

ВЛИЯНИЕ УЛЬТРАЗВУКА НА ПЕНООБРАЗУЮЩИЕ КОМПОЗИЦИИ РЕАГЕНТОВ, ИСПОЛЬЗУЕМЫХ ПРИ ФЛОТАЦИИ РУД

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В статье рассмотрено влияние ультразвуковой обработки различной акустической мощности пенообразующих композиций флотореагентов (раствор солянокислого амина, раствор солянокислого амина с добавлением полиэтиленгликоля 200М и раствор солянокислого амина с добавлением триэтиленгликоля) на свойства двухфазных пен: пенообразование, кратность пен, влажность, устойчивость и средняя скорость разрушения пен; а также на изменения поверхностного натяжения и размера флокул собирателя в пенообразующих композициях реагентов. Установлено, что ультразвуковая активация пенообразующих композиций реагентов при акустической мощности 420 Вт увеличивает кратность пен и пенообразование на 10,3 и 12,1% соответственно, при этом пены становятся более «сухими» на 15,2% (в случае раствора солянокислого амина), 15,6% (в случае раствора солянокислого амина с добавлением полиэтиленгликоля 200М) и 13,3% (в случае раствора солянокислого амина с добавлением триэтиленгликоля). Кроме того, наблюдается повышение стабильности пенного слоя при увеличении акустической мощности ультразвуковой обработки до максимальной (420 Вт) на 63,1% (в случае раствора солянокислого амина), что свидетельствует о возможности регулирования устойчивости и других характеристик пены (пенообразование, кратность и влажность пены) с помощью акустического метода. Благодаря ультразвуковому диспергированию, флокулы собирателя (раствор солянокислого амина) более эффективно распределяются по всему объему эмульсии, в т.ч. на поверхности пенообразующей композиции реагентов. Установлено, что ультразвук изменяет вышеуказанные характеристики пены за счет снижения поверхностного натяжения пенообразующих композиций реагентов и уменьшения размера мицелл раствора солянокислого амина.

Ключевые слова: пена, пенообразующая композиция, ультразвук, собиратель, вспениватель, устойчивость пен, пенообразование, кратность пен, влажность пен, поверхностное натяжение

EFFECT OF ULTRASOUND ON REAGENT COMPOSITIONS FOAMING PROPERTIES USED IN MINERAL FLOTATION

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The article considers the effect of foam-forming compositions of flotation reagents (hydrochloric amine solution, hydrochloric amine solution with addition of polyethylene glycol 200M and hydrochloric amine solution with addition of triethylene glycol) ultrasonic treatment on the properties of two-phase foams. It was determined that ultrasonic activation of reagent compositions increases a foam specific volume and foam capacity by 10.3 and 12.1%, respectively, while the foams become drier by 15.2% (in the case of the hydrochloric amine solution), 15.6% (in the case of the hydrochloric amine solution with addition of polyethylene glycol 200M) and 13.3% (in the case of the hydrochloric amine solution with addition of triethylene glycol). In addition, there is an increase in the foam layer stability with an increase in the ultrasonic treatment power by 63.1% (in the case of the hydrochloric amine solution), which indicates the ability to control the stability and other characteristics of the foam using the acoustic method. Due to ultrasonic dispersion, amine floccules are more efficiently distributed over the entire volume of the emulsion, including the surface of the liquid. It was found that ultrasound changes the above foam characteristics by reducing the surface tension of foam-forming compositions and decreasing collector micelles.

Key words: foam, foam-forming composition, ultrasound, collector, frother, foam stability, foam capacity, foam specific volume, foam density, surface tension

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INTRODUCTION

Froth flotation is the main technology used in potash enrichment plants to separate valuable minerals from waste rock based on surface wettability differences due to the addition of collectors [1-3]. In this process, an important role is played by frothers that can disperse the collector micellar structures, for example, during cationic flotation of water-soluble minerals, and also contribute to the formation of small air bubbles in the pulp volume, which are necessary for the attachment of hydrophobic mineral particles, and to the formation of the stable foam layer on the surface of the pulp [4-8].

The process of mineral flotation is difficult to research because the solid, liquid, and gaseous phases take part in the interaction. The properties of these phases and the results of their interaction under certain conditions determine the final indicators of flotation: the quantity and quality of the foam concentrate in the flotation machine [9, 10].

The foam's stability affects the intensity of particles shedding, however, the foam, when removed

from the flotation machine cell, should be easily destroyed and not cause technological complications in the subsequent stages of enrichment [11-15]. The foam destruction is caused by the outflow of the interfilm fluid from the Plateau-Gibbs channels separating the bubble's air shells, which is called syneresis [16, 17]. Diffusion transfer of gas also occurs in the foam due to the pressure difference in different size bubbles, since bubbles in the foam have different dispersity [6, 18]. This process contributes to the increase or decrease in the air bubbles size, changing the particle size distribution of the foam. Air bubbles formed in the flotation pulp volume actively coalesce when colliding with each other since any thermodynamic system tends to a minimum of internal energy, which is facilitated by the liquid outflow from the foam and gas diffusion [19-21]. The foam stability depends on the strength of their film frame: foams with liquid films are generally unstable [14, 22]. The quantitative characteristic of any foam is foam specific volume (the ratio of the foam volume to the liquid volume forming the sides of the bubbles), which affects the particle shedding, and foam density [23-25].

The recovery efficiency and the quality of the flotation product depend on the foam properties. In some conditions, the results of flotation do not meet industrial requirements due to such factors as low foam stability, low foam specific volume, low dispersion of air bubbles in the pulp volume.

To improve the specified characteristics of foam systems, there are the following methods: chemical (using combinations of various reagents: collectors and frothers) [7, 20, 26, 27], mechanical [18, 28] and physical (thermal, acoustic, etc.) [29-32] methods for controlling the processes of foam formation and foam destruction.

A promising method for increasing flotation efficiency is the use of ultrasound. Due to ultrasonic treatment, in particular cavitation, many physicochemical properties of flotation systems, including foam characteristics, can be changed [32-34].

However, when researching foams, significant difficulties arise due to the complexity of their experimental study, the variety of foams properties, as well as the phase state of foams, in connection with which this article investigates the properties of two-phase foams, which gives a general understanding of the processes occurring after ultrasonic treatment of flotation emulsions, and the possibility of modeling the processes occurring in the flotation foam layer.

It should be noted that the research of the effect of ultrasound on the change in the foam characteristics is of both theoretical and practical interest since the results of the studies can be used in the technology of potash ores and other minerals flotation enrichment. There are few studies on this topic. For example, the papers [35, 36] show the effect of ultrasonic treatment of a hydrochloric amine solution used as the collector in potash flotation on the foam capacity and assessment of foam stability, while, as noted by the authors, in the future, the effect of ultrasonic treatment on the "collector-frother" composition should be researched, since it is the composition that is used in the flotation enrichment of many minerals. In addition, the articles do not indicate the reasons for the change in the characteristics of foams after ultrasonic treatment of the collector.

The purpose of this article is to research the effect of ultrasonic treatment of foam-forming compositions of flotation reagents («collector-frother» composition) on such basic foam characteristics as foam capacity, foam specific volume, foam density, and foam stability, also to determine the causes for the change in the listed foam characteristics after ultrasonic treatment of flotation emulsions.

MATERIALS AND METHODS

Foam-forming compositions

Three different emulsions were used to study the effect of ultrasonic activation on the foam-forming composition: 1 – hydrochloric amine solution (HCA), which is used as the collector in, for example, potash flotation; 2 – emulsion of the HCA with the addition of polyethylene glycol 200M (HCA-PEG); 3 – emulsion of the HCA with the addition of triethylene glycol (HCA-TEG). PEG and TEG are used as flotation frothers in sylvite flotation [7].

To prepare the first solution (HCA), primary, distilled and granular amines of the C17-C20 fraction were used as amines. HCA was prepared according to the following technique: solid distilled amine (previously ground in a mortar) is added to distilled water, the concentration of amine in the solution is 0.8 wt. %, and chemically pure hydrochloric acid in an amount 15% higher than necessary for the neutralization of the amine. The prepared solution, with stirring, is placed in a thermostat with a set temperature of 70 °C for 90 min. Next, the temperature of the solution is reduced to a working temperature of 60 °C.

The second (HCA-PEG) and third (HCA-TEG) emulsions are prepared similarly to the method described above, with the difference that after lowering the HCA temperature to 60 °C, to add with stirring PEG or TEG 30% by mass of dry amine, respectively.

Ultrasound treatment of foam-forming compositions

Ultrasonic activation of the foam-forming composition was carried out using the laboratory ultrasonic installation, shown in Fig. 1. As a source of ultrasonic vibrations, an ultrasonic generator with a piezoelectric oscillatory system with a developed radiating surface made of titanium alloy in a metal case with forced air cooling, model UZTA-0.8/22-OMU (series "Volna") from the "Tsentr ul'trazvukovykh tekhnologiy" (Russia) was used.

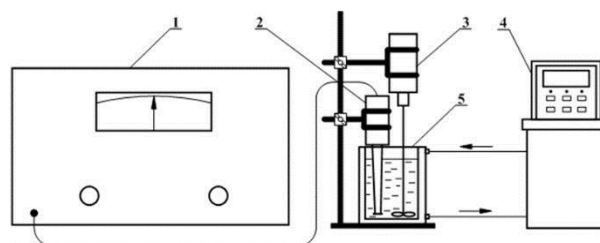


Fig. 1. The laboratory setup for ultrasonic treatment of foam-forming composition: 1 – US-generator; 2 – US radiating surface; 3 – mechanical stirrer; 4 – thermostat; 5 – jacketed reactor
Рис. 1. Установка для УЗ-обработки пенообразующей композиции: 1 – УЗ-генератор; 2 – УЗ-излучающая поверхность; 3 – механическая мешалка; 4 – термостат; 5 – реактор с рубашкой

The laboratory installation has a nominal operating frequency of 22 ± 1.65 kHz and radiation intensity

of at least 3.5 W/cm². Electronic generator with timer and power regulator from 40 to 100%. At 100% power ultrasonication, the total power is 1600 V·A, the active power is 650 W, and the acoustic power of 420 W is introduced into the medium. By changing the power setting, the total, active and acoustic power changes. The changes are proportional.

The operating temperature of an emulsion was maintained using the thermostat (4). A foam-forming composition in a volume of 500 ml was placed in the reactor (2) and it was ultrasonicated at various modes of acoustic power (from 168 to 420 W in steps of 84) and treatment duration of 150 s. Control experiments without ultrasonic treatment were carried out under identical conditions.

Measurement of amine floccules

The particle size of the amine was measured using a Zetasizer Nano ZS nanoparticle detection system from Malvern Panalytical (Netherlands, Great Britain). The analysis is carried out according to the method of dynamic light scattering using non-invasive backscattering technology.

To determine the amine floccules size, 1.5-1.7 ml of the activated or non-activated by ultrasonic treatment emulsion was sampled. The sample was placed in a special clean cuvette (disposable polystyrene cuvette DTS0012) installed in the device cell and heated to a working temperature of 60 °C. The water viscosity values used to measure the collector particle size were taken as reference values at a given temperature. The refractive index of foam-forming compositions was 1.4407.

10 consecutive measurements of one sample were made on the device, after which the values were averaged.

Measurement of foam-forming composition surface tension

The surface tension of emulsions activated and non-activated by ultrasound was measured using the Wilhelmy plate method on a Kruss K100C-MK2 tensiometer (Germany).

Surface tension (σ , mN/m) is calculated from the measured force (F , mN), wetted surface length (L , m) and contact angle (θ). The standard plate is made of platinum ($\theta = 0^\circ$). Surface tension (σ) was calculated using formula (1):

$$\sigma = \frac{F}{L \cdot \cos\theta} \quad (1)$$

Assessment of foam capacity, foam stability, and foam density of foam-forming compositions

Assessment of foam capacity, foam stability, and foam density of activated and non-activated by ultrasonic treatment of the emulsion were carried out using a Kruss DFA100 foam analyzer (Germany).

A sample of 50 ml was taken from the emulsion activated and non-activated by ultrasound with a syringe and injected into the glass measuring column of the foam analyzer. Using an air flow meter, the required amount of gas supply was set (0.3 l/min), air was passed through the emulsion for 10 s. The duration of each measurement was 360 s.

During the analyzer operation, the following indicators were measured:

- Foam capacity (mL/mL) is the ratio of the foam volume to the passed gas volume, which characterizes foaming based on gas binding.
- Foam density (mL/mL) is the ratio of the liquid part in the foam to the foam volume when the foaming process ends, which characterizes the moisture content in the foam.
- Foam specific volume (mL/mL) is the ratio of the foam volume to the liquid volume in the foam when the foaming process ends, describing the foaming properties of the liquid.
- Assessment of the degree of foam destruction (assessment of foam stability) by the modified Ross-Miles method ISO 696 (RMI) [37], according to which the remaining foam volume is measured after 30 (RMI 30), 180 (RMI 180), and 300 (RMI 300) s.

In addition to the indicators listed above, the average foam destruction rate (v_{av}) was calculated using formula (2):

$$v_{av} = \frac{V_{max} - RMI300}{300}, \quad (2)$$

where v_{av} – the average foam destruction rate (mL/s), V_{max} – maximum foam volume (mL), RMI 300 – foam volume after 300 s measurement (mL), 300 – time elapsed since the start of the measurement (s).

RESULTS AND DISCUSSIONS

Assessment of foamability and foam density of the foam-forming compositions

The foamability of the foam-forming compositions was assessed by foam capacity and foam specific volume, foam density – by the moisture content in a foam. The results of these characteristics research are shown in Table 1.

From the analysis of the Table 1 (Foam capacity), it can be seen that the addition of PEG or TEG to the HCA increases foam capacity from 1.57 to 1.66 and 1.70 mL/mL, respectively, compared with the HCA without frothers and ultrasonic treatment. Ultrasonic treatment of all emulsion types increases foam capacity almost linearly with increasing acoustic power up to a maximum of 420 W. In this case, most of the foam is formed using the HCA-TEG emulsion. It is important to note that the ultrasonic treatment of the HCA with

the power of 252-420 W is able to increase foam capacity to 1.70, 1.73, and 1.76 mL/mL, respectively, which is numerically equal to or exceeds the foam capacity of HCA with TEG or PEG additives and without ultrasonic treatment.

Table 1

Effect of ultrasonic treatment of foam-forming compositions on foam characteristics

Таблица 1. Влияние ультразвуковой обработки пенообразующих композиций на характеристики пены

Foam-forming composition	Acoustic power, W	Foam capacity, mL/mL	Foam specific volume, mL/mL	Foam density, mL/mL
HCA	0	1.57	3.10	0.33
	168	1.69	3.24	0.31
	252	1.70	3.26	0.30
	336	1.73	3.31	0.29
	420	1.76	3.42	0.28
HCA-PEG	0	1.66	3.20	0.32
	168	1.71	3.30	0.30
	252	1.76	3.40	0.29
	336	1.77	3.50	0.28
	420	1.78	3.53	0.27
HCA-TEG	0	1.70	3.27	0.30
	168	1.72	3.37	0.29
	252	1.75	3.60	0.28
	336	1.78	3.65	0.27
	420	1.82	3.70	0.26

The analysis of Table 1 (Foam specific volume and Foam density) indicates that with the use of ultrasound treatment of emulsions and a gradual increase in ultrasound power, the ratio of the foam volume to the liquid part in the foam (foam specific volume) increases, while the content of moisture in foam is reduced, that is, the foam becomes more "dry". Most foam specific volume is observed in the HCA-TEG, and the same composition has the least wet foam both with and without ultrasonic treatment. "Dry-type" foams are effective when high-quality concentrates are required, however, in this case, the yield of a product may decrease [38].

Foam stability assessment of foam-forming compositions

Fig. 2 and Table 2 show the experimentally obtained dependences of the foam destruction degree (foam stability) by the modified Ross-Miles method (ISO 696) and the average foam destruction rate from ultrasound treatment of foam-forming compositions.

From the analysis of the curves in Fig. 2, it can be seen that with the use of ultrasonic treatment of all

foam-forming compositions, the foam stability increases - the remaining foam volume after a certain time period is more than without ultrasound activation. At the same time, with an increase in the ultrasonic power from 168 to 420 W, the foam stability increases in all foam-forming compositions.

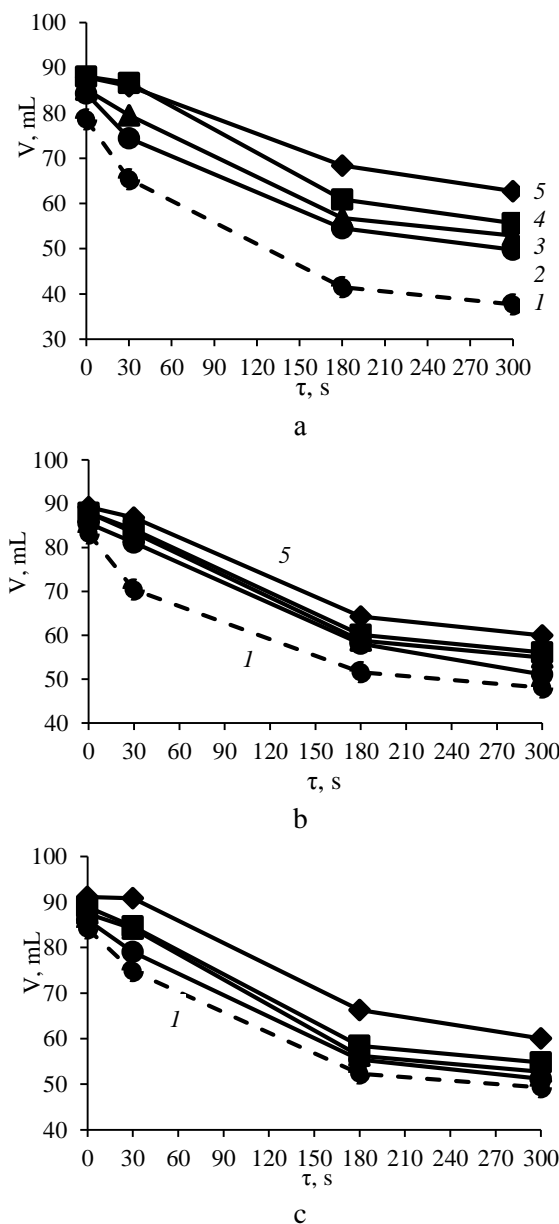


Fig. 2. Assessment of foam stability (remaining foam volume – V) by the modified Ross-Miles method (ISO 696) using ultrasound activation of foam-forming compositions: a – HCA; b – HCA-PEG; c – HCA-TEG. 1 – 0 W; 2 – 168 W; 3 – 252 W; 4 – 336 W; 5 – 420 W

Рис. 2. Влияние ультразвуковой обработки пенообразующих композиций на изменение устойчивости пен (оставшийся объём пены - V) методом Росс-Майлса (ISO 696): а – раствор солянокислого амина; б – раствор солянокислого амина с добавлением ПЭГ 200М; с – раствор солянокислого амина с добавлением триэтиленгликоля. 1 – 0 Вт; 2 – 168 Вт; 3 – 252 Вт; 4 – 336 Вт; 5 – 420 Вт

Table 2
Effect of ultrasonic treatment of foam-forming compositions on average foam destruction rates

Таблица 2. Влияние ультразвуковой обработки пенообразующих композиций на среднюю скорость разрушения пены

Foam-forming composition	Acoustic power, W	Average foam destruction rates, mL/s
HCA	0	0.137
	168	0.115
	252	0.108
	336	0.105
	420	0.084
HCA-PEG	0	0.117
	168	0.115
	252	0.110
	336	0.106
	420	0.097
HCA-TEG	0	0.116
	168	0.116
	252	0.116
	336	0.114
	420	0.104

As can be seen from Table 2, with an increase in the power of the ultrasonic treatment of foam-forming compositions, the average rate of foam destruction decreases. At the same time, the average rates of foam destruction of the HCA-TEG and HCA-PEG decrease smoothly with an increase in the ultrasonic treatment power, while the HCA has the greatest depends on the average rate of foam destruction from ultrasonic treatment, which is associated with a more stable foam (Fig. 2a) in comparison with HCA-PEG (Fig. 2b) and HCA-TEG (Fig. 2c) foams.

However, for a successful flotation process, foam with optimal stability is required: low-stability foam does not provide a high recovery of a floated product, and high-stability foam can cause technological complications at subsequent stages of the process (for example, reduced filtration efficiency and loss of flotation concentrate during thickening). Therefore, enrichment plants strive to obtain foams that would not collapse before being removed from the flotation cell, but would quickly distract in a flotation machine chutes.

The main role in the foam stability is played by the strength of the film frame, which depends on the moisture content in the foam (liquid films are unstable) and the foam specific volume. These foam characteristics, in turn, depend on the surface tension of a foam-forming composition. As shown above (Table 1), when foam-forming compositions are treated to ultrasound, the foam specific volume increases, while the moisture

content of the foam decreases, while the foam stability increases (Fig. 2 and Table 2).

The change in surface tension from ultrasonic treatment of foam-forming compositions is shown below, and the phenomenon of this change is explained.

Change in surface tension of foam-forming compositions and collector floccules size

The change in surface tension and amine floccule size of foam-forming compositions from the ultrasonic treatment is considered in Fig. 3 and Table 3, respectively.

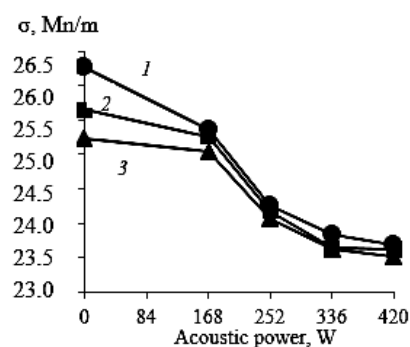


Fig. 3. Effect of ultrasonic treatment of foam-forming compositions on change in surface tension: 1 – HCA; 2 – HCA-PEG; 3 – HCA-TEG

Рис. 3. Влияние ультразвуковой обработки пенообразующих композиций на изменение поверхностного натяжения: 1 – раствор солянокислого амина; 2 – раствор солянокислого амина с добавлением ПЭГ 200М; 3 – раствор солянокислого амина с добавлением триэтиленгликоля

As can be seen from Fig. 3, with an increase in the power of ultrasonic treatment of foam-forming compositions, the surface tension at the liquid-gas phase boundary decreases. At the same time, the ultrasound treatment on the HCA with a power of 420 W reduced the surface tension by 10%. The maximum decrease in surface tension is observed upon ultrasonic activation of HCA-TEG with a power of 420 W, which is 23.51 mN/m.

It is known [33, 34] that ultrasonic treatment of colloidal solutions and flotation emulsions can disperse surfactant floccules, which in turn leads to a decrease in size and an increase in the number of floccules. Table 1 shows the results of the analysis of the amine floccules size in various foam-forming compositions.

With an increase in the power of ultrasonic treatment on foam-forming compositions, a decrease in the amine floccules size is observed due to the cavitation effect. The smallest sizes are observed at the maximum power of ultrasonic treatment of foam-forming compositions containing TEG or PEG. This is explained by the fact that frothers, along with ultrasonic cavitation, can disperse amine floccules [7, 39].

It should be noted that due to ultrasonic dispersion, amine floccules are more efficiently distributed over the entire volume of the emulsion, incl. the surface of the liquid. In combination with frothers, the concentration of surfactants at the interface liquid-gas will increase, as a result of which the surface tension will decrease (Fig. 4), which is confirmed by the data in Fig. 3, and the research [20], which studied the effect of concentrations of flotation reagents (collectors and frothers) on changes in surface tension.

Table 3

Effect of ultrasonic treatment of foam-forming compositions on the amine floccules size

Таблица 3. Влияние ультразвуковой обработки пенообразующих композиций на размер флоккул амина

Foam-forming composition	Acoustic power of ultrasound treatment, W	Amine floccules size, nm
HCA [34]	0	10.5±1.6
	168	5.8±0.9
	252	5.2±0.9
	336	4.3±0.6
	420	4.1±0.5
HCA-PEG	0	7.5±1.3
	168	6.0±0.3
	252	5.2±0.1
	336	4.9±0.8
	420	3.4±0.8
HCA-TEG	0	7.0±1.0
	168	6.6±0.3
	252	5.8±0.4
	336	4.1±0.2
	420	3.5±0.1

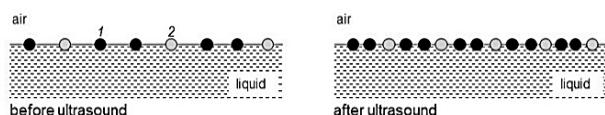


Fig. 4. Location of collector and frother molecules in the surface layer: 1 – collector; 2 – frother

Рис. 4. Расположение молекул собирателя и вспенивателя в поверхностном слое: 1 – собиратель; 2 – вспениватель

Concentrating on the surface of the phases liquid-gas, ultrasonic-activated foam-forming compositions more effectively reduce the surface tension of water and form a hydrate layer around the air bubble, which in turn reduces the coalescence of air bubbles, allowing them to maintain their original dispersion, also increase the foam stability and foaming properties.

CONCLUSION

Determined that ultrasonic activation of foam-forming compositions increases foam capacity and foam specific volume. In addition, the foam becomes less wet.

It was found that with the increase in the ultrasonic treatment power of foam-forming compositions, the foam stability increases, and the average foam destruction rate decreases.

It has been proven that with the use of ultrasonic treatment of foam-forming compositions, the surface tension decreases, which is associated with the cavitation dispersion of amine floccules, which are more efficiently distributed on the surface of the emulsion.

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DECLARATION OF INTERESTS

The authors declare the absence a conflict of interest warranting disclosure in this article.

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