

The Role of Physically Adsorbing Species of Oxyhydriyl Reagents in Flotation Aggregate Formation

S. A. Kondratyev, O. I. Ibragimova

Abstract—The authors discuss the collecting abilities of desorbable species (DS) of saturated fatty acids. The DS species of the reagent are understood as species capable of moving from the surface of the mineral particle to the bubble at the moment of the rupture of the interlayer of liquid separating these objects of interaction. DS species of carboxylic acids (molecules and ionic-molecular complexes) have the ability to spread over the surface of the bubble. The rate of their spreading at pH 7 and 10 over the water surface is determined. The collectibility criterion of saturated fatty acids is proposed. The values of forces exerted by the spreading DS species of reagents on liquid in the interlayer and the liquid flow rate from the interlayer are determined.

Keywords—Criterion of action of physically adsorbed reagent, flotation, saturated fatty acids, surface pressure.

I. INTRODUCTION

COLLECTING abilities and selectivity of collecting agents are considered to depend mostly on the features of adsorption of the agents at the solid–liquid interface. In a series of studies, it is shown that collecting abilities of such collecting agents as saturated fatty acids and amines can be materially affected by their property to reduce surface tension at the gas–liquid interface. The correlation between the surface tension in the saturated fatty acid film on the free liquid surface and the floatability of a useful mineral has been proved experimentally [1]–[3]. Quast states that the value of the surface pressure of a reagent film not always characterizes collecting abilities of saturated fatty acid [3]. For example, pH range of increased extraction of hematite by tetradecanoic acid disagrees with the pH range of high surface pressure. On that ground, the author in [3] thinks that the surface properties of this acid have no influence on flotation performance.

The correlation between the surface pressure in a reagent film and the floatability of quartz has been proved for cation reagents [4]. Finch and Smith [5] show that combination of adsorbed ion–molecular groups of amines at a mineral particle surface and high surface tension of “fresh” bubbles promotes better flotation results. The use of bubbles with the lower

surface tension resulted in the decreased efficiency of flotation.

It is found that collecting abilities of dialkyl dithiophosphate depend on the dynamic activity of the latter relative to the gas–liquid interface [6]. As in the case of saturated fatty acids and amines, with the higher dynamic activity, the collecting abilities of dialkyl dithiophosphates enhance. In the capacity of a collectibility criterion, the authors suggest the dynamic surface tension of a reagent.

The mechanism of influence exerted by reagents that are active relative to the gas–liquid interface on the formation of particle–bubble aggregate has been put forward in [7], [8]. According to this mechanism, bubbles adsorb surface active reagents and transport them to a mineral particle surface. Transition of active reagents from the air bubble to a mineral particle reduces the surface tension at the particle–bubble interface and accelerates formation of a flotation aggregate. It is supposed that bubbles that carry surface active flotation agents should have lower surface tension. However, the proposed mechanism disagrees with the conclusions drawn in the study [5] where the necessity of high surface tension of “fresh” bubbles is illustrated.

The mechanism of active species of flotation agents with their return from a mineral particle to the air bubble has been proposed by Kondratyev [9]–[11]. The researcher shows that, at the moment of the particle and bubble collision, local rupture of an interlayer between them takes place, and a meniscus with an advancing angle is formed. At the same moment, the gas–liquid interface contacts the particle surface. The surface active associates of, for instance, saturated fatty acids $(RCOO)_2H^+$ or amines $(RNH_3^+) \cdot (RNH_2)$ adsorbed at the mineral particle go to the meniscus surface and, owing to high spreading rate, entail water from the interlayer between the subjects of interaction. The spreading velocity of a reagent film and the absence of the hydrophobic sliding of the film on the water surface are the factors that govern the time of the water removal from the interlayer.

In some studies, it is shown that an adsorption coat consists of a few layers. The lower layer is attached chemically. Physical adsorption is possible as a result of interaction between counter ions Na^+ and anion of a reagent [12]. This layer is well-organized and compacted thanks to the hydrophobic interaction of hydrocarbon chains. The layer imparts strong hydrophobic property to the mineral particle surface [13]. The upper layer forms only on the mineral surface coated with a chemically or physically adsorbed

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reagent, and is represented by calcium and sodium oleates and neutral molecules of saturated fatty acid. The upper layer imparts no extra hydrophobic properties to the mineral. According to [9]–[11], it is supposed that at the moment of the interlayer rupture, the upper layer can desorb from a mineral particle and transfer to the bubble surface. High spreading velocity of DS of agents is possible when bubbles with high surface tension, or “fresh” bubbles as per Finch and Smith terminology, are used. The quantity of a collecting agent transferred to the gas–liquid interface depends on the surface activity of the agent and on the density of its adsorption at the mineral. This layer attached to a hydrophobized surface is assumed to be a DS of a reagent.

Let us discuss the applicability of the proposed behavior of a flotation agent, physically adsorbed at the gas–liquid interface and desorbable from it, to the floatability–surface pressure relations obtained in [3]. It seems interesting to study the behavior of collecting agents from the class of saturated fatty acids in view of their common use in flotation of non-sulfide ores. These agents feature notable activity at the gas–liquid interface. To find a collecting agent with the optimal properties, it is required to relate the values of the surface tension of a solution pregnant with the active associates and the force exerted by the film of the agents on the liquid inside the interlayer.

The aims of this study are to assess collecting abilities of a physically adsorbed collecting agent based on the mechanism proposed in [9]–[11], to develop a criterion for the comparison of the collectibilities of agents belonging in the same homologous series, and to test the criterion based on the known experimental and practical data. Another objective of the study is the numerical determination of the collecting force of DS species from the series of saturated fatty acids upon the assumption that this force is effective under removal of water from the interlayer. With this end in view, the author solves the problem on water removal from the interlayer by the flowing species of saturated fatty acids.

II. MATERIALS AND EQUIPMENT

In the determination of spreading velocities of acids over water surface, acids of technical grade Ch were used: hexanoic acid (Technical Specification TS 6–09–126–75), tetradecanoic acid (TS 6–09–127–75) and palmitic acid (TS 6–09–4132–75). In octanoic, decanoic and dodecanoic acids, the blending agent content made 98%. Filming used high-speed camera CasioEXLIMEX-F1 (300 snapshots per second). The tests used distilled water. Values of pH were measured by addition of NCl and NaOH.

In the tests of flotation of fluorite, both experiment and checkout, the ore from the Yaroslavl Mining and Processing Works was used. Flotation was carried out on flotation machine 189 FL with the volume of 0.5 l. The size of the floating material was $-44 \mu\text{m}$ to the extent of 88%. The collecting agent was decanoic acid at the consumption of 4000 g/t. The depressor for calcite was soluble glass at the consumption of 1500 g/t. For the foam formation, reagent T-80 was used. The physically adsorbed layer was removed from

the mineral surface using alcohol at the consumption of 20 ml per 200 g of ore.

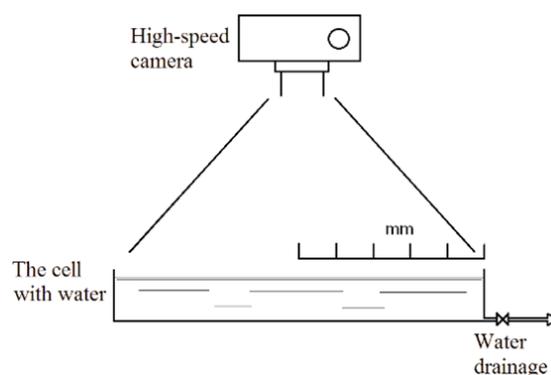


Fig. 1 The plant to determine spreading velocity of saturated fatty acids over water surface

III. EXPERIMENT

The spreading velocity of saturated fatty acids over water surface was determined using a plant represented by a cell filled with water (Fig. 1). The cell was placed under the camera to make video file of flow of saturated fatty acid species active relative to the gas–liquid interface. The spreading velocity of saturated fatty acids over water surface was determined for pH values of 7 and 10 (Figs. 2 and 3, respectively).

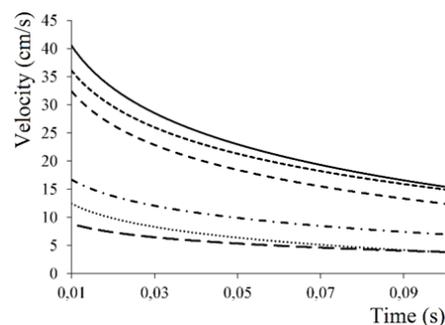


Fig. 2 Time dependence of spreading velocity of saturated fatty acids over water surface at pH = 7.0: — hexanoic, ---- octanoic, - - - decanoic, - · - · - dodecanoic, ···· tetradecanoic, — · — hexadecanoic

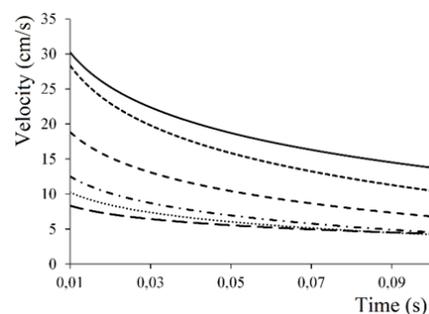


Fig. 3 Time dependence of spreading velocity of saturated fatty acids over water surface at pH = 10.0. The legend in Fig. 2

Regarding the flotation by the physically adsorbing reagents, the experiment on decanoic acid flotation of fluorite was carried out. The series was composed of two tests. The first one, checkout test, was decanoic acid flotation of fluorite. In accordance with [12], [13], the adsorption coat is made by the chemical and physical adsorption. In the second test, the adsorption coat contains single layer of chemically attached anions of decanoic acid. In the experimental test, after agitation, the ore was subjected to decantation and alcohol wash.

The decanoic acid at the consumption of 400 g/t acted as the collecting agent. It was assumed that alcohol would remove the desorbable layer of the adsorption coat on the mineral.

The agitation time was 15 min in the both tests. The flotation duration was 16 min. Table I describes the results of the flotation test.

TABLE I
DATA ON FLOTATION OF FLUORITE BY DECANOIC ACID WITHOUT WASH OF THE MATERIAL AFTER AGITATION AND WITH ALCOHOL WASH OF THE MATERIAL AFTER AGITATION WITH THE REAGENT

Product	Yield γ , %	Content of CaF_2 , %	Recovery of CaF_2 , %
Without wash			
Froth product	47.5	41.43	81.1
Middlings	52.5	8.75	18.9
Total	100	24.27	100
With alcohol wash after agitation with reagent			
Froth product	31.6	35.16	42.5
Middlings	68.4	21.98	57.5
Total	100	26.15	100

IV. RESULTS AND DISCUSSION

In the study [3], the author emphasizes correlation between the values of the surface pressure of octanoic, decanoic, and dodecanoic acids and the recovery of hematite. The correlation is interrupted in case of long-chain reagents: tetradecanoic, hexadecanoic, octadecanoic acids. The total lack of the said correlation for the long-chain acids allowed the author [3] to state that the value of the equilibrium surface pressure is not always a good indication of the efficiency of a flotation collector. According to the mechanism of the flotation agent behavior proposed in [9]–[11], the pressure calculated as the difference between the equilibrium surface tensions of water and a reagent solution fails to display the physical aspect of the formation of a flotation aggregate. For the flotation aggregate to be formed, of importance is the local value of the surface pressure at a short moment of the interlayer rupture and the spreading velocity of reagent species. For this reason, for the long-chain reagents, it is required to account for a decrease in the spreading velocity due to the cohesion of their hydrocarbon fragments. It follows from Fig. 2 that the initial spreading velocity of tetradecanoic acid is 2.5 times lower than the initial spreading velocity of decanoic acid. Given the equal surface pressures of these acids, it can be expected that the collecting ability of tetradecanoic acid is 2.5 times lower as compared with the decanoic acid. The experimental

flotation of hematite has shown that decanoic acid recovery of hematite exceeds hematite recovery by tetradecanoic acid by 2.5 times.

The short-chain fatty acids with the hydrocarbon chain composed of less than 8 hydrocarbon atoms have high spreading velocity over water surface (Figs. 2 and 3). Their water solubility is considerably higher than solubility of the long-chain acids. Incidentally, adsorption of the short-chain fatty acids at a mineral surface is insignificant. The gradient of the surface tension, or, in other words, the surface pressure of the film of the reagent at the moment of rupture of the water interlayer is also low, which limits the velocity and mass of DS of the reagents.

Based on the obtained data, a criterion is proposed for the numerical determination of change in the collecting ability under replacement of a saturated fatty acids acid with a certain hydrocarbon fragment for another acid with different length of the hydrocarbon chain. Such criterion of the collectibility of DS species of saturated fatty acids can be the thickness of the surface flow of DS species. In this case, the surface flow thickness in a given section is understood as the total energy of the flow in this section per unit time. At different points in the flow cross section, associates of DS species of reagents have different energy due to transfer of some energy to the adjacent water layers. The flow thickness can be expressed in terms of the product of the surface flow energy at a point and the mass flow rate: $Q_m: N = \pi_0 Q_m$. Here, π_0 is the surface pressure, N/m Q_m – is the surface flow of DS species of reagent, m^2/s . Apart from the value of the surface pressure, the criterion takes into account the spreading velocity of DS species of reagent over water surface. The criterion is applicable for the comparative assessment of collecting agents belonging in the same homologous series of chemical compounds.

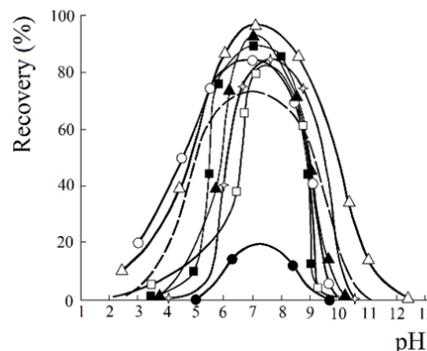


Fig. 4 Floatability of minerals with oleic acid (1 kg/t) versus pH:
—□—columbite, —◇— zircon, —▲— tantalite, —■— ilmenite,
—○—rutile, —△— garnet and tourmaline, —●— albite, — — perovskite
[15, p. 266]

The variation of pH in a flotation system will change the thickness of the surface flow. The flow thickness will be changed due to deceleration of spreading of a reagent and owing to a decrease in the mass of its DS on a mineral surface. The spreading velocity of films of saturated fatty acids at pH

~10 is shown in Fig. 3. For decanoic acid, the spreading velocity at pH 10 lowers by 1.8 times as compared with the range of neutral pH. It is worthy of accounting for the potential wash-out of the molecular form of saturated fatty acids from mineral surface by alkali solution in the alkali range of pH. In [14], it was found that some DS of reagents can be washed by water. Oleate desorption from the surface of hematite and magnetite makes 20 and 35% at pH 6.3 and 45 and 65% at pH 10, respectively. All in all, reduction in spreading velocity of saturated fatty acids and removal of DS species of a reagent from a mineral particle surface results in the depression in flotation process in the alkali range of pH (Fig. 4).

According to the available information [12], an adsorption coat consists of two layers: the lower layer with the chemically attached reagent and the upper layer composed of calcium and sodium oleates and neutral molecules of saturated fatty acid or ion-molecular complexes. By Abramov's test data, alcohol readily removes oleic acid molecules and fails to move away calcium oleate [16]. In this connection, after washing of feed ore with alcohol, it is plausible that mineral surface only keeps chemically adsorbed reagent. Recoveries of fluorite in case of the single mode of sorption (chemical) and with the both modes of reagent adsorption (chemical and physical (hydrophobic) adsorption) essentially differ. The experiment has shown that both modes of adsorption are important. High performance of flotation is only reached when a mineral surface contains both chemically and physically adsorbed reagent. The absence of any kind of adsorption greatly worsens flotation results. According to the experimental data in [3], it is assumable that readily DS of a flotation agent at the moment of formation of a flotation aggregate can entail liquid and can remove it from the interlayer between a mineral particle and the air bubble. Removal of liquid from the interlayer lifts the kinetic restriction of the formation of flotation aggregates.

V. COLLECTING FORCE OF FATTY ACIDS

We calculated numerically collecting force of DS species for saturated fatty acids with six to 18 carbon atoms in the chain. With this point in view, we determined the force exerted by the film of spreading DS species of reagent on liquid in the water layer using the solution to the problem on liquid removal from the water layer with the surface tension gradient on the water layer surface [11]. It was assumed that physically adsorbed DS species of reagent were on the mineral surface. Solution of the DS species possesses low surface tension. After the local rupture of the water layer, physically adsorbed DS species get access to the gas-liquid interface, where the value of surface tension is factually equal to the surface tension of water ("fresh" bubbles). If there is the non-uniform surface tension, capillary forces will be generated and affect the reagent film along a tangential to the free surface of the liquid, thus forcing the reagent to spread [17]. The reagent film flow entails the adjacent layers of liquid and generates a convective flow named as Marangoni convection.

A thin layer of viscous incompressible liquid (interlayer) is

bounded by a hard surface $z = 0$ from below and by a free boundary $z = H(r, t)$ from above. To describe the axial-symmetric flow, the Navier-Stokes equations in a cylindrical coordinate system (r, θ, z) were used [18]

$$\rho \left(\frac{\partial v_r}{\partial t} + v_r \frac{\partial v_r}{\partial r} + v_z \frac{\partial v_r}{\partial z} \right) = -\frac{\partial p}{\partial r} + \mu \left[\frac{1}{r} \frac{\partial}{\partial r} \left(r \frac{\partial v_r}{\partial r} \right) - \frac{v_r}{r^2} + \frac{\partial^2 v_r}{\partial z^2} \right] \quad (1)$$

$$\rho \left(\frac{\partial v_z}{\partial t} + v_r \frac{\partial v_z}{\partial r} + v_z \frac{\partial v_z}{\partial z} \right) = -\frac{\partial p}{\partial z} - \rho g + \mu \left[\frac{1}{r} \frac{\partial}{\partial r} \left(r \frac{\partial v_z}{\partial r} \right) + \frac{\partial^2 v_z}{\partial z^2} \right] \quad (2)$$

$$\frac{1}{r} \frac{\partial r v_r}{\partial r} + \frac{\partial v_z}{\partial z} = 0. \quad (3)$$

μ = kinematic viscosity factor; ρ = liquid density; p = pressure; g = acceleration of gravity in the negative direction of the z axis; the velocity factor components (v_r, θ, v_z) . Equations (1)–(3) were simplified down to the equations of the theory of lubrication. The simplification assumed that the relation between the characteristic vertical dimension (thickness of the liquid film on the mineral surface at initial time, H_0) and the characteristic horizontal dimension (e.g., initial radius of surfactant spot, R_0) was much less than 1.

The calculations involved: $\mu = 1.052 \cdot 10^{-3} \text{ Pa} \cdot \text{s}$; $\rho = 10^3 \text{ kg/m}^3$; $g = 10.0 \text{ m/s}^2$; $D_S = 10^{-8} \text{ m}^2/\text{s}$; $S = \sigma_w - \sigma_m$ - surface pressure, N/m; $\sigma_w = 72.3 \cdot 10^{-3} \text{ N/m}$; σ_m was varied depending on the length of hydrocarbon fragment of saturated fatty acids molecules, and was evaluated using the data from the paper [3] and experimentally.

Fig. 5 shows the profile of the water layer surface $h(r, t)$, the surface concentration $\Gamma(r, t)$ of the reagents and the volume flow rate Q_m as the functions of radial distance at the time $t = 4.0 \cdot 10^{-5} \text{ s}$.

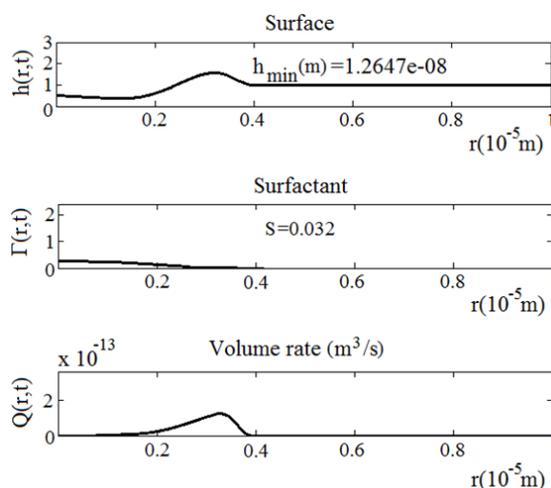


Fig. 5 Calculation results for liquid flow from the water layer due to spreading of fatty acid film with 10 carbon atoms in the chain: *a* — free surface profile; *b* — reagent concentration; *c* — flow rate of liquid as the functions of radial coordinate at the time $t = 4.0 \cdot 10^{-5} \text{ s}$; $\sigma_m = 40.3 \times 10^{-3} \text{ N/m}$

From the comparison of the interlayer thickness h behind the front of the spreading reagent film and the liquid flow rates, it

follows that the rate of the liquid removal grows in line of the following sequence of acids: hexanoic, dodecanoic, octanoic and decanoic (Table II). In accordance with the same sequence, the collecting abilities of the saturated fatty acids enhance [3]. The calculated values for the collecting forces F of DS of saturated fatty acids under assumption that these forces are effective in liquid removal from the interlayer appropriately characterize collectibility of the acids as per [3].

The proposed mechanism refines the conclusions drawn in [3]. The pH range of the surface pressure of tetradecanoic acid disagrees with the range of the maximum floatability of hematite. On this ground, the author [3] arrives to the conclusion that it is not always valid that the value of the surface pressure of a reagent film characterizes collecting abilities of saturated fatty acids. In accordance with the proposed mechanism, precipitation of DS species of tetradecanoic acid (ion–molecular complexes) results in the increase in the density of the acid adsorption at a mineral surface and, after rupture of the interlayer, in the higher gradient of the surface tension. For this reason, creating conditions for extraction of agent species that are active relative to the gas–liquid interface from solution, and for their precipitation on a mineral surface is one of the major factors of high performance flotation. As per the proposed criterion, namely, reagent DS surface flow thickness, a reagent, aside from the high surface pressure, should be capable to spread at a high velocity over the gas–liquid interface. Fig. 2 implies that the spreading velocity of tetradecanoic acid is 2.5 times lower than the spreading velocity of decanoic acid. That is the reason for tetradecanoic acid to exhibit weaker collectibility than decanoic acid. The statement that the surface pressure does not always characterize the collecting properties of saturated fatty acids implicates that an incorrect criterion is selected for the evaluation of the collecting abilities of a flotation agent.

The mechanism proposed in [9]–[11] confirms the conclusions made in [5] on the requirement of “fresh” bubbles for flotation. High surface pressure of “fresh” bubbles and low surface tension of the solution of ion–molecular complexes or DS species of reagents work towards generation of high local surface pressure at the moment of rupture of the liquid interlayer. High spreading velocity of DS species of a reagent results in rapid removal of liquid from the interlayer and induces formation of a flotation aggregate.

TABLE II

VALUES OF SURFACE PRESSURE S , FLOTATION REAGENT FORCE F , WATER LAYER THICKNESS h AND MAXIMUM FLOW RATE Q_m AT $R = 0.2 \cdot 10^{-5}$ M FOR SATURATED FATTY ACIDS WITH SIX TO 18 CARBON ATOMS IN THE CHAIN

Parameters	Saturated fatty acids			
	Hexanoic	Octanoic	Decanoic	Dodecanoic
$S = \sigma_w - \sigma_m$ (mN/m)	3.0	15	32.0	28.0
h (m)	$2.68 \cdot 10^{-8}$	$1.62 \cdot 10^{-8}$	$1.26 \cdot 10^{-8}$	$1.3219 \cdot 10^{-8}$
F (H)	$7.57 \cdot 10^{-6}$	$4.57 \cdot 10^{-5}$	$9.78 \cdot 10^{-5}$	$8.6896 \cdot 10^{-5}$
Q_{max} (m ³ /s)	$1.6 \cdot 10^{-14}$	$6.8 \cdot 10^{-14}$	$1.2 \cdot 10^{-13}$	$1.097 \cdot 10^{-13}$
t_m (s)	$4.0 \cdot 10^{-5}$			

VI. CONCLUSIONS

Formation of a flotation aggregate depends on the value of local surface pressure at the moment of rupture of interlayer. The equilibrium pressure calculated as the difference of surface tensions of water and a reagent solution is not always reflective of the physical nature of the process of flotation aggregate formation. In case of long-chain reagents, it is required to account for the decrease in their spreading velocity due to cohesion of their hydrocarbon fragments.

The authors have proposed the criterion for the comparative evaluation of collectibilities of reagents belonging in the same homogenous series: thickness of surface flow of DS of reagents.

The flotation force of DS species is calculated for saturated fatty acids on the assumption that major restriction on the formation of a flotation aggregate is the liquid interlayer between a mineral particle and the air bubble after local rupture of reagent film. It has been found that the order of change in the calculated collecting forces F for the DS of saturated fatty acids coincides with the line of change in the flotation activity of these acids.

The mechanism of liquid removal from the interlayer by the spreading DS species of reagents confirms the conclusion drawn in [5] on the need of “fresh” bubbles for flotation. The high surface tension of “fresh” bubbles and the low surface tension of the solution of ion–molecular complexes of DS species of reagents promote generation of high local surface pressure at the moment of rupture of the liquid interlayer.

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