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# СВОЙСТВА ПЕН, СОДЕРЖАЩИХ ГИДРОФОБИЗОВАННЫЙ ДИОКСИД ТИТАНА

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***В работе изучены свойства пен, стабилизированных гидрофобизованным диоксидом титана. Показана зависимость времени жизни данных дисперсных систем от водородного показателя (pH). Наиболее устойчивые дисперсные системы образовывались при значении pH=5, которое близко к изоэлектрической точке оксида. Пены, полученные из суспензии состава 20% $TiO_2$ + 76,6 ммоль/л гексиламина, сохраняли устойчивость в 4 сут. и более при pH=5 и изменении относительной гидрофобности поверхности от 0,383 до 1,27 ммоль/г. При значительном повышении степени гидрофобизации поверхности до 4,6 ммоль/г и таком же значении pH пенообразование было незначительным (высота слоя составляла 0,2 см), а время жизни порядка 10 сек. Вероятно, закрепление частиц на границе раздела жидкость-газ является необратимым процессом только при достижении некоторой определенной степени гидрофобности. Для объяснения возможных причин изменения устойчивости пен, содержащих диоксид титана, от водородного показателя было изучено распределение частиц по их размерам методом седиментационного анализа. Установлено, что при значении pH = 10 50% всех частиц суспензии приходится на мелкие частицы радиусом 3 мкм, одновременно максимальный радиус составлял 21 мкм. Значение краевого угла смачивания частиц было равно 40,80. В кислой среде (pH=2-3) и при pH=5 процентное содержание фракции со средним радиусом частиц 5 мкм составляло 29% и 30% соответственно. Однако низкие (14,680) значения краевого угла их смачивания при pH=2 могут являться причиной малой устойчивости пен в кислой среде. Высокоустойчивые пены были получены при изменении вязкости дисперсионной среды в суспензиях гидрофобизованного диоксида титана. При добавлении 2,0% глицерина к суспензии состава 9,0% $TiO_2$ + 0,7% гексиламина, pH=9 были получены гелеобразные пены, которые не разрушались в течение 10 сут. Однако, в кислой среде (pH=3) при добавлении такого же количества глицерина образования устойчивой дисперсной системы не наблюдали.***

**Ключевые слова:** пены, гидрофобизованный оксид титана, гексиламин, радиус частиц

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# FOAMS STABILIZATION BY HYDROPHOBIZED OXIDES OF VARIOUS CHEMICAL NATURE

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***The properties of foams stabilized by hydrophobized titanium oxide and silica are studied. It was shown that when hexylamine was added to silica suspensions (in an amount of ≥ 10 mmol/l), the values of the hydrogen index corresponded to the alkaline region. Moreover, the stability of such foams increases with the increasing concentration of hexylamine and an increase in the relative degree of hydrophobization to ng = 2.75 mmol/g. However, rapid destruction of disperse systems is possible when a certain degree of hydrophobization is achieved (for example, ng≥3 in aerosil suspensions), which is due to aggregation of particles in the initial suspension. The dependence of the lifetime of these dispersed systems on the hydrogen index (pH) is shown. The most stable disperse systems were formed at the pH value of 5, which is close to the isoelectric point of the oxide. Foams obtained from the suspension of the composition: 20% titanium oxide + 76.6 mmol/l hexylamine remained stable for four days or more at pH = 5 and a change in the relative hydrophobicity of the surface (ng) from 0.383 to 1.27 mmol/g. With a significant increase in the degree of hydrophobization of the surface to 4.6 mmol/g and the same pH value, foaming was insignificant (the layer height (h) was 0.2 cm) and life time t≈10 sec. Probably fixing the particles at the liquid-gas interface is an irreversible process only when a certain degree of hydrophobicity is achieved. To explain the possible causes of change stability of the foam containing the titanium dioxide from the pH we have investigated the distribution of particles according to their sizes by the method of sedimentation analysis. It was found that at pH=10 the fractional composition of the suspension with an average radius of 3 μm was equal to 50%, at the same time the maximum radius was 21 μm; the value of the wetting angle of the particles was equal to 40.8°. In an acidic medium (pH=2-3) and at pH=5, the fraction content with an average particle radius of 5 μm was 29% and 30%, respectively. However, low (14.7°) values of the wetting angle at pH=2 can cause low stability of foams in an acidic environment. Highly stable foams were obtained by changing the viscosity of the dispersion medium in suspensions of hydrophobized titanium oxide. When adding glycerol to the suspension composition: 9.0% titanium dioxide + 0.7% hexylamine, pH=9, gel foam was obtained, which was not destroyed within 10 days. However, in an acidic medium with the addition of the same amount of glycerol, the formation of a stable dispersed system was not observed.***

**Key words:** foam, hydrophobized titanium oxide, modified silica, hexylamine, the radius of the particles

The intensive study of foams stabilized by solid particles is due to their diverse technological applications [1-3]. It is known that in such dispersed systems it is possible to completely stop the process of liquid evolution (syneresis), diffusion gas transfer and significantly increase the lifetime [4, 5]. The study of the properties of foams containing solid additives is the scientific basis for many well-known technological processes: the development of foaming compositions for the process of drilling oil wells, the production of foams, cosmetics and detergents, drugs. It is known to use porous sorbents based on hydrophobized silica to remove hydrocarbon films from a water surface. Colloidal solids (without surfactants) can also be used to purify water from hydrocarbons.

At present, new possibilities of using such foams are considered in [6, 7]. In particular, stabilization of foams in oil recovery processes EOR (enhanced oil recovery [6]) is an urgent task. In such processes, polymers are usually added to surfactant solutions:

such as hydrolyzed polyacrylamide, guar gum. It was found that a mixture of an anionic surfactant (sodium dodecyl sulfate) and a cationic polymer (polyvinylamine), although it improves the stability of the foam, but complicates the process of foaming. Foams stabilized by micro and nanoparticles are promising substitutes for polymers. The experimental results of [6] show that the addition of nanoparticles to solutions of alpha-olefin sulfonate (AOS) and guar gum can significantly increase the stability of the foam. That is, under the same conditions, dispersed systems based on a polymer and surfactants are less stable than foams based on a surfactant and nanoparticles. The antiviral effect of $TiO_2$ nanocomposites on a culture model of cells infected with the influenza virus was noted in [8]. It was found that nanocomposites based on this oxide exhibit a pronounced antiviral effect. The analysis shows that the study of disperse systems containing solid hydrophobized phases of various oxides is an important scientific and practical task. It is relevant to study the properties of such foams depending on the nature and concentration of solid additives, the degree of hydrophobicity of stabilizing particles, the conditions for their aggregation, and the pH of the dispersion medium. In addition, the nature and concentration of the components included in the foaming solution determines in the future the properties of the foams obtained and the possibility of their practical applications.

Typically, clay, coal, silica, glass, oxides and hydroxides and many metals (iron, titanium, calcium, zinc, and others) are used as the most common stabilizing solids [9-16]. It is known that particles of solids can be adsorbed at the water-air interface, forming an interfacial layer that slows down the process of diffusion transport of gas in the foam. The main reasons for the stability of foams in the presence of a solid phase, considered in [12-19] are: adsorption hydrophobized solid particles and the formation of interphase layers on the surface of foam bubbles; capillary pressure in the foam film containing solid phase; steric or electrostatic interactions in the adsorption layers; mechanical properties of the structures ( strength and elasticity), which are formed in the dispersion medium usually results from the interaction of surfactants, solid particles and the dispersion medium. The formation of a dense layer of particles is determined by the adsorption energy of a single particle on the interfacial surface and is considered in [4, 5]:

$$\Delta G = \pi R_p^2 \sigma_{w/g} (1 \pm \cos\theta)^2, \tag{1}$$

where $R_p$ is the particle radius, $\sigma_{w/g}$ – surface tension at the interface liquid-gas; θ – edge angle, which is formed by the arrangement of the solid particles at the interface liquid-gas.

## MATERIALS AND METHODS OF RESEARCH

A cationic surfactant, hexylamine $C_6H_{15}N$ (99%, Acros Organics, with a density of 0.766 g/cm$^3$) was used as a solid particles modifier. To stabilize the foams, titanium (IV) oxide (polymorphic modification - rutile) was used; 79,87 g/mol, a density of 4.235 g/cm$^3$. Two types of colloidal silica were used: Aerosil-380 – a powder with a particle diameter of 12 nm and 41% sol (in terms of $SiO_2$) Ludox – HS with a particle diameter of 15 nm.

### *Method for determination of foam stability*

The life time ($\tau_p$) of disperse systems, depending on their stability and the nature of their destruction, was evaluated in two ways:

a) for foams stabilized by hydrophobized titanium oxide (IV), the period of destruction of the column of foam of a certain height in the gravitational field was determined [16];

b) the lifetime of foams containing hydrophobized silica was determined under conditions of accelerated syneresis [16]. In this case, the foam was placed in a special glass cell (with a porous partition). The space under the partition was connected to a container with a reduced (compared to atmospheric) pressure (Fig. 1). The value of the applied pressure drop ΔP was equal to:

$$\Delta P = P_f - P_{atm},$$

where $P_f$ – is the air pressure under the filter; $P_{atm}$ – atmospheric pressure over the foam.

The applied pressure drop ΔP was measured with a U-shaped water gauge or vacuum gauge.

### *Method for determining the contact angle*

It is known that to determine the contact angles, solid particles are placed on a plate onto which a drop of liquid is applied (sedentary drop method) or a drop is pressed (pressed drop method). This method includes estimating the contact angle using a gas bubble, which is created in a cuvette filled with the initial suspension (Fig. 1). Using a syringe, the vial is placed on a hard surface. The image enlarged by a microscope is transferred to a computer. Using the obtained photographs, the values of the contact angles are estimated [16].

### *pH Method*

The pH was monitored using a universal pH-105 ionomer. To establish the desired pH value, a solution of hydrochloric acid with a concentration of $C_{HCL}$ = 1 mol/l was used.

### *Sedimentation method*

The fractional composition of suspensions containing hydrophobized particles of medium radius ($R_{av}$) was determined by sedimentation analysis [16]. Using the sedimentation rate (v) in the gravitational field, observing the clarification of the sol (moving the sol / water interface), the radius of aggregates of micrometric size is calculated. For dynamic viscosity of water $\eta = 10^{-3}$ Pa·s, water density $\rho_w = 10^3$ kg/m$^3$, silica $\rho_s = 2{,}2\cdot10^3$ kg/m$^3$, and titanium oxide $\rho_m = 4{,}235\cdot10^3$ kg/m$^3$ (at sedimentation rate, expressed in cm/min) the average radius of the aggregates in micrometers was determined by the formula:

$$R_{агр} = \sqrt{\frac{9\eta\cdot v}{2\cdot(\rho_p - \rho_w)\cdot g}}. \qquad (2)$$

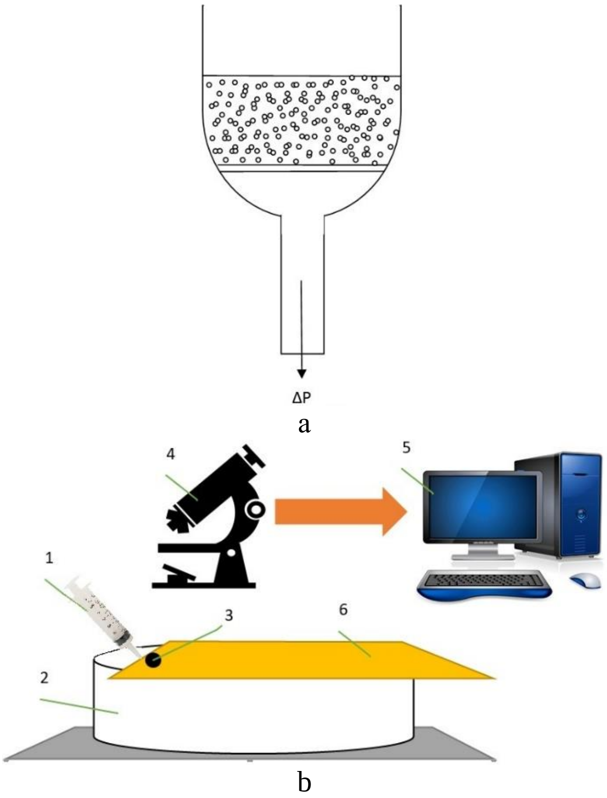

Fig. 1. Research methods: a) scheme of the setup for stability studies in conditions of accelerated syneresis; b) determination of the contact angle using the pressed bubble method: 1 - syringe, 2 - vessel with the test suspension, 3 - air bubble, 3 -plate, 4 - microscope, 5 - computer
Рис. 1. Методы исследования: а) схема установки для исследования стабильности в условиях ускоренного синерезиса; б) определение угла контакта методом прессованного пузырька: 1 - шприц, 2 - сосуд с тестовой суспензией, 3 - воздушный пузырь, 3 - пластина, 4 - микроскоп, 5 - компьютер

## RESULTS AND DISCUSSION

It was previously established [17, 18] that the stability of foams obtained from Ludox suspensions with a solids content of 2% naturally increased with an increase in the degree of hydrophobization of particles ($n_g = C_s/C_{sol}$, mmol/g; $C_s$ – surfactant concentration, $C_{sol}$ – the content of titanium oxide in the aqueous phase, g/l) from 0.54 to 4.77. Foams obtained from suspensions with a solids content of 20% and a degree of hydrophobization less than 0.21 collapsed within 4 h in a gravitational field, and with a degree of hydrophobization of more than 0.5, no liquid was released in the absence of any external influences and was highly stable. With increasing hexylamine concentration above 160 mmol/l ($n_g \geq 0.8$), the formation of a gel-like system was observed.

Foams with an Aerosil content of 2% and $n_g = 0.9$-2.75 live for more than 2 months, provided that there are no external influences [19]. However, the rapid destruction of disperse systems was established with an increase in the relative degree of hydrophobization of solid particles in silica suspensions to $n_g \geq 3$. For example, with an increase in the concentration of hexylamine to 60 mmol/l, $n_g = 3$, the lifetime of the foams decreased to 2 days, and with the further growth of hexylamine $n_g \geq 4.75$, the foams were destroyed instantaneously. Accelerated destruction due to the increase in the average radius of the aggregated particles (up to 50 microns). For foams obtained from more concentrated suspensions, for example, 4% $SiO_2$, with an increase in the degree of surface hydrophobization to $n_g = 1.63$, a transition of the foam to a gel is observed, which lives in the gravitational field for an arbitrarily long time. But with a further increase in the amount of the surfactant (more than 120 mmol/l, $n_g > 3$), the viscosity of the system begins to decrease, and as a result a mobile suspension is formed, with shaking which results in a volume, but quickly collapsing foam. With a mass content of luduox of 20%, disperse systems that are very resistant to syneresis could be obtained with a degree of hydrophobization of more than 0.5.

The stability of Aerosil foams (2 wt.% , ng = 0.9-2.75, lifetime 2 months or more) can greatly decrease with an increase in the relative degree of hydrophobization of $n_g \geq 3$, both due to an increase in the average particle radii to 100 microns, and due to the reduction of the viscosity of the suspension.

It should be noted that the studied forms of silica (aerosil and ludox) are spherical, non-porous particles of amorphous silica with a density of $2.2\cdot10^3$ kg/m$^3$. It is known that for these forms of silicon oxide, the isoelectric point and the point of zero charge correspond to the pH value of pH = 1.8-2.2. Accordingly, at pH < 2, the particles are positively charged, and at pH > 2 it is negatively charged. When hexylamine was added to silica suspensions (in the smallest amounts of 10 mmol/l), the values of the hydrogen index corresponded to the alkaline region. In particular, in suspensions of ludox with a mass content of silica of 3% and at concentrations of water repellent 10; twenty; 40 mmol/l values of the hydrogen index corresponded to the alkaline region and were equal to 8, 10, 11, respectively. That is, in all the experiments studied, the interaction of hexylamine occurred with a negatively charged surface of silica.

The analysis of the properties of foams containing hydrophobized silica allows us to conclude that a regular increase in the stability of these dispersed systems with an increase in the stabilizer concentration to certain values was observed only at a mass content of ludox 2% in the initial suspension. With a mass content of ludox of 20%, disperse systems very resistant to syneresis could be obtained with a degree of hydrophobization of more than 0.5 mmol/g.

The study of the stability of titanium oxide-containing foams showed the dependence of the lifetime of these dispersed systems on the pH value (Table 1). The most stable systems were formed at pH values of pH = 5, which is close to the isoelectric point (i.p.) of the oxide. The value of i.p. is 6.3 and 5.8 (±0.2) when the concentration of $KNO_3$ electrolyte varies from 0.001 mol/l to 0.1 mol/l [20]. The dependence of the electrokinetic potential of the titanium oxide sol on pH with the addition of a 0.2 M solution of lithium hydroxide was studied in [21]. It was established that with increasing pH, the sol changes the charge (the $H^+$ counterion changes to $OH^-$) and passes through the isoelectric point at pH = 5. It is obvious that an increase in pH above the isoelectric point not only leads to a recharge of the surface, but may also be accompanied by peptization.

***Table 1***

**Stability of titanium dioxide stabilized foams obtained at pH = 5 (suspensions 1-4); pH = 2, 3 (suspension 5, 6); pH = 9 (suspension 7)**

***Таблица 1.*** **Устойчивость пен, стабилизированных диоксидом титана и полученных при pH = 5 (суспензии 1-4); pH = 2, 3 (суспензии 5, 6); pH = 9 (суспензия 7)**

| The composition of the initial suspension | Relative hydrophobization $n_g$, mmol/g | Foam life time | Height of the column, cm |
|---|---|---|---|
| T | | ≥ 4 days | 1 |
| 2 | | days | 1 |
| 3 | | days | 2 |
| 4 | | days | 0 |
| 5) 19.2% $TiO_2$ + 122, mmol/l hexylamine | 0.64 | min | 0 |
| 6) 20% $TiO_2$ + 91 mmol/l hexylamine | 4.6 | s | 0 |
| 7) 19.2% $TiO_2$ + 122.5 mmol/l hexylamine | 0.64 | 10 s | cm |

As can be seen from the above table, the foam remained stable for three days or more with a constant value of pH of 5, and a change in the relative hydrophobicity of the surface from 0.383 to 1.27 mmol/g. With a significant increase in the degree of surface hydrophobization to 4.6 mmol/g and the same value of pH, foaming was insignificant (h = 0.2 cm), and the layer lifetime t ≈ 10 s. It is likely that particles will be fixed at the interface of a liquid-gas process by an irreversible process only when a certain degree of hydrophobicity ($n_{g,max}$) is reached. A similar phenomenon was observed earlier in [17, 18].

Perhaps, above the specified value ($n_{g,max}$), the degree of hydrophobicity no longer affects the distribution of particles between the volume and the interface. Table 1 also shows that the foam was unstable at pH values of 2,3 and 9 (suspensions 5, 6 and 7, respectively). Similarly, the most stable systems were formed at pH values that are close to the isoelectric point of aluminum hydroxide. It is known that solutions of aluminum salts (aluminum sulphate, aluminum chloride) form sols of various charges when changing the hydrogen index. In particular, at pH = 5, an aluminum hydroxide sol is formed. The zero charge point of $A1(OH)_3$ particles prepared by leaching of water-soluble aluminum salts is pH = 6.7 [22]. Note also that the foam was obtained only by adding butyric acid to a solution of aluminum sulfate and a further change in pH to 4.8-5 with sodium hydroxide solution. Thus, Table 1 shows a significant change in the stability of foams containing modified titanium oxide when the pH value changes from 2 to 9. The sedimentation method has been used to study the particle size distribution, which is formed by the interaction of titanium oxide with hexylamine at the different values of hydrogen indicators (Table 2).

***Table 2***

**The radii of the modified titanium oxide particles. The composition of the suspension: $TiO_2$ (0.74 wt %) + 7.5 mmol/ l of hexylamine, ng = 1. Q% is the percentage of the fraction with an average radius of particles Rav.**

**Таблица 2. Фракционный состав суспензий состава $TiO_2$ (0,74% масс.) + 7,5 ммоль/л гексиламина, ng=1**

| pH | Q% | $R_{av}$, μm | $R_{min}$, μm | $R_{max}$, μm |
|---|---|---|---|---|
| 2-3 | 29 | 5 | 4,3 | 13 |
| 5 | 30 | 5 | 2 | 11.8 |
| 9-10 | 50 | 3 | 1-2 | 21 |
| 2-3, electrolyte NaCl | 45 | 3 | 2 | 9.2 |

To explain the possible reasons for the change in the stability of foams containing titanium dioxide from the pH, the particle size distribution of their sizes was studied by sedimentation analysis. It was established that at pH = 10 the fractional composition of the suspension with an average radius of $R_{av}$ = 3 µm was 50% at the same time the maximum radius was 21 µm; the value of the wetting angle of the particles was equal to 40.8°. In [18], the analysis of the effect of particle size on the stability of dispersed systems containing silica. It was shown that particles of an average radius of 4-7 µm took part in the stabilization of foam films and foams. In particular, the thicknesses of bilayer foam films from a suspension of composition 2% LudoxHS-40 + hexylamine + 0.1 mol/l NaCl were equal to 8.2 and 12.9 µm with increasing degree of hydrophobization of the particle surface from 0.55 to 2.75 and a corresponding change in the contact angle from 25 to 52 degrees. Note that the thickness of the same films with a lower concentration of electrolyte (5 mmol/l) was 8.9 microns. Similarly, in suspensions of 20% LudoxHS-40 + hexylamine + 0.1 mol/l NaCl, an increase in the degree of hydrophobization of particles from 0.055 to 0.11 resulted in the formation of films with a thickness of 7.4 and 13.1 µm. Probably high (50%) content of hydrophobized titanium oxide particles with an average radius of 3 microns does not provide stable foam.

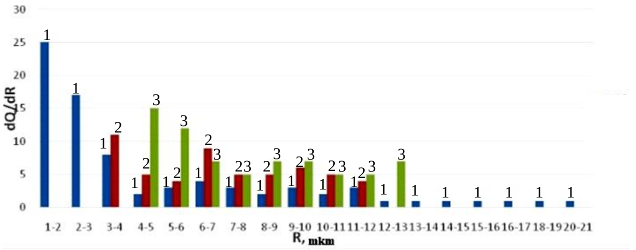

Fig. 2. Fractional composition of suspensions of the composition $TiO_2$ (0.74% wt.) + 7.5 mmol/l hexylamine, ng = 1. 1- pH=10, 2- pH=5, 3- pH=2

Рис. 2. Фракционный состав суспензии состава $TiO_2$ (0,74% мас.) + 7,5 ммоль/л гексиламина, ng = 1. 1- pH=10, 2- pH=5, 3- pH=2

In an acidic environment (pH = 2-3) and at pH = 5, the percentage of the fraction with an average particle radius of 5 µm was 29% and 30%, respectively. However, low (14.680) values of their wetting angle at pH = 2 can cause low stability of foams at the indicated value of pH. In particular, at pH = 2-3 and an increase in the degree of hydrophobization from 0.15 to 4.6, foaming was insignificant, and the life of the foam layer did not exceed 10 minu. It was previously established that a change in the viscosity of the dispersion medium affects the stability of the resulting foams. It is also known that increasing viscosity slows down the process of syneresis and increases the stability of the resulting foam. Highly resistant foams were obtained by changing the viscosity of the dispersion medium in suspensions of hydrophobized titanium oxide. In particular, by adding 2.0% glycerol to the suspension of the composition: 9.0% $TiO_2$ + 0.7% hexylamine, pH = 9, highly stable gel foam foams were obtained, which were not destroyed for 10 days. However, in an acidic medium (pH = 3), adding the same amount of glycerol did not observe the formation of a gel-like disperse system.

Thus, it was found in the work that when hexylamine was added to silica suspensions (in an amount of ≥ 10 mmol/l), the values of the hydrogen index corresponded to the alkaline region. The stability of the studied foams increased with an increase in the concentration of hexylamine and an increase in the relative degree of hydrophobization to $n_g$ = 2.75 mmol/g. The rapid destruction of such dispersed systems upon reaching a certain degree of hydrophobization is due to the aggregation of particles in the initial suspension. The formation of stable foams containing titanium oxide was observed at a certain value of pH (pH = 5) and with a change in the relative degree of hydrophobization of the solid surface from 0.38 to 1.37. Particles with an average radius of 3 µm and less did not lead to the formation of a stable dispersion system in an alkaline medium. The low (less than 15°) degree of hydrophobization of solid particles is one of the possible reasons for the low stability of foams at pH = 2. A change in the viscosity of the dispersion medium at the degree of hydrophobization of solid particles corresponding to the contact angle ≈ 40° led to the formation of highly stable foams in an alkaline medium.

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