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ФОРМИРОВАНИЕ И УСТОЙЧИВОСТЬ ПЕН В ВОДЕ, СОДЕРЖАЩЕЙ ДИЗЕЛЬНОЕ ТОПЛИВО, В ПРИСУТСТВИИ МОДИФИЦИРОВАННОГО КРЕМНЕЗЕМА

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Формирование пен в присутствии органических жидкостей и изучение их свойств и устойчивости представляет важную научную и практическую задачу. Целью исследования является изучение свойств пен, которые формируются в воде, насыщенной дизельным топливом (DF), в присутствии модифицированных частиц кремнезема. В качестве твердой фазы использовали диоксид кремния двух видов: Aerosil, Ludox HS-40 и модификатор – гексан-1-амин (99%) плотностью 0,766 г/см^3. При формировании пены использовали воду, насыщенную дизельным топливом. Для ее получения смешивали равные объемы дистиллированной воды и дизельного топлива (DF), перемешивали в течение 4-5 мин, через 1 сут. водную фазу отделяли с помощью делительной воронки. Исследовали пенообразование при смешении определенного объема V_2 растворенного DF и суспензии модифицированных твердых частиц объемом V_1. Исследована максимальная высота слоя пены (L), которая образуется в водной среде, содержащей дизельное топливо, в присутствии модифицированных частиц диоксида кремния, а также корреляция ее устойчивости со степенью модификации поверхности диоксида кремния и концентрацией органической жидкости. Показано, что устойчивые к разрушению слои пены высотой 2 см образуются из модифицированного золя Ludox HS-40 с процентной концентрацией твердой фазы 20% и воды, насыщенной дизельным топливом, взятых в равных объемных соотношениях. При добавлении к суспензии гидрофобизированного Ludox HS-40 растворенного дизельного топлива в большем (в 2-4 раза) объеме формировались неустойчивые пены с большей высотой столба L = 9-11 см. Показано, что пенообразование в системе гидрофобизированный аэросил – растворенное дизельное топливо было незначительным (L≤3 см); разрушение пен обусловлено их диффузионным укрупнением. Образование устойчивой пеноэмульсии, полученной из суспензии гидрофобизированного аэросила и дизельного топлива возможно при соотношении их объемов, равном 3:1.

Ключевые слова: модифицированный кремнезем, гидрофобизация поверхности, дизельное топливо, пена, устойчивость пены, краевой угол

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FORMATION AND STABILITY OF FOAMS IN WATER CONTAINING DIESEL FUEL IN THE PRESENCE OF HYDROPHOBIZED SILICA

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The formation of foams in the presence of organic liquids and the study of their properties and stability is an important scientific and practical task. The purpose of this work is to study the properties of foams that are formed in water saturated with diesel fuel (DF) in the presence of hydrophobized silica particles. Two types of silica were used as the solid phase: Aerosil-380, Ludox HS-40 and hydrophobizer-hexane-1-amine (99%) with a density of 0.766 g/cm^3.Water saturated with diesel fuel was used to produce the foam. To obtain it, 50 ml of distilled water was added to 50 ml of diesel fuel (DT), stirred for 3-5 min and left in a dividing funnel at a temperature of 25 °C. After 24 h, the water was separated. Foaming was investigated when mixing a certain volume (V2) of dissolved diesel fuel and a suspension of hydrophobized solid particles with volume V1. The maximum height of the foam layer (H), which is formed in an aqueous medium containing diesel fuel in the presence of hydrophobized silica, as well as the change in its stability with an increase in the degree of hydrophobization of silica and the concentration of organic liquid, was studied. It was found that resistant to destruction foam layers (L~ 2 cm) are formed from hydrophobized Ludox HS-40 sol with a mass content of 20% and water saturated with diesel fuel, taken in equal volume ratios. When hydrophobized Ludox HS-40 was added to the suspension, dissolved diesel fuel in a larger (2-4 times) volume formed unstable foams with a higher column height H = 9-11 cm. It is shown that the foaming in the hydrophobized aerosil – dissolved diesel fuel system was insignificant (L ≤ 3 cm). The destruction of foams is due to their diffusion enlargement. The formation of a stable foam emulsion obtained from a suspension of hydrophobized aerosil and diesel fuel is possible with a volume ratio of 3:1.

Key words: modified silica, surface hydrophobization, diesel fuel, foam, foam stability, contact angle

The formation of foams in the presence of organic liquids and the study of their stability is an important scientific and practical problem. It is well known that the efficiency of a number of technological processes: the separation of oil and oil products from wastewater, defoaming, fire extinguishing is determined by the stability of the resulting dispersed systems, which can be formed both in the presence of surface-active substances (surfactants) and particles of the solid phase [1-11]. In the study [1], experiments were carried out to study disperse systems stabilized by various surfactants in the presence of isomeric hydrocarbons, which showed the dependence of foam stability on the type of stabilizer, viscosity and density of the oil. In [2], the relationship between the content of surfactants, gel-like solid particles, electrolytes and the stability of aqueous disperse systems containing petroleum products is shown. Foam formation and foam stability in the presence of saturated hydrocarbons for various surfactant stabilizers was studied in [3]. It was shown that the stability of the foam and the volume of liquid in it increased with increasing concentration of the surfactant. The main stages of foam destruction are identified: at the first stage, gravitational syneresis prevailed with the column height being preserved. At the second stage, the destruction of the foam column occurred as a result of coalescence. The destabilizing effect of oil was more pronounced for alkanes with shorter carbon chains.

In [4], the dependence of the efficiency of wastewater treatment from oil products by froth flotation on the salt concentration, bubbling rate, and hydraulic fluid retention time is shown. One of the main problems in the use of foam for enhanced oil recovery is the effect of oil on its stability [5]. In this article, the foamability and foam stability in the absence and presence of alkane-type oils for various commercial surfactants used in the petroleum industry (Dowfax 8390, C12-15 Enordet, Petrostep SB and C14-16 AOS alpha-olefin sulfonate) were investigated. C14-16 AOS was found to provide the greatest foam stability in the presence of oil. In [6, 7], the conditions for the effective isolation of DF dissolved in water were determined by the flotation method using sodium dodecyl sulfate (DDSNa) and gelatin. An increase in the concentration of dissolved hydrocarbons correlates with an increase in surface tension at the aqueous solution (DDSNa + + gelatin)/air interface and is determined by the ratio of the concentrations of gelatin and DDSNa.

The study of the effect of organic substances on the stability of foams is usually carried out in the study of defoaming processes [9, 10] and in fire fighting [11, 12]. In [9], when describing the mechanism of foam stability in the presence of an organic liquid, a significant role is assigned to asymmetric foam films. It was established in [11, 12] that the rate of destruction of water films is determined by the intensity of diffusion penetration of alcohol from mixed organic fuel.

It is known that solid hydrophobized particles can act as defoamers and good foam stabilizers under certain conditions [13-23]. The influence of such powders on the properties of Pickering foams is due to various factors: mechanical, adsorption, steric, etc. The energy factor plays a special role, since the presence of hydrophobic solid particles in the initial suspension from which the foam is obtained leads to a decrease in surface tension at the liquid-air interfaces. Experimental confirmation of the relative decrease in tension with a change in the contact angle is given in [17]. The greatest change in the relative interfacial tension was observed at a contact angle of wetting the silica surface of 45°, which corresponded to the most stable foams. However, an increase in the concentration of the water repellent in accordance with an increase in the contact angle can reduce the stability of foams and prevent their formation as a result of coagulation of solid phase particles.

Reducing the stability of foams and preventing foaming is possible with the simultaneous use of oil and solid particles [22, 23]. In [22], the effect of oil and solid phase on the stability of foams was studied. The use of oil as an antifoaming agent performed worse than a mixture of oil and solids. In [23], mixed defoamers are considered. The explanation of the mechanism is expressed by the analysis of the formation of contact angles of wetting at the solid-water-oil and solid-water-air interfaces. In this case, the main role of solid particles in mixed defoamers is the destruction (penetration) of an asymmetric film, which is formed at the oil-water-air interface.

An analysis of these works shows that hydrophobized particles can be good foam stabilizers under certain conditions: the degree of particle surface modification, contact angle, pH, gelation in the dispersion medium of the film and foam. The lifetime of such dispersed systems can be determined in years in the absence of external influences. However, the dispersed solid phase, when obtained from a hydrophobic material (Teflon) or with certain processing of particles that are hydrophilic in nature, can prevent foaming. At the same time, the development of defoamers containing both oil and solid particles is relevant in many technological processes. A possible practical model that allows one to study the simultaneous effect of an organic liquid and solid particles on the foam stability is the study of the properties of such systems in the presence of an organic liquid contained in a dispersion medium.

The aim of this study is to study the formation and stability of Pickering foams that form in water saturated with diesel fuel (DF) in the presence of hydrophobized silica particles.

MATERIALS AND METHODS OF RESEARCH

When obtaining foams, hexane-1-amine or hexane-1-amine (99%) with a density of 0.766 g/cm^3 and two types of silica were used: Aerosil and Ludox HS-40. Ludox HS-40 (Merck) is a silica sol with a SiO_2 particle concentration of 41%. Aerosil -380 (Evonik) is a bluish amorphous airy powder that produces dust when poured. The surface of silica particles is hydrophilic. The particle surface was modified by chemical adsorption of cationic surfactants for its hydrophobization.

Silica slurry was prepared at a concentration of 2 to 20% by mixing solid particles with a calculated volume of distilled water. Hexane-1-amine (0.02-0.2 ml) was added dropwise to the suspension and stirred for 2-3 min. The degree of relative modification of the surface of solid particles d_g (mmol/g), was calculated by the formula:

$$d_g = C_{hex}/C$$

where C_{hex} – surfactant concentration, mmol/l, C – concentration of silicon dioxide in the aqueous phase, g/l. The foam was obtained by shaking a suspension of a given composition in a stoppered tube for 30 s. The height of the foam column above the liquid in the test tube, measured with a ruler in centimeters, was taken as the height of the foam layer.

To obtain water saturated with diesel fuel, distilled water and DF were mixed in a 1:1 volume ratio, stirred for 4-5 min, after a day the aqueous phase was separated using a separating funnel.

The setup used to determine the contact angle by the pressed bubble method is schematically shown in Fig. 1. Air bubble 5 is passed into the cuvette with initial suspension 3 by syringe 2 so that it touches glass plate 4 and does not touch the walls of the cuvette. Using a photograph of such a bubble, the value of the contact angle (θ) is determined graphically.

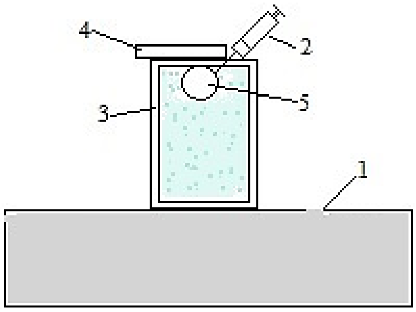

Fig. 1. Installation diagram for determining the contact angle by the pressed bubble method: 1 – table; 2 – syringe; 3 – cell with the studied suspension; 4 – glass plate; 5 – air bubble

Рис. 1. Схема установки для определения краевого угла методом прижатого пузырька: 1 – стол; 2 – шприц; 3 – кювета с исследуемой суспензией; 4 – стеклянная пластина; 5 – пузырек воздуха

The role of solid particles in foam systems is determined by their degree of hydrophobicity or contact angle. Particles whose surface is characterized by $\Theta \approx 90°$ behave as stabilizers of a dispersed system, and in the case with a contact angle of more than 90°, particles play the role of antifoam agents [22, 23].

The influence of the degree of hydrophobization of a solid surface on the stability of dispersed systems was studied in [16-18]. It has been established that an increase in the content of hexyl-1-amine changes the contact angle Θ for the Ludox HS-40 or Aerosil silica phase. The maximum value of Θ in all experiments at all studied concentrations of silicon dioxide took a value of 45 degrees. Hydrophilic silica particles in the absence of hexyl-1-amine are not able to fix at the “water solution/air” interface, and therefore are not able to stabilize the disperse system, just like hexyl-1-amine, being a short-chain surfactant, is a poor stabilizer, giving small amounts of foam, disintegrating in a few seconds. Modified solid particles form stable foams that practically did not collapse for several months at a concentration of hexyl-1-amine corresponding to the optimal degree of surface modification (Table 1).

Table 1

Degradation degree of foams stabilized with 20% Ludox HS-40, %

***Таблица 1.* Степень деструкции пен, стабилизированных 20% Ludox HS-40**

C_{hex}, mmol/l	Degree of foam destruction, %		
	2 h after receiving it	5 days after receiving it	4 months after receiving it
21.4	95	–	–
63.8	29	31	30
95.3	13	12	12
127.6	0	0	1

The degree of foam destruction was defined as the ratio of the destroyed foam volume to the initial foam volume in percent.

Similarly, foams containing Aerosil 2%, when the concentration of hexane-1-amine is changed from 18 to 55 mmol/l, live more than 2 months. It was previously noted that the maximum stability of the foam layers is observed at the maximum degree of modification of the surface of the solid phase, corresponding to contact angles of 45-50 degrees. Further modification of the surface of silica particles with hexane-1-amine, exceeding a contact angle of 500, was impossible as a result of their coagulation. But work [24] shows the possibility of phase reversal in disperse systems stabilized by solid particles, as a result of which the foam turns into “dry” water or powder, inside the particles of which water is contained.

Table 2 shows the ability to form foams from a suspension of 20% Ludox HS-40 + hexane-1-amine in water containing diesel fuel.

From Table 2 it follows that an increase in the concentration of the water repellent from 45.96

to 61.3 mmol/l was accompanied by an increase in the height of the foam column from 5 to 11 cm. However, the dispersed system was unstable: 92% and 59% of the foam layer was destroyed in 4-5 min.

Table 3 shows the correlation of the maximum layer height of foam stabilized with modified silica in the presence of DF with the relative degree of surface modification of the solid phase.

Table 2

Stability of foams stabilized with modified Ludox HS-40 in the presence of DF at a ratio of V_2:V_1 = 8:3, where V_1 is the volume of silica slurry; V_2 is volume of water with DF

***Таблица 2.* Устойчивость пен, стабилизированных модифицированным Ludox HS-40, в присутствии DF при соотношении V_2:V_1 = 8:3, где V_1 – объем суспензии кремнезема; V_2 – объем воды с DF**

N	Composition of the initial disperse system	Foam column height (L), cm	Experiment time (t), min	Degree of foam destruction (D), %
1	20% Ludox HS-40+22,98 mmol/l hexane-1-amine	5	5	84
2	20% Ludox HS-40+45,96 mmol/l hexane-1-amine	9	4	92
3	20% Ludox HS-40+61,3 mmol/l hexane-1-amine	11	4	59

Table 3

Stability of foams obtained from silica of various degrees of modification in water containing DF

***Таблица 3.* Стабильность пен, полученных из диоксида кремния различной степени модификации в воде, содержащей DF**

N	Composition of the initial disperse system	Ratio V_2:V_1	t, min	D, %	L, cm	d_g, mmol/g
1	20% Ludox HS-40 + 28,7 mmol/l hexane-1-amine	4:1	6	93	5,4	0.14
2	20% Ludox HS-40 + 28,7 mmol/l hexane-1-amine	2.7:1	5	84	5	0.14
3	20% Ludox HS-40 + 76,5 mmol/l hexane-1-amine	2.7:1	4	59	11	0.38
4	2% aerosol + 76,5 mmol/l hexane-1-amine	0.78	60	35	2	3.8

It follows from the Table (experiments 1 and 2) that at a low degree of modification of the solid surface d_g = 0.14 (which corresponds to $\theta \leq 300$), unstable foams 5-5.4 cm high are formed. In the absence of a modifier in the initial suspension, low foaming was observed (the height of the foam column did not exceed 2 mm) in combination with extremely low stability (lifetime no more than a minute).

An increase in the degree of particle surface modification correlated with an increase in foaming. So, with an increase in dg to a value of 0.38 in the third experiment (Table 3), a twofold increase in the height of the foam column was observed compared to experiments 1 and 2.

The reasons for the change in the stability of foams at various degrees of modification of the silica surface were discussed in [13-17]. It was noted that the foams obtained from the Ludox HS-40 sol with a solids content of 20% by weight in the absence of DF were characterized by increased stability. Such foams at concentrations of hexyl-1-amine 63.8-95.3 mmol/l remained stable for several months. The data in Table 3 indicate that the stability of dispersed systems containing modified silicon dioxide in the presence of dissolved DF is also determined by the ratio of the concentrations of the modifier and the solid phase in the initial suspensions.

The content of DF and the relative degree of modification of the surface of the particles had an impact on the stability of the foam.

For example, at the degree of hydrophobization of the surface of the solid phase (d_g = 0.42) from 1 ml suspension of 20% Ludox HS-40 + 84 mmol/l hexane-1-amine and 7 ml of water saturated with DF (V_2:V_1 = 7:1), a rapidly degrading foam It was obtained. Its lifetime was 1.5 min, the height of the foam column was 0.6 cm. On the contrary, when 4 ml of the above suspension was added to 4 ml of the above suspension and shaking the resulting suspension, a foam column 2.2 cm high was formed, the stability of which is shown by curve 2 in Fig. 1. From 20% Ludox HS-40 with the addition of 108.8 mmol/l of hexane-1-amine (d_g = 0.54) with a significant dilution with water with DF (V_2:V_1 = 7:1), a foam layer with a foam height of 0.5 cm was obtained, the lifetime of which was 4.5 min. With an increase in the amount of hydrophobized particles V_2:V_1 = 4:4, the height of the foam column was 2.3 cm, Fig. 1.

An increase in foam formation (L = 7 cm) and the formation of a more stable foam were observed when using a suspension of the composition: 2% Ludox HS-40 solution + 19.15 mmol/l hexane-1-amine with a higher degree of surface hydrophobization (d_g = 0.96). With a volume ratio of V_2:V_1 = 1:9, about 64% of the layer remained stable for 70 min (curve 1, Fig. 2).

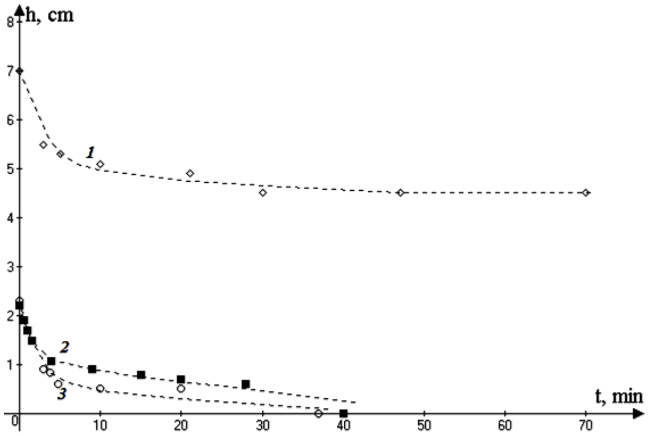

Fig. 2. Destruction curves of foams obtained from Ludox HS-40 sol: ◊ – 2% Ludox HS-40 +19.15 mmol/l hexane-1-amine, $v_2:v_1 = 1:9$ (curve 1), ■ – 20% Ludox HS-40 + 108.8 mmol/l hexane-1-amine, $v_2:v_1 = 4:4$ (curve 2), ○ – 20% Ludox HS-40 + 84 mmol/l hexane-1-amine, $v_2:v_1 = 4:4$ (curve 3)

Рис. 2. Кривые разрушения пен, полученных из золя Ludox HS-40: ◊ – 2% Ludox +19,15 ммоль/л гексиламина, $v_2:v_1 = 1:9$ (кривая 1), ■ – 20% Ludox HS-40 + 108,8 ммоль/л, $v_2:v_1 = 4:4$ (кривая 2), ○ – 20% Ludox HS-40 + 84 ммоль/л, $v_2:v_1 = 4:4$ (кривая 3)

Foams stabilized with hydrophobized silica (Aerosil) have been studied. Note that in this case, foaming was not intense, and the layer height did not exceed 3 cm. For example, a 10% suspension of Aerosil + 61.3 mmol/l of hexane-1-amine was mixed with dissolved diesel fuel in a volume ratio of $V_2:V_1 = 8:3$. Upon shaking, a foam 2.5 cm high was formed, which completely collapsed within 50 min. In all cases, the destruction was accompanied by diffusion enlargement of bubbles; the formation of foam cells with a diameter of about 4 mm was observed. The coarse structure of such foams is shown in Fig. 3.


a
b

Fig. 3. Foam stabilized with hydrophobized aerosil in the presence of dissolved diesel fuel: a) ratio $V_2:V_1 = 8:3$;
b) ratio $V_2:V_1 = 3:1$ (7 days after its formation)

Рис. 3. Пена, стабилизированная гидрофобизированным аэросилом в присутствии растворенного дизельного топлива: а) соотношение $v_2:v_1 = 8:3$; б) соотношение $V_2:V_1 = 3:1$ (через 7 сут. после ее формирования)

Thus, in the work it is established:

- destruction-resistant foam layers ~ 2 cm high can be formed from hydrophobized Ludox HS-40 sol with a mass content of 20% and water saturated with diesel fuel, taken in equal volume ratios;

- when adding dissolved diesel fuel to the suspension of hydrophobized Ludox HS-40 in a larger volume (2-4 times), unstable foams were formed with a higher column height L = 9-11 cm;

-foaming in the hydrophobized aerosil system - dissolved diesel fuel was insignificant (layer height did not exceed 3 cm); the destruction of foams is due to their diffusion enlargement.

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

Авторы заявляют об отсутствии конфликта интересов, требующего раскрытия в данной статье.

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