

SYNTHESIS AND PROCESSING OF POLYMERS
AND POLYMERIC COMPOSITES

СИНТЕЗ И ПЕРЕРАБОТКА ПОЛИМЕРОВ
И КОМПОЗИТОВ НА ИХ ОСНОВЕ

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RESEARCH ARTICLE

Stabilisation of cosmetic compositions using combined emulsifiers

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Abstract

Objectives. This study investigated the surface properties and micelle formation of combined stabilizers, which are a mixture of ionic and nonionic surfactants or different nonionic surfactants, to establish a correlation between the composition of stabilizers and the colloidal–chemical properties of direct emulsions obtained in their presence.

Methods. The surface tension at the interface between the aqueous solutions of the combined stabilizers with air and toluene was measured using a digital tensiometer. The sedimentation stability of the emulsions was assessed by the volume of the exfoliated water and oil phases for seven days. The particle sizes of the dispersed phase were determined using an Olympus CX3 bright field microscope equipped with a universal serial bus video camera connection. The rheological properties of the emulsions were evaluated using a rotary viscometer.

Results. According to the isotherms of the surface tension of aqueous surfactant solutions at the interface with air and toluene at emulsion preparation temperatures of 50 and 65°C, a mixture of nonionic surfactants exhibited a higher surface activity and lower critical micelle concentration at the interface with toluene. The optimal amount of stabilizers providing stability to the compositions for one month was 4 mass % for a mixture of anionic surfactants and nonionic surfactants and 7 mass % for mixtures of different nonionic surfactants. Emulsions obtained in the presence of a mixture of anionic and nonionic surfactants exhibited higher kinetic sedimentation stability values due to the formation of electrostatic and steric stabilization factors in the system. The developed compositions were microheterogeneous systems, the average droplet diameter of which varied within the range of 1.0–5.7 μm. In terms of rheological properties, emulsions were classified as liquid-like structured systems with coagulation structures; the strength of single contacts between particles of the dispersed phase was $(1.6–27.0) \times 10^{-10}$ N.

Conclusions. A comparison of the physicochemical characteristics of the compositions obtained in the presence of organic emulsifiers showed that emulsions stabilized using a mixture of ionic and nonionic surfactants, which form mixed adsorption layers, exhibited the best set of properties.

Keywords: nonionic surfactant, anionic surfactant, surfactant mixture, adsorption, critical micelle concentration, emulsification, particle size, viscosity, kinetic sedimentation stability

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НАУЧНАЯ СТАТЬЯ

Стабилизация косметических композиций комбинированными эмульгаторами

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Аннотация

Цели. Изучить поверхностные свойства и мицеллообразование комбинированных стабилизаторов, являющихся смесью ионных и неионных поверхностно активных веществ (ПАВ) или смесью неионных ПАВ. Установить корреляцию между составом стабилизаторов и коллоидно-химическими свойствами прямых эмульсий, полученных в их присутствии.

Методы. Поверхностное натяжение на границе раздела фаз водных растворов комбинированных стабилизаторов с воздухом и толуолом измеряли на цифровом тензиометре. Седиментационную устойчивость эмульсий оценивали по объему отслоившихся водной и масляной фаз в течение 7 дней. Размеры частиц дисперсной фазы определяли с использованием микроскопа Olytrius CX3, предназначенным для работы в светлом поле, снабженным соединением для USB видеокамеры. Реологические свойства эмульсий оценивали с помощью ротационного вискозиметра.

Результаты. По изотермам поверхностного натяжения водных растворов ПАВ на границе с воздухом и толуолом при температурах приготовления эмульсий (50 и 65 °С) установлено, что большей поверхностной активностью и меньшей критической концентрации мицеллообразования на границе с толуолом обладает смесь неионных ПАВ. Оптимальное количество стабилизаторов, обеспечивающих устойчивость композиций в течение 1 месяца, составляет 4 мас. % смеси АПАВ и НПАВ и 7 мас. % смеси НПАВ. Эмульсии, полученные в присутствии смеси анионных и неионных ПАВ, имеют более высокие значения кинетической седиментационной устойчивости за счет формирования в системе электростатического и стерического факторов стабилизации. Разработанные композиции являются микрогетерогенными системами, средний диаметр капель которых изменяется в пределах 1.0–5.7 мкм. По реологическим свойствам эмульсии относятся к жидкообразным структурированным системам с коагуляционными структурами, прочность единичных контактов между частицами дисперсной фазы которых составляет $(1.6–27.0) \times 10^{-10}$ Н.

Выводы. Сравнение физико-химических характеристик композиций, полученных в присутствии органических эмульгаторов, показало, что лучшим комплексом свойств обладают эмульсии, стабилизированные смесью ионных и неионных ПАВ, образующих смешанные адсорбционные слои.

Ключевые слова: неионные ПАВ, анионные ПАВ, смеси ПАВ, адсорбция, критическая концентрация мицеллообразования, эмульгирование, размер частиц, вязкость, кинетическая седиментационная устойчивость

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INTRODUCTION

Emulsions comprising two immiscible liquids are the primary materials for the production of products in various industries, particularly for the production of cosmetic and hygiene products [1], in which various substances of natural origin and those synthesized from natural raw materials are used [2, 3]. However, thermodynamic and sedimentation instability or delamination is a problem of all emulsion compositions, ultimately leading to their destruction [4].

To ensure the stability of the system, surfactants are introduced to the emulsion, and their role is to 1) reduce interfacial tension to 5 mJ/m²; 2) create the ability to quickly adsorb on droplets, creating a thin layer that does not change when droplets collide and prevent coalescence; 3) provide good solubility in a dispersion medium; 4) provide the emulsion a degree of electrokinetic potential; 5) influence the viscosity of the emulsion; 6) introduce emulsifying properties even at low concentrations [5, 6].

To stabilize emulsions for various purposes, surfactant compositions of different or identical nature are commonly used. At specific component ratios, these compositions show the synergistic effect of reducing critical micelle concentration (CMC) and increasing surface activity due to the formation of mixed micelles. Adsorption layers of surfactant mixtures at the oil–water boundary include molecules of all surfactant components, ensuring the high stability of dispersed systems [7, 8]. Currently, in the production of cosmetic emulsions, there is a tendency to use readymade complex semifinished emulsifiers. Information regarding the colloidal–chemical properties of combined emulsifiers, which

are mixtures of various natural surfactants, and the physicochemical characteristics of the final product, eliminate the requirement of performing a large number of experiments to predict the final results, thus remarkably simplifying the procedure for developing compositions that meet consumer properties.

MATERIALS AND METHODS

In this work, industrial-combined emulsifiers were used as stabilizers for direct emulsions: Blanova Muls Eco 2277, a mixture of anionic and nonionic surfactants (*Azelis Rus*, Russia), and Remiwx SE containing a composition of nonionic surfactants (*Revada*, Russia).

Table 1 presents the information regarding the components that constitute the combined emulsifiers.

The surface and interfacial tensions of the aqueous solutions of surfactant mixtures at emulsion preparation temperatures were measured using a K9 digital tensiometer (*Krüss AG*, Germany). The stability of the emulsions was evaluated by the volume of the exfoliated water and oil phases within seven days. The colloidal stability of emulsions was determined using a Type 310 b high-speed centrifuge (*Mechanika Precyzyna*, Poland). The particle sizes of the dispersed phase were determined using an Olympus CX31 microscope (*Olympus*, Japan), which is designed for working within a bright field and is equipped with a connection for a universal serial bus video camera. The rheological properties of emulsions were studied using a Polymer RPE-1M (*Khimpribor-1*, Russia) rotary viscometer. For preparing the compositions, the so-called “hot/hot” standard method was selected for preparing emulsions. Emulsions were prepared at 50°C (mixture of nonionic surfactants) and 65°C

Table 1. General characteristics of the emulsifier components

International Nomenclature of Cosmetic Ingredients – INCI Registry number CAS	Hydrophilic-lipophilic balance – HLB	M, g/mol
A mixture of anionic and nonionic surfactants (Blanova Muls Eco 2277). Molecular weight ~346 g/mol		
Glyceryl Stearate CAS Number 123-94-4	3–6	358
Stearyl Alcohol CAS Number 112-92-5	15.5	270
Sodium Stearoyl Lactylate CAS Number: 25383-99-7	8.3	450
Glyceryl Stearate Citrate CAS Number: 50825-78-0	3.4	306
Nonionic surfactant mixture (Remiwx SE PF). Molecular weight ~600 g/mol		
Glyceryl Stearate CAS Number: 31566-31-1	2.5	358
Ceteareth-20, Ceteareth-12 CAS Number: 68439-49-6	13.2 15.5	1136 784
Cetearyl Alcohol CAS Number: 67762-27-0	15.5	242.2
Cetyl Palmitate CAS Number: 540-10-3	10	480

(mixture of anionic and nonionic surfactants) using a Polytron PT 1200 E homogenizer (*Kinematica AG*, Switzerland). The mixing speed was 6000 rpm, and the homogenization time was 3 min. The ratio of the oil and water phases was 1 : 4. The concentration of the emulsifier was varied from 0.5–7% (wt).

RESULTS AND DISCUSSION

The type and stability of emulsions depend on the ratio (balance) of hydrophilic and hydrophobic (or lipophilic) functional groups among the surfactant molecules. To obtain direct «oil-in-water» emulsions, using emulsifiers with hydrophilic–lipophilic balance (HLB) numbers ranging from 8–13 was necessary [9]. The numbers of HLB stabilizers were experimentally determined using the Davies method. For a mixture of nonionic surfactants, this number was equal to 10.5; for a mixture of anionic and nonionic surfactants, it was 13.5. An ester of polyglyceride and fatty acids with an HLB number of 4 was used as a surfactant with a known HLB number. Based on the data obtained,

refined sunflower oil with a close HLB number was selected as the nonpolar phase [5].

A K9 digital tensiometer was used to measure the surface tension of the combined stabilizers at the aqueous solution–gas and aqueous solution–toluene interface, which was selected as a model for the nonpolar phase. The surface isotherms, interfacial tension of nonionic surfactant mixtures, and the mixtures of anionic and nonionic surfactants at emulsion preparation temperatures are shown in Fig. 1.

The obtained surface and interfacial tension isotherms were used to determine the surface activity and CMC, and the parameters for the monolayers of stabilizers were calculated [10]. The results are presented in Table 2.

The data presented indicates that at the aqueous solution–air interface, the mixture of anionic and nonionic surfactants will have higher surface activity values, maximum adsorption, and a lower CMC value compared with a mixture of nonionic surfactants nondissociating in water, which is associated with the

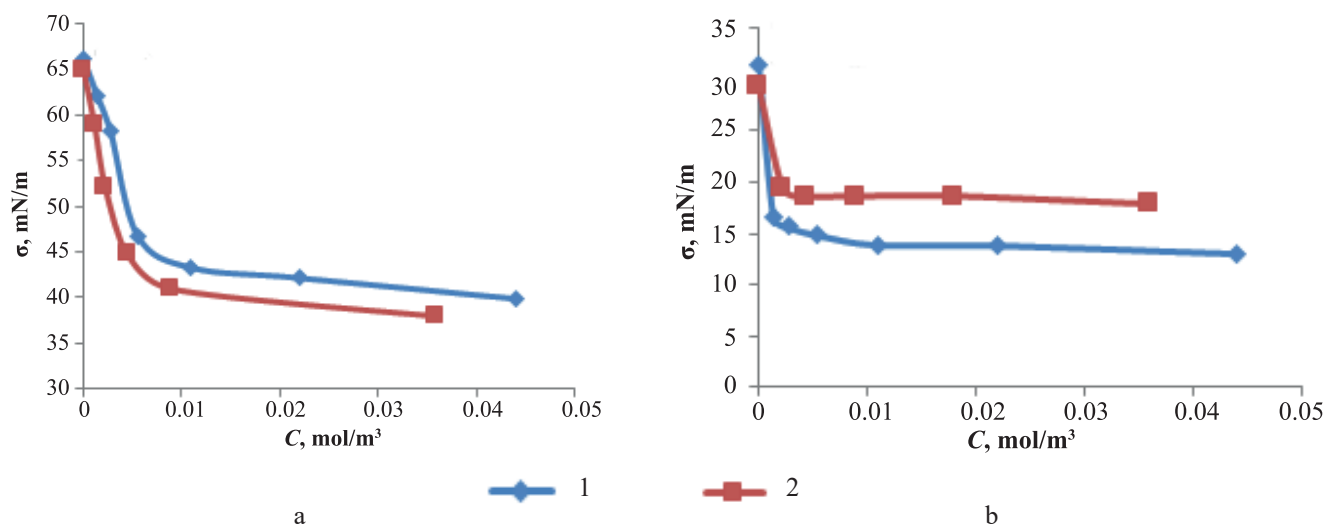


Fig. 1. Surface tension isotherms of surfactants in aqueous solutions containing mixtures of different nonionic surfactants (1); anionic and nonionic surfactants (2) at different interfaces, i.e., (a) air and (b) toluene.

Table 2. Surface activity values, critical micelle formation concentration, and the parameters of the adsorption layers of surfactant mixtures

Name of surfactants	Adsorption characteristics of surfactants					
	G , J·m/mol	$\sigma_{\min} \times 10^3$, J/m ²	$\Gamma_{\max} \times 10^6$, mol/m ²	$S_0 \times 10^{19}$, m ²	$\delta \times 10^9$, m ²	C_{CCM} , mol/m ³
Aqueous solution–air boundary						
A mixture of anionic and nonionic surfactants	4.86	37.0	5.10	3.25	1.76	0.0045
A mixture of nonionic surfactants	3.80	40.0	7.80	2.95	2.94	0.0064
Aqueous solution–toluene boundary						
A mixture of anionic and nonionic surfactants	4.70	17.8	4.60	3.60	1.60	0.0037
A mixture of nonionic surfactants	8.80	13.0	6.80	2.44	4.08	0.0025

Note: G – surface activity;

$\sigma_{\min}$ – minimum surface tension;

$\Gamma_{\max}$ – maximum adsorption value;

S_0 – area occupied by a surfactant molecule in a saturated monolayer;

δ – the thickness of the adsorption layer;

C_{CCM} – the value of the critical concentration of micelle formation.

formation of mixed micelles in the solution [6]. At the boundary of the aqueous solution–toluene, an emulsifier with the best surface-active properties is a mixture of nonionic surfactants.

Notably, low values of $\sigma_{\min}$ at the interface between toluene and aqueous solutions of surfactant mixtures contributed to the formation of stable direct emulsions.

To calculate the temperature coefficients of the surface tension of the combined stabilizers, the σ values for aqueous solutions of surfactant mixtures of the same concentration were measured at different temperatures. For a mixture of anionic and nonionic surfactants, the value of $d\sigma/dT$ was -0.044 mJ/m^2 ; for a mixture of nonionic surfactants, the value of $d\sigma/dT$ was -0.037 mJ/m^2 . The values of the temperature coefficient of surface tension allowed for predicting the behavior of the emulsifier at different temperatures and enabled selecting the conditions for mixing the phases.

The sedimentation stability of the emulsion was assessed by the volume of the exfoliated water and oil phases for seven days. The colloidal stability of the emulsions was determined according to the standard method¹ in an ultracentrifuge at a speed of 6000 rpm, from which it followed that the emulsion would be considered stable if, after centrifugation, in a test tube, no more than one drop of the aqueous phase and/or 0.5 cm of the oil layer were released.

The compositions differed in terms of the type of emulsifier and its concentration. Based on the results obtained, histograms of the ratios of the emulsion and exfoliated phases were plotted according to the concentration in the compositions stabilized with a mixture of anionic and nonionic surfactants and a mixture of different nonionic surfactants (Figs. 2 and 3).

With an increase in the concentration of the added stabilizer for all samples, the amount of stable emulsion increased. Compositions had high stability with the introduction of an emulsifier, starting from 3% for a mixture of anionic and nonionic surfactants and 5% for a mixture of different nonionic surfactants.

The influence of negative temperatures on the stability of emulsions was investigated, which enabled determining the stability of the emulsion at low temperatures and predicting the behavior of finished product during transportation in adverse weather conditions. It was found that at temperature drops, the loss of stability was determined to be 1–2%, which did not affect the consumer properties of the product.

As a result of the experiments, the following outcomes were observed:

- The optimal percentage of the input of the emulsifier for preparing the cosmetic composition was 4% by weight for an anionic and nonionic surfactant mixture and 7% weight for a nonionic surfactant mixture. Emulsions with these properties were sufficiently stable to withstand mechanical stress and did not collapse when exposed to a centrifugal field and negative temperatures.

¹ GOST 31460-2012. Interstate standard. Cosmetic creams. General specifications. Moscow: Standartinform; 2013.

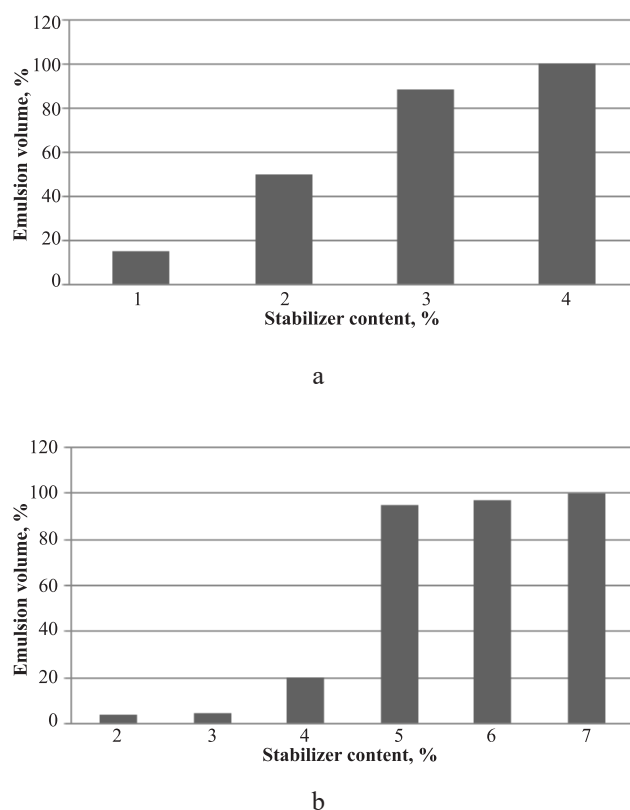
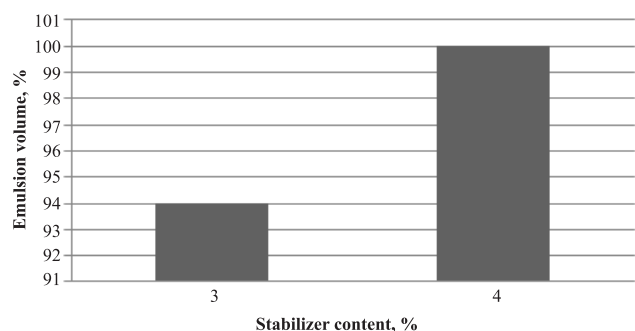


Fig. 2. Influence of the concentration of stabilizers comprising a mixture of anionic and nonionic surfactants (a) and a mixture of nonionic surfactants (b) on the stability of emulsions in a gravitational field.

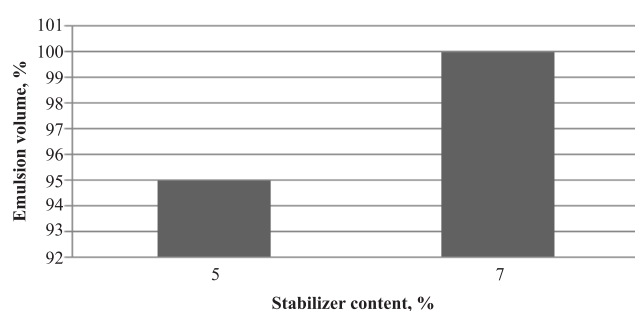
- The use of a stabilizer in the form of a mixture of nonionic surfactants enabled obtaining cosmetic products for various purposes; with the introduction of a 2–3% emulsifying base, a liquid emulsion was obtained that could be used to prepare cosmetic milk and lotions. To obtain cosmetic creams and masks while preparing emulsions, the percentage of input should range from 5 to 7%.

- The high stability of the obtained emulsions in the presence of anionic and nonionic surfactant mixtures resulted from confounding stability factors in dispersed systems; the presence of ionic surfactants in a system provided electrostatic stabilization, and the presence of a nonionic surfactant provided steric stabilization (electrosteric stabilization) [5]. When using a combined stabilizer, i.e., a mixture of nonionic surfactants, only the steric factor of stability was realized in the system [11, 12].

Particle size in the dispersed phase is one of the defining characteristics of emulsion compositions, which was determined using the light microscopy method. Figure 4 shows the differential curves of the particle size distribution of emulsions obtained in the presence of nonionic surfactants. Similar data were obtained for compositions stabilized with a mixture of anionic and nonionic surfactants.



a



b

Fig. 3. Influence of the concentration of stabilizers comprising a mixture of anionic and nonionic surfactants (a) and a mixture of nonionic surfactants (b) on the stability of emulsions in a centrifugal field.

The presented data show that the obtained emulsions were polydispersed microheterogeneous systems. The diameters of various fractions varied from 1 to 5 μm for a mixture of anionic surfactants and nonionic surfactants, and from 3.3 to 16.6 μm for a mixture of different nonionic surfactants. The degree of polydispersity, equal to the ratio of the maximum diameter to the most probable diameter ranged from 1.75 to 4.25.

Rheological characteristics that determined the consumer properties of cosmetic compositions (absorbency, spreadability, moisturizing ability) [13, 14] were measured using a Polymer RPE-1M rotational viscometer. The flow curves of emulsions stabilized using different amounts of a mixture of ionic and nonionic surfactants and the dependence of viscosity on shear stress are shown in Figs. 5 and 6.

Based on the presented graphs, the developed compositions were liquid structured systems with a yield point. Similar dependences of the shear stress on the strain rate were obtained for emulsions stabilized with a mixture of nonionic surfactants. The rheological

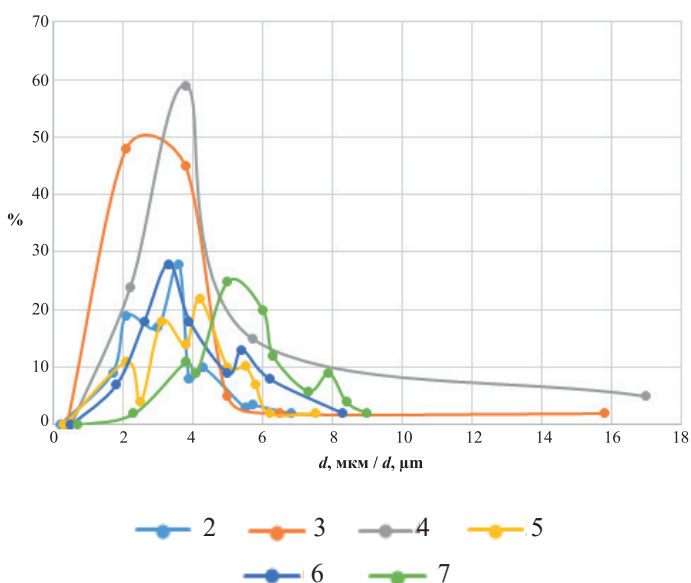


Fig. 4. Drop-size distribution of emulsions stabilized with various amounts of a mixture of nonionic surfactants (2–7 g per 100 mL of the composition).

behavior of such emulsions can be described using the Ostwald–de Waele and Herschel–Bulkley equations [15], which include the value of the ultimate shear stress (P_{str}), the form of which is expressed as follows:

$$P = P_{\text{str}} + \eta_{\text{pl}} \cdot \gamma^n, \quad (1)$$

where P_{str} is shear yield stress; η_{pl} is plastic viscosity; γ is strain rate; n is a constant that characterizes the degree of deviation in fluid properties from a Newtonian liquid.

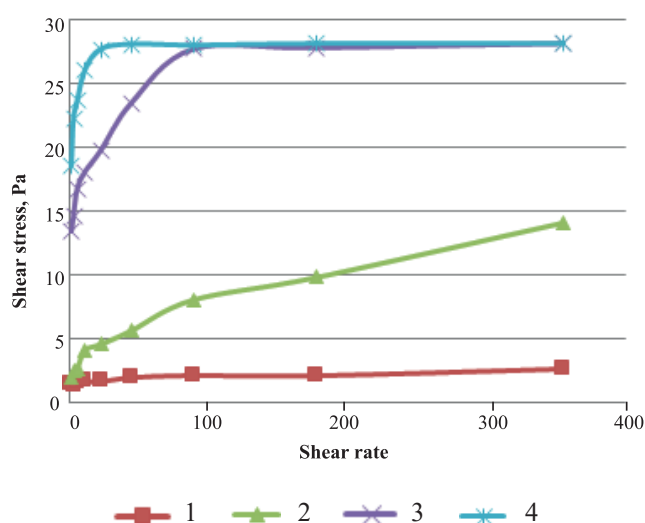


Fig. 5. Curves of the flow of emulsions stabilized using various amounts of a combination of anionic and nonionic surfactants (1–4 g per 100 mL of composition).

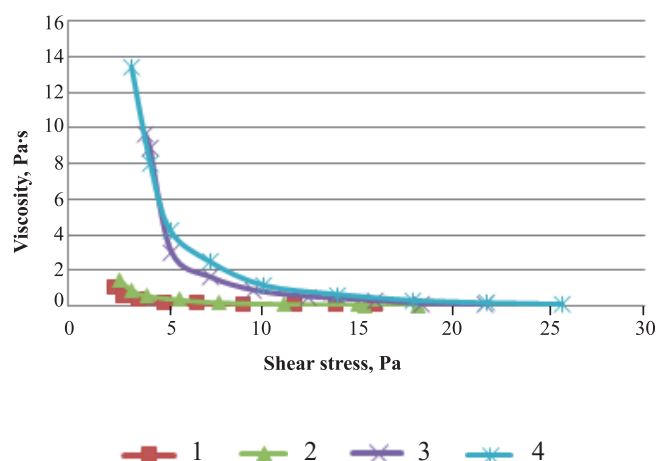


Fig. 6. Influence of the stabilizer content from a mixture of anionic and nonionic surfactants on the viscosity of emulsions.

The structural and mechanical properties of emulsions depended on the strength of a single contact between particles of the dispersed phase, which determined the nature of the contact (phase or coagulation). The availability of data on the strength of a single contact determines the ability of the system to resist destruction under the influence of external forces. According to Kuhn's model [16], which considers the yield point, the strength of a single contact was estimated for the compositions developed by us as follows:

$$F_1 = \frac{P_{\text{str}} 3\pi d^2}{2\varphi}, \quad (2)$$

where F_1 is single contact strength (N); φ is volume fraction; d is emulsion droplet diameter (m).

The strength values of single contacts (Table 3) showed that coagulation contacts were formed in emulsions, ensuring the restoration of the structure after destruction.

Table 3. Structural-mechanical and molecular-kinetic properties of emulsions

No.	Stabilizer content, g/100 mL	Yield strength P_{str} , single contact strength F_1 , and plastic emulsion viscosity η_2			Sedimentation rate U and kinetic sedimentation stability (KSS) of emulsions			
		P_{str} , Pa	$F_1 \times 10^{10}$, N	η_2 , Pa·s	$U_1 \times 10^7$, m/s	$\text{KSS}_1 \times 10^{-7}$, s	$U_2 \times 10^{10}$, m/s	$\text{KSS}_2 \times 10^{-10}$, s
A mixture of anionic and nonionic surfactants								
1	1.0	1.4	1.59	0.90	2.01	4.91	2.23	4.41
2	2.0	2.0	1.88	1.45	1.66	5.92	1.17	8.73
3	3.0	13.4	3.15	8.00	0.41	2.41	5.20	1.91
4	4.0	18.5	6.27	13.40	0.59	1.60	4.47	2.24
A mixture of nonionic surfactants								
1	2.0	1.8	4.61	1.31	4.53	2.16	3.74	2.62
2	3.0	2.4	6.53	1.78	4.81	2.04	2.70	3.63
3	4.0	2.7	9.67	1.95	6.33	1.55	2.98	3.28
4	5.0	3.3	1.30	2.40	6.99	1.40	2.91	3.37
5	6.0	3.3	1.24	2.40	6.62	1.48	2.68	3.66
6	7.0	3.6	2.75	2.60	10.80	0.90	4.15	2.36

Note: index 1 refers to values calculated considering the viscosity of the dispersion medium; index 2 refers to the values calculated considering the viscosity of the compositions.

The speed of movement of individual drops under the action of gravitational forces was proportional to the difference in the densities of the dispersed phase and the dispersion medium, ρ_1 and ρ_2 , as well as the square of the radius of the drops, and it was inversely proportional to the viscosity of the dispersion medium [4]. Kinetic sedimentation stability (KSS) was the reciprocal of the sedimentation constant.

Using the experimentally obtained values of the particle size and viscosity of the developed compositions, the reverse sedimentation rates of direct emulsions were calculated, considering only the viscosities of water and taking into account the viscosity of real systems, as well as the KSS of emulsions (Table 3).

The calculations made enabled quantifying the effect of the stabilizer on the stability of the compositions. As concluded from the data given in Table 3, the rate of emulsion sedimentation in the presence of a stabilizer decreased, and the KSS increased 1000 times. The presence of combined stabilizers considerably increased the viscosity of emulsions, providing an additional hydrodynamic stability factor, and made enabled obtaining compositions stable for almost an unlimited time.

Analysis of the rheological characteristics of direct concentrated emulsions showed that these compositions were pseudoplastic systems with a yield point, the value of which depended on the type and amount of stabilizer. Varying the content of the emulsifier allowed gaining a line of cosmetics including different products ranging from milk to creams for various purposes. An emulsion obtained using stabilizer that is a mixture of anionic and nonionic surfactants, and which form mixed adsorption layers providing electrostatic and steric stability factors, is preferable in terms of structural and mechanical properties. The calculated values of the strengths of single contacts were in the range of 10^{-10} – 10^{-9} N, which confirmed the presence of coagulation contacts between the particles of the dispersed phase.

CONCLUSIONS

The parameters of the adsorption layers of various natural surfactant mixtures were calculated. The maximum adsorption value at the interface with

toluene and the thickness of the saturated adsorption layer of the nonionic surfactant mixture at this interface increased compared with the analogous value at the solution–air interface, which indicated an increase in the hydration layer around the oil droplets. The interfacial tension was reduced to 15 mN/m, which contributed to an increase in the stability of emulsions.

The optimal content of the emulsifier was established as 4% for a mixture of ionic and nonionic surfactants and 7% for a mixture for different nonionic surfactants. In the presence of stabilizers, the values of KSS increased by 1000 times.

Compositions stabilized using a combination of anionic and nonionic surfactants were microheterogeneous systems with an average droplet diameter of 1.0–5.0 μm , based on the surfactant content; for a mixture of nonionic surfactants, this value ranged from 4.0 to 8.0 μm and included a small number of fractions with sizes ranging from 10.0 to 16.5 μm .

The compositions were pseudoplastic fluids with a yield point. The calculated strength of single contacts between particles of the dispersed phase indicated the presence of coagulation contacts in dispersed systems.

A comparison of the physicochemical characteristics of the compositions obtained in the presence of industrial organic emulsifiers showed that emulsions stabilized using a mixture of ionic and nonionic surfactants, which provided stability because of electrostatic and steric stability factors, exhibited the best combination of properties.

Authors' contribution

V.V. Korypaeva – conducting an experiment to study the colloidal-chemical properties of surfactant mixtures, studying the rheological properties of emulsions, collecting and processing material, writing the text of the article.

E.F. Bukanova – scientific consulting at all stages of the work.

E.V. Eskova – consultation during the individual stages of research and processing of the results obtained.

V.A. Sokhraneva – conducting an experiment to obtain emulsions and study their stability.

The authors declare no conflicts of interest.

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