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CHOICE JUSTIFICATION OF DAIRY RAW MATERIALS ACCORDING TO INDICATORS OF THEIR STRUCTURE FOR OBTAINING SELENIUM-PROTEIN DIETARY SUPPLEMENTS

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Abstract

Aim. To substantiate the choice of raw materials for selenium-protein dietary supplements, namely the type of whey as a matrix for their production, and to determine the subsequent possibility of using additives in the technology of culinary products for special purposes. Methods. The determination of the structural characteristics of whey lactose samples was carried out using a Quattro ESEM electron microscope at a magnification of 10,000 and 1,000 times. The analysis of the quantitative and dimensional characteristics of the investigated particles was carried out using the MS Excel software. Results. The method and mode of whey thickening have been substantiated, and the homogeneity analysis of lactose-free and lactose-containing whey samples under different thickening modes has been carried out. The ratio of the constituents of selenium-protein dietary supplements and the nature of the interaction of the components with each other have been determined. Conclusions. Analysis of the structure of model systems with the use of low-lactose milk whey and selenium salts condensed by the contact method made it possible to establish the rational content of the latter in the range of 0.0087...0.0176 %, at which the formation of a homogeneous finely dispersed microstructure is noted. This allows foreseeing high technological properties, stability during storage and use of done selenium-protein dietary supplements.

Keywords: lactose; milk whey; microstructure; granulometric composition; selenium-protein dietary supplements.

ОБҐРУНТУВАННЯ ВИБОРУ МОЛОЧНОЇ СИРОВИНИ ДЛЯ ОДЕРЖАННЯ ДОБАВОК ДІЄТИЧНИХ СЕЛЕН-БІЛКОВИХ ЗА ПОКАЗНИКАМИ ЇЇ СТРУКТУРИ

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Анотація

Мета. Обґрунтувати вибір сировини для селен-білкових дієтичних добавок, а саме виду сироватки в якості матриці для їх виробництва, і визначити подальшу можливість використання добавок в технології кулінарних виробів спеціального призначення. Методи. Визначення структурних характеристик зразків молочної сироватки проводили за допомогою електронного мікроскопа Quattro ESEM при збільшенні в 10000 і 1000 разів. Аналіз кількісних і розмірних характеристик досліджуваних частинок проводився з використанням програми MS Excel. Одержані результати. Обґрунтовано спосіб і режим згущення сироватки, проведено аналіз однорідності зразків сироватки безлактозної і з її вмістом при різних режимах згущення. Визначено співвідношення складових селен-білкових добавок і характер взаємодії їх компонентів. Висновки. Аналіз структури модельних систем з використанням низьколактозної молочної сироватки і солей селену, конденсованих контактним способом, дозволив встановити раціональний вміст останніх в діапазоні 0.0087...0.0176 %, при якому спостерігається утворення однорідної дрібнодисперсної мікроструктури. Це дозволяє передбачати високі технологічні властивості, стабільність стуктури готових селен-білкових добавок при їх зберіганні і застосуванні.

Ключові слова: лактоза; молочна сироватка; мікроструктура; гранулометричний склад; селен-білкові дієтичні добавки.

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Introduction

Scientists have found that the complete exclusion of dairy products in the case of lactose malabsorption can cause concomitant diseases associated with a deficiency of Ca, Mg and vitamin D. To solve the problem, a number of researchers suggest the possibility of complete or partial removal of lactose from milk raw materials, its further processing and use [1–8].

Protein-carbohydrate raw milk, in particular milk whey, is promising from this point of view. One of the ways of its processing, which has found practical application, is thickening. The thickening process of whey can be accompanied by crystallization of lactose, what will contribute to the formation of a powdery structure. In order to prevent the crystallization of lactose in dairy products, the method of its enzymatic hydrolysis is used. This method of processing whey successfully solves the problem of creating low-lactose dairy products. Condensed milk whey with a reduced lactose content can be used as a basis for the production of food products or semi-finished products in the diet of patients suffering malabsorption.

Previous studies have substantiated the methods and modes of whey lactose fermentolysis and its thickening, the expediency of a combination with selenium salts in the production of a semi-finished product for selenium-protein dietary supplements [9].

Every year at the European Union, the dairy industry produces 75 million tons of whey, about 40% of the volume is utilized. To solve the problem of marketing whey, as a by-product of the dairy industry, a Special Program was adopted by the European Whey Association (EWPA) [10]. That is why the processing of secondary milk raw materials, namely whey, is an urgent task. Research in this direction is promising and their successful completion will allow developing a number of new effective technologies for food products.

One of the ways to increase the amount of nutrients in whey is achieved by concentration (freeze drying, thickening, ultrafiltration, etc.). But it is known that the quality of food systems is determined by such factors as the shape, size and uniformity of the distribution of particles in a dispersed system. Therefore, the characteristics of the microstructure of its components are important in the process of creating structured culinary products [11].

It was found that the crystallization of lactose is influenced by its concentration in the serum, the pH level, the presence of denatured protein, the degree of supersaturation as a result of thickening, the presence of impurities, the stirring rate and the cooling rate [12].

Since condensed milk whey is a polydisperse system, it is important to determine the size and shape of dispersed particles. These parameters determine the properties of dispersed systems, and, consequently, the area of their application. Therefore, the issue of developing technologies for semi-finished products and food products for patients with lactose malabsorption is currently relevant.

Results and discussion

To determine the nature of the texture of the sample, microphotographs were analyzed at a multiple magnification of ×10000 and ×1000 times (fig. 1, a, b). The crystallization concentration in 1 cm^2 of the product is 450 thousand crystals. The value of the calculated average numerical diameter of the crystals is 3.84 μm with a coefficient of variation of 1.35 %. The system is characterized as fairly homogeneous, since crystals are formed up to 10 μm in size during thickening.

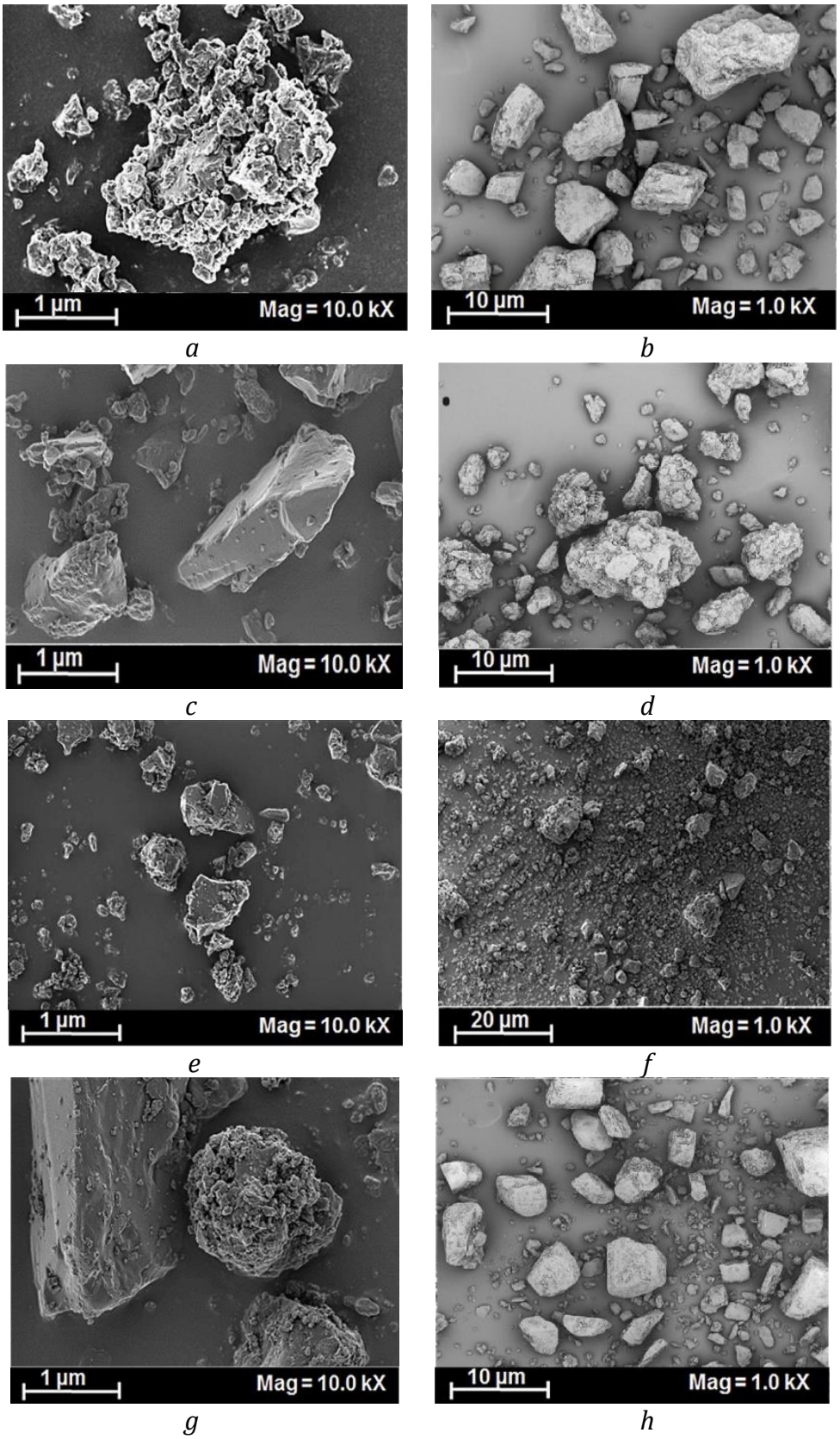
The sample №2 (non-fermented milk whey (NFW)) was not subjected to preliminary fermentolysis, and therefore it was characterized by a high lactose content of 5%. Microphotography of the sample №2 was carried out at multiple magnifications of ×10000 and ×1000 times (fig. 1, c, d), since it had a larger crystal size comparing to the sample №1.

Thickening was carried out in the same way as in the previous sample, under conditions of weak rarefaction at P = –0.1 Pa at a reduced boiling point.

The concentration of crystallization in 1 cm^2 of the product is 92 thousand crystals, the calculated value of the average number diameter is 15.54 μm with a coefficient of variation of 5.1%. The system is characterized by a mealy consistency, which is confirmed by the calculated values. Mealy has already began to be felt at a crystal size above 11 μm and negatively affects the consistency of the resulting product. The continuation of crystal formation was also noted when the sample was cooled.

The sample №1 was subjected to preliminary enzymatic hydrolysis of lactose with subsequent thickening of low-lactose whey by the contact method with an open evaporation surface (LLWCM). Due to the uneven temperature distribution in the entire volume of the product and its relatively higher values, melanoidin

formation was observed, as a result of which the color of the product from white with a shade of green was transformed into a rich cream.



a *b* *c* *d* *e* *f* *g* *h*

Fig. 1. The sample № 1 (LLW) at a multiple increase in: *a* – ×10000 times; *b* – ×1000 times; the sample № 2 (NFW) at a multiple increase in: *c* – ×10000 times; *d* – ×1000 times; the sample №3 (LLWCM) at multiple magnification in: *e* – ×10000 times; *f* – ×1000 times; the sample №4 (NFWCM) at multiple magnification in: *g* – ×10000 times; *h* – ×1000 times

The magnification factor during microphotography of LLWCM was ×10000 and ×1000 times (fig. 1, e, f), as well as the low-lactose sample had a smaller comparative crystal size than NFWsample.

The concentration of crystallization in 1 cm^2 of the product is 271 thousand crystals. The value of the calculated number average crystal diameter is 6.7 μm with a coefficient of variation of 4.63%. The investigated sample №3 was characterized by the presence of powdery, which begins to be felt at a crystal size of 11–30 μm. Upon cooling, the crystallization process proceeded more rapidly.

The sample №2 was not subjected to enzymatic hydrolysis of lactose (NFWCM). Thickening was carried out by a contact method with an open evaporation surface, in connection with which the product in the process changed color to a rich caramel.

Microphotography of the sample №2 was performed at a multiple magnification of ×10000 and ×1000 times (fig. 1, g, h), since the sample had a large crystal size in comparison with the samples LLW and LLWCM.

The concentration of crystallization in 1 cm^2 of the product is 27 thousand crystals, the calculated value of the average number diameter is 26.15 μm with a coefficient of variation of 6.19%. The sample №2, with crystal sizes of more than 31 microns, has a coarse sandy consistency that is strongly felt.

Analyzing the samples of view at a magnification of ×1000 times it is seen that the samples №1 and №3 are characterized by a sufficiently homogeneous structure. The samples №2 and №4 are characterized by an uneven distribution of dispersed particles and their grouping into complexes. At the micro level, there is an alternation of smooth and granular areas of the micron level (0.1...5 μm), which probably indicates the high viscoelastic properties of the system. This, in turn, suggests that the resulting food systems are highly reactive and can be used for the production of structured food products.

Taking into account the calculated values, the granulometric composition of the studied systems of condensed milk whey was determined, what makes it possible to establish the percentage of particles with a certain size interval. The percentage of individual fractions is presented in the form of histograms of the studied samples, which is a stepped graph of the dependence of the relative content of particle fractions on their size.

For a visual representation of the degree of polydispersity of the analyzed systems and the content of each fraction in them, the histogram was constructed in the form of a diagram consisting of several rectangles. The number of drawn rectangles corresponds to the number of fractions in the system.

The bases of the rectangles are between $d_i - \Delta d_i/2$ and $d_i + \Delta d_i/2$ values of diameters. Due to the equal values of the diameters in the fractions, the constructed histograms give a visual representation of the degree of polydispersity of the analyzed systems and the content of each fraction in them. When constructing differential curves of the particle size distribution, the values of their diameters are counted along the abscissa axis, and their distribution density along the ordinate axis.

When assessing the degree of crystallization of lactose, it is not enough to provide a visual description of the structure of the system. The prototypes contain particles, the minimum and maximum sizes of which differ several times. Therefore, the determination of the average particle size is considered insufficient, and for a more complete description, the granulometric composition of lactose crystals in the samples of condensed whey was determined (fig. 2).

The properties of a dispersed system are described by the distribution function of the number of crystals on their average size, the numerical values of the distribution density of crystals ($\Delta Q/\Delta d_i$, $\%/\mu m$) plotted along the ordinate, along the abscissa plot the values of the average diameters (Δd_i, μm). In accordance with the calculated data, a histogram of the granulometric composition of the sample №1 (LLW) was constructed using a differential curve (fig. 2).

The sample №1 is characterized by crystals of smaller size and the smallest range of size values, which indicates a high homogeneity of the system.

The sample №2, despite the relatively small size range of lactose crystals, is marked by a high concentration of particles with a size of 5–10 μm. That is, the sample №2 has a lack of mealy, which is determined by the organoleptic.

The granulometric composition of the crystals of sample №3 is shown in the histogram in fig. 2. The sample №3 contains a large number of crystals with diameter of 4–9 μm, but at the same time, it is distinguished by a high scatter of particle sizes from 0.5 to 4 μm, which impart heterogeneity to the structure and affect the consistency. In addition, due to such a dimension,

it is difficult to predict the result of interaction with other substances, because lactose particles, depending on their size, differ in different reactivity.

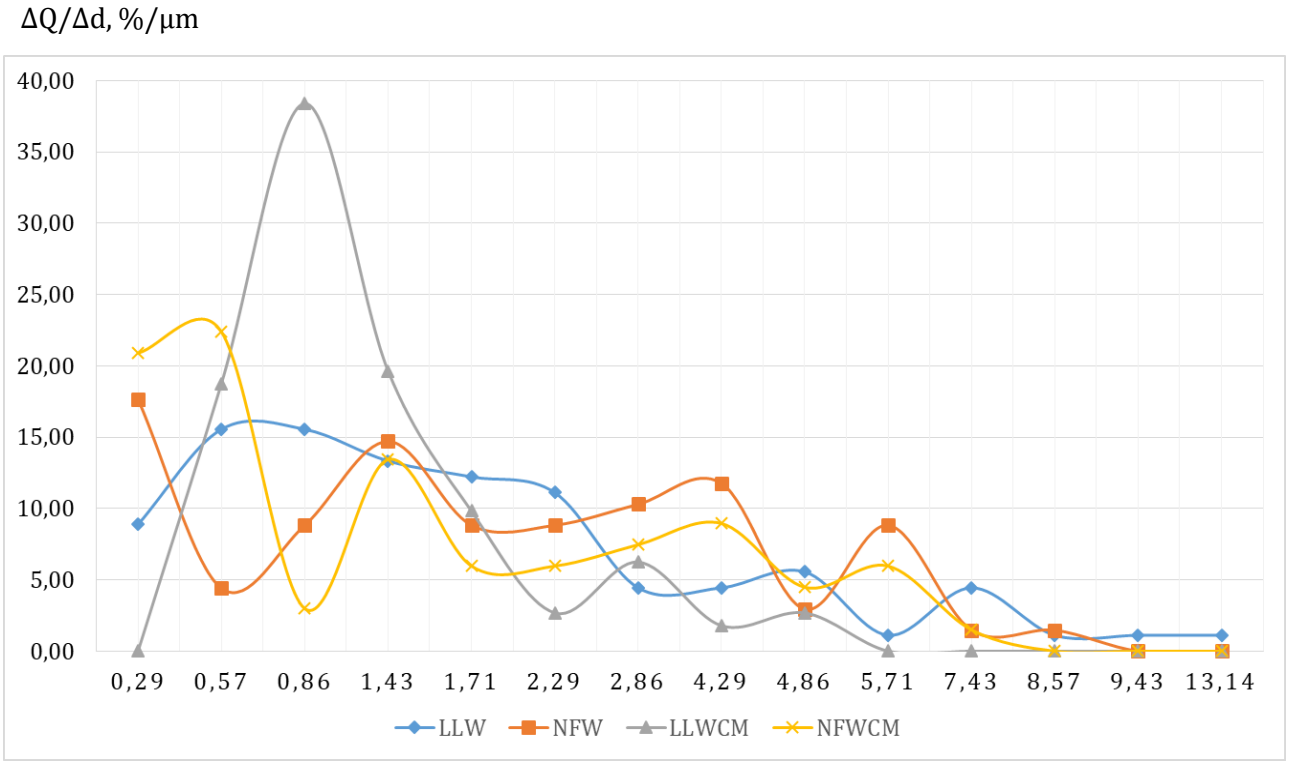


Fig. 2. Granulometric compositions of the samples №1 (LLW), №2 (NFW), №3 (LLWCM) and №4 (NFWCM)

Based on the data obtained during the experiment, a histogram of the granulometric composition of the investigated sample №4 was constructed (fig. 2). The sample №4 is characterized by the content of lactose crystals, the average size of which is in the range of 3–8 μm. This can significantly affect the rheological properties of model systems.

Accordingly, the only sample that has a uniform consistency is the sample №3 – low-lactose milk whey condensed by a contact method. It can be recommended for use as part of multicomponent food systems, as it has good organoleptic characteristics and functional and technological properties.

As can be seen from fig. 2, with a decrease in the degree of dispersion, the width of the differential distribution curves becomes narrower, and their maximum increases.

It is known that dispersed systems, depending on the particle size distribution, can be restructured over time. This means that the granulometric composition of the systems can vary during storage under the same conditions, which will manifest itself in the form of clumping, powdery or sandiness [13–17]. The granulometric composition of lactose crystals during storage of prototypes for 30 days was investigated. The research results are shown in the table 1.

Table 1

Dynamics of changes in the dimensional characteristics of lactose crystals

Sample	Crystal fraction size, μm	The ammount of crystals in 1 cm², % Day			
		1	10	20	30
№ 1 LLW	≤2	66	37	30	24
	3 – 5	26	45	49	52
	6 – 8	6	12	13	14
	9 – 11	2	4	6	7
	≥12	1	2	2	3
№ 2 NFW	≤2	88	69	55	44
	3 – 5	12	18	19	23
	6 – 8	–	13	24	25
	9 – 11	–	–	2	7
	≥12	–	–	–	1
№ 3 LLWCM	≤2	100	62	43	21
	3 – 5	–	34	39	46

Continued Table 1

	6 – 8	–	4	18	28
	9 – 11	–	–	–	4
	≥12	–	–	–	1
№ 4 NFWCM	≤2	93	66	46	23
	3 – 5	7	23	25	34
	6 – 8	–	11	23	28
	9 – 11	–	–	6	13
	≥12	–	–	–	–

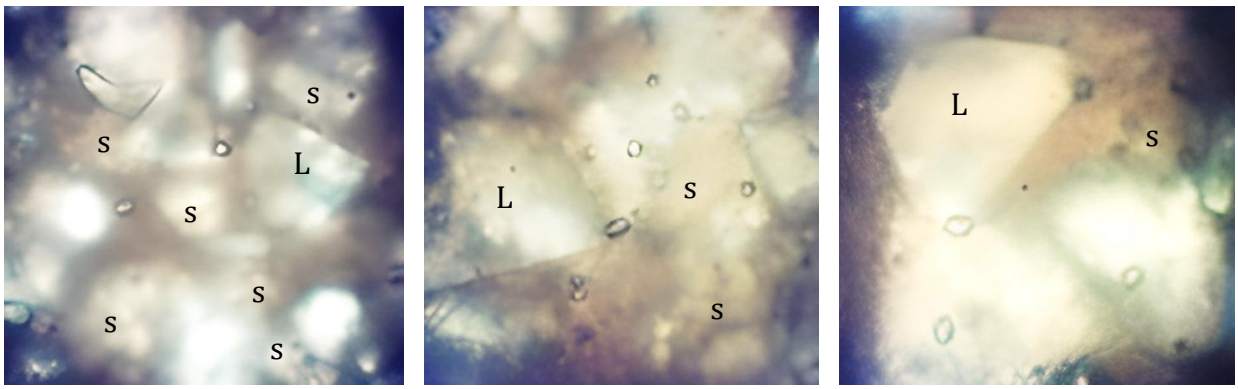
The results of studying the dynamics of changes in the dimensional characteristics of lactose crystals presented in the table 1, showed that lactose crystals in all samples enlarged. As a result of consolidation they were combined into complexes. Already after 10 days of storage, crystals of large initial values are observed in all samples. On day 30, the sample №3 was characterized by a decrease in the homogeneity of the system, while the sample №1 partially lost its fluidity due to a significant increase in the centers of crystallization. The samples №2 and №4 were noted by an increase in the centers of crystallization, which contributed to an increase in the signs of mealy. The reason for the increase in the crystallization of lactose after 10 days of storage is the completion of the induction (latent) period, when the concentration of lactose remains constant, but the formation of embryos of a new phase occurs. At the second stage, a rapid increase in its concentration and crystal growth begins.

For further studies, the sample №3 was used, which has the maximum uniformity in size and distribution of particles.

The information obtained from the analysis of the microstructure allows us stating that the low-lactose whey condensed by the contact method has a fairly uniform fine-crystalline structure at different storage times. Unlike other samples, this allows us recommending it for use in production technology of the selenium-protein dietary supplements.

The microstructure of model systems, in which LLWCM and selenium salts (SS) were used in the amount of 0.0087…0.05 %, was investigated (fig. 3).

Analysis of the structure of the model system with a LLWCM content of 0.0087 ... 0.0176% (fig. 3, *a*) showed that it is characterized by a decrease in the possibility of visualizing dispersed phases, which is associated with the formation of a more homogeneous finely dispersed microstructure, and, probably, the formation of multiple Selenium-protein complexes [18–22].



a *b* *c*

Fig. 3. Microstructure of model systems of Selenium-protein dietary supplements at 23.3 ºC (increase in 300 times) with a mass fraction of Selenium salts:
***a* – 0,0087…0,0176 %; *b* – 0,018…0,036%; *c* – 0,037…0,05%,**
where L – LLWCM, s – Selenium-protein complexes

At the same time, the structure of the model system with an LLWCM content of 0.018...0.036 % (fig. 3, *b*) is characterized by the homogeneity of the distribution of protein globules in the protein-plant mixture and the absence of Selenium-protein complexes. In particular, the microstructure of the sample is represented mainly by small particles of cellulose with a width of 3–4 μm, and an insignificant amount of isolated rounded particles with a size of 1–1.5 μm [23]. At the same time, the microstructure of a sample with an LLWCM content of 0.037...0.05 %% (fig. 3, *c*) is represented exclusively by isolated rounded

particles with a size of 0.5-1 μm with impaired visibility without clear contours [24].

Experimental part

Cheese-separated milk whey was the subject of the study. For the production of the sample №1, low-lactose whey (hereinafter – LLW) was concentrated in vacuum, and for the sample №2 – non-fermented one (hereinafter – NFW). In the sample №1 the amount of dry matter is 40%, the lactose content is 2 %. In the sample №2 dry matter is also 40 %, while the lactose content is 28 %. For the preparation of the sample № 3 low-lactose milk whey was concentrated by the contact method (hereinafter referred to as LLWCM). In the sample №3 the amount of dry matter is 40 %, and the lactose content is 2 %. Non-fermented milk whey for the sample №4 was concentrated by the contact method (hereinafter referred to as NFWCM). Thickening was carried out until the amount of dry matter in the sample №4 reached 40 %, with lactose content of 28 %. Anti-crystallizers were not used.

The uniformity of crystallization was assessed by the value of the average crystal diameter. Determination of the size of lactose crystals was carried out by microscopy using a microscope Quattro ESEM. The clearance of the optical microscope is 0.1–0.2 μm. The results were recorded by microphotography. The consistency was assessed by optical microscopy using a bright field, according to which a uniformly illuminated field in the image plane can be obtained in transmitted light. The image of an object becomes visible as a result of partial absorption and rejection of the incident light by its individual elements. During the preparation of the analysis, the sample was not heated or diluted in order to prevent the destruction of milk sugar crystals. The uterine solutions were prepared by applying a thin drop of the sample to a glass slide and covered with a glass. The research was carried out in a freshly prepared product and during its storage [25].

The crystallization concentration was calculated from the value of the equivalent crystal diameter per 1 cm² of the product [26]. The equivalent diameter was determined using the length and width of the particle:

$$\bar{d} = \frac{(l+b)}{2}. \quad (1)$$

The concept of equivalent diameter is used in connection with the measurement of a large number of particles in their random arrangement and orientation. It is the equivalent diameter that was used to estimate the specific surface area of the particles. Particle size analysis results obtained by microscopy are expressed by the numerical particle size distribution.

In order to calculate the average particle size, the method for determining the average numerical diameter $\overline{d_n}$ was used. The value of $\overline{d_n}$ corresponds to the diameter of the particles of such a monodisperse system, which, for the same number of particles, has the same sum of diameters as in the given disperse system. Calculations are made according to the formula:

$$\overline{d_n} = \sum_i \frac{n_i}{\sum_i n_i} \times d_i = \sum_i f_{ni} \times d_i ,$$

were n_i is a number of particles in a *i*-th fraction of d_i diameter;

$\sum_i n_i$ is a total number of particles in the system;

is a numerical fraction of the *i*-th fraction.

Due to the impossibility of calculating the concentration of crystallization in the entire volume of the product, the coefficient of variation was calculated to characterize the degree of polydispersity of the system under study:

$$K_n = \frac{\sigma}{\overline{d_n}} \times 100\% \quad (3)$$

were σ is a standard deviation, which characterizes the breadth of the particle size distribution.

The numerical value of the standard deviation was calculated for the number average diameter by the formula:

$$f_{ni} = n_i / \sum_j n_j$$

$$\sigma = \left\{ \sum_i f_{ni} \left(d_i - \overline{d_n}\right)^2 \right\}^{\frac{1}{2}} \quad (4)$$

The application of such a methodology will allow a more accurate study of the microstructure of selenium-protein dietary supplements and can be applied to similar groups of products and food environments.

It is known that the consistency of condensed dairy product depends on the number of lactose crystals and their size [27]. Estimation of the degree of crystallization of lactose is determined by the average size of its crystals in the prototypes. The degree of crystallization is calculated per 1 cm² of the sample. Based on the calculated numerical values, it is possible to objectively evaluate the structure of the samples under study.

The sample №1 (low-lactose whey (LLW)) was subjected to preliminary fermentolysis of lactose with subsequent thickening in vacuum at P = –0.1 Pa and reduced boiling point 50±2 ºC [28]. The sparing mode of processing allows keeping color of a product, prevents coagulation of proteins and destruction of biologically active substances.

Conclusions

Analysis of the structure of model systems with the use of low-lactose milk whey and selenium salts condensed by the contact method made it possible to establish the rational content of the latter in the range of 0.0087...0.0176%, at which the formation of a homogeneous finely dispersed microstructure is noted. This allows ensuring high technological properties, stability during storage and use of done selenium-protein dietary supplements.

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