaction. In: L.L. Augsburger, S.W. Hoag (Eds.), *Pharmaceutical Dosage Forms – Tablets, 3rd Edition*, CRC Press, Boca Raton, https://doi.org/10.1201/b15115

CDER (2015), *Guidance for Industry: Size, Shape, and Other Physical Attributes of Generic Tablets and Capsules*, United States Food and Drug Administration.

Commission BP (2021), *British Pharmacopoeia* 2021, Stationery Office.

ERWEKA GmbH (2024), TAR II – advanced friability and friability tester – ERWEKA GmbH. *Home – ERWEKA GmbH*. Available at: https://www.erweka.com/products/physical-testers/friability-tester.html

Etzler F.M., Bramante T., Deanne R., Sienkiewicz S., Chen F. J. (2011), Tablet tensile strength: an adhesion science perspective, *Journal of Adhesion Science and Technology*, 25(4–5), pp. 501–519, https://doi.org/10.1163/016942410X525687.

European pharmacopoeia. Available at: https://www.edqm.eu/en/web/edqm/european-pharmacopoeia.

Giannis K., Schilde C., Finke J.H., Kwade A. (2021), Modeling of high-density compaction of pharmaceutical tablets using multi-contact discrete element method, *Pharmaceutics*, 13(12), 2194, https://doi.org/10.3390/pharmaceutics13122194

Goots V., Guts O., Gubenia O. (2016), Reodynamical theory of viscous-elastic system deforming, Proceedings of University of Ruse, 55(10.2), pp. 18–22.

Gordiienko O.I., Vronska L., Melnyk O.A., Groshovyi T.A. (2015), Current state of creation, production and research of tablet medicinal products, *Pharmaceutical Review*, 1, https://doi.org/10.11603/2312-0967.2015.1.3780

Gubenia O., Goots O. (2010), Modeling of cutting of food products, *Journal of EcoAgriTourism*, 1, pp. 67–71.

Herasymenko D., Zomenko O., Gubenia O. (2024), Influence of tablet press rotor speed on tablet friability performance, *90th International Scientific Conference of Young*

*Scientist and Students "Youth Scientific Achievements to the 21st Century Nutrition Problem Solution"*, April 11–12, 2024, NUFT, Kyiv.

Natoli D., Levin M., Tsygan L., Liu, L. (2017), Development, optimization, and scale-up of process parameters: Tablet compression, In: Y. Qiu, Y. Chen, G.G.Z. Zhang, L. Yu, R.V. Mantri (Eds.), *Developing Solid Oral Dosage Forms*, Elsevier, pp. 917–951, https://doi.org/10.1016/B978-0-12-802447-8.00033-9

Peeters E., Silva A.F.T., Fonteyne M., De Beer T., Vervaet C., Remon C. (2018), Influence of extended dwell time during pre- and main compression on the properties of ibuprofen tablets, *European Journal of Pharmaceutics and Biopharmaceutics*, 128, pp. 300–315, https://doi.org/10.1016/j.ejpb.2018.05.007.

Pharmaeducation (2024), *Quality control tests of tablets or Evaluation of tablets,* Available at: https://pharmaeducation.net/quality-control-tests-of-tablets-or-evaluation-of-tablets

Pitt K., Heasley M. (2013), Determination of the tensile strength of elongated tablets, *Powder Technology*, 238, pp. 169–175, https://doi.org/10.1016/j.powtec.2011.12.060.

Podczeck F. (2012), Methods for the practical determination of the mechanical strength of tablets – From empiricism to science, *International Journal of Pharmaceutics*, 436(1-2), pp. 214–232, https://doi.org/10.1016/j.ijpharm.2012.06.059.

Rösler J., Harders H., Bäker M. (2007), *Mechanical Behaviour of Engineering Materials: Metals, Ceramics, Polymers, and Composites*, Springer.

Salawi A. (2022), Pharmaceutical coating and its different approaches, a review, *Polymers*, 14(16), 3318, https://doi.org/10.3390/polym14163318

Siiriä S.M. Antikainen O., Heinämäki J., Yliruusi J. (2011), 3D simulation of internal tablet strength during tableting, *American Association of Pharmaceutical Scientists*, 12(2), pp. 593–603, https://doi.org/10.1208/s12249-011-9623-0.

Sinka I.C., Pitt K.G., Cocks A.C.F. (2007), The strength of pharmaceutical tablets. In: A.D. Salman, M. Ghadiri, M.J. Hounslow (Eds.), *Handbook of Powder Technology, Volume 12,* pp. 941–970, https://doi.org/10.1016/s0167-3785(07)12025-x

Swarbrick J. (2007), *Encyclopedia of pharmaceutical technology, Volume 6*, Taylor & Francis Group.

Trudko S.S., Gubenia O.O., Desyk M.G., Gavva O.M. (2024), Packaging of medicines in blister packs, *Packaging,* 3, pp. 28–31.

Tye C.K., Sun C., Amidon G.E. (2005), Evaluation of the effects of tableting speed on the relationships between compaction pressure, tablet tensile strength, and tablet solid fraction, *Journal of Pharmaceutical Sciences*, 94(3), pp. 465–472, https://doi.org/10.1002/jps.20262

# Vacuum distillation in the technology of non-alcoholic wines from Isabella grapes

**Olha Uspalenko[1,2], Marina Bilko[1], Volodymyr Kucherenko[1,2], Matthias Schmitt[3], Italiano Lorenzo[3]**

*1 – National University of Food Technologies, Kyiv, Ukraine*
*2 – Research and Production Centre "Ukrainian Wine Institute", Kyiv, Ukraine*
*3 – Hochschule Geisenheim University, Institute of Enology, Geisenheim, Germany*

**Keywords:**

Wine
Non-alcoholic
Vacuum distillation
Dealcoholization
Cryoconcentrate

**Article history:**

Received 18.04.2024
Received in revised form 14.06.2024
Accepted 28.06.2024

**Corresponding author:**

Olha Uspalenko
E-mail: olhauspalenko@gmail.com

**DOI:**
10.24263/2310-1008-2024-12-1-7

## Abstract

**Introduction.** The aim of the study was to develop the technology of non-alcoholic wine from Isabella grapes by vacuum dealcoholization with maximum preservation of varietal sensory properties of the initial wine material and harmonization of taste by adding grape must cryoconcentrate.

**Materials and methods.** The wine material was unprocessed dry red from the Isabella grape variety (Ukraine) cryoconcentrate of grape must. Dealcoholization was carried out using vacuum distillation. Physico-chemical indicators of dealcoholized wine were determined using chromatography. Sensory properties were evaluated using special methods.

**Results and discussion.** In the process of dealcoholization, the content of ethyl alcohol decreased from 9.8% to 0.4% by volume, which was accompanied by a decrease in the concentration of volatile acids, aldehydes, and esters while preserving a significant part of phenolic compounds (catechins, proanthocyanins), important for the antioxidant activity of the drink. Studies have shown that vacuum distillation reduces not only the content of ethanol, but also methanol by up to 30%, which increases the level of safety of non-alcoholic wine consumption.

Dealcoholization by vacuuming leads to an increase in the concentration of organic acids, tartaric and malic, which negatively affects the taste characteristics. To correct the taste properties, cryoconcentrate of grape must was added to the dealcoholized wine in different dosages. Sensory evaluation, carried out according to the methodology of the International Organization of Grapes and Wine, made it possible to note that this approach contributed to obtaining the taste harmony of the drink. The sweetness of the drink reduced the feeling of excessive acidity, which helped to balance the taste of non-alcoholic wine.

**Conclusions.** The results confirmed that the application of vacuum distillation in combination with the use of grape must cryoconcentrate is an effective way of obtaining non-alcoholic wine with pleasant sensory characteristics, ensuring its appeal to consumers and preservation of useful properties.

## Introduction

Non-alcoholic wine Dealcoholization Sensory indicators Vacuum distillation Cryoconcentrate

The global non-alcoholic wine market is over USD 10 billion and is estimated to still grow at a significant CAGR (compound annual growth rate) of over 7% between 2019 and 2027, reaching a revenue share of over USD 30 billion (Sarkissian and Liganenko, 2020).

The first references to the production of alcohol-free wines date back to 1869, when the American Thomas Bramwell Welch, being a strong supporter of the movement for the moderation of alcohol consumption, produced this product by the pasteurization method (Sawler et al., 2013). Later, in 1908, the German scientist Carl Jung patented the technology of alcohol-free wine by applying a vacuum to lower the distillation temperature to 35 °C (Gornostay, 2011).

Dealcoholization of wine can be achieved by several methods. These methods can be classified into three groups based on the principle or mechanism of ethanol reduction and removal at various stages of wine production, including reduction of fermentable sugars (pre-fermentation stage), reduction or limitation of ethanol production (fermentation stage), and ethanol removal by membrane separation or heat treatment (post-fermentation stage) (Uspalenko et al., 2024).

In literary sources, there are studies of various ways of reducing the alcohol content in wines. The simplest of them is diluting the wort with water or adding juice from unripe grapes to the wort, which is called "green wort". In countries such as South Africa, New Zealand, Australia, and the USA (except California), water is allowed only as an aid in the preparation of stabilizing materials for the processing of wine materials, and the use of "green must" leads to negative changes in the sensory quality of wines (Kroiruddin et al., 2018).

To reduce the fermentable sugars in the wort, juice filtration is used using nanofiltration, ultrafiltration or reverse osmosis membranes, which have very small pore sizes and can retain sugar. At the same time, these methods affect the reduction of the content of polyphenols, anthocyanins and color intensity, which negatively affects the sensory properties and biological value of wine (Blackman et al., 2017).

Another way to reduce alcohol in wine is the use of glucose oxidase, which reduces glucose in grape juice before fermentation (Ebert et al., 2016). The producer of the enzyme is the fungus *Aspergillus niger.* Glucose oxidase converts β-D-glucose to D-gluconolactone in the first step of the reaction, releasing hydrogen peroxide, and catalyzes the conversion of D-gluconolactone to gluconic acid in the second step of the reaction. These reactions cause the oxidation of glucose in the must, which, accordingly, leads to a decrease in the alcohol level in the wine. The increased content of gluconic acid gives the wine excessive acidity and has a negative effect on the manifestation of fruit aroma and the reduction of varietal characteristics of the aroma (Di Renzo et al., 2014).

The use of yeast of the genus *Metschnikowia* or modified yeast strains with a low ability to produce alcohol contributes to the reduction of alcohol content by synthesizing a higher content of secondary fermentation products. This method only partially reduces the level of ethyl alcohol in wine (Suo et al., 2019).

Hydrophobic absorbents such as zeolites can also absorb ethanol from wine by absorption and filtration. This method can be used to produce alcohol-free wines with an ethanol content of up to 0.5% by volume (Akyerenko et al., 2021). But extraction methods of alcohol reduction are expensive and rarely used in the production of low-alcohol and alcohol-free wines.

Modern methods of dealcoholization of wines include vacuum and osmotic distillation, nanofiltration, and reverse osmosis (Cachon et al., 2006; Schelezki et al., 2020). These techniques minimize the loss of important volatile aromatic compounds.

The formation of harmonious sensory characteristics in non-alcoholic wines during dealcoholization or the use of other methods that contribute to reducing the alcohol content and harmonizing the balance is extremely important.

The purpose of the research was to develop the technology of non-alcoholic wine from Isabella grapes by vacuum dealcoholization with maximum preservation of varietal sensory characteristics of the initial wine material and harmonization of taste by adding cryoconcentrate of grape must.

## Materials and methods

### Materials

Unprocessed dry red wine made from grapes of the Isabella variety, cultivated in the Kyiv region, with an alcohol content of 9.8% vol. and cryoconcentrate of grapes must with a sugar content of 50-55%.

### Experimental setup and dealcoholization conditions

Dealcoholization was carried out by vacuum distillation. Usually, in this method, the wine is placed in a strong vacuum. The wine was gently heated to boil off the alcohol at temperatures between 20 °C and 38 °C, maintaining a pressure of less than 0.1 bar, since vacuum is crucial for effective dealcoholization while preserving the initial sensory properties of the wine material (Figure 1) (Uspalenko et al., 2024).

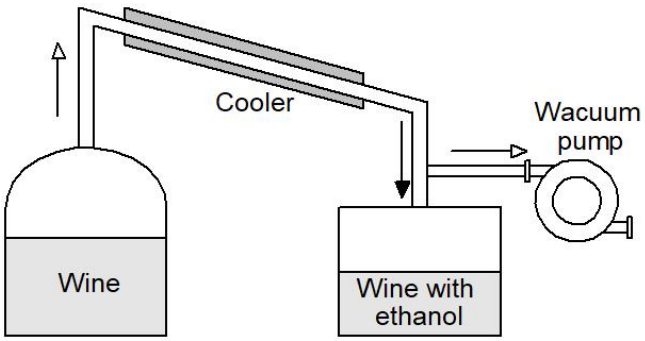


**Figure 1. Scheme of the vacuum distillation method**

But,since the Isabella grape variety is known for its high methanol content, dealcoholization was carried out in a vacuum apparatus, maintaining a pressure of 6 kPa and heating the wine to a temperature of 65 °C, under such conditions, the vapors passed into the gas phase and entered another tank, where they condensed and collected. Dealcoholization lasted 15 minutes, where 4.5 l of distillate and 13.5 l of dealcoholized wine were obtained from 18.00 l of wine. After that, the alcohol-free wine was filtered through laboratory filter paper.

To reduce acidity and to harmonize the taste of dealcoholized wine, a cryoconcentrate was added at the rate of 5-25 g/l and 30-80 g/l to bring non-alcoholic wine to the level of semi-dry and semi-sweet, respectively.

**Setting up the experiment**

During the experiment, the physico-chemical and sensory quality indicators of non-alcoholic wines obtained by the vacuum distillation method were investigated in comparison with the control sample, and a sensory analysis of the tested wine sweetened with cryoconcentrate to different levels of sweetness was also carried out.

- **control sample** (Control) – wine with an alcohol content of 9.8% vol.
- **sample 1** – dealcoholized wine by vacuum distillation with an alcohol content of 0.4% vol.
- **sample 2** – dealcoholized wine by vacuum distillation with an alcohol content of 0.4% vol. sweetened with cryoconcentrate up to 20 g/l
- **sample 3** – dealcoholized wine by the vacuum distillation method with an alcohol content of 0.4 % vol. sweetened with cryoconcentrate up to 75 g/l

At the first stage of the experiment, dealcoholized wine was produced by vacuum distillation. At the second stage, a physical and chemical analysis was carried out using the chromatography method. To harmonize the taste of dealcoholized wine, grape must cryoconcentrate was added to the level of semi-dry and semi-sweet wine.

The final stage was sensory analysis by a specialized tasting commission.

**Determination of sensory properties**

Tastings took place in a special room equipped with individual booths and air conditioning at 20°C. The testing was conducted by two independent tasting commissions.

For wine tasting, a standard tasting glass made of thin, clean, transparent glass with a capacity of 210-220 ml was used, which makes it possible to operate 60-70 ml of wine for a comprehensive sensory assessment of all elements of quality.

Wine with alcohol content and de-alcoholized wine were evaluated according to the Standards of the International Organization of Vine and Wine (Resolution OIV/Competition ECO 332A/200). The maximum tasting rating of the experimental samples was 100 points and was determined as the sum of points for each indicator: appearance (transparency, color) – 14; aroma (authenticity, intensity, quality of aroma) - 30; taste (authenticity, intensity, harmonic stability, taste quality) - 44; harmony (general impression) – 11.

To create the aromatic profiles of the experimental samples, a descriptive method was used according to a 10-point rating scale based on the following descriptors: citrus, fruity, nutmeg, floral, sweetness, acidity, bitterness, body/fullness, intensity, astringency.

Graphical representations of experimental data were performed using Microsoft Excel 2010.

Comparing the profiles of the experimental samples made it possible to determine their differences and draw conclusions about the change in wine quality during dealcoholization.

**Determination of physical and chemical indicators**

Determination of physical and chemical parameters was carried out in accordance with the methods and regulations of the International Organization of Grapes and Wine (OIV). Standard protocols and analytical methods recommended by the OIV were used to conduct the research, ensuring the accuracy and reproducibility of the results obtained.

**Processing research results**

Determination of physico-chemical indicators of non-alcoholic wine samples was carried out in two repetitions. Results are shown as mean ± standard deviation.

Statistical data processing was performed, including determination of mean content and standard deviation (±SD), with four replicates (four collections) for the main compounds identified in all four collections analyzed.

## Results and discussion

**Changes in the physical and chemical parameters of wine during dealcoholization**

During dealcoholization of wine, complex physicochemical processes take place, in which, in addition to the removal of ethyl alcohol, other components of wine are also changed - such as volatile ones - aldehydes, acids, higher alcohols, ethers, acetates, organic acids, and amino acids (Diban et al., 2008). During the experiment, the alcohol content was reduced from 9.8% vol. (control) up to 0.4% vol. It should be noted that due to the removal of alcohol from wine, the concentration of wine components occurs, because of which the concentration of non-volatile compounds increases (Table 1).

**Table 1**

**Physico-chemical parameters of dealcoholized wine from Isabella grapes**

| Indicator | Control | Sample 1 |
|---|---|---|
| Ethanol content, % vol. | 9.80 ± 0.40 | 0.40 ± 0.02 |
| Mass concentration of sugars, g/l: | 3.00 ± 0.15 | 4.20 ± 0.20 |
| *Organic acids, mg/l:* | | |
| titrated acids | 6.10 ± 0.30 | 7.80 ± 0.30 |
| volatile acids | 0.20 ± 0.01 | 0.15 ± 0.01 |
| tartaric acid | 4.90 ± 0.20 | 4.20 ± 0.20 |
| citric acid | 0.015 ± 0.007 | 0.010 ± 0.005 |
| malic acid | 1.00 ± 0.05 | 2.90 ± 0.15 |
| *Alcohols, mg/l:* | | |
| methanol | 84.00 ± 4.20 | 25.20 ± 1.26 |
| glycerol | 3020.0 ± 151.00 | 3020.0 ± 151.00 |
| acetaldehyde | 19.00 ± 0.95 | 16.00 ± 0.80 |
| ethyl acetate | 42.00 ± 0.21 | 40.00 ± 0.20 |
| higher alcohols (isoamyl and isobutyl) | 309.70 ± 15.28 | 301.30 ± 15.06 |
| furfural | 2.80 ± 0.15 | 2.00 ± 0.10 |
| 5-hydroxymethylfurfural | 13.20 ± 0.66 | 12.90 ± 0.65 |
| Hydroxymethylfurfural (HMF), mg/l | 2.50 ± 0.12 | 2.10 ± 0.10 |

The total content of organic acids undergoes significant changes during dealcoholization, this is due to the change in concentration caused by the removal of ethanol and water. Organic acids are an important group of compounds in wines because they affect the physicochemical and microbiological stability of wines, as well as their sensory properties (Coelho et al., 2018).

Content of titrated acids increased by 1.28 times. At the same time, the concentration of representatives of organic acids varied in different ways. The content of volatile acids decreased by 1.33 times, which is characteristic of dealcoholization. The mass concentration of tartaric acid decreased by 1.17 times and citric acid by 1.5 times, while the content of malic acid increased by 2.9 times. Such transformations lead to a change in the general taste profile of non-alcoholic wine, which is expressed in an imbalance of taste and disharmony in the sensory perception of wine. Thus, some researchers noticed that dealcoholized wines have higher acidity and less bitterness compared to the original (control) sample (Corona et al., 2019).

However, it should be noted that organic acids have the ability to interact with alcohols, forming complex esters that add a rich aroma and taste to wines. They create harmony of taste, which is extremely important for consumers' perception of wine quality.

A slight decrease, compared to other acids, was observed in tartaric acid, which helps maintain acidity, lower pH, inhibit bacterial growth and preserve freshness in wine for a long time. In addition, tartaric acid is necessary to improve the structure and flavor profile of wine (Yang, 2021).

As shown in Table 1, malic acid increased 2.9-fold during dealcoholization. This can lead to the formation of lactic acid, which negatively affects the stability of the wine and can introduce an unpleasant bitter taste. Therefore, it is important to control the level of malic acid during dealcoholization.

Concentrations of volatile substances also changed after dealcoholization of wines. For example, when applying vacuum distillation, the content of acetaldehyde slightly decreased by 16%. It should be noted that aldehydes play an important role in the formation of sensory indicators of wine (aroma, taste). One of the main aldehydes formed during fermentation is acetaldehyde. Increasing the content of this aldehyde gives the wine a sharp smell with hints of toasted bread, but the decrease resulted in less expressive fruit aromas and flavors, making it less saturated and complex in sensory perception (Sam et al., 2021).

The dealcoholization process also affects other volatile compounds that affect the aroma and taste of the wine. Wine contains more than 1000 volatile compounds of various chemical classifications (alcohols, esters, fatty acids, aldehydes, terpenes, ketones, sulfur compounds), where about 400 volatile compounds are formed during wine fermentation (Esteras-Saz et al., 2021). Higher alcohol in wine are formed in the process of alcoholic fermentation, which is carried out by yeast. They are by-products of yeast metabolic processes. Higher alcohol can be formed from amino acids by the so-called Ehrlich pathway. During this process, amino acids are deaminated to their corresponding aldehydes, which are then reduced to higher alcohol.

A representative of monoatomic alcohols is methyl alcohol, which is synthesized before and during alcoholic fermentation due to the hydrolysis of pectins by methyl-pectinesterase, which is contained in grapes. Pectinase catalyzes the cleavage of ester bonds in pectin, which leads to the formation of methyl alcohol and pectic acid (Kucherenko and Bilko, 2020). It is known that methanol is a toxic substance that can accumulate in the body and have a harmful effect on various organs and systems. In red wines, the methanol content is up to 250 mg/l, the results of the study showed that the methanol content in the control sample was 84.00 mg/l, which is 33.6% less than the maximum permissible norm, and the methanol content in the non-alcoholic sample is even lower - 25.2 mg/l. This indicates that methanol is removed along with ethyl alcohol during the wine dealcoholization process.

Other indicators of volatile compounds, such as ethyl acetate and higher alcohols, remain almost unchanged, demonstrating the stability of the wine's aroma after treatment.

The concentration of hydroxymethylfurfural (HMF) decreased by 1.19 times 2.1 mg/l versus 2.5 mg/l, and the mass concentration of higher alcohols decreased by 1.03 times 301.3 mg/l in non-alcoholic wine versus 309.7 mg/l. The content of furfural decreased by 1.4 times to 2.0 mg/l versus 2.8 mg/l, and 5-hydroxymethylfurfural decreased by 1.02 times to 12.9 mg/l in the non-alcoholic sample versus 13.2 mg/l. Hydroxymethylfurfural and furfural, which are markers of heat treatment, have slightly reduced concentrations in the dealcoholized sample, which indicates the preservation of the naturalness of the product.

Therefore, when switching to a non-alcoholic version of wine, there are both significant increases in individual indicators and decreases in other parameters, which can significantly affect the sensory properties of the product.

**Changes in sensory parameters of wine during dealcoholization**

During the study, slight changes in the color of the wine were recorded compared to the initial (control) sample. Sample 1, obtained by the method of vacuum distillation, retained its deep red color with a purple tint and transparency, but also added a light brick shade, which was formed during dealcoholization (Figure 2).

**Control** **Sample 1**

**Figure 2. Comparison of the color of the control sample with the dealcoholized sample**

However, the greatest impact of dealcoholization was found on the aromatic and taste characteristics of the wine, which emphasizes the importance of choosing processing methods and improving the balance to preserve the sensory properties of the product. The results of the comparative sensory analysis of the studied wine made it possible to establish their sensory characteristics (Figure 3).

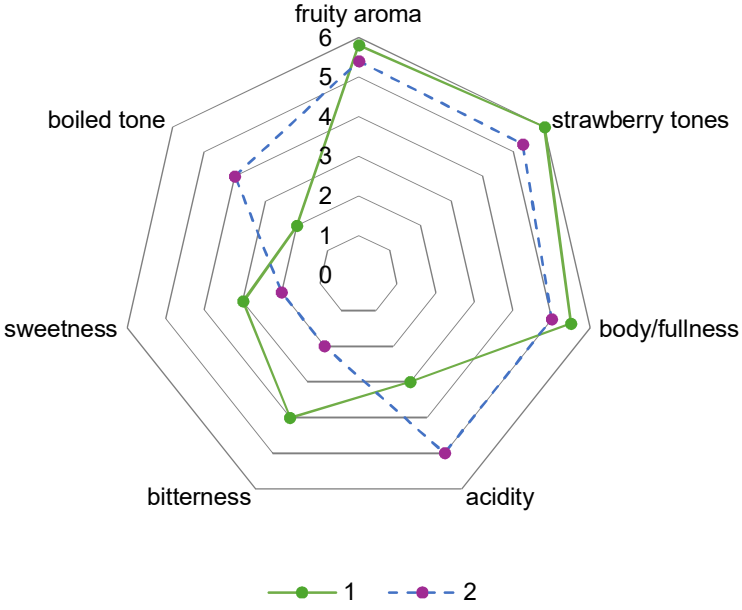


**Figure 3. Profilogram of red wine from the Isabella grape variety: 1 — alcoholic wine (control), 2 — dealcoholized wine (sample 1)**

A careful analysis of the changes in the main descriptors of the experimental samples made it possible to establish the following. Dealcoholization of wine is usually associated with the loss of volatile aromatic components (Kumar et al. 2024). On the other hand, decreasing the ethanol level improves the perception of aromatic components, which can compensate for the loss of aroma (Goldner et al. 2009). In this case, both effects probably resulted in the perception of the strawberry flavor not changing. The processed wine showed a similar expression of fruit tones. Dealcoholized samples are usually described as having a slightly reduced but slightly altered intensity of this descriptor.

The investigated dominant aromatic properties, such as strawberry tones, were evaluated almost equally for the dealcoholized wine and did not differ significantly from the original untreated wine. Perception of sweet was clearly altered because of dealcoholization. The de-alcoholized wines had significantly less sweetness compared to the original wine, but the dealcoholized wine sweetened with cryoconcentrate had a higher perceived sweetness (Figure 4).

Based on this, it becomes clear why non-alcoholic wines are usually produced with an increased level of sugar, that is, semi-dry or semi-sweet. To compensate for this change due to dealcoholization, a cryoconcentrate was added to sweeten the wine to a semi-dry 25 g/l, thereby rebalancing the balance. Acidity perception was rated significantly higher for still wines without sweetening. Therefore, when choosing wines for dealcoholization, this factor should be taken into account and wines with lower-than-normal acidity values should be chosen to obtain a balanced product or sweeteners such as cryoconcentrate should be added.

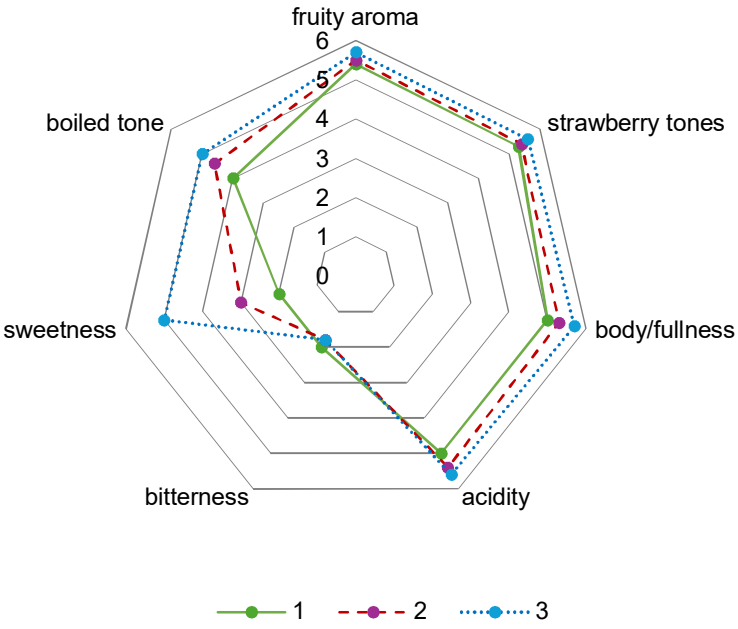


**Figure 4. Profilogram of non-alcoholic red wines from grapes of the Isabella variety with the addition of cryoconcentrate:**
**1 — dry (control without the addition of cryoconcentrate);**
**2 – semi-dry,**
**3 – semi-sweet**

The bitterness of the original wine was significantly higher in the control wine compared to the dealcoholized variants. The direct effect of ethanol in wine is to increase bitterness.

The perception of body and fullness of wine is clearly correlated with alcohol content (Grainger, 2009). The higher the alcohol content, the higher this perception. The results of this study show a significant difference between the processed variant and the original wine. Since body and fullness are clearly considered positive sensory characteristics of wine, oenological strategies such as the addition of cryoconcentrate, mannoproteins or tannins can partially compensate for the reduction of these sensory parameters due to dealcoholization.

The study shows that dealcoholization of wine significantly affects its sensory characteristics. The sensory characteristics of the aromatic parameters were changed less than the attributes affecting the perception of the wine on the palate. The main effects of dealcoholization are consistent with the complex sensory characteristics of ethanol in wine.

## Conclusions

1. The process of vacuum distillation allows you to effectively reduce the content of ethyl alcohol in wine to the level of 0.4% vol. A decrease in the concentration of volatile acids, aldehydes and esters is observed within limits that do not critically affect the overall quality of the product, ensuring its chemical stability.

2. As a result of the dealcoholization process, there is an increase in the concentration of organic acids, such as tartaric and malic, which creates a certain imbalance in the sensory properties of the drink.
3. Adding grape juice cryoconcentrate allows you to adjust the sensory properties of the drink, creating a balance between acidity and sweetness. This contributes to the formation of a pleasant taste profile, softening of bitterness and improvement of the aromatic component, which is confirmed by sensory evaluation.
4. The combination of vacuum distillation and the use of cryoconcentrate is a promising technology to produce high-quality non-alcoholic wine. The obtained results demonstrate the possibility of creating a drink that meets modern consumer demands, preserves the benefits of natural components and provides an attractive taste profile.

## References


Akyereko Y.G., Wireko-manu F.D., Alemawor F., Adzanyo M., Patarata L. (2021), Effects of production methods on flavor characteristics of nonalcoholic wine, *Journal of Food Quality*, pp. 1–10, http/ doi.org/ 10.1155/2021/3014793

Blackman J.W., Longo R., Rogiers S.Y., Schmidtke L.M., Torley P.J. (2017), Changes in volatile composition and sensory attributes of wines during alcohol content reduction, *Journal of the Science of Food and Agriculture,* 97, pp. 8–16, https://doi.org/10.1002/jsfa.7757

Cachon R., Dequin S., Heux S., Sablayrolles J.M. (2006), Engineering a *Saccharomyces cerevisiae* wine yeast that exhibits reduced ethanol production during fermentation under controlled microoxygenation conditions, *Applied and Environmental Microbiology,* 72(9), pp. 5822–5828, https://doi.org/10.1128/AEM.00750-06

Coelho EM, Padilha CV, Miskinis GA, Sá AG, Pereira GE, Azevedo L., Lima MD (2018), Simultaneous analysis of sugars and organic acids in wine and grape juices by HPLC: Method validation and characterization of products from northeast Brazil, *Journal of Food Composition and Analysis*, 66, pp. 160–167, https://doi.org/10.1016/j.jfca.2017.12.017

Corona O., Liguori L., Albanese D., di Matteo M., Cinquanta L., Russo P. (2019), Quality and volatile compounds in red wine at different degrees of dealcoholization by membrane process, *European Food Research and Technology,* 245(11), pp. 2601–2611, https://doi.org/10.1007/s00217-019-03376-z

Diban N., Athes V., Bes M., Souchon I. (2008), Ethanol and aroma compounds transfer study for partial dealcoholization of wine using membrane contactor, *Journal of Membrane Science*, 311(1–2), pp. 136–146, https://doi.org/10.1016/J.MEMSCI.2007.12.004

Di Renzo L., Carraro A., Valente R., Iacopino L., Colica C., De Lorenzo A. (2014), Intake of red wine in different meals modulates oxidized ldl level, oxidative and inflammatory gene expression in healthy people: a randomized crossover trial, *Oxidative Medicine and Cellular Longevity,* 2014, 681318, https://doi.org/10.1155/2014/681318

Ebert K., Grossmann M., Pasch L. , Röcker J., Schmitt M. (2016), The use of glucose oxidase and catalase for the enzymatic reduction of the potential ethanol content in wine, *Food Chemistry,* 210, pp. 660–670, https://doi.org/10.1016/j.foodchem.2016.04.093

Esteras-Saz J., de la Iglesia Ó., Peña C., Escudero A., Téllez C., Coronas J. (2021), Theoretical and practical approach to the dealcoholization of water-ethanol mixtures and red wine by osmotic distillation, *Separation and Purification Technology*, 270, 118793, https://doi.org/10.1016/j.seppur.2021.118793

Goldner M.C., Zamora M.C., Dileo P., Gianninoto H., Bandoni A. (2009), Effect of ethanol level in the perception of aroma attributes and the detection of volatile compounds in red wine, *Journal of Sensory Studies,* 24(2), pp. 243–257, https://doi.org/10.1111/J.1745-459X.2009.00208.X

Gornostay P. (2011), *Jung Carl Gustav*, Political Encyclopedia*,* Parliamentary publishing house, Kyiv.

Grainger K. (2009), *Wine Quality: Tasting and Selection,* Chichester/ England: John Wiley and Son's Ltd. https://doi.org/10.1002/9781444301687

Khoiruddin K., Mangindaan D., Wenten I.G. (2018), Beverage dealcoholization processes: Past, present, and future, *Trends in Food Science and Technology,* 71, pp. 36–45, https://doi.org/10.1016/j.tifs.2017.10.018

Kucherenko V., Bilko M. (2020), Quantitative content of methanol in wines of Isabel grape varieties and its impact on consumer health, *Scientific Works of NUFT*, 26(6), pp. 16–23, https://doi.org/10.24263/2225-2924-2020-26-6-4

Kumar Y., Ricci A., Parpinello G.P., Versari A. (2024), Dealcoholized wine: A scoping review of volatile and non-volatile profiles, consumer perception, and health benefits, *Food and Bioprocess Technology*, 17, pp. 3525–3545, https://doi.org/10.1007/s11947-024-03336-w

Sam F.E., Ma T.Z., Salifu R., Wang J., Jiang Y.M., Zhang B., Han S.Y. (2021), Techniques for dealcoholization of wines: their impact on wine phenolic composition, volatile composition, and sensory characteristics, *Foods,* 10, 2498, https://doi.org/ 10.3390/foods10102498

Sarkissian A., Liganenko M. (2020), Analysis of the innovative environment for the development of wineries as an infrastructure component of wine routes, European Scientific Journal of Economic and Financial Innovation, 2(6), pp. 55–65, https:/ /doi.org/10.32750/2020-0205

Sawler J., Reisch B., Aradhya M.K., Prins B., Zhong GY., Schwaninger H., Simon C., Buckler E., Myles S. (2013), Genomics assisted ancestry deconvolution in grape, *PLoS One,* 8(11), e80791, https://doi.org/ 10.1371/journal

Schelezki O.J., Deloire A., Jeffery D.W. (2020), Substitution or dilution? Assessing pre-fermentative water implementation to produce lower alcohol Shiraz wines, *Molecules,* 25, 2245, https://doi.org/10.3390/molecules25092245

Suo H., Tian R., Li J., Zhang S., Cui Y., Li L., Sun B. (2019), Compositional characterization study on high-molecular - Mass polymeric polyphenols in red wines by chemical degradation, *Food Research International,* 123, pp. 440–449, https://doi.org/10.1016/j.foodres.2019.04.056

Uspalenko O., Bilko M., Kucherenko V. (2024), Methods of reducing and removing alcohol from grape wines, *Scientific Works of NUFT*, 30(4), pp. 154–175, https://doi.org/ 10.24263/2225-2924-2024-30-4-14

Yang S. (2021), Discussion on organic acids in wine and their determination, *China Food Safety Magazine,* 29, pp. 187-188, https://doi.org/10.16043/j.cnki.cfs.2021.29.113

# Instructions for Authors

**Dear colleagues!**

The Editorial Board of scientific periodical «**Ukrainian Journal of Food Science**» invites you to publish of your scientific research.

A manuscript should describe the research work that has not been published before and is not under consideration for publication anywhere else. Submission of the manuscript implies that its publication has been approved by all co-authors as well as by the responsible authorities at the institute where the work has been carried out.

It is mandatory to include a covering letter to the editor which includes short information about the subject of the research, its novelty and significance; state that all the authors agree to submit this paper to Ukrainian Food Journal; that it is the original work of the authors.

**Manuscript requirements**

Authors must prepare the manuscript according to the guide for authors. Editors reserve the right to adjust the style to certain standards of uniformity.

Title page, references, tables and figures should be included in the manuscript body.

Language – English

Manuscripts should be submitted in Word.

Use 1.0 spacing and 2 cm margins.

Use a normal font 14-point Times New Roman for text, tables, and sings on figures, 1.0 line intervals.

Provide tables and figures in the text of the manuscript.

Consult a recent issue of the journal for a style check.

Number all pages consecutively.

Abbreviations should be defined on first appearance in text and used consistently thereafter. No abbreviation should be used in title and section headings.

Please submit math equations as editable text and not as images (It is recommend software application MathType or Microsoft Equation Editor)

Minimal size of the research article (without the Abstract and References) is 10 pages; for the review article minimal size is 25 pages (without the Abstract and References).

**Manuscript should include:**

**Title** (should be concise and informative). Avoid abbreviations in it.

**Authors' information**: the name(s) of the author(s); the affiliation(s) of the author(s), city, country. One author has been designated as the corresponding author with e-mail address. If available, the 16-digit ORCID of the author(s).

**Declaration of interest**

**Author contributions**

**Abstract**. The **abstract** should contain the following mandatory parts:

**Introduction** provides a rationale for the study (2-3 lines).

**Materials and methods** briefly describe the materials and methods used in the study (3-5 lines).

**Results and discussion** describe the main findings (23-26 lines).

**Conclusion** provides the main conclusions (2-3 lines).

The abstract should not contain any undefined abbreviations or references to the article.

**Keywords.** Immediately after the abstract provide 4 to 6 keywords.

**Text of manuscript**

**References**

**Manuscripts should be divided into the following sections:**

- **Introduction**
- **Materials and methods**
- **Results and Discussion**
- **Conclusions**
- **References**

**Introduction.** Provide a background avoiding a detailed review of literature and declare the aim of the present research. Identify unexplored questions, prove the relevance of the topic. This should be not more than 1.5 pages.

**Materials and methods.** Describe sufficient details to allow an independent researcher to repeat the work. Indicate the reference for methods that are already published and just summarize them. Only new techniques need be described. Give description to modifications to existing methods.

**Results and discussion.** Results should be presented clearly and concisely with tables and/or figures, and the significance of the findings should be discussed with comparison with existing in literature data.

**Conclusions.** The main conclusions should be drawn from results and be presented in a short Conclusions section.

**Acknowledgments**(if necessary)**.** Acknowledgments of people, grants, or funds should be placed in a separate section. List here those persons who provided help during the research. The names of funding organizations should be written in full.

Divide your article into sections and into subsections if necessary. Any subsection should have a brief heading.

**References**

Please, check references carefully.

The list of references should include works that are cited in the text and that have been published or accepted for publication.

All references mentioned in the reference list are cited in the text, and vice versa.

Cite references in the text by name and year in parentheses. Some examples:

(Drobot, 2008); (Qi and Zhou, 2012); (Bolarinwa et al., 2019; Rabie et al., 2020; Sengev et al., 2013).

Reference list should be alphabetized by the last names of the first author of each work: for one author, by name of author, then chronologically; for two authors, by name of author, then name of coauthor, then chronologically; for more than two authors, by name of first author, then chronologically.

If available, please include full DOI links in your reference list (e.g. "https://doi.org/abc").

**Reference style**

**Journal article**

Please follow this style and order: author's surname, initial(s), year of publication (in brackets), paper title, *journal title (in italic)*, volume number (issue), first and last page numbers, e.g.:

> Ivanov V., Shevchenko O., Marynin A., Stabnikov V., Gubenia O., Stabnikova O., Shevchenko A., Gavva O., Saliuk A. (2021), Trends and expected benefits of the breaking edge food technologies in 2021–2030, *Ukrainian Food Journal*, 10(1), pp. 7-36, https://doi.org/10.24263/2304-974X-2021-10-1-3

The names of all authors should be provided. Journal names should not be abbreviated.

**Book**

Deegan C. (2000), *Financial Accounting Theory*, McGraw-Hill Book Company, Sydney.

**Book chapter in an edited book**

Fordyce F.M. (2013), Selenium deficiency and toxicity in the environment. In: O. Selinus (Ed.), *Essentials of Medical Geology*, Springer, pp. 375–416, https://doi.org/10.14453/10.1007/978-94-007-4375-5_16

**Online document**

Mendeley, J.A., Thomson, M., Coyne, R.P. (2017), *How and when to reference*, Available at: https://www.howandwhentoreference.com

**Conference paper**

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.

**Figures**

All figures should be made in graphic editor using a font Arial.

The font size on the figures and the text of the article should be the same.

Black and white graphic with no shading should be used.

The figure elements (lines, grid, and text) should be presented in black (not gray) colour.
Figure parts should be denoted by lowercase letters (a, b, etc.).
All figures are to be numbered using Arabic numerals.
Figures should be cited in text in consecutive numerical order.
Place figure after its first mentioned in the text.
Figure captions begin with the term Figure in bold type, followed by the figure number, also in bold type.
Each figure should have a caption describing what the figure depicts in bold type.
Supply all figures and EXCEL format files with graphs additionally as separate files.
Photos are not advisable to be used.
If you include figures that have already been published elsewhere, you must obtain permission from the copyright owner(s).

**Tables**

Number tables consecutively in accordance with their appearance in the text.
Place footnotes to tables below the table body and indicate them with superscript lowercase letters.
Place table after its first mentioned in the text.
Ensure that the data presented in tables do not duplicate results described elsewhere in the article.

**Suggesting / excluding reviewers**

Authors are welcome to suggest reviewers and/or request the exclusion of certain individuals when they submit their manuscripts.
When suggesting reviewers, authors should make sure they are totally independent and not connected to the work in any way. When suggesting reviewers, the Corresponding Author must provide an institutional email address for each suggested reviewer. Please note that the Journal may not use the suggestions, but suggestions are appreciated and may help facilitate the peer review process.

**Submission**

**Email for all submissions and other inquiries:**

**ukrfoodscience@meta.ua**

## Ukrainian Journal of Food Science

**Ukrainian Journal of Food Science** публікує оригінальні наукові статті, короткі повідомлення, оглядові статті, новини та огляди літератури.

Тематика публікацій в **Ukrainian Journal of Food Science:**

Харчова інженерія
Харчова хімія
Мікробіологія
Властивості харчових продуктів
Якість та безпека харчових продуктів
Нанотехнології
Процеси та обладнання
Економіка і управління
Автоматизація процесів
Упаковка для харчових продуктів
Здоров'я

**Періодичність журналу 2 номери на рік** (червень, грудень).

Результати досліджень, представлені в журналі, повинні бути новими, мати зв'язок з харчовою наукою і представляти інтерес для міжнародного наукового співтовариства.

**Ukrainian Journal of Food Science** індексується наукометричними базами**:**

EBSCO (2013)
Google Scholar (2013)
Index Copernicus (2014)
Directory of Open Access scholarly Resources (ROAD) (2014)
CAS Source Index (CASSI) (2016)
FSTA (Food Science and Technology Abstracts) (2018)

**Ukrainian Journal of Food Science** включено у перелік наукових фахових видань України з технічних наук, в якому можуть публікуватися результати дисертаційних робіт на здобуття наукових ступенів доктора і кандидата наук (Наказ Міністерства освіти і науки України № 793 від 04.07.2014)

**Рецензія рукопису статті**. Наукові статті, представлені для публікації в «**Ukrainian Journal of Food Science**» проходять «подвійне сліпе рецензування» (рецензент не знає, чию статтю рецензує, і, відповідно, автор не знає рецензента) двома вченими, призначеними редакційною колегією: один є членом редколегії, інший – незалежний учений.

**Авторське право.** Автори статей гарантують, що робота не є порушенням будь-яких існуючих авторських прав, і відшкодовують видавцю порушення даної гарантії. Опубліковані матеріали є правовою власністю видавця «Ukrainian Journal of Food Science», якщо не узгоджено інше.

**Політика академічної етики.** Редакція «**Ukrainian Journal of Food Science**» користується правилами академічної етики, викладеними в праці Miguel Roig (2003, 2006) "Avoiding plagiarism, self-plagiarism, and other questionable writing practices. A guide to ethical writing". Редакція пропонує авторам, рецензентам і читачам дотримуватися вимог, викладених у цьому посібнику, щоб уникнути помилок в оформленні наукових праць.

# Редакційна колегія

## Шановні колеги!

Редакційна колегія наукового періодичного видання
«**Ukrainian Journal of Food Science**»
запрошує Вас до публікації результатів наукових досліджень.

### Вимоги до оформлення статей

Мова статей – англійська.

Мінімальний обсяг статті – **10 сторінок** формату А4 (без врахування анотацій і списку літератури)**.**

Для всіх елементів статті шрифт – **Times New Roman**, кегль – **14**, інтервал – 1.

Всі поля сторінки – по 2 см.

### Структура статті:

1. **Назва статті.**
2. Автори статті (ім'я та прізвище повністю, приклад: Денис Озерянко).
3. *Установа, в якій виконана робота.*
4. Анотація. **Обов'язкова** структура анотації:
   - Вступ (2–3 рядки).
   - Матеріали та методи (до 5 рядків)
   - Результати та обговорення (пів сторінки).
   - Висновки (2–3 рядки).
5. Ключові слова (3–5 слів, але не словосполучень).

**Пункти 2–6 виконати англійською і українською мовами.**

6. Основний текст статті. Має включати такі обов'язкові розділи:
   - Вступ
   - Матеріали та методи
   - Результати та обговорення
   - Висновки
   - Література.

За необхідності можна додавати інші розділи та розбивати їх на підрозділи.

7. Авторська довідка (Прізвище, ім'я та по батькові, вчений ступінь та звання, місце роботи, електронна адреса або телефон).
8. Контактні дані автора, до якого за необхідності буде звертатись редакція журналу.

Рисунки виконуються якісно. Скановані рисунки не приймаються. Розмір тексту на рисунках повинен бути **співрозмірним** (!) тексту статті. **Фотографії можна використовувати лише за їх значної наукової цінності.**

Фон графіків, діаграм – лише білий. Колір елементів рисунку (лінії, сітка, текст) – чорний (не сірий).

Рисунки та графіки EXCEL з графіками додатково подаються в окремих файлах.

Скорочені назви фізичних величин в тексті та на графіках позначаються латинськими літерами відповідно до системи СІ.

У списку літератури повинні переважати англомовні статті та монографії, які опубліковані після 2010 року.

## Оформлення цитат у тексті статті:

| Кількість авторів статті | Приклад цитування у тексті |
|---|---|
| 1 автор | (Arych, 2019) |
| 2 і більше авторів | (Bazopol et al., 2021) |

**Приклад тексту із цитуванням:** It is known (Bazopol et al., 2006; Kuievda, 2020), the product yield depends on temperature, but, there are some exceptions (Arych, 2019).

У цитуваннях необхідно вказувати одне джерело, звідки взято інформацію.
Список літератури сортується за алфавітом, літературні джерела не нумеруються.

## Правила оформлення списку літератури

В Ukrainian Food Journalвзято за основу загальноприйняте в світі спрощене оформлення списку літератури згідно стандарту Garvard. Всі елементи посилання розділяються **лише комами**.

**1. Посилання на статтю:**

**Автори А.А. (рік видання), Назва статті, *Назва журналу (курсивом)*, Том (номер), сторінки, DOI.**

Ініціали пишуться після прізвища.

Всі елементи посилання розділяються комами.

1. Приклад:
   Ivanov V., Shevchenko O., Marynin A., Stabnikov V., Gubenia O., Stabnikova O., Shevchenko A., Gavva O., Saliuk A. (2021), Trends and expected benefits of the breaking edge food technologies in 2021–2030, *Ukrainian Food Journal*, 10(1), pp. 7–36, https://doi.org/10.24263/2304-974X-2021-10-1-3

**2. Посилання на книгу:**

**Автори (рік), *Назва книги (курсивом)*, Видавництво, Місто.**

Ініціали пишуться після прізвища.

Всі елементи посилання розділяються комами.

Приклад:

2. Wen-Ching Yang (2003), *Handbook of fluidization and fluid-particle systems*, Marcel Dekker, New York.

**Посилання на електронний ресурс:**

Виконується аналогічно посиланню на книгу або статтю. Після оформлення даних про публікацію пишуться слова **Available at:** та вказується електронна адреса.

Приклади:

(2013), *Svitovi naukovometrychni bazy*, Available at: http://www.nas.gov.ua/publications/q_a /Pages/scopus.aspx

Cheung T. (2011), *World's 50 most delicious drinks*, Available at: http://travel.cnn.com/explorations/drink/worlds–50-most-delicious-drinks–883542

Список літератури оформлюється лише латиницею. Елементи списку українською та російською мовою потрібно транслітерувати. Для транслітерації з українською мови використовується паспортний стандарт.

Зручний сайт для транслітерації з української мови: http://translit.kh.ua/#lat/passport

**Детальні інструкції для авторів розміщені на сайті:**

**http://ukrfoodscience.nuft.edu.ua**

**Стаття надсилається за електронною адресою:**

**ukrfoodscience@meta.ua**

**Наукове видання**

# Ukrainian Journal of Food Science

## Volume 12, Issue 1
## 2024

## Том 12, № 1
## 2024

*Рекомендовано Вченою радою*
*Національного університету*
*харчових технологій*
*Протокол № 5 від 21.12.2023*

**Адреса редакції:**

Національний університет
харчових технологій
Вул. Володимирська, 68
Київ
01601
Україна

**E-mail**:

**Ukrfoodscience@meta.ua**

Підп. до друку 03.09.2024 р. Формат 70x100/16.
Обл.-вид. арк. 2.35. Ум. друк. арк. 2.96.
Гарнітура Times New Roman. Друк офсетний.
Наклад 100 прим. Вид. № 22н/24.

НУХТ 01601 Київ–33, вул. Володимирська, 68

Свідоцтво про державну реєстрацію
друкованого засобу масової інформації
КВ 19324–9124Р
видане 23 липня 2012 року.