Influence of organic and inorganic precipitators on the efficiency of surfactants extraction from wastewater by flotation

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Abstract. The chemical structure of surfactants causes their accumulation on the surface of water sources and leads to the formation of stable foam, which prevents the access of oxygen to the water and leads to the death of hydrobionts and deterioration in water quality. Therefore, the extraction of surfactants from wastewater is an urgent task. Increase the efficiency of flotation extraction of cationic surfactants, namely hexadecyl pyridinium chloride $[C_{16}H_{33}NC_5H_5]Cl$ (CHDP) and surfactants based on it - cetazol, by introducing organic – (sodium alkyl sulfate (SAS), $C_nH_{2n+1}OSO_3Na$, n=10-16), and inorganic - potassium hexacyanoferrate (II, III) precipitators. The solubility of the products of interaction of cationic surfactants with SAS (sublates) was determined, which are extracted from solutions at different temperatures and impurities of electrolytes. The Gibbs free energy ($\Delta G^0_{ads}$), enthalpy ($\Delta H^0_{dis}$), and entropy ($\Delta S^0_{dis}$), which take place in the process of dissolution, are calculated. The low value of the heat of dissolution (2.1-16.6 kJ/mol) indicates a small effect of temperature on the solubility of sublates. The thermodynamic analysis of the process of formation of sublates allows determining the optimal length of the hydrocarbon radical of the precipitator. It is established that the extraction of the studied cationic surfactants should be extracted from water solutions and wastewater by the method of precipitation flotation using organic and inorganic precipitators. The presence of electrolytes and an increase in temperature leads to an increase in the degree of extraction hexadecyl pyridinium alkyl sulphonates sublates.

1. Introduction

Currently, water quality and safety have become one of the most troubling environmental issues. A third of the world’s drinking water demand is provided by surface sources: lakes, rivers, and canals [1, 2]. At the same time, these sources of freshwater also serve as a reservoir for domestic and industrial wastes. At the same time, surface water is used as a source of drinking and domestic water [3]. In developing countries, wastewater is treated and reused for agricultural irrigation, surface water recharge, and even as drinking water [1, 4]. Therefore, surface water must be cleaned of contaminants before use, and water reuse requires higher quality wastewater to ensure water safety, especially for the control of microbiological pollutants, surfactants, metals, and other harmful substances [2, 5].

The main sources of water pollution are untreated and incompletely treated wastewater. Emissions of industrial wastewater contaminated with technical surfactants (surfactants) have led to a critical state of surface water sources in Ukraine, which has been formed for a long time
due to neglect of objective laws of development and reproduction of natural resources of Ukraine. When entering the water, the most widespread surfactants interfere with biologic oxidation processes and inhibit their self-purification. As a result, sanitary and chemical indicators of water quality deteriorate - dry matter increases, oxidation, and chemical oxygen demand increase. In addition, the water acquires an unpleasant taste and odor if it contains surfactants [6,7]. Surface water is one of the most impacted ecosystems on Earth, and its changes have led to extensive environmental degradation, such as reduced water quality and availability, species extinction, waterlogging, reduced biodiversity [8].

According to current international standards, a lot of surfactants are classified as the third and fourth class of hazard and therefore their content in open water and wastewater, which are used for biological treatment, is regulated. There is a need for improvement of existing and development of new methods of wastewater treatment of technical wastewater, which is quite a complex technological task. The flotation method deserves special attention, as it is notable for its simplicity, cost-effectiveness, high productivity and widely implemented in practice [9]. Flotation is known as a separation process based on the introduction of gas bubbles as a transport medium. The suspended particles, being hydrophobic or hydrophobic conditioned, then attach themselves to the bubbles and move toward the surface of the aqueous solution [8, 10]. This method has a number of modifications both by the type of bubble formation and by the presence of additional components (reagent and nonreagent). Initially, this method was used in the mining industry, but later the method has found wide application in other industries and demonstrates high results of wastewater treatment from various pollutants [8]. In this regard, the development of ideas about the patterns of flotation removal of technical ionic surfactants is relevant from both scientific and practical points of view and allows to expand basic knowledge and provide a scientific basis for improving wastewater treatment technology contaminated with harmful substances.

The aim of the work is to improve the flotation extraction of cationic surfactants based on N-hexadecyl pyridinium from aqueous solutions and wastewater, by introducing inorganic and organic reagents, and thermodynamic justification of their choice.

2. Research methods and materials

2.1. Object of research and staging of the experiment

The objects of research are N-hexadecylpyridinium chloride \(C_{16}H_{33}NC_5H_5Cl\) (CHDP) and technical surfactants based on it - cetazole, which is widely used in industry. Sodium alkylsulfates (SAS) with the formula \(C_nH_{2n+1}OSO_3Na\) (where \(n=10-16\), were used as precipitators. Model surfactant solutions were prepared by dilution in bidistilled water. The solubility of sparingly soluble products of the interaction of cationic surfactants with sodium alkyl sulfates (sublates) was determined by the turbidimetric method. Below are the results of the research aimed at determining the basic regularities of the flotation separation process of hexadecyl pyridinium chloride \((1.5-5)*10^{-4} \text{ mol/dm}^3\) and cetazol \((50 \text{ mg/dm}^3\) precipitated by sodium alkyl sulfates at a concentration below the critical concentration of myceloid formation. Their choice was conditioned by their availability and biological quality [9]. To determine the effect of electrolytes on the solubility of sublates, a salt \((NaCl)\) solution with a concentration of \(5,0*10^{-4} \text{ mol/dm}^3\) was used as an electrolyte. SAS solutions were injected into CHDP and cetazol solutions at continuous mixing in the quantity required for the formation of sparingly soluble substances \(C_{16}H_{33}C_5H_5\text{NOSO}_3R_1\), where \(-R_1\) is a hydrocarbon radical \((CH_3−CH_2−)\).
2.2. Flotation extraction parameters and evaluation of its efficiency

Flotation treatment of surfactant solutions was performed on the unit, the main element of which was a 0.140 m high glass column of 0.045 m diameter. The bottom of the column and air disperser was a glassy crusty plate. The air was supplied to the column at a speed of 2.5-5.8*10^{-7} m^3/s. The volume of surfactant solution poured into the column was 3*10^{-2} dm^3, flotation time 600 s. The efficiency of the flotation separation process of surfactants was evaluated by the degree of their separation from the solution (\(\alpha\)).

\[
\alpha = \left(\frac{c_0 - c_p}{c_0}\right) \times 100
\]  

(1)

where \(c_0\) and \(c_p\) - surfactant concentration in the solution, before and after flotation.

Each measurement was repeated 5-6 times to obtain statistically significant results. The measurement error of the degree of flotation purification of HDP and cetazole \(\alpha\) did not exceed 5 %.

2.3. Determination method of surfactant content in solution

The surfactant content in the solution was performed by photometric method which is based on the interaction of cationic surfactants with methyl orange and the formation of a chloroform-soluble yellow complex. The color intensity of the chloroform solution is proportional to the concentration of the methyl orange-cationic surfactant complex and is measured photometrically. To determine the surfactant concentration, aliquots of the test solution are placed in a separatory funnel and diluted with distilled water to 50 ml. Add 1 ml of methyl orange (0.1 %), 5 ml of citrate-phosphate buffer solution (\(pH = 3.0\)) and 25 ml of chloroform. Close the separating funnel with a stopper and shake it for 30 seconds. Allow the sample to stand for 20 minutes. The chloroform layer is separated from the water and is at the bottom. Place the filter funnel over a clean cuvette (thickness of the absorbed layer 5 sm) and filter the chloroform layer through fiberglass to remove water from it. Collect chloroform extract in a cuvette. Determine the optical density of the chloroform solution at \(\lambda = 415\) nm relative to pure chloroform. The equivalent surfactant content is determined by a calibration curve constructed for cetyldimethylbenzylammonium chloride [11].

3. Results and discussion

The solubility of mixed micelles sublates is low (table 1), and is determined by the length of the SAS hydrocarbon radical which is involved in their formation. And much less solubility of the compounds formed by the deposition of alkylpyridinium ions by inorganic precipitants [9] and with increasing temperature in the range of 20-40 °C decreases slightly, and then with increasing to 60 °C practically does not change. This can be explained by an increase in sublate particles formed by the interaction of hexadecylpyridinium chloride and cetazole with sodium alkyl sulfates, which dehydration of the particles causes [12]. At all temperatures from the solutions by flotation can be removed that part of the surfactant, which when introduced into the precipitator solution becomes part of the dispersed phase of the colloidal solution formed. With increasing temperature from 20 to 40 °C, solubility decreases slightly, then, with increasing to 60 °C practically does not change. The values of the free Gibbs dissolution energy (\(\Delta G_0^{\text{dis}}\)), calculated according to the equation:

\[
\Delta G_0^{\text{dis}} = -RT\ln S P
\]

are in the range 48.3-67.5 kJ/mol and increase with increasing temperature and the length of the SAS carbohydrate radical; enthalpy

\[
\Delta H_0^{\text{dis}} - \ln S = -\Delta H_0^{\text{dis}}/(RT) + \text{const}
\]

(3)
in the range - (2.1-16.6) kJ/mol; entropy
\[ \Delta S^0_{dis} - \Delta S_{dis} = (\Delta H^0_{dis} - \Delta S^0_{dis})/T \] (4)
in the range -140.2 - 165.3 J/mol*K and decrease with increasing temperature.

Table 1. Values of $S$ (solubility), $\Delta G^0_{dis}$, $H^0_{dis}$ and $\Delta S^0_{dis}$ for the process of dissolving the products of the interaction of hexadecylpyridinium chloride and cetazole with sodium alkyl sulfates. **Note.** *:- SOS - sodium octyl sulfate; SS - sodium decyl sulfate; SDS - sodium dodecyl sulfate; STS - sodium tetradecyl sulfate; SHS - sodium hexadecyl sulfate; CZ - cetazole

| Sublate | T,K   | $S * 10^{-5}$, $mol/dm^3$ | $\Delta G^0_{dis},kJ/mol$ | $\Delta H^0_{dis},kJ/mol$ | $-\Delta S^0_{dis},J/mol*K$ |
|---------|-------|---------------------------|-----------------------------|-----------------------------|-----------------------------|
| *SOS    | 293   | 5.0                       | 48.3                        | 2.1                         | 157.8                       |
|         | 303   | 4.8                       | 50.1                        | 2.1                         | 158.5                       |
|         | 313   | 4.7                       | 51.8                        | 2.1                         | 158.8                       |
|         | 323   | 4.6                       | 53.6                        | 2.1                         | 159.5                       |
|         | 333   | 4.5                       | 55.4                        | 2.1                         | 160.2                       |
|         | 293   | 3.0                       | 50.7                        | 2.1                         | 144.7                       |
|         | 303   | 2.9                       | 52.6                        | 2.1                         | 146.2                       |
| *SS     | 313   | 2.7                       | 54.7                        | 8.3                         | 148.2                       |
|         | 323   | 2.7                       | 56.5                        | 8.3                         | 149.2                       |
|         | 333   | 2.7                       | 58.2                        | 8.3                         | 149.8                       |
| *SDS    | 293   | 2.7                       | 51.5                        | 9.7                         | 142.6                       |
|         | 303   | 2.6                       | 53.2                        | 9.7                         | 143.5                       |
|         | 313   | 2.5                       | 55.4                        | 9.7                         | 145.9                       |
|         | 323   | 2.5                       | 56.9                        | 9.7                         | 146.0                       |
|         | 333   | 2.5                       | 58.7                        | 9.7                         | 147.1                       |
| *STS    | 293   | 0.9                       | 56.5                        | 16.6                        | 136.1                       |
|         | 303   | 0.8                       | 59.1                        | 16.6                        | 140.2                       |
|         | 313   | 0.7                       | 61.8                        | 16.6                        | 144.6                       |
|         | 323   | 0.7                       | 63.7                        | 16.6                        | 145.7                       |
|         | 333   | 0.7                       | 65.7                        | 16.6                        | 147.5                       |
| *SHS    | 293   | 0.7                       | 57.9                        | 12.5                        | 155.1                       |
|         | 303   | 0.6                       | 60.6                        | 12.5                        | 158.5                       |
|         | 313   | 0.5                       | 63.5                        | 12.5                        | 158.5                       |
|         | 323   | 0.5                       | 65.5                        | 12.5                        | 158.5                       |
|         | 333   | 0.5                       | 67.5                        | 12.5                        | 158.5                       |
| *SDS+CZ | 293   | 1.2                       | 55.2                        | 8.3                         | 160.0                       |
|         | 303   | 1.2                       | 57.2                        | 8.3                         | 161.3                       |
|         | 313   | 1.1                       | 58.9                        | 8.3                         | 161.6                       |
|         | 323   | 1.2                       | 60.8                        | 8.3                         | 162.5                       |
|         | 333   | 1.3                       | 62.3                        | 8.3                         | 162.1                       |

The dependence of the solubility of the sublimate on the number of atoms (n) of carbon in the carbohydrate radical SAS is described by the equation:

\[ \ln S = A - (16 + n)B \] (5)
Numerical values of coefficients A and B were determined experimentally and calculated theoretically:

\[ A = \frac{(\Delta G_p^0 + \Delta G_p^{0\prime})}{(2RT)}, \quad B = -\frac{\Delta G_{CH_2}^0}{2RT} \]  

(6)

(where \( \Delta G_p^0 \), \( \Delta G_p^{0\prime} \) - the contribution to the Gibbs energy of the polar atom group of sublate formation; \( \Delta G_{CH_2}^0 \) - contribution to the methylene group of sublate formation), the values of 1.84 and 0.32 are well matched and equal, respectively. The obtained results indicate that SAS are quite effective precipitators and at the same time flotation collectors of the studied surfactants, which allows them to be used as precipitators at the same time flotation collectors of cationic surfactants [9]. For all members of the SAS homologous series \(|\Delta G_{form,subl}^0| > |\Delta G_{form,collect}^0|\) (therefore, they are able to interact with surfactants with the formation of sublates – mixed micelles), then \(|G_{form,subl}^0| > |\Delta G_{ads}^0|\) (as a result, their flotation extraction takes place in the mode of foam flotation).

The point of intersection of the lines that describe the dependence of the intrinsic Gibbs energy of the SAS creation \((\Delta G_{form,collect}^0)\) and the intrinsic Gibbs energy of adsorption \((\Delta G_{ads}^0)\) of SAS, depending on the length of the carbohydrate radical, which corresponds to sodium tetradecyl sulfate \((n=14)\), coincides with the maximum point on the curve of the dependence of the degree of flotation isolation of HDPAS on the length of the carbohydrate radical SAS (figure 1).

This can be explained based on the notion of adsorption-mechanical energy ratio (AMER) of surfactants, which is described by the ratio of Gibbs free energy of surfactant adsorption at the solution-air interface to Gibbs free energy of their micelle formation in solution (AMER \(= \frac{\Delta G_{ads}^0}{\Delta G_{micel}^0}\)), according to which the efficiency of the collecting action of the SAS is maximal at AMER=1 [13].

It should be noted that for the first time flotation purification of water from surfactants using molecular analogues (potassium alkylcarboxylates) of SAS as precipitators and flotation collectors of surfactants, was presented in Skrilov et al., 1998 [14]. His method allows to achieve the highest surfactant recovery in the foam fraction and reduce flotation time. Comparison of the obtained experimental values of using SAS with the prototype Skrylov et al. shows higher efficiency (up to 95 %) than the prototype using potassium alkylcarboxylates (80-90 %). In addition, the alkyl carboxylates used by the Skrylev group have higher biological rigidity, cost and low biodegradation. They also do not describe the effect of the presence of electrolytes on α [14].

Introduction to surfactant solutions containing HGDP sublates a small amount \((1.0-3.5*10^{-4}mol/dm^3)\) of electrolytes into sublate solutions, somewhat reduces their solubility. The solubility of HDPAS is affected by both the concentration of the counterion (Na⁺ - ions) and the nature of the electrolyte, which is associated with varying degrees of their dissociation (table 2).

One of the advantages of flotation extraction of surfactants in the form of sparingly soluble compounds, in contrast to their extraction in ionic or molecular form, is the ability to expand the range of pH values optimal for their flotation extraction, namely its displacement into the neutral area, which is always technologically feasible. The degree of flotation extraction of CHDP and cetazole in the form of sparingly soluble compounds practically does not change with pH varying, because the pH shift to strongly acidic or alkaline area does not significantly affect the solubility of compounds that are practically not hydrolyzed [12], and only changes in the colloid-chemical properties of sublates are observed.

The use of SAS as precipitators and at the same time flotation collectors, makes it possible to reduce the flotation time up to 5-6 minutes instead of 10 minutes, which on an industrial scale can save material resources. The Skrylev method with co-authors also involves reducing the flotation time to these values (5-6 min) [14]. The speed constants of the flotation extraction
Figure 1. Influence of the number of carbon atoms (n) in the SAS carbohydrate radical on:
- degree of flotation extraction (a) CHDP (1) and b - the Gibbs volatile energy of the SAS 
adsorption at the interface of the fluid-air phase $\Delta G_{ads}^0$ (2), the Gibbs volatile energy of the 
SAS formation $\Delta G_{form, colec.}^0$ (3), free Gibbs energy adsorption sublate at the interface phase 
solution-air $\Delta G_{ads, subl}^0$ (4) and the free Gibbs energy of substrate formation $\Delta G_{form, subl.}^0$ (5). 

Based on the first obtained values of solubility of products of interaction of 
hexadecylpyridinium chloride with sodium alkyl sulfates without and in the presence of 
electrolytes in solution, we concluded that the process of flotation extraction of cationic 
surfactants from aqueous solutions using organic precipitants (sodium alkyl sulfates) is effective 
because: the degree of flotation extraction of hexadecylpyridinium chloride is maximum when 
introduced into a solution of sodium tetradecyl sulfate, the AMER of which is equal to one; 
reducing the solubility of hexadecylpyridinium-alkyl sulfates leads to an increase in the degree 
of flotation extraction of HDP$^+$ ions. n particular, it is established that the obtained laws 
allowed to move to scientifically sound methods of wastewater treatment from surfactants, as 
as well as to identify and establish such methods of flotation that provide the greatest efficiency of 
the treatment process.

The efficiency of flotation extraction of the studied technical cationic surfactants can be 
increased by switching from foam fractionation to precipitating flotation, by introducing 
inorganic precipitators. Thus, the introduction into the surfactant solutions of electrolytes 
(potassium hexacyanoferrates III and II) (table 4), which form sparingly soluble compounds
Table 2. The effect of electrolyte solution (NaCl) additives on the solubility (S) of the interaction products (sublates) of alkyl sulfates (SAS) and cetazole (50 mg/dm$^3$).

| Sublate         | $C, mg/dm^3$ | $S\times 10^{-5}, mol/dm^3$ |
|-----------------|--------------|-------------------------------|
| SOS + cetazole  | 0            | 4.3                           |
|                 | 100          | 4.0                           |
|                 | 200          | 3.8                           |
|                 | 300          | 3.5                           |
|                 | 400          | 3.3                           |
|                 | 500          | 3.0                           |
| SS + cetazole   | 0            | 2.6                           |
|                 | 100          | 2.2                           |
|                 | 200          | 2.0                           |
|                 | 300          | 1.8                           |
|                 | 400          | 1.6                           |
|                 | 500          | 1.4                           |
| SDS + cetazole  | 0            | 1.2                           |
|                 | 100          | 1.0                           |
|                 | 200          | 0.8                           |
|                 | 300          | 0.6                           |
|                 | 400          | 0.5                           |
|                 | 500          | 0.4                           |
| STS + cetazole  | 0            | 0.4                           |
|                 | 100          | 0.4                           |
|                 | 200          | 0.3                           |
|                 | 300          | 0.3                           |
|                 | 400          | 0.3                           |
|                 | 500          | 0.3                           |
| SHD + cetazole  | 0            | 0.3                           |
|                 | 100          | 0.2                           |
|                 | 200          | 0.2                           |
|                 | 300          | 0.2                           |
|                 | 400          | 0.1                           |
|                 | 500          | 0.1                           |

Table 3. Values of rate constants of extraction of hexadecylpyridinium chloride and cetazole with sodium alkyl sulfates

| Sublate     | SS  | SDS | STS | SHS | CHDP | SDS+cetazole |
|-------------|-----|-----|-----|-----|------|--------------|
| Speed constants,$\times 10$ min$^{-1}$ | 4.6 | 5.8 | 6.3 | 6.9 | 4.2  | 5.2          |

with surfactants, intensifies the process of their removal by precipitation flotation (the degree of flotation recovery increases to 90 %, the volume of foam switched to solution, decreases to 1-5 % of the total volume of the solution, reduces the flotation time for technical cationic surfactants
to 5 minutes, the range of pH values favorable for flotation extraction expands.

Table 4. The effect of potassium hexacyanoferrates III and II content on the degree of flotation extraction of cetazole. 

Note. Flotation time - 5 minutes.

| Electrolyte          | $C \times 10^{-2}$, mol/dm$^3$ | $\alpha$, % |
|----------------------|-------------------------------|-------------|
| $K_3[Fe(CN)_6]$      | 0                             | 75.0        |
|                      | 0.5                           | 93.0        |
|                      | 1.0                           | 92.0        |
|                      | 2.0                           | 100.0       |
|                      | 3.0                           | 100.0       |
|                      | 4.0                           | 100.0       |
|                      | 5.0                           | 100.0       |
| $K_4[Fe(CN)_6]$      | 0                             | 75.0        |
|                      | 0.5                           | 94.0        |
|                      | 1.0                           | 97.0        |
|                      | 2.0                           | 98.0        |
|                      | 3.0                           | 98.0        |
|                      | 4.0                           | 100.0       |
|                      | 5.0                           | 100.0       |

Elucidation of physicochemical regularities of the process of extraction of the studied cationic surfactants and thermodynamic analysis showed that they should be removed from aqueous solutions and wastewater by precipitation flotation using organic (SAS) and inorganic (potassium hexacyanoferrates III and II) precipitators. The presence of electrolyte ($NaCl$) and an increase in temperature leads to an increase in the degree of extraction of HDPAS sublates. The advantages of using precipitation flotation can also include a large pH range of purified water.

Thus, the method of precipitation flotation can be recommended for wastewater treatment containing both cationic surfactants and anionic after mixing, which does not require additional costs.

4. Conclusions

The solubility of the products of the interaction of cationic surfactants with sodium alkyl sulfates (sublates), which are extracted from solutions at different temperatures and impurities of electrolytes, has been established; the Gibbs free energy ($\Delta G^{0}_{ads}$), enthalpy ($\Delta H^{0}_{dis}$) and entropy ($\Delta S^{0}_{dis}$), which take place in the process of dissolution, are calculated. The low value of dissolution heat (2.1-16.6 $kJ/mol$) indicates a small effect of temperature on the solubility of sublates. The thermodynamic analysis of the process of formation of sublates allows to determine the optimal length of the hydrocarbon radical of the precipitator. The high efficiency of the method for extracting surfactants from model solutions 75 – 100 % has been established, which in combination with the low cost of equipment, allows to recommend it for wastewater treatment from this type of pollutants.

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