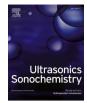


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# The effects of ultrasonic treated whey on the structure formation in food systems based on whey in combination with pectin and agar-agar

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## ABSTRACT

We studied the effects of ultrasonicated whey in food systems with the structure-forming additives such as pectin and agar-agar. The high-intensity (45KHz, 40 W with cavitation) ultrasonic treated whey was used. The conditions and optimal modes of cavitation based ultrasonic processing of curd milk whey have been determined. The mechanism of structure formation has been studied in detail.

From the studies carried out, the scientific basis for the choice of structure-forming agents in food systems was established along with the range of rational concentrations of pectin and agar-agar. It was shown that in the case of processing milk curd whey by the cavitation method, the concentration of the structure former can be reduced by 2 times compared to using non-sonicated whey in the food system thus saving costs on the raw materials.

It was established that high-purity cavity treatment minimizes gel-like food systems set time up to 20% compared to the control within 15 min. The duration of high-purity treatment within 15 min contributes to an increase in penetration pressure, which characterizes the texture of the gel-like food two times.

## 1. Introduction

The scientific basis for the modern strategy of food production and processing is the search for new resources of essential components (protein, vitamins, etc.), the use of non-traditional types of raw materials and the creation of new progressive technologies that allow to increase the nutritional and biological value of the product. These new methods also give the food specified properties thereby significantly increasing the shelf life.

The range of dairy products available in the market is increasing at a rapid pace, as consumer demand for it is growing, and, consequently, the volume of whey as a secondary byproduct in milk processing sector is increasing [1].

The increased whey production problem calls for the rational use of secondary dairy raw materials. A promising direction in solving this problem could be the introduction of "green" technologies in food systems, in the recipe of which whey will become the dominant component. The advantage of products based on milk cottage cheese whey, is that they have a functional benefit to the consumer, contribute to improving the functioning of the gastrointestinal tract, restore or strengthen the gut microbiota composition with good bacteria, have a strengthening effect on the body which is especially important during the COVID-19 pandemic, with which all mankind is fighting [2].

It is known that the main functional and technological properties of milk proteins include solubility, moisture binding, viscosity, structure formation, emulsification, foaming, diphilicity, thermal stability, etc. [3]. Organoleptic and qualitative characteristics of dairy products result from these properties. The role of milk proteins in structured dairy products is well established. Structure formation is one of the most important functional properties of food systems. It is characterized by the ability of proteins and denatured proteins to form, during the interaction of protein-protein, gels that bind a certain amount of water [4]. The main role in the formation of the proteins-based gels is attributed to serum globulins. It is well-known [5] that whey protein solutions form gels at a dry matter concentration of 5% and above. To achieve this type of solutions. the whey must be heated to a temperature of at least 85 °C with a holding time of 5 min. When heated to 120 °C with a holding of 20 s, the gel is formed in milk. During subsequent storage of the product, the gel is destroyed with the restoration of the original viscosity, that depends not only on the presence or absence of a gel, but

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Fig. 1. Laboratory installation for high-intensity (45 KHz) cavitation processing of native milk curd whey.

## Table 1

Table 2

Physicochemical parameters of native milk whey.

Product (native milk whey (cottage cheese) The name of the physical and chemical indicator	Value	
fat content,%	$0.05\pm0.02$	
protein content,%	$0.46\pm0.04$	
dry matter content,%	$5.72\pm0.04$	
pH	$4.65\pm0.05$	
Calciumcontent (mg/100 g)	$53.94\pm2$	
Density (kg/m <sup>3</sup> )	$1023.70\pm1$	
Viscosity Pa*s	$(1.55 \pm 0.1)*103$	
Titrated acidity, °T	$68.0 \pm 2$	
Contentof lactose, %	$\textbf{4.10} \pm \textbf{0.02}$	

also on other factors. Certain functional properties of food systems are achieved by the addition of certain food additives of directed action such as: emulsifiers, stabilizers, and structure-forming agents.

As a rule, structure-formers, due to the uniqueness of their nature (chemical, physical), demonstrate versatility, the use of other properties is possible only in a specific direction.

In the production of food systems, several types of structure-forming agents of various origins are used. Priority is the use of natural structure-forming agents. They are a mixture of hydrocolloids consisting of potassium, sodium, magnesium and calcium-sulfate esters of galactose [6]. For example, the stabilizer agar-agar, is a mixture of two poly-saccharides contained in the cell walls of red seaweed. In the structure of agar-agar, the agar fraction with the greatest gelling ability is isolated, the remaining fractions are chemically related to agaropectin. The solution of agar-agar gives dense gels, hardening at a temperature of 36-42 °C. The structure formation of solutions occurs even at low concentrations of agar-agar in a solution of 0.04% [7].

Among the innovative methods for intensifying the processing of milk whey are ultra-high-intensity processing in continuous and pulsed mode, membrane technologies, ultraviolet and infrared radiation, electrical contact heating, treatment in an electrostatic field, acoustic treatment, and others [8]. The existing literature indicates that ultrasound exposure is a particularly promising technology for the dairy industry [9].

The advantage of ultrasonic processing is due to the fact that in many food processing plants, it is possible to combine ultrasonic equipment with the existing ones. This makes it possible to introduce new processing methods for increasing the production efficiency without additional material costs. Ultrasonic transducers are easily integrated into already operating technological equipment, which makes it possible to modernize existing production [10].

When processing raw materials for food production with ultrasound, it is possible to eliminate the use of consistency stabilizers and preservatives, or significantly reduce their amount in the finished product [8].

Currently, there is a need for complete and deep processing of milk whey to protect the environment, because once it gets into wastewater stream, it causes serious harm to the environment [3].

Based on the arguments provided, expediting the development of scientific approaches for the creation of food systems, including structured ones, based on milk whey processed using cavitation action, is obvious and relevant.

## 2. Experimental details

## 2.1. Materials

We used milk curd whey obtained in real production at a dairy plant (Moscow), which meets all the requirements of regulatory documents – GOST 34352-2017 "Milk whey – raw materials". The sampling of milk whey was carried out in accordance with the requirements of GOST 33957-2016 "Milk whey and drinks based on it. Acceptance rules, sampling, and control methods".

Citrus pectin, produced by Andre Pectin APC, manufacturer of the PRC, as well as agar-agar, manufactured by the company Greenagar (PRC), were used as structure-forming agents.

## 2.2. Experimental methods

For cavitation processing of milk whey, a high-intensity (45 KHz) ultrasonic device "UZI-150" (Avangard, Russia) was used (Fig. 1). The radiated power of the device is 40 W, the useful power is  $12.3 \pm 1.8$  W.

The organoleptic properties of milk whey were determined in accordance with GOST R 53438-2009 "Whey. Raw materials. "Mass fraction of fat and protein, expressed in% – by an instrumental express method for determining physical and chemical identification indicators using an infrared analyzer MilkoScan RT-120 in accordance with GOST 32255-2013; mass fraction of lactose, expressed in% – by the iodometric method according to GOST R 54667-2011; mass fraction of dry substances, expressed in% - refractometric method according to GOST R 54669-2011; active acidity, °T-indicator method according to GOST R 54669-2011; active acidity (pH) – by potentiometric method according to GOST 32892-2014; calcium content, expressed in mg% -complexometric method according to GOST 10398-2016; density, expressed in kg /m<sup>3</sup> – by the hydrometric method according to GOST R 54758-2011; viscosity, expressed in Pa-s using an Oswald viscometer [11]. Structural and mechanical indicators – using a texture analyzer from

Physicochemical parameters of serum after high intensity ultrasonic exposure for various time periods.

Indicators Processing Time	Fat content, %	Protein content,%	dry content Substances %	pН	titrated acidity, $^\circ T$	Lactose content, %
15 min	$0.03\pm0.01$	$0.36\pm0.04$	$5.72\pm0.04$	$4.56\pm0.05$	$53\pm2.00$	$\textbf{4.10} \pm \textbf{0.02}$
18 min	$0.03 \pm 0.01$	$0.34\pm0.03$	$5.75\pm0.05$	$\textbf{4.56} \pm \textbf{0.04}$	$53 \pm 1.80$	$4.57\pm0.03$
20 min	$0.03\pm0.01$	$0.35\pm0.02$	$5.85\pm0.03$	$\textbf{4.55} \pm \textbf{0.06}$	$53\pm2.10$	$4.57\pm0.04$
25 min	$0.03 \pm 0.01$	$0.30\pm0.03$	$5.80\pm0.04$	$\textbf{4.53} \pm \textbf{0.05}$	$54\pm2.60$	$4.55\pm0.02$
30 min	$0.03\pm0.01$	$0.35\pm0.03$	$5.70\pm0.03$	$4.50\pm0.04$	$54\pm2.30$	$4.50\pm0.03$

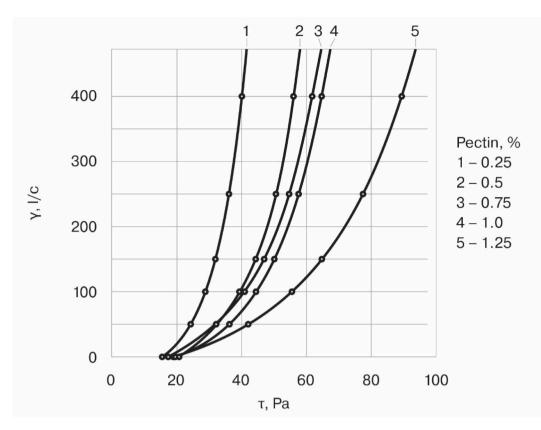


Fig. 2. Dependence of the shear velocity gradient on the shear stress for structured milk product without high-intensity cavitation treatment with pectin content: 1–0.25; 2–0.5; 3–0.75; 4–1.0; 5–1.25, %.

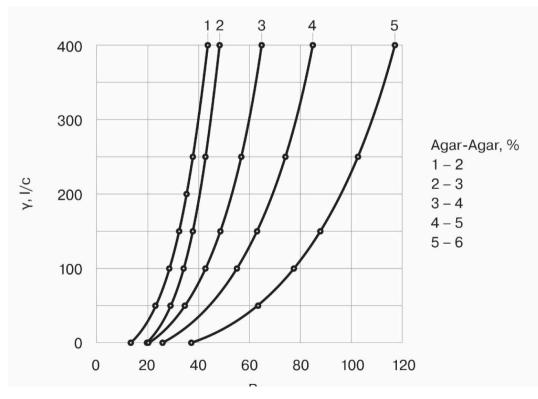


Fig. 3. Dependence of the shear velocity gradient on the shear stress for structured milk product without high-intensity cavitation treatment with agar-agar content: 1–2.0; 2–3.0; 3–4.0;4–5.0;5–6.0, in %.

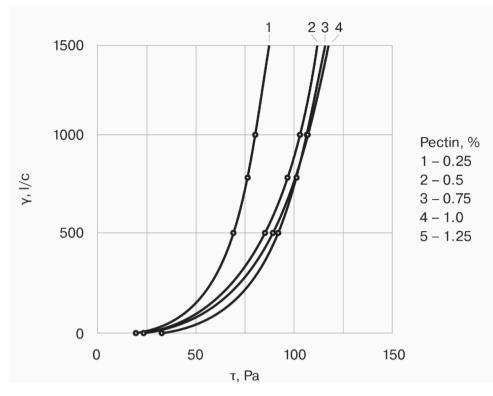
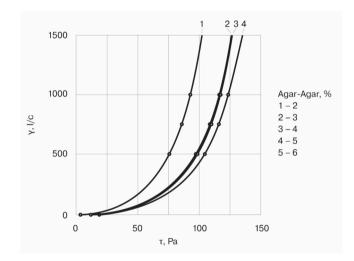


Fig. 4. Dependence of the shear velocity gradient on the shear stress for structured milk product using high-intensity cavitation treatment with pectin content: 1–0.25; 2–0.5; 3–0.75; 4–1.0; 5–1.25, in %.



**Fig. 5.** Dependence of the shear velocity gradient on the shear stress for the structured milk product using high-intensity cavitation treatment with agaragar content: 1–2.0; 2–3.0; 3–4.0; 4–5.0;5–6.0, in %.

Brookfield CT-3. The gelling ability is based on the determination of the critical concentration of gelling (CGC) [7]. Salt content in milk serum by capillary electrophoresis [12].

## 3. Research results and discussion

At the first stage of the research, rational modes of exposure to highintensity cavitation treatment on changes in the constituent components of curd whey were determined. The initial physicochemical parameters of the native curd whey are presented in Table 1.

The processing of whey was carried out under the following exposure

modes: frequency 40 kHz, power 45 W, exposure time - 15, 18, 20, 25 and 30 min.

The results of measuring physicochemical parameters after treatment of serum with high-intensity ultrasound under various modes are presented in Table 2.

The results obtained (Table 2) shows that at different exposure time and fixed power the changes in the chemical composition of whey, pH, titratable acidity and lactose content do not occur. The optimal exposure time is under 15–20 min and responds to organoleptic object characteristics. Under the high-cavity treatment above 20 min there is a negative change in the indicators of taste and aroma [10,13,21].

The production of structured whey food systems (hereinafter referred to as SSPS) is the most dynamically developing sector of the dairy industry. The unifying idea for the development of new SSPS was the creation of low-calorie products enriched with food functional ingredients, balanced in composition, and having a consistency that meets the traditionally established tastes of consumers.

Considering the above context, we studied the gel-forming ability of the structure-forming agents using the structuring agents such as pectin and agar-agar. We carried out experiments to determine the critical concentration of gelation (CGC). The gel-forming ability of pectin is characterized by its critical concentration and gelation time, in addition, it is determined by the degree of esterification, pH of the medium, the sugar content for highly esterified pectins, the presence of polyvalent metal ions, in particular, calcium ions. We found that, in fact, the process of gelation in aqueous solutions is observed at a concentration of 0.25%, and the CCG for the studied samples of structure-forming agents was in the range of 0.25–0.5%.

The optimal concentration of agar-agar solution in water, providing stable gelation at a temperature of  $25^{\circ}$  C, was 1.5–2.0%. Our choice was based on the data from literature [4,7,14], as well as the results of our own research.

The analysis of the flow curves of structured gels without and with the use of high-intesity cavitation treatment of native milk curd whey was carried out based on a large array of experimental conditions in the

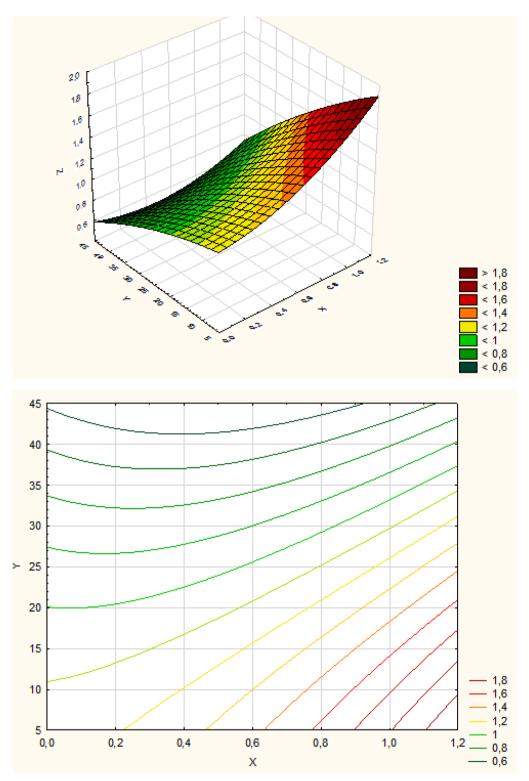


Fig. 6. Response surfaces and cross-section isolines for viscosity with a undestructed structure (A).

established concentration range (Figs. 2-5). It was found that when using cavitation treatment, it is possible to reduce the concentration of structure-forming agents, practically, by a factor of two.

cosity and structuring index for serum gels are given by variants.

$$D_r = \frac{1}{\eta} \tau^N \tag{1}$$

It is established that the nature of the flow curves of the studied  $D_r$  – shear speed,  $c^{-1};\,N$  is the index of liquid structuring;  $\tau\text{-}$  Shearstructure-forming agents is inherent in pseudoplastic systems and can be voltage, Pa; – viscosity, Pa-s or H-s/m  $\eta^2$ . described by the Oswald-de-Villa Eq. (1). As an example, the de-After processing the data for multivariate experiments, regression pendencies of the shear velocity gradient on the shear stress and vis-

equations were obtained that adequately describe the changes in the viscosity of the structure-forming agents depending on the

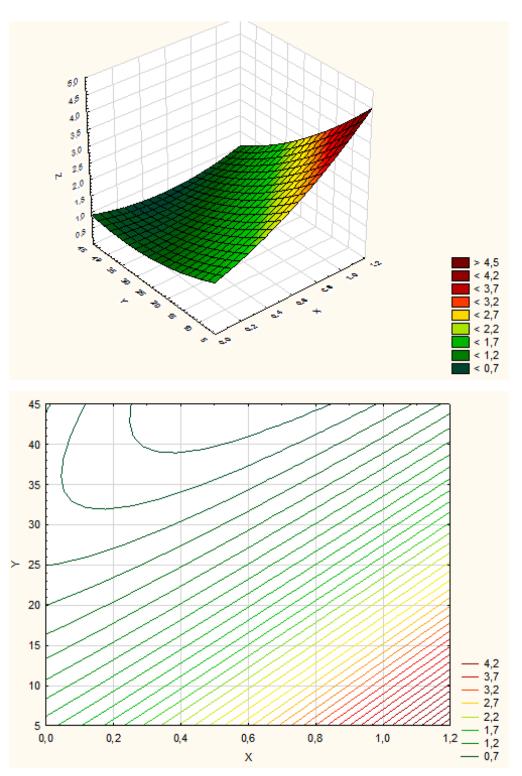


Fig. 7. Response surfaces and cross-section isolines for viscosity with a undestructed structure (B).

concentration and temperature with and without cavitation treatment. For pectin, these dependencies are:

 $Z=1.18+0.20x\text{-}0.01y+0.41x^2\text{-}0.01xy$  without high-intensity cavitation treatment (A).

 $Z=1.67\,+\,1.1x\text{-}0.05y\,+\,0.67x^2\text{-}0.04xy$  with high-intensity cavitation treatment (B).

For agar-agar:

 $Z = 1.83 + 1.55x-0.06y + x^2-0.06xy$  without high-intensity

cavitation treatment (C).

 $Z=3.63\,+\,17.71x\text{-}0.41y\text{-}31.48x^2\text{-}1.13xy\,+\,0.1y^2\text{with}$  with high-intensity cavitation treatment (D).

where: Z-viscosity Pa\*s; x-concentration, %; y-temperature, °C.

To obtain patterns of structure formation in food systems of serum gels with and without cavitation treatment of whey, graphs of output parameter response surfaces were constructed (Figs. 6-9).

The use of the Oswald-de-Villa formula (1) made it possible to carry

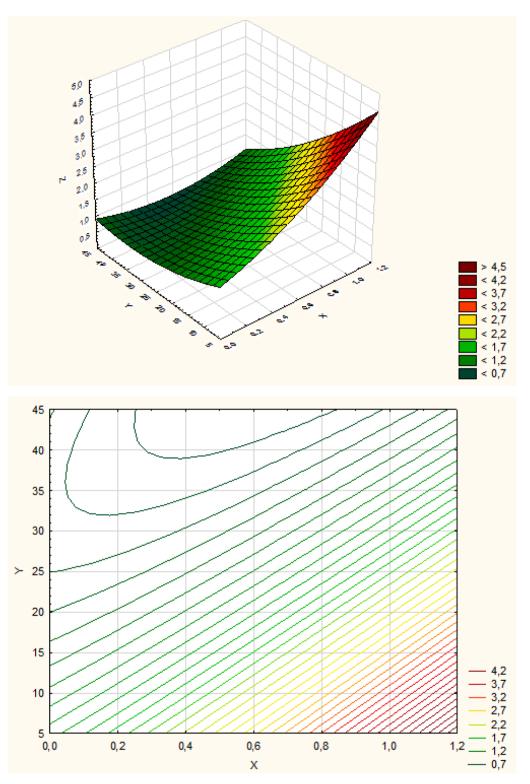


Fig. 8. Response surfaces and cross-section isolines for viscosity in undestructed structure (C).

out an adequate description of the experimental curves in the case of different concentrations of structure-forming agents at different temperatures and methods of exposure (with or without cavitation treatment). The obtained correlation coefficients are close to 1 and in most cases exceeded the value of 0.99, which indicates the correctness of using the Oswald-de-Villa formula (1). As our studies have shown (Figs. 1-4), in full accordance with existing concepts, serum structured gels with various concentrations of structure-forming agents exhibit the

properties of a pseudoplastic medium. Their viscosity changes depending on the shear rate: with an increase in the shear rate, it decreases significantly, which is associated with partial or complete destruction of the structure of the food system. Thixotropy was determined at a shear rate corresponding to its temperature and mechanical effects, which structured dairy products are subjected to during filling (up to 180 rpm) and characterized by its thixotropy coefficient (the ratio of viscosity at the same shear rate with increasing and decreasing load). For example,

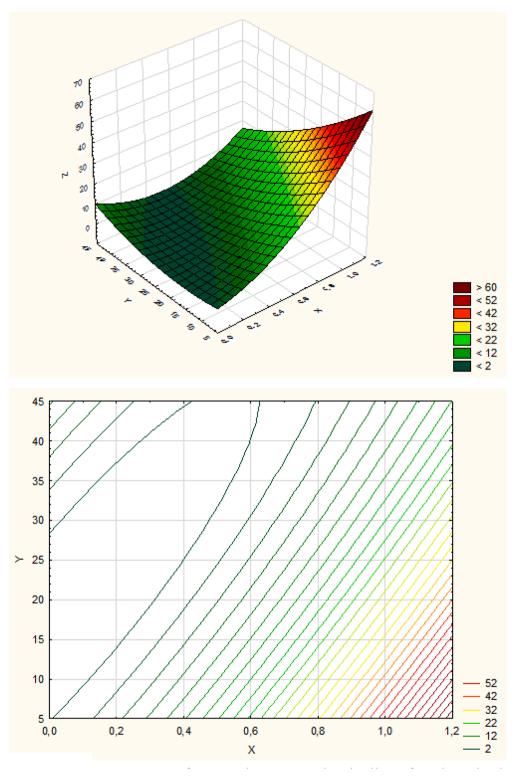


Fig. 9. Response surfaces and cross-section isolines for viscosity in undestructed structure (D).

in the variant with pectin N, the index of structure formation in whey gels after cavitation treatment and structured whey gels without treatment at a concentration of 0.25% increases 1.7 and 1.1 times, respectively; at a concentration of 0.5% - 1.8 and 1.2 times; at a concentration of 0.75% – by 2.2 and 1.3 times, at a concentration of 1.0% – by 2.4 and 1.3 times, respectively. Similar results were obtained using agar-agar as a structure-forming agent.

reduced by 2 times. Considering their high cost of ingredients, the economic efficiency of the cavitation method of exposure is beyond doubt. Thus, from the studies carried out, a scientific proof for the choice of

Subsequently, agar-agar was used in the experiments as a structure-

type of possible structure-forming agents of food systems was carried

out, rational concentrations of pectin and agar-agar were established,

and it was shown that in the case of processing milk curd whey by the

cavitation method, the concentration of the structure former can be

#### Table 3

The results of measuring the structural and mechanical parameters of samples of gel-type food systems with agar-agar based on milk whey.

Samples	Hardening time, min	Penetration pressure, g/cm <sup>2</sup>	Work Destruction MJ
Control sample (without whey processing)	10	$\textbf{98.00} \pm \textbf{2.30}$	$\textbf{3.04} \pm \textbf{0.32}$
Prototype No. 1(high-inten- sity cavitation treatment of whey lasting 15 min)	8	$209.00\pm3.45$	$4.35\pm0.35$
Prototype No2(high- intensity cavitation treatment of whey lasting 20 min)	10	$105.00\pm2.45$	$3.16\pm0.40$

## Table 4

Optimal recipe for gel food systems based on whey with the addition of fruit filler.

Name of the sample of food systems	Milk whey, %	Agar-agar, %	Raspberry puree, %
Samplecontaining cavitation- treated whey	86.60	1.90	11.50
Whey-based sample without cavitation	84.70	3.80	11.50

### Table 5

Structural and mechanical indicators of structured food systems with gel-like appearance, developed according to the optimal recipe.

Index Sample	Hardening time min	Penetration pressure, g/cm <sup>2</sup>	Work Destruction MJ
Control sample (without processing)	10.0	$\textbf{86.00} \pm \textbf{4.50}$	$1.03\pm0.20$
Prototype No1 (with the use of high-intensity cavitation treatment of whey lasting 15 min)	7–8	$166.00\pm3.8$	$3.92\pm0.30$

## Table 6

Total salt composition of gel-like samples based on whey.

Name of the indicator	Relative % Deviation in Measurements	Control sample (without whey processing),	Prototype No1 (duration of cavitation processing of whey 15 min)
Chloride content, mg/kg	(±6.0%)	916.8	823.0
Sulfate content, mg/kg	(±6.0%)	51.03	23.07
Citrate content, g/ kg	(±6.0%)	3.88	2.84
Phosphate content, g/ kg	(±6.0%)	1.46	1.22
Nitrate content, mg/kg	(±18.0%)	3.74	2.271

forming agent for food systems based on milk whey for reasons of economy (the cost of 1 kg of agar-agar on the Russian market, on average, is 60% lower compared to the cost of pectin).

To determine the optimal mode of cavitation action on dairy curd whey to obtain stable gel-like food systems (with the addition of a fruit filler), studies were carried out to identify patterns between the dry

## Table 7

The results of evaluating the quality indicators of gel-type food systems with a
fruit filling based on whey.

Index Sample	Humidity,	Total	Fat
	%	acidity, °T	content, %
Control sample (without whey processing) Prototype No. 1(high-intensity cavitation treatment of whey for 15 min)	$\begin{array}{l} 97.55 \pm \\ 1.75 \\ 94.82 \pm \\ 1.20 \end{array}$	$\begin{array}{l} 59.00 \pm \\ 2.20 \\ 63.00 \pm \\ 1.80 \end{array}$	$\begin{array}{l} 0.03 \pm \\ 0.01 \\ 0.03 \pm \\ 0.01 \end{array}$

matter content in serum and the density of the gel, which were evaluated using structural and mechanical parameters.

The experimental results on the structural and mechanical parameters of samples food systems with a gel-like consistency are presented in Table 3, their formulation was as follows, in %: native milk curd whey-84.7, agar-agar-3.8, raspberry puree-11.5.

From Table 3, it can be noted that when determining the characteristics of the sample, which contained serum after 15 min of cavitation treatment, the penetration pressure indicator increased approximately 2 times, and, therefore, the gel density increased by the same value.

When the structure-forming agent of agar-agar is added to food systems with a gel-like nature, in the amount of 3.8 kg per 100 kg of the finished product, in accordance with the recipe, the density of the gel is 2 times higher than necessary to ensure the indicators of the reference consistency for this type of food systems produced in production conditions [15]. This can be explained by an increase in the degree of its swelling in cavitation-treated milk whey due to a decrease in the total salt content in it after its high-intensity ultrasonic treatment and the associated increase in the level of hydration of biopolymers. From this it follows that a possible decrease in the amount of expensive a structureforming agent for agar-agar in food systems containing milk whey processed using cavitation action.

Based on the results obtained (Table 3), for further studies, the mode of action of high-intensity cavitation on native milk curd whey with a duration of 15 min was chosen as the optimal one.

The optimal recipe for gel food systems based on whey after adjusting the amount of agar-agar and adding a flavoring filler (raspberry puree) is shown in Table 4.

The results of a comparison of the structural and mechanical characteristics of gel-like food systems, developed using milk whey exposed/ or not exposed to high-intensity cavitation, are presented in Table 5.

The results obtained (Table 5) can be theoretically explained by a decrease in the concentration of mineral salts in the serum after its highintensity ultrasonic treatment [12] lasting 15 min. The results of the analysis of the total salt composition of samples of food systems, obtained using the method of capillary electrophoresis, made it possible to confirm these theoretical assumptions (Table 6).

The results on the salt composition of samples of food systems, presented in Table 6, indicate that the use of ultrasonic high-intensity cavitation effects for the processing of milk whey leads to a decrease in the content of chlorides in it by 11%, citrates by 27%, phosphates 16% and nitrates by 40%. compared to a sample containing untreated curd whey. Sulfates contained in milk curd whey are especially susceptible to destruction – their amount after processing decreased by 55%, which once again confirms the working hypothesis of a decrease in the concentration of salts in curd whey after its high-intensity ultrasonic treatment.

The results obtained correspond with the results of the work of Y. Artemova, [16], who noted a decrease in the concentration of salts in water after its cavitation treatment, as well as Australian scientists, Ashokkumar M., B. Zisu, and others, who studied the behavior of whey proteins under cavitation. [17,18].

It has also been established that gel-type food systems based on milk whey subjected to high-intensity ultrasonic treatment have improved organoleptic characteristics – consistency, taste and aroma, in comparison with food systems containing untreated milk whey.

It is known that in the dairy industry there is a problem of undesirable foaming of milk raw materials, since this process contributes to a decrease in the yield of the product, deterioration of its consumer characteristics and acceleration of oxidative processes, which leads to a reduction in the shelf life of the product [19,20].

The results obtained confirm that the degree of foaming decreases (by about 45–50%) when mixing curd whey after applying highintensity ultrasonic treatment, which improves the consumer characteristics of gel-like food systems.

The upper row shows samples made using untreated milk whey; in the bottom row, samples made using milk whey, treated with highintensity cavitation for 15 min.

It is noted that gel-like food systems containing milk whey subjected to high-intensity cavitation treatment for 15 min after adding a fruit filler (in particular, raspberry puree) have a more intense bright color compared to a sample containing untreated milk whey and fruit filler.

Based on the foregoing, it can be concluded that 15-minute highintensity ultrasonic treatment has a positive effect on improving the color characteristics of food systems, as well as reducing the degree of foaming in the finished product, which, in combination, helps to improve their consumer characteristics.

The results on the physicochemical parameters of samples of gel-type food systems with a fruit filler are presented in Table 7. They indicate a decrease in the value of the moisture index and an increase in the value of the acidity index, which causes an increase in the shelf life of gel-type food systems based on milk whey subjected to high-intensity cavitation treatment within 15 min.

## 4. Conclusions

The experimental evidence indicates the effectiveness of highintensity cavitation treatment for the structuring of food systems based on milk curd whey.

To improve the structuring process of milk-based food systems, it is recommended to use pectin and agar-agar as a structure-forming agent, and their concentration can be halved when using high-intensity cavitation action lasting no more than 15 min.

The obtained results on measuring the total salt content of food systems based on milk curd whey indicate a significant decrease in chlorides -11%, sulfates -55%, citrates -27%, phosphates -16%, nitrates -40% in systems containing cavitation processed milk whey compared to food systems with untreated whey, which contributes to the increase in the degree of swelling of structure-forming agents in food systems, and, as a consequence, a decrease in their concentration and an increase in the level of hydration.

The use of high-intensity cavitation for the processing of curd whey helps to increase the efficiency of its use in food systems (for example, in the production of marmalade, milk desserts), which has a beneficial effect on sustainable production.

## **Declaration of Competing Interest**

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Olga Krasulya reports financial support was provided by Ministry of Science and Higher Education of the Russian Federation.

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