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ANALYTICAL CHEMISTRY OF WATER

Floatation–Extraction Recovery of U(VI) by Means of Thin Emulsified Solutions of Trialkylamine in White Spirit

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Abstract—We have established expediency and high efficiency of using thin emulsified solutions of trialkylamine in white spirit as floatation collectors of U(VI) during its floatation–extraction recovery from nitrate, sulfate, and carbonate solutions. We have also determined optimal conditions of running the process (the pH, consumption of the collector, floatation time) and depending on the ionic composition of the solution and the pH of the medium its mechanism was suggested.

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Since the prospects of the development of Ukraine's uranium–extraction industry look promising [1] the issue of developing the effective and accessible methods of recovering small amounts of uranium from large volume of solutions of the various ionic composition that form during mining and hydrometallurgical processing of uranium ores [2]. In addition, wastewaters containing uranium feature high chemical toxicity (the uranium MAC in the water of open water bodies equals 0.05 mg/dm^3 [31]), therefore prior the discharge to open water bodies they should be thoroughly cleaned.

The principle methods of isolating uranium from diluted aqueous solutions are sorption, extraction, and ionic exchange [4, 5]. However, it should be noted that the most effective method of isolating uranium from diluted aqueous solutions is ionic floatation featuring economy, simplicity, and high capacity, especially when processing depleted solutions. One of the setbacks of ionic floatation should be considered frequent fouling of the froth product with a solution being treated and with impurities contained in it. In this case ionic floatation is diametrically opposite to extraction whose main advantage [6] is the obtaining of final products and a drawback is low efficiency when processing diluted solutions due to an increase of losses of extragents. Photoextraction [7] is a combination of ionic floatation and liquid extraction and combines positive aspects of the both methods.

The objective of the present paper is the study of the regularities of the process of floatation–extraction recovery of U(VI) from nitrate, sulfate, carbonate solutions by means of thin emulsified solutions of trialkylamide (TAA) in white spirit.

EXPERIMENTAL

Nitrate, sulfate, and carbonate solutions of U(VI) prepared from chemically pure salt $UO_2(CH_3COO)_2 \cdot 2H_2O$ and containing 50 mg (2.1×10^{-4} mol) of metal in 1 dm^3 were used as objects of research. When choosing main objects of research of solutions enumerated above we proceeded from the fact we had to deal with similar solutions when purifying diluted process solutions and wastewaters of uranium-extraction industry [2, 4, 8] after sulfuric or sodium alkalization of uranium ore and also after extraction removal of uranium from clarified solutions. The concentration of anions in solutions varies within broad limits (depending on the composition of the uranium ore and the flowchart of its processing): from 0001 to 0.1 and on the average constitutes 0.02 mol/dm^3.

We used a 0.1% solution of TAA in white spirit. Trialkylamide (TAA) presents [9] a technical product, which is a blend of tertiary, secondary, and primary aliphatic amines containing in the alkyl chain of 7–9 atoms of carbon. The mass fraction of the said aliphatic amines constitutes respectively 70, 25, and 5%. White spirit is a fraction of gasoline of direct distillation [10–12], with organic acids, whose bulk consists of naphthenic acids and phenols, which are always present in white spirit. During long storage their content increases as a result of white spirit oxidation.

Acidity of white spirit used in the experiment constituted 0.8 mg CFU/100 cm^3. It was determined in [11] by titration of a hot (after boiling) blend of equal amounts of white spirit and 96% ethanol by a 0.008 M solution of potassium hydroxide.

The collector was introduced to the U(VI) solutions in quantities corresponding to molar ratios of TAA : U from 0.1 : 1 to 2.5 : 1. After introducing the collector to the U(VI) solutions the systems were subjected to ultrasound dispersion (emulsifying) by means of an UZDN-2T ultrasound disperser with a strictor frequency 44 kHz for 15 min. After emulsifying we established a necessary pH value by means of a 0.1 M solution of nitric, sulfuric, and hydrochloric acids respectively for nitric, sulfate, and carbonate solutions of uranium and also by means of 0.1 M solutions of potassium hydroxide. The pH values were measured by a universal EV-74 ion meter with a glass electrode.

Floatation treatment of emulsions that formed was carried out by way of passing through the solution of air dispersed by a porous material on a plant for floatation, which is a glass column for floatation 110 mm high and 30 mm in diameter. A glass porous plate (Schotta filter no. 4) was used simultaneously as an air disperser and as a bottom of the column. The volume of emulsion poured into the column equaled 50 cm^3; floatation time—15 min. The air to the column was fed from below through a porous plate at the rate 25 cm^3/min. The experiments were conducted at room temperature. The emulsions in the course of floatation were periodically analyzed for the content in them of U(VI). The analysis was conducted photocolorimetrically as a reagent of arsenaso III [13]. The efficiency of the floatation process was judged by the degree of uranium extraction, %:

$$\alpha = \frac{C_S^0 - C_S}{C_S^0} \cdot 100, \quad (1)$$

where $C_s{}^0$ and C_s is U(VI) concentration in emulsion (solution) respectively before and after floatation.

The radius of the emulsion drops was measured by means of a microscope fitted with the Goryaev grid [14]. The electrokinetic ξ-potential of the emulsion drops was determined microelectrophoretically [14].

It is found that during emulsifying of the collector in nitrate and carbonate solutions of U(VI) polydispersion emulsions were formed in which the drops with the radius 1.0–1.3 μm were formed (Fig. 1). In contrast, in the case of sulfate solutions of U(VI) there formed monodispersion emulsions with the drop radius close to 5 μm. The resultant emulsions were concentrated [14] (the concentration of the dispersion phase was 0.19–7.6 vol %), they distinguished by high stability not splitting for 1–10 days. Stability of the emulsions was determined by anion nature: the most stable were emulsions obtained in nitrate solutions of U(VI), while the least stable—in carbonate solutions.

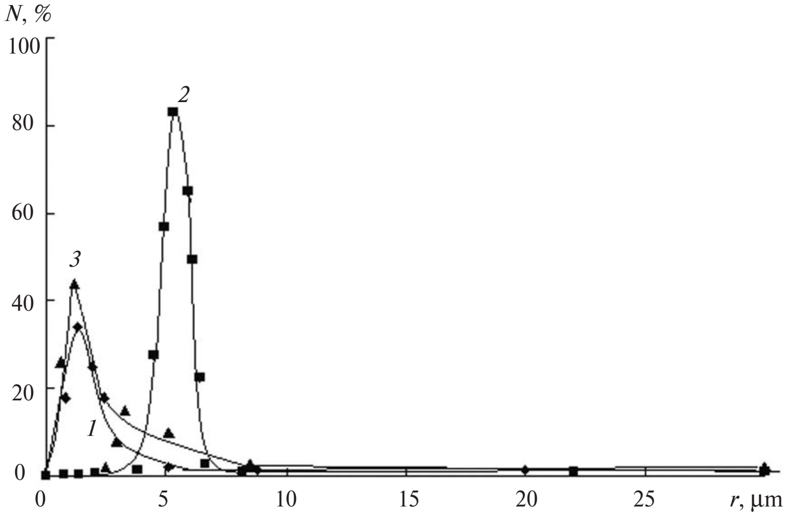


Fig. 1. Distribution of particles (*N*) by the sizes of the dispersion phase of the emulsions containing uranium in nitrate (*1*), sulfate (*2*), and carbonate (*3*) solutions of U(VI).

RESULTS AND DISCUSSIONS

The research conducted demonstrated that the finely emulsified solutions TAA in white spirit may be used as effective floatation extraction collectors of U(VI) from nitrate, sulfate, and carbonate solutions (Figs. 2–4).

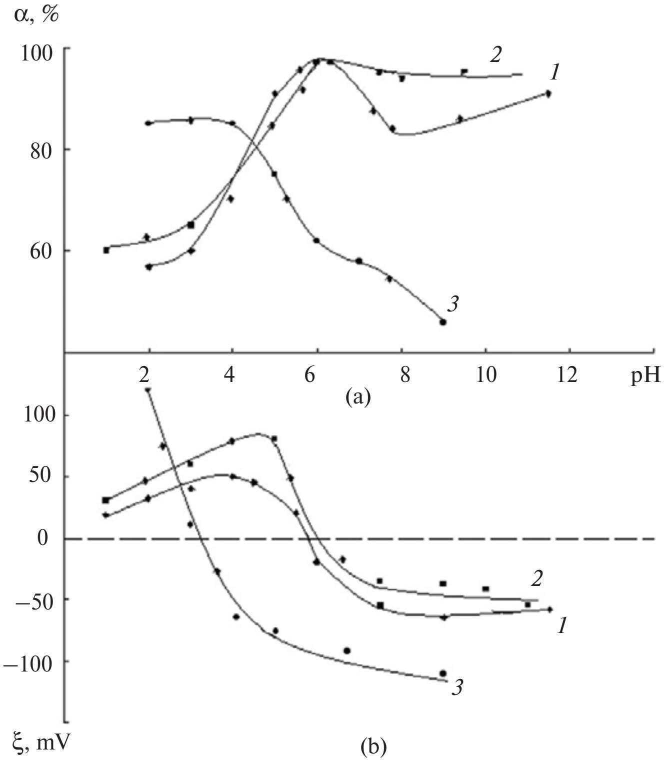


Fig. 2. Impact of the medium pH on the degree of floatation–extraction recovery of U(VI)) (a) from its nitrate (*1*), sulfate (*2*), and carbonate (*3*) solutions and also on the ξ-potential of the particles of the dispersion phase of the emulsions containing uranium (b). Molar ratio of the components TAA:U = 1 : 1.

The maximum degree of floatation recovery of uranium from nitrate solutions (95%) takes place within the interval of the pH values 5.5–6 from sulfate (95–100%)—within the interval 6–11, while from initial carbonate ones (75–78%)—within the interval 2–4. In all the cases the maximum degree of uranium recovery was observed at the pH values close to the isoelectric state of the emulsion drops containing uranium (see Fig. 2).

For establishing the mechanism of floatation extraction recovery of uranium from various media the authors of [15, 16] calculated the forms of finding uranium in nitrate, sulfate, and carbonate solutions within the pH interval 1–11 (Table 1) taking into account simultaneous course of the reactions of complexation and hydrolysis. In this case stepwise constants of hydrolysis (K_h) of the uranyl ion [17], constants of instability [18] of nitrate, sulfate, and carbonate complexes of uranyl and dissociation stepwise constants [18] of carbonic acid ($K_{H_2CO_3} = 3.3 \times 10^{-7}$; $K_{HCO_3^-} = 5.0 \times 10^{-11}$). The calculations were made for the initial uranium concentration 2.1×10^{-4} mol/dm^3 and the initial concentration of anions 0.02 mol/dm^3.

With the account of the form of finding U(VI) in nitrate solutions one may assume that in the case of isolating U(VI) from nitrate solutions at pH 2–6 there occurs, by and large, cation–exchange extraction of the UO_2^{2+} and $UO_2(OH)^+$ ions with naphthenic acid RCOOH (where R is a hydrophobic part of a series of cyclopentane and cyclohexane) incorporated into white spirit:

at pH 2–5

$$2RCOOH_{org} + UO^{2+}_{2\,wat} \Leftrightarrow UO_2(RCOO)_{2\,org} + 2H^+_{wat}; \quad (2)$$

at pH 3–7

$$RCOOH_{org} + UO_2(OH)^+_{wat} \Leftrightarrow UO_2(OH)RCOO_{org} + H^+_{wat}. \quad (3)$$

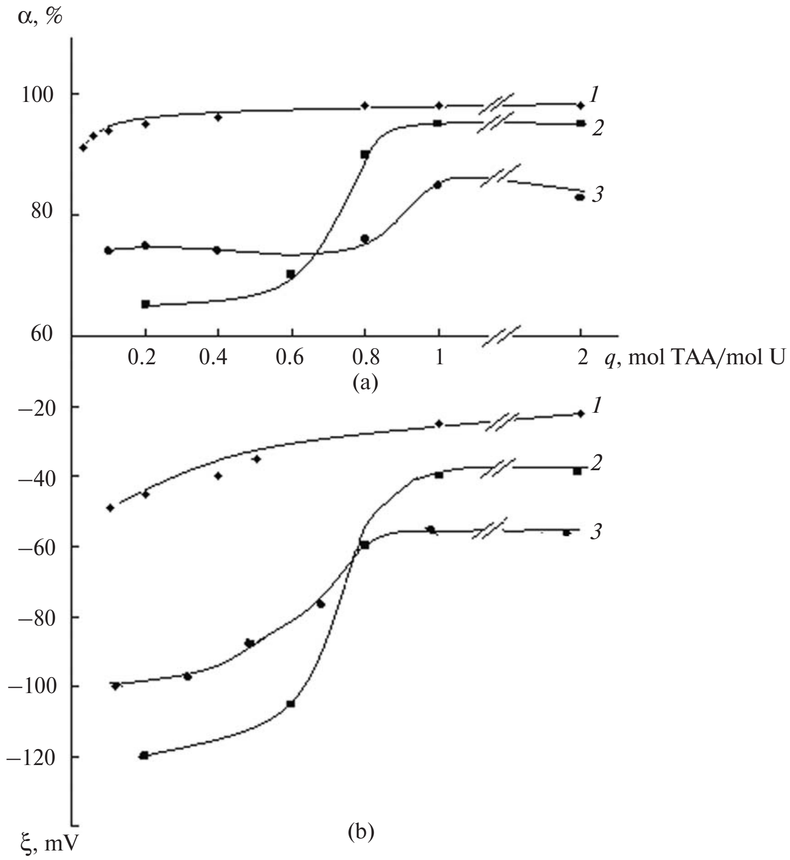


Fig. 3. Impact of the TAA amount (*q*) introduced to nitrate (*1*), sulfate (*2*), carbonate (*3*) uranium solutions on the degree of floatation–extraction recovery of U(VI) (a) and the ξ-potential of particles of the dispersion phase of the emulsions containing uranium (b). The pH value of the emulsions: 6 (*1*), 8 (*2*), and 4 (*3*).

With the pH interval 5–7 within which there exists a sufficient number of ionized groups $RCOO^-$, there is a possibility for the formation of hardly soluble products on the surface of emulsion drops (the liquid–liquid interface):

$$RCCOO^-_{bound} + UO_2(OH)^+_{wat} \Leftrightarrow UO_2(OH)RCOO_{bound} \quad (4)$$

Floatation recovery of uranyl hydroxide (pH > 5) is possible as a result of its interaction with carboxylic groups of naphthenic acids, while in strong alkaline media (pH 11)—also with amino groups of TAA due to the formation of donor–acceptor and hydrogen bonds between polar groups of SAS and $UO_2(OH)_2$ [19].

In the case of recovery of U(VI) from sulfate solutions, most likely, there takes place both anion–exchange extraction of sulfate complexes of uranyl (see Table 1) with the TAA sulfates [20] within the pH interval 1–5

$$2R_3N_{org} + H_2SO_{4\,wat} \Leftrightarrow (R_3NH)_2SO_{4\,org}; \quad (5)$$

$$(R_3NH)_2SO_{4org} + [UO_{4\,org}(SO_4)]^0_{wat} \Leftrightarrow (R_3NH)_2[UO_2(SO_4)_2]_{org}; \quad (6)$$

$$(R_3NH)_2SO_{4\,org} + [UO_{4org}(SO_4)_2]^{2-}_{wat} \Leftrightarrow (R_3NH)_2\,[UO_2(SO_4)_2]_{org} + SO^{2-}_{4\,wat}, \quad (7)$$

and cation–exchange extraction of the UO_2^{2+} and $UO_2(OH)^+$ ions (pH 1–6) with naphthenic acids incorporated into white spirit, by Eqs. (2) and (3). Within the interval of the pH values of sulfate solutions 6–11 uranium, just as in the case of nitrate solutions, is isolated in the form of uranyl hydroxyl.

Taking into consideration the forms of finding U(VI) in carbonate solutions (see Table 1) one may assume that from the solutions with pH 2–5 uranium is isolated in the form of UO_2^{2+} by the mechanism described by Eq. (2); from the solutions with pH 4–5 in the form of UO_2^{2+} and $UO_2(OH)^+$—by mechanisms (2) and (3); from the solutions with pH 6–9—by the mechanism

$$[UO_{4org}(CO_3)_2]^{2-}_{wat} + (R_3NH)_2CO_3 \Leftrightarrow (R_3NH)_2[UO_2(CO_3)_2]_{org} + CO^{2-}_{3\,wat}, \tag{8}$$

where as from the solutions with pH ≥ 8—by the mechanism

$$2(R_3NH)_2CO_{3\,org} + [UO_2(CO_3)_3]^{4-}_{wat} \Leftrightarrow (R_3NH)_4[UO_2(CO_3)_3]_{org} + 2CO^{2-}_{3\,wat}. \tag{9}$$

Table 1. Content (%) of different forms of finding U(VI) in nitrate, sulfate, and carbonate solutions depending on the pH

Form of finding U(VI)	Hydrolysis constants; constants of instability of complexes	pH										
		1	2	3	4	5	6	7	8	9	10	11
		Concentration of U(VI), %										
		Nitrate solutions										
UO_2^{2+}	–	99.0	98.9	98.0	90.0	48.2	5.8	0.1	–	–	–	–
$[UO_2OH]^+$	$K_h = 10^{-5}$	–	0.1	1.0	9.0	48.2	57.8	13.8	1.6	0.2	–	–
$UO_2(OH)_2$	$K_{h2} = 6.61\times10^{-7}$	–	–	–	0.1	3.1	36.4	86.1	98.4	99.8	100	100
$[UO_2NO_3]^+$	$K_1 = 2$	1.0	1.0	1.0	0.9	0.5	–	–	–	–	–	–
		Sulfate solutions										
UO_2^{2+}	–	46.3	46.3	–	44.2	30.9	5.5	0.1	–	–	–	–
$[UO_2OH]^+$	$K_h = 10^{-5}$	–	–	–	4.5	31.1	54.0	13.7	1.6	0.2	–	–
$UO_2(OH)_2$	$K_{h2} = 6.61\times10^{-7}$	–	–	–	–	2.0	34.1	86.1	98.4	99.8	100	100
$[UO_2(SO_4)]$	$K_1 = 5.00\times10^{-2}$	46.3	46.3	–	44.2	30.9	5.5	0.1	–	–	–	–
$[UO_2(SO_4)_2]^{2-}$	$K_{1,2} = 2.86\times10^{-3}$	6.5	6.5	–	6.2	4.5	0.8	–	–	–	–	–
$[UO_2(SO_4)_2]^{4-}$	$K_{1,2,3} = 4.00\times10^{-4}$	0.9	0.9	–	0.9	0.6	0.1	–	–	–	–	–
		Carbonate solutions										
CO_3^{2-}		–	–	–	–	3.38×10^{-9}	2.59×10^{-7}	7.76×10^{-6}	9.68×10^{-5}	9.50×10^{-4}	6.67×10^{-3}	1.67×10^{-2}
UO_2^{2+}		100	100	99.0	90.9	47.5	0.4	–	–	–	–	–
$[UO_2OH]^+$	$K_h = 10^{-5}$			1.0	9.1	47.5	3.8	–	–	–	–	–
$UO_2(OH)_2$	$K_{h2} = 6.61\times10^{-7}$			–	–	3.0	2.4	0.3	–	–	–	–
$[UO_2(CO_3)_2]^{2-}$	$K_{1,2} = 2.70\times10^{-16}$			–	–	2.0	93.4	99.6	87.0	60.0	0.2	0.1
$[UO_2(CO_3)_3]^{4-}$	$K_{1,2,3} = 1.72\times10^{-23}$			–	–	–	–	0.1	13.0	40.0	99.8	99.9

The comparison of the proposed mechanism of the process and the degree of floatation agrees well with the data of [21] about the fact that extraction capacity of salts of tertiary amines with respect to highly charged ions is much lower than with respect to single charged ions and does not contradict the results of the experiments on the impact of the pH value of the medium on the ξ-potential of the drops of the emulsions containing uranium.

The efficiency of floatation extraction recovery of uranium from nitrate solutions slightly depends on the TAA consumption (Fig. 3) with emulsified white spirit possessing collection capacity even in the absence of TAA with respect to uranium under the given conditions of the experiments (the degree of floatation recovery of U(VI) recovery by means of emulsified white spirit—76%). When conducting a similar experiment with pure hydrocarbon (pentadecane) uranium recovery is absent. This confirms collection capacity of SAS impurities present in white spirit (in particular, naphthenic acids) with respect to particles containing uranium. Therefore, white spirit may be used not only as TAA solvent, but also as a polar collector during floatation–extraction recovery of U(VI). The use of TAA emulsions in white spirit containing the ever ascending amounts of TAA (from 0.025 to 2 moles per 1 mol of uranium) results in an increase of the degree of floatation–extraction recovery of uranium from 76 to 92–97% (see Fig. 3). During recovery of U(VI) from sulfate and carbonate solutions for achieving the maximum degree of recovery it is necessary to introduce 1 of mole of TAA per 1 mole of uranium.

For explaining the obtained results we calculated the surface of the drops of emulsions, which may be occupied by TAA amino groups when introducing 0.5; 1 and 2 moles of TAA per 1 mole of uranium under condition that the whole introduced TAA is concentrated on the surface of a drop, i.e., at the liquid–liquid interface (Table 2). From the said Table it can be seen that only 7–40% of the surface of the drops of the emulsions may be covered by the TAA amino groups. In addition, it is necessary to take into account the role of white spirit in the given process. On the one hand it performs the function of a solvent (thinner) of TAA, and, on the other, of an apolar floatation collector [22] containing in the form of impurities [10–12] naphthenic acids capable of responding with the component of a solution. The use of the apolar collector makes it possible to reduce the adhesion time of an emulsion drop to an air bubble, to avoid redundant frothing, and to improve the structure of the floatation froth; apolar collectors produce a flocculation effect on thin disperse particles.

Table 2. Share of the surface of the drops of the emulsions covered with TAA amino groups

Anion	Radius of emulsion drop, m	Area of surface of drops of emulsion, m^2	Number of emulsion drops in 100 cm^3 of emulsion	Amount of TAA				Share of TAA adsorbed on the surface emulsion drops (of possible), %		
				adsorbed on the surface of emulsion drops, mol	introduced to 100 cm^3 of uranium solution, mol TAA/mol U(VI)					
					0.5	1.0	2.0	0.5	1,0	2,0
NO_3^-	1.3×10^{-6}	5.3×10^{-12}	4.0×10^{11}	2.3×10^{-4}	1×10^{-5}	2×10^{-5}	4×10^{-5}	4.6	9.2	18.4
SO_4^{2-}	5.0×10^{-6}	7.9×10^{-11}	1.2×10^{10}	1.0×10^{-4}	1×10^{-5}	2×10^{-5}	4×10^{-5}	10.0	20.0	40.0
CO_3^{2-}	1.0×10^{-6}	3.1×10^{-12}	8.8×10^{11}	3.0×10^{-4}	1×10^{-5}	2×10^{-5}	4×10^{-5}	3.3	6.7	13.4

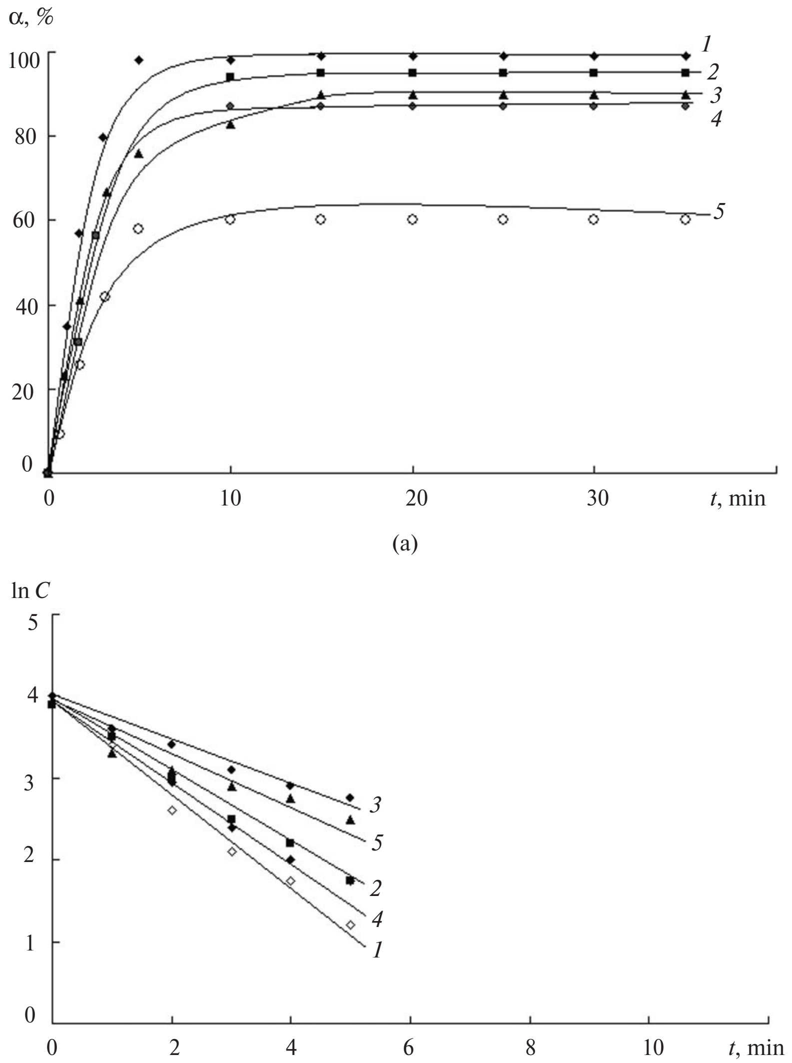
The TAA role is reduced to neutralization of the negative ξ-potential of the drops of white spirit emulsions, of an increase of stability of emulsions, and also of interaction of its amino groups with anionic complexes of uranyl. As the amount of introduced TAA increases the negative ξ-potential of the drops of the emulsions decreases (see Fig. 3), which is explained by a growth of the amount of positively charged amino groups adsorbed on the surface of the drops of the emulsions (see Table 2). This leads to a reduction of the electrostatic barrier of repulsion occurring between the bubbles of the air and the drops of the emulsions in the course of floatation.

The kinetics of the process of floatation recovery of U(VI) collected by means of finely emulsified solutions of TAA in white spirit depends on the nature of anions (NO_3^-, SO_4^{2-}, and CO_3^{2-}) present in solutions and on the pH value of the medium, but depends little on the TAA amount introduced into the solution containing uranium (Fig. 4, Table 3). The time necessary for achieving the maximum (under given conditions of the experiments) degree of floatation–extraction recovery of uranium constitutes 10–20 min (see Fig. 4).

The kinetics of the process in all the cases are described by the equation similar to the reaction equation of the first order:

$$\ln C = \ln C_0 - \mathrm{K} \cdot t, \tag{10}$$

where C and C_0 is the concentration (mol/dm^3) of uranium in an emulsion (solution) respectively at the initial instant of time and at the time instant t.



(a)

(b)

Fig. 4. Impact of floatation time of nitrate (*1–3*), sulfate (*4*), and carbonate (*5*) solutions of U(VI) on the degree of floatation–extraction recovery of U(VI) (a) and the value lnC (b). Molar ratio of the components TAA : U is 1 : 1 (*1, 4, 5*); 0.1 : 1 (*2*). The pH value of the emulsions: 6 (*1, 2, 4, 5*), 4 (*3*).

The values of the constants of the process rate found by the way of graphic solutions (see Fig. 4) of Eq. (10) are given in Table 3 from which it follows that the maximum rate takes place in the case of floatation–extraction recovery of uranium from nitrate solutions with pH 6 in the case of introducing 1 mole of TAA per 1 mole of uranium.

Table 3. The values of the constants of the rate (K) of the process of floatation–extraction recovery of uranium collected by means of thin emulsified TAA solutions in white spirit

Characteristic of aqueous phase		Molar ratio TAA : U	K, min^{-1}
anion	pH		
NO_3^-	6	1 : 1	0.56
SO_4^{2-}	6	1 : 1	0.27
CO_3^{2-}	6	1 : 1	0.18
NO_3^-	4	1 : 1	0.21

Table 3. (Contd.)

NO_3^-	6	1 : 1	0.56
NO_3^-	6	0.1 : 1	0.48
NO_3^-	6	1 : 1	0.56

During floatation–extraction recovery of uranium by means of fine emulsified TAA solutions in white spirit the limiting stages of the process are the diffusion stages of the drops of the emulsions to the surface of the air bubbles and adsorption of the drops at the liquid–gas interface therefore the process under study is described by the kinetic equation of first order (10).

CONCLUSIONS

Thus, in the experimental way we demonstrated the possibility and efficiency in principle of using TAA fine emulsified solutions in white spirit as floatation–extraction collectors of U(VI) from its nitrate, sulfate, and carbonate solutions. The optimal conditions of the studied process (Table 4) have been determined.

Table 4. Optimal conditions for floatation–extraction recovery of uranium from nitrate, sulfate, and carbonate solutions

Indicator	Uranium solutions		
	nitrate	sulfate	carbonate
pH of emulsions	5.5–6	6–10	2–4
Dose of TAA, mole of TAA per 1 mole of uranium	0.2–2	1–2	1–2
Floatation–extraction, time, min	5–10	10	15

The use of TAA fine emulsified solutions in white spirit as flotation collectors of uranium compared with individual cationic SAS—chlorides of primary and secondary aliphatic amines, alkyl pyridinium salts [23] makes it possible to bring down the costs of the collector, to expand the area of the pH optimal values, and to improve the process characteristics. Essential is also the fact that TAA being used and white spirit are accessible reagents whose price is much lower that that of individual SAS.

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