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MODIFICATION OF THE PROPERTIES OF MILK-FAT EMULSIONS WITH THE PHASE STRUCTURE OF "OIL IN WATER" IN THE DEPENDENCE ON THE MASS PART OF THE LIPOID AND THE STABILIZING SYSTEMS

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ABSTRACT

The composition and properties of cream with fat levels from 30% to 70% were investigated. It has been established that the decrease of fat level and, accordingly, the increase of plasma level in the cream leads to significant changes in the physicochemical parameters of the fat emulsion. Accordingly, the production of low-fat dairy products requires adjustment of the cream properties. It has been shown that using different doses of structure stabilizers: QNA colloid as a consistency stabilizer in the amount of from 1 to 2% and the Dimodan emulsifier U/G – from 0 to 1 % (Danisco, Denmark) changes the physicochemical properties (effective viscosity, sedimentation stability) and organoleptic properties (consistency, taste) of cream with fat level 40%. It has been conducted mathematical modeling, aimed to calculate dosing for improving the properties of milk-fat emulsions, structure formation and further ensuring of the necessary consistency of cream pastes. Optimal doses of colloid QNA as a consistency stabilizer and a U/G Dimodan emulsifier have been determined. It has been established that their content should be 1,0% and 0,5%, relatively, for pastes with a fat level of 40%. The effectiveness of the joint action of consistency stabilizers and emulsifiers in obtaining milk-fat emulsions were defined by the level of effective viscosity. Moreover, the use of the emulsifier had less effect on the effective viscosity of the milk-fat emulsions. It has been found that using the structure stabilizers could improve the formation of low-fat products and the formation of the desired paste-like consistency of high-fat cream with a fat level of 70%. Thus, it is possible to adjust the composition and properties of raw material as a basis for milk-fat emulsions by optimizing the ratio of structure stabilizers. Comparative evaluation of the physicochemical properties of milk-fat emulsions and high-fat cream makes it possible to predict their potential for conversion into a creamy paste with a given consistency.

Keywords: cream; milk and fat emulsions; stabilizers; emulsifiers; effective viscosity

INTRODUCTION

In the current conditions of the current shortage of milk raw materials and the full utilization of the secondary raw material potential of the dairy industry and dairy enterprises of Ukraine today are focusing on the creation of a new group of dairy products. This group is based on the low content of milk fat, the effectiveness of various conditions of thermomechanical treatment for the conversion of milk fat emulsions into a quality product.

In particular, oil and cream pastes are promising for improving the efficiency of the dairy industry and for providing various consumer needs. According to world standards **(CAS 279, 1971; CAS 253, 2006**), these products are called dairy spreads or semi-fat oils. Considering the increased content of milk plasma in these products, the formation of the proper structural-mechanical properties and their stability during storage requires the involvement of functional-technological ingredients, in particular stabilization systems. These systems combine well with the milk base and allow the most effective use of the properties of their constituents for the formation of stable complex emulsions. In the writings of scientists **(Krasulya and Zaitseva, 1986; Ipsen, 2017)**, such approaches are revealed that all us to obtain products with a given paste-like structure, which is close to the structure of classical butter.

The development of low-fat dairy products with a fat content of 30-40% is complicated by the deterioration of the thermomechanical treatment of low-fat milk-fat emulsions. The complication is caused by a change in their physical and chemical properties **(Mollakhalili, et. al., 2014; Nguyen, et. al., 2015; Rios, et. al., 2014).** his was especially noted in the works **(Vyshemirsky, Topnikova, Kuchnova 1997; Gulyaev-Zaitsev 1988)**, which revealed the essential properties of reducing viscosity and instability. Several researchers **(Bayés-García, et. al., 2015; Patel, et. al., 2010; Fredrick, et. al., 2009)** consider that the stability of the emulsion is impeded by the processes of coalescence, delamination, and flocculation.

Therefore, the manufacture of a high-quality emulsion product with a high content of milk plasma, first of all, involves the choice and the right combination of stabilization systems: consistency stabilizers and emulsifiers. The action of emulsifiers is due to their ability to accumulate at the boundary of two liquid phases, reducing interfacial tension and creating a protective layer around the droplets that prevents coagulation and coalescence. We agree with the authors (**Topnikova, 2004; Topnikova, 2005)** that the type of emulsifier and its amount depend on the mass fraction of fat in the emulsion product, the modes of technological process, the type of raw material used, the degree of milk fat replacement, the conditions and shelf life of the product.

Typically, to stabilize low-fat emulsions, it is necessary to use mixed-type emulsifiers that combine hydrophilic and hydrophobic compositions. They have maximum functionality and allow them to create a wide range of emulsion products with specified properties. The most common emulsifiers include mono- and diglycerides of dietary fatty acids, lecithin and phospholipids, emulsifiers based on polyglycerol, and others. In the writings of the authors **(Zobkova, 2007; Dorozhkina and Buhmet, 2012; Ipatova, Zadorozhnaya and Malchenko, 1999)** is noted that due to hydrophilicity and stronger moisture retention at elevated phospholipid temperatures, their additional attraction improves the functional properties of the emulsifiers.

Also, they must meet certain requirements: be physiologically safe, promote the retention of moisture in the product during machining in the production process, stabilize high- and low-dispersion emulsions, increase their stability, and ensure the stability of the product during storage. When choosing an emulsifier take into account the melting point, iodine number, characterizing the degree of saturation. For example, in studies **(Zobkova, 2007; Dorozhkina and Buhmet, 2012; Ipatova, Zadorozhnaya and Malchenko, 1999; Fedyakina, Gorshkova and Rubina, 2007)** state that monoglycerides with an iodine value of 50 – 90 g I_2/100 g are used in the production of low-fat emulsions.

The authors **(Vyshemirsky, Topnikova and Kuchnova 1997; Topnikova, 2004**, **Topnikova, 2005**) have found that the use of structure stabilizers causes changes in the physicochemical properties and structural and mechanical parameters of milk-fat emulsions. Their nature of change depends on the type and amount of stabilizer used.

Several authors **(Vyshemirsky, Topnikova and Kuchnova, 1997; Topnikova, 2004; Bogdanova, 2013)** suggest the use of gelatin, cellulose derivatives, pectin, complex stabilization systems Hamulsion, Palsgaard to stabilize the structure of low-fat products. They include hydrocolloids – carrageenan, xanthan and guar gums, modified starch, etc., which allows you to get pastes with a different structure.

Scientific hypothesis

Ensure the stability of the direct type emulsion needs to use surfactants, in which there is a dispersion of the fat phase, increase of the organoleptic and physicochemical characteristics of the finished product.

The process of forming the properties of milk-fat emulsions with the phase structure of "fat in the water" under the influence of structure stabilizers for cream pastes is aimed at the creation of the new technologies of low-fat butter-making products. Direct control of the composition and properties of the raw material – cream with a fat content of 40%, depending on the dose of the structure stabilizers could ensure a stable structure.

It has been determined physical and chemical properties of milk-fat emulsions as a basis for the production of cream pastes for the realization of this hypothesis and achievement of this goal.

MATERIAL AND METHODOLOGY

The objects of this study were milk fat emulsions with a fat level of 30% with adding of the structure stabilizers and cream with fat level from 30% to 70% and, accordingly, a solids level from 2.5% to 9.3%. High-fat cream was obtained by the method of double separation.

The physicochemical properties of cream were determined according to the recommendations of the author (**GOST 3626, 1973)** Determination of the mass fraction of milk solids non-fat (MSNF) was conducted by GOST 3626-73, the mass fraction of fat **(GOST 5867, 1990),** the mass fraction of total nitrogen by the Kjeldahl method – by DSTU ISO 8968:2005-part 2 **(ISO 8968-2, 2005)** ("Fisher Bioblock Scientific", France).

The physicochemical properties of cream and cream with added structure stabilizers were investigated by the sedimentation resistance of the emulsions to delamination, according to the method **(Vaitkus, Kazlauskaite and Malakauskiene, 1981)** of fat settling.

The effective viscosity of cream and cream with added structure stabilizers was determined by the rotary viscometer "Rheotest II" with a cylinder/cylinder measuring system (S/S1, S/S2, S/S3) for different viscosities according to the instructions. In the gap between the cylinders made cream, in which under the influence of the rotation force of the rotor was shifting one layer of the bunch relative to another. The rheological characteristics of the product were determined by the speed of rotation of the rotor and the force of resistance of its rotation. The measurements were performed at a strain rate of 48.6 s^{-1}. The parameters of the instrument were translated into the shear stress (PA) at a given strain rate by the formula:

$$\tau = Z \cdot \alpha,$$

where:
τ – shear stress, Pa;
α – indicators of the device;
Z – cylinder constant, Pa / unit. scale instrument.

The effective viscosity of the test samples was determined by the formula:

$$\eta_{eff} = \tau / Dr .$$

Where:
η_{eff} - effective viscosity, Pa
Dr – shear stress, Pa^2/sec. Dr=constfor each mode of clot destruction.

Milk-fat emulsions were prepared based on skim milk and high-fat cream with a fat content of 70% by weight. For this purpose, the stabilizer was separately dissolved in skim milk, heated at (45 ±5 ºC), and the emulsifier in high fat cream at temperature. The components in the quantities determined in the experimental conditions were combined to obtain normalized cream with a mass fraction of fat, respectively, 30% and 40%. The resulting mixture was heated to (80 ±1°C) with continuous stirring, followed by cooling to (15 ±1°C). Component ratios were varied to normalize cream 30% and 40%.

A software package STATISTICA© v 5 for Windows (StatSoft Inc., USA) was used to processing of the obtained experimental data according to the recommendations of the author **(Lapach, Chubenko and Babich, 2001)**.

The results of the studies were evaluated according to the significance level of *p*-value. The replication of the experiments was threefold. The results were considered credible at the trust level $p < 0.05$.

Graphic processing of the results is done using the software Microsoft Excel 2010. Inc, Mathsoft Mathcard Enterprise Edition ON V11. A.

Statistical analysis

Also, a multivariate regression analysis of the experimental data was performed to evaluate the effect on the studied performance of each of the factors introduced into the model at a fixed position at the average of the other factors. An important condition is the lack of a functional link between the factors. For this purpose, a mathematical model was constructed in the form of an analytical expression that best reflected the relationship of factor traits with the resultant one. To obtain stable milk-fat emulsions, mathematical modeling was performed using a two-factor linear regression model, which in general looks like:

$$Y = b_0 + b_1 \cdot x_1 + b_2 \cdot x_2 + \varepsilon$$

Where:

b_0 – a free member that defines values Y, when all independent variables x_i equal 0.

b_1, b_2 – shows how much the result will change if you change the unit of measurement of each independent factor x_1 and x_2.

To obtain stable milk-fat emulsions:

x_1 – a mass fraction of consistency stabilizer in cream in the range from 1.0 to 2.0 %;

x_2 – a mass fraction of emulsifier in cream in the range from 0 to 1.0 %;

Y_1 – effective viscosity; Y_2 – taste, and smell; Y_3 – consistency; Y_4 – sedimentation stability;

ε – a random variable characterizing the deviation of the factors x_1, x_2 from regression line (residual variable).

In the study, the mathematical expectation of random deviation ε_i is 0 for all observations ($M(\varepsilon_i) = 0$).

To evaluate unknown parameters b_0, b_1, b_2 the least squares method. According to the method, unknown function parameters are chosen such that the sum of the squares of the deviations of the experimental (empirical) values Y_i from their calculated (theoretical) Y_{ip} values were minimal, that is:

$$S = \sum_{i=1}^{n}(Y_i - Y_{ip})^2 = \sum_{i=1}^{n}(Y_i - \varphi(X_i, b_0, b_1, ..., b_k))^2 \to \min$$

The modeling and processing of the experimental data were performed using a mathematical package MathCad. For emulsions with a mass fraction of fat 40% the equations of mathematical models have the form:

1) The equation that describes quantitatively effective viscosity:

$$Y_1 = 2{,}71 + 23{,}89 \cdot x_1 + 8{,}61 \cdot x_2$$

2) An equation that quantitatively describes taste and smell:

$$Y_2 = 5{,}99 - 0{,}46 \cdot x_1 - 0{,}87 \cdot x_2$$

3) The equation that describes quantitatively consistency:

$$Y_3 = 3{,}89 - 0{,}02 \cdot x_1 + 0{,}21 \cdot x_2$$

4) Equation quantitatively describing sedimentation stability:

$$Y_4 = 89{,}22 - 3{,}09 \cdot x_1 - 0{,}65 \cdot x_2$$

The adequacy of the models was verified by the coefficients of determination R^2_{Y1}=96 %, R^2_{Y2}=98 %, R^2_{Y3}=91 %, R^2_{Y4}=92 %, which testify to the high-quality character of the correlation of the system coefficients, as well as the F-test (Fisher's F-test) and the Student's t-distribution to evaluate the reliability of the correlation coefficients.

For a comprehensive analysis of the change in the properties of cream with a fat content of 40%, depending on the dose of the stabilizers of the structure and to determine the rational ranges of the stabilizers, a multidimensional regression analysis of the experimental data was performed.

Based on the results of the experimental studies, a two-dimensional approximation of the mathematical dependence was performed f = φ(x,y) – a mathematical model that reproduces with sufficient accuracy the studied regularity y = f(x,y) – experimental data.

Functional dependencies in the form of two-dimensional second-degree polynomials are described, which describe the technological process fairly accurately. The simulation results are presented in Figure 1.

Analysis of the studies showed that the optimal indicators for Y_1 are 50.36 Pa·s; for Y_2 is 4,7 points; for Y_3 is 5.0 points and Y_4 is 82.2 % by making 1.0 % of stabilizer and 0.5 % of emulsifier.

Equations that adequately describe the result of the experiment for a 40% mass fraction of fat have the following form:

$$Y1(x,y) := 0.07 + 27.47 \cdot x - 1.05 \cdot x^2 + 8.98 \cdot y - 0.05 \cdot y^2 - 2.13 \cdot x \cdot y$$

$$Y2(x,y) := 0.13 + 6.63 \cdot x - 2.28 \cdot x^2 - 0.11 \cdot y - 0.16 \cdot y^2 - 0.39 \cdot x \cdot y$$

$$Y3(x,y) := -0.12 + 4.86 \cdot x - 1.47 \cdot x^2 + 1.67 \cdot y - 2.45 \cdot y^2 + 0.67 \cdot x \cdot y$$

$$Y4(x,y) := 2.7 + 110.09 \cdot x - 35.2 \cdot x^2 + 21.02 \cdot y - 4.6 \cdot y^2 - 11.5 \cdot x \cdot y$$

The results of the researches made it possible to determine the influence of all independent factors on the formation of physical-structural and organoleptic parameters.

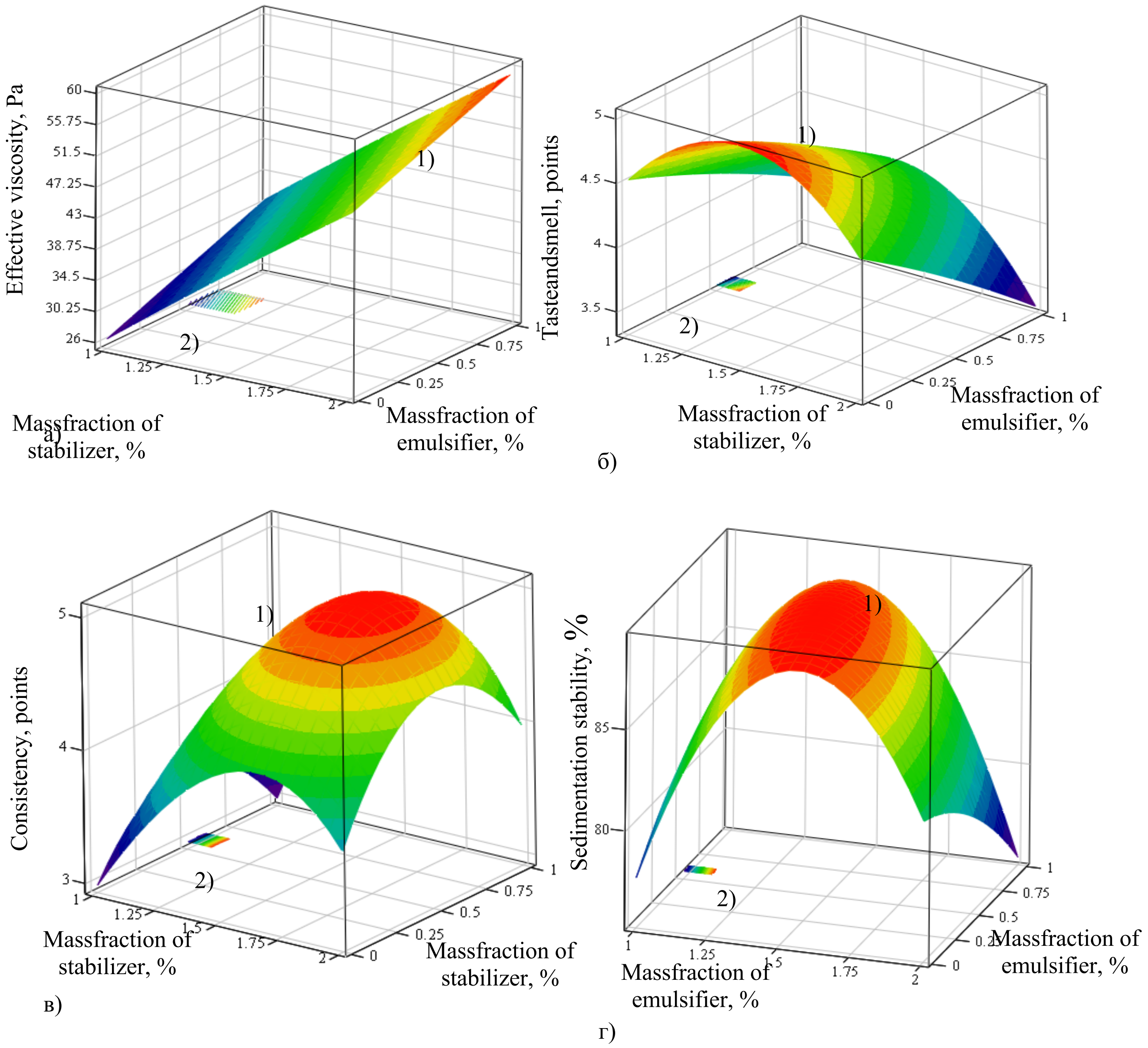

Figure 1 Graphic 3D models (a, b, c, d – 1) and rational ranges (a, b, c, d – 2) of the influence of independent factors x and y on the basic physical-structural and organoleptic parameters of milk-fat emulsions with a fat content by weight of 40%.

RESULTS AND DISCUSSION

The decrease in the fat level in the production of low-fat pastes (30 – 40% of fat) causes a decrease in their viscosity, the stability of structuring, regardless of the hardware design process. Several researchers **(Vishemirsky, et. al., 1997; Gulyaev-Zaitsev, 1986)** report that the reason for this is a change in the composition and physicochemical properties of cream **(Vyshemirsky, Topnikova and Kuchnova, 1997; Gulyaev-Zaitsev, 1986)**. Studies on the composition and physicochemical properties of cream with the fat level of 30-50% as the basis for milk-fat emulsions in the production of cream pastes, showed that a decrease in the fat level leads to a decrease of the ratio fat level milk solids non-fat (MSNF) in cream, effective viscosity and sedimentation resistance (Table 1).

In particular, a decrease in the fat mass from 70 to 30% in the cream resulted in a decrease in their viscosity from 13.789 to 0.036 Pa·s. Cream with a mass fraction of fat 30-40% by this indicator approached each other and was in the range of 0.036-0.072 Pa·s. The stability of fat emulsions is the inverse of the amount of fat and directly proportional to the ratio in them Fat/ DSMR. Sedimentation resistance with mass fraction of fat 50%, 40%, 35% and 30% was lower respectively on 10.5%, 11.4%, 22.4%, 33.3%, than in cream with a mass fraction of fat 70% (Table 1).

The data obtained indicate that a decrease in the fat content of the cream and, consequently, an increase in the plasma, leads to significant changes in the indicators, which impair the ability of the cream to turn into oily products and stabilize their structure To improve the ability of the cream to machining and the formation of a homogeneous stable structure of the products of low-fat content requires changes in the properties of the original cream **(Rybak, 1997; Ronholt, Mortensen and Knudsen, 2013; Leal-Calderon, 1997)**.

Table 1 Physico-chemical indicators of cream of different fat content as raw material for paste production.

Mass fraction of fat cream, %	The ratio of mass fraction of fat / MSNF	Precipitate of fat, %	Effective viscosity, Pa·s
70	27.91	96.0 ±1.0	13.789 ±0.50
50	11.96	85.9 ±0.8	0.818 ±0.009
40	7.97	85.1 ±1.0	0.072 ±0.008
35	4.99	74.5 ±1.0	0.054 ±0.006
30	3.22	64.0 ±0.9	0.036 ±0.006

Table 2 The change in the properties of cream with a mass fraction of fat 40% depending on the dose of the stabilizers of the structure.

Mass part of the stabilizer, %	Mass fraction of emulsifier, %	Effective viscosity, Pa·s	Taste and smell, points	Consistency, points	Sedimentation stability,%
0.5	0	24.3 ±0.5	4.5	3.0	85.1
0.5	0.5	32.3 ±0.9	4.5	3.7	86.0
0.5	1	35.5 ±0.9	3.5	3.0	86.4
1	0	48.6 ±0.8	5.0	4.5	83.2
1	0.5	50.4 ±1.6	5.0	5.0	82.2
1	1	56.1 ±1.6	4.0	2.5	88.8
1.5	0	52.4 ±1.6	4.5	4.2	82.7
1.5	0.5	56.1 ±1.6	4.5	4.0	82.3
1.5	1	68.9 ±1.6	3.0	3.0	80.6

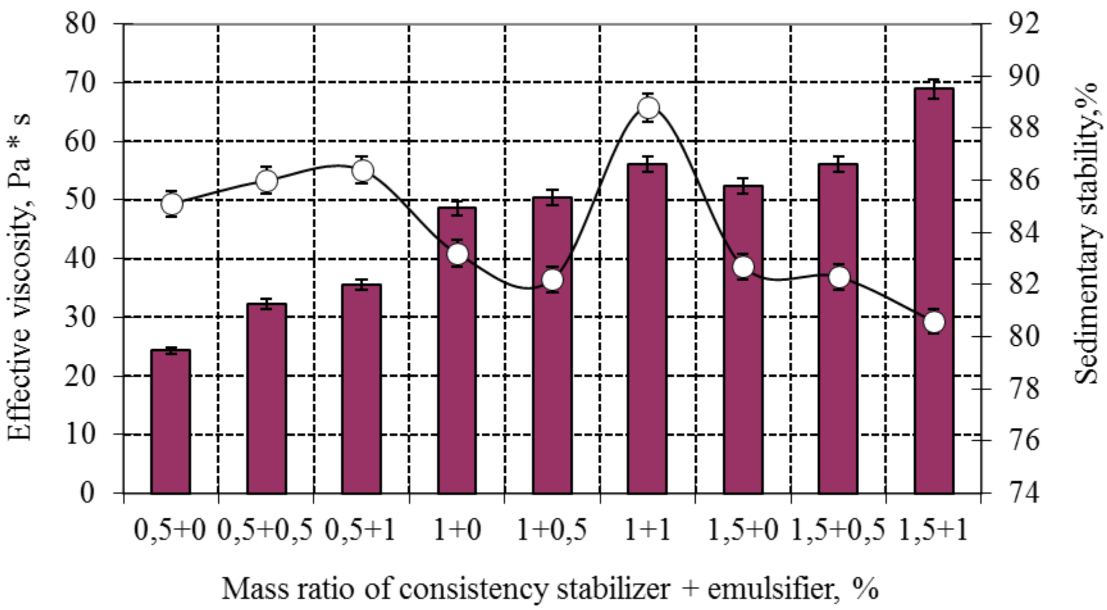

Figure 1. Dependency of change in effective viscosity and sedimentation resistance to increasing the dose of stabilizers in milk-fat emulsions.

In this regard, for direct control of the properties of milk-fat emulsions (mass fraction of 40% fat as a basis for the production of pastes and optimization of doses of structure stabilizers relative to the mass fraction of fat, which would provide milk-fat emulsions physical indicators close to the control cream (70% fat by weight)) were studied the effectiveness of the joint action of consistency stabilizers on the example of QNA colloidal in the amount of 1 to 2.0% and the emodulator of the dimodan U / G - from 0 to 1.0% and the dependences of changes in physicochemical and organoleptic properties were determined.

The results of the analysis of changes in the properties of cream with a fat content of 30 – 40% using a stabilizer and emulsifier are presented in Table 2.

It has been found that with increasing doses of structure stabilizers, effective viscosity increases in milk-fat emulsions. And sharing emulsifier and consistency stabilizer is more effective. It should also be noted that adding 40% fat to the emulsifier had less effect on the effective viscosity of the milk-fat emulsions. Significant changes in this indicator occurred due to the introduction of the stabilizer cream.

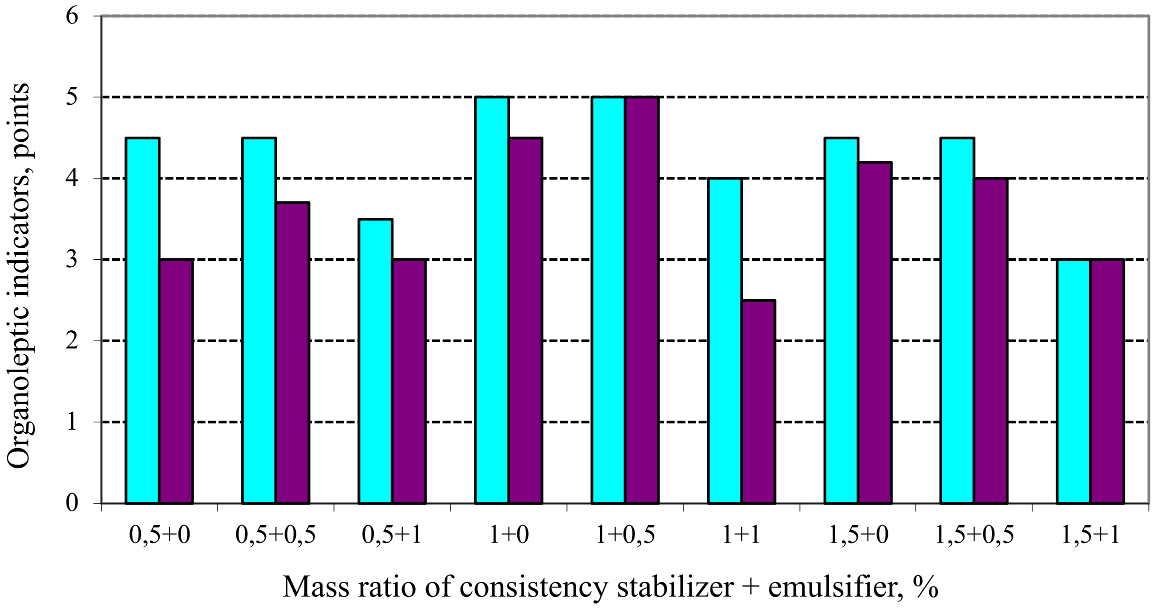

Figure 3 Dependence of organoleptic parameters of milk-fat emulsions on mass particles of stabilizer and emulsifier: taste and smell, consistence.

Table 3 Scale of organoleptic evaluation of milk-fat emulsions to obtain cream pastes.

No.	Characteristic	Points
	Taste and smell	
1	Pure, creamy, without the foreign flavors and odors of structure stabilizers	5.0
2	The creamy taste and smell are not pronounced enough	4.0
3	Not expressed creamy, slightly foreign	3.0
4	Slightly unclean, with a foreign taste and the smell of consistency stabilizers and emulsifiers	2.0
5	Strange flavors and smells of stabilizers and emulsifiers of structure are pronounced, bitterness is felt	1.0
	Consistence	
1	Dense, plastic, homogeneous	5.0
2	Too tight, homogeneous, slightly powdery	4.0
3	Pasty, not dense, creamy, homogeneous, low fatness	3.0
4	Pasty, heterogeneous, sticky, refractory or sour cream, very viscous, sandy	2.0
5	Liquid, unstable	1.0

Obtaining stable milk-fat emulsions with corresponding structural and mechanical properties has a narrow range of a mass fractioni of the stabilizer of consistency (x_1) and the emulsifier (x_2). The values of these ranges significantly affect the performance of viscosity, taste, and consistency. Figure 1 shows the dependence of the change in effective viscosity on the dose of stabilizers in milk and fat emulsions. Adding of the emulsifier partially affected the effective viscosity of milk-fat emulsions compared to the addition of stabilizer in the cream. The optimal ranges of values of mass fraction of the stabilizer of consistency (x_1) and the mass fraction of the emulsifier (x_2) were 1% and 1%.

The viscosity of cream with a fat level of 40%, even with 0,5% of the stabilizer addition was in 1.7 times higher than the viscosity of the control cream with a fat level of 70% and values 13.789 Pa·s (tab. 2). Instead, the addition of 1 and 2% of stabilizer resulted in a 3.5, and 3.8 times increase in the viscosity of the cream. Accordingly, compared to the control cream results were in diapason ranged within 48.6-52.4 Pa·s.

There was no clear trend in the adding of structure stabilizers in cream with 40% fat content. In particular, some samples showed an increase in sedimentation resistance of up to 4.4%, while in others a decrease in sedimentation resistance was observed.

A certain pattern in the study of the sedimentation stability of cream, depending on the doses of stabilizers and emulsifiers was not found by another researcher

(Topnikova, 2002), which used in its studies other stabilizers of the structure: gelatin and monoglycerides distilled P-0291 **(Topnikova, Oborina and Vyshemirsky, 2001)**.

Taste and odor, as well as the consistency of the samples of milk and fat emulsions, were determined on a specially designed 5-point scale (Table 3) and evaluated respectively in the range of 3.0-5,0 points and 2.5-5.0 points. Graphic representation of organoleptic parameters is presented in Figure 3.

In all investigated variants of milk-fat emulsions using structure stabilizers, there was a tendency to improve the consistency due to the increase of their plasticity. The highest consistency was milk-fat emulsions with a fat content by weight of 40%, obtained with the use of stabilizer and emulsifier in quantities of 1.0% and 0.5%, respectively. Milk-fat emulsions with the only stabilizer in quantity were also highly rated 1.0 %. Milk and fat emulsions using stabilizer up to 1.0 – 1.5% and emulsifier 0.5% by taste were acceptable. Increasing the emulsifier to 1.0% worsened the taste of the emulsion due to the foreign taste of bitterness.

Thus, the results of studies on the effect of stabilizers and emulsifiers on the viscosity of milk-fat emulsions with a fat content of 40% suggest that adding stabilizers of the structure makes it possible to approximate the structural and mechanical properties of the cream 70% fat.

Thus, the data obtained by us indicate that low-fat cream with a high plasma content is significantly different in physicochemical properties from high-fat cream, which are a key factor in the formation of plastic fat bases **(Viriato, et. al., 2016, Ribeiro, et. al., 2015)**. Our findings are consistent with the results of the authors' research **(Oborina, Vyshemirsky and Topnikova, 2003)**. Such milk-fat emulsions can serve as the basis for the development of technologies for new types of fat products with necessary properties. Other researchers are inclined to this opinion **(Halukh, et. al., 2016)**.

CONCLUSION

It has been established the dependence of changes in the physicochemical properties of cream with fat level of 40% by the dose of the stabilizers of the structure. In particular, with increasing doses of stabilizers of the structure and mass fraction of fat in milk-fat emulsions, the effective viscosity is increased and the consistency score is increased by increasing their plasticity. It should be noted that common use of emulsifiers and consistency stabilizers are more effective. According to the research results, it has been established that the selected parameters affect on the efficiency of the milk-fat emulsion. However, the formation of the structure is more influenced by consistency stabilizers than emulsifiers.

Thus, optimized doses of structure stabilizers will provide physicochemical and organoleptic indicators close to the control cream in the original low-fat cream. In particular, for milk-fat emulsions with a fat level of 40%, the QNA colloid stabilizer and the dimodan U/G emulsifier are respectively in the amounts of 1.0% and 0.5%.

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