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АЛКИЛБЕНЗОЛСУЛЬФОКИСЛОТА КАК РЕАГЕНТ ДЛЯ ФЛОТАЦИОННОГО ИЗВЛЕЧЕНИЯ ИОНОВ ЛАНТАНА(III), САМАРИЯ(III) И ТЕРБИЯ(III)

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Анионогенное поверхностно-активное вещество алкилбензолсульфокислота предложено в качестве реагента для ионной флотации редкоземельных элементов. Предварительно изучена устойчивость пен при различных концентрациях алкилбензолсульфокислоты. При встряхивании 5 мл растворов с содержанием реагента 0,1 или 0,01 моль/л в течение 30 с образуется 15 мл пены. Объем пены сохраняется до 10 мин, после чего она разрушается в течение 1,5 ч. При концентрации алкилбензолсульфокислоты 0,001 моль/л, близкой к критической концентрации мицеллообразования, объем образующейся пены в два раза меньше и ее разрушение происходит медленнее. Дальнейшее уменьшение содержания алкилбензолсульфокислоты приводит к образованию скудных и неустойчивых пен. Рассмотрена возможность флотационного извлечения трехзарядных ионов редкоземельных элементов (лантана, самария и тербия) алкилбензолсульфокислотой из их водных растворов с концентрацией 0,001 моль/л. Исследовано влияние мольного соотношения ионов лантанидов и алкилбензолсульфокислоты, а также времени флотирования на качественные и количественные характеристики процесса флотации. Определение степени извлечения редкоземельных элементов проводили по их остаточному содержанию в растворе после флотации. Алкилбензолсульфокислота в изучаемых процессах преимущественно выступает в роли осадителя. Оставшиеся в растворе ионы поверхностно-активного вещества способствуют пенообразованию. При флотации в течение 5 мин максимальная степень извлечения лантана (III) составила 97,21% при соотношении металл : реагент 1:3,5. Для самария и тербия оптимальным является трехкратный избыток алкилбензолсульфокислоты. Извлечение тербия (III) при этом достигает 91,72% в течение 5 мин, самария (III) – 86,84% в течение 10 мин.

Ключевые слова: алкилбензолсульфокислота, лантан, самарий, тербий, ионная флотация

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ALKYLBENZENESULFONIC ACID AS A REAGENT FOR FLOTATION EXTRACTION OF LANTHANUM (III), SAMARIUM (III) AND TERBIUM (III) IONS

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The anionogenic surfactant alkylbenzenesulfonic acid is proposed as a collector for ionic flotation of rare-earth elements. Stability of foams at various concentrations of alkylbenzenesulfonic acid has been preliminarily studied. Shaking 5 mL of solutions with 0.1 or 0.01 mol/L reagent containing for 30 s produces 15 mL of foam. This volume persists for up to 10 min, after which it degrades within 1.5 h. At the alkylbenzenesulfonic acid concentration of 0.001 mol/L, close to the critical micelle concentration, the volume of the formed foam is half as much and its destruction is slower. Further reduction of the alkylbenzenesulfonic acid content leads to the formation of scanty and unstable foams. The possibility of flotation recovery of three-charged ions of rare earth elements (lanthanum, samarium and terbium) with alkylbenzenesulfonic acid from their aqueous solutions with 0.001 mol/L concentration was considered. The influence of the molar ratio of lanthanide ions and alkylbenzenesulfonic acid, as well as the flotation time on the qualitative and quantitative characteristics of the flotation process has been studied. Determination of rare earth elements recovery rate was carried out by their residual content in the solution after flotation. Alkylbenzenesulfonic acid predominantly acts as a precipitant in the studied processes. The surfactant ions remaining in the solution contribute to foaming. During flotation for 5 minutes the maximum recovery of lanthanum (III) was 97.21% at the metal vs. reagent ratio 1:3.5. For samarium (III) and terbium (III) a threefold excess of alkylbenzenesulfonic acid is optimal. The extraction of terbium (III) in this case reaches 91.72% for 5 min, samarium (III) – 86.84% for 10 min.

Key words: alkylbenzenesulfonic acid, lanthanum, samarium, terbium, ionic flotation

INTRODUCTION

Materials containing rare-earth elements (REE) have found use in many industries: in the chemical and metallurgical industries, electronics, medicine, engineering, nuclear power, laser materials, and high-temperature superconductors. They are used for the development of modern and advanced systems of military equipment, rocket, space and shipbuilding technologies [1-3]. The introduction of REE into the composition of manufactured products completely changes their properties.

Since rare-earth elements are mainly found as impurities in various minerals, it is advisable not to search for and develop new deposits, but to extract them from already processed ores [4]. In this case, the problem of searching for effective methods of REE extraction from recycled raw materials becomes relevant. Depending on the production conditions precipitation methods [5], selective oxidation or reduction [1], ion-exchange sorption [6], and liquid extraction [7-9] are used for the production of concentrates containing REE, their separation and impurity treatment. Also of great interest is the method of ionic flotation, the main advantages of which are high efficiency at relatively low concentrations of recoverable metal, high rate of the process and its environmental safety. Ion flotation makes it possible to extract rare metals from relatively poor solutions (10-100 mg/L of REE).

Since lanthanides in solutions are mainly in cationic form, the anionic surfactants capable of forming low-soluble complexes with them and stabilizing the heterogeneous system to remove the resulting product from the solution are chosen as a reagent for their flotation. The team of researchers at St. Petersburg Mining University has carried out extensive research on the flotation processes of rare-earth element ions with sodium dodecylsulfate [10, 11]. The forms of

REE existence in aqueous solutions depending on pH were considered [12, 13]; it was established that flotation extraction of lanthanides (Ln) begins at pH 4.5 and becomes quantitative at pH ≥5; thermodynamic parameters of their ionic flotation from nitric acid and chloride solutions were studied [14-18], conditions of REE separation in their joint presence were found [19-21].

In order to expand the list of reagents used in ionic flotation of rare earth elements, it was proposed to use an anionogenic surfactant similar in composition to sodium dodecylsulfate – alkylbenzenesulfonic acid (ABSA). This surfactant has been proposed in separation and concentration methods as a component of delaminating systems for extraction [22, 23] and as a foaming additive in apatite enrichment flotation [24]. It was established that ABSA forms with lanthanide ions white amorphous precipitates of the composition $Ln(C_nH_{2n+1}C_6H_4SO_3)_3$ (confirmed by chemical analysis, thermogravimetry and elemental analysis methods [25]). The resulting compounds are slightly soluble in water, soluble in sulfuric acid and ethanol. The equilibrium pH value of the solutions after precipitation, depending on the concentration of the added ABSA solution, is in the range from 1.5 to 5.0.

In order to use flotation effectively, the conditions under which it is carried out must be selected cautiously. An important criterion for flotation recovery is foam stability.

Foam properties depend on bubble size, bubbling rate, collector and solution pH. It should not collapse too easily so that there is no loss of extractable compound. Stable foams are also undesirable because they complicate the sublate analysis and contribute to the entrainment of large amounts of stock solution, which reduces the efficiency of the concentration, separation and extraction processes. With that, the concentration of the used surfactant is important, which in ionic flotation is tried to maintain less than the critical micelle concentration (CMC) [26]. First of all, it is economically feasible, because it reduces the cost of reagents. Secondly, the presence of micelles can lead to competition of two processes: solubilization of products of interaction between surfactants and inorganic ions and their adsorption on the "liquid-gas" surface. The amount and stability of the foam depends on the solution media and the collector. The most desirable case is when the foam is stable and collapses at approximately the same rate as it forms.

In this case, one of the tasks of this study was to select the ABSA concentration, at which the resulting foam will be most convenient for the ionic flotation of REE.

MATERIALS AND METHODS

Reagents. Anionogenic surfactant alkylbenzene sulfonic acid grade A (LLC "KINEF") TU 2481-026-05766480-2006, the general formula $C_nH_{2n+1}C_6H_4SO_3H$, where n = 10-14, average molecular weight 320.9 g/mol, content of main substance 96.8%. 0.1 mol/L solution was prepared by dissolving a sample of ABSA in distilled water. The exact concentration of surfactant was established potentiometrically and by titration with NaOH solution in the presence of bromocresol green [27]. Solutions with concentrations of 0.01; 0.001; 0.0001 mol/L were obtained by appropriate dilution.

Nitrates of lanthanum $La(NO_3)_3$, samarium $Sm(NO_3)_3$ and terbium $Tb(NO_3)_3$, were analytical grade. Solutions with an REE ion concentration of 0.1 mol/L were prepared by dissolving an exact sample in distilled water. The exact concentration of lanthanide ion was established by complexometric titration with xylenol orange in the presence of urotropine buffer solution with pH 5.1 [28].

Instruments. The shaking method was used to study foam stability over time depending on ABSA concentration [29]. 5 ml of the studied solution with ABSA concentrations, mol/L: 0.1; 0.01; 0.001; 0.0001, were injected into graduated test tubes for 25 ml with lapped stoppers. The solution was shaken for 30 s, the foam volume immediately after shaking and its change over time were noted.

The ionic flotation tests were carried out on a FML 0.3 mechanical flotation machine with a camera volume of 200 ml. Foam collection was automatic. 2 ml of 0.1 mol/L REE ion solution was put into the 200 ml capacity measuring flask, and water was added to the mark. The obtained solution was put into the flotation cell, the calculated amount of 0.1 mol/L ABSA solution was injected, the mixture was incubated for 3 min for precipitation maturation with constant stirring at a speed of 25 rps, an air supply of 40 ml/min was turned on and the foam containing sublate was collected. At the end of flotation, pH_{eq} and the residual content of REE ions were determined, respectively, on Multiteest-101 ionometer and spectrophotometrically with Arsenazo III on spectrophotometer UNICO 1201 in the chamber solution [30].

The recovery rate of REE ions (R,%) into the foam was calculated by the formula:

$$R, \% = 100 \cdot \left(1 - \frac{C_{Ln}}{C_{Ln}^{o}}\right)$$

where C_{Ln}^{o} – initial concentration of REE ions, mol/L; C_{Ln} – concentration of REE ions after flotation, mol/L.

RESULTS AND DISCUSSION

Foams stability of ABSA solutions. The dependence of foam volume on time at different ABSA concentrations is shown in Fig. 1.

The shape of the obtained curves for ABSA solutions with concentrations of 0.1 and 0.01 mol/L has a similar character: the first 10 min the foam volume practically does not change, then there is a gradual decrease, it takes 3 h for complete destruction. The ABSA content in these solutions exceeds the critical micelle concentration, equal to $2.3 \cdot 10^{-3}$ mol/L [31]. Reducing the concentration of ABSA to 0.001 mol/L, close to the CMC, the volume of formed foam decreases markedly, the rate of its destruction decreases and the curve of falling foam volume has a flatter character. For the 0.0001 mol/L ABSA solution, the maximum volume of foam is 0.6 ml and its complete destruction occurs within 9 min.

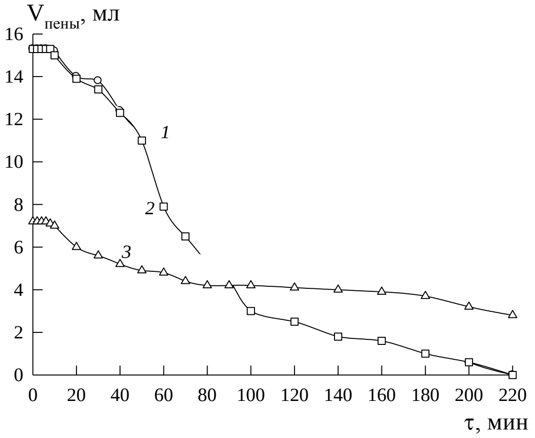

Fig. 1. Dependence of foam stability of ABSA solutions on a time. C_{ABSA}, mol/L: *1* – 0.1; *2* – 0.01; *3* – 0.001

Рис. 1. Зависимость устойчивости пены растворов АБСК от времени. $C_{АБСК}$, моль/л: *1* – 0,1; *2* – 0,01; *3* – 0,001

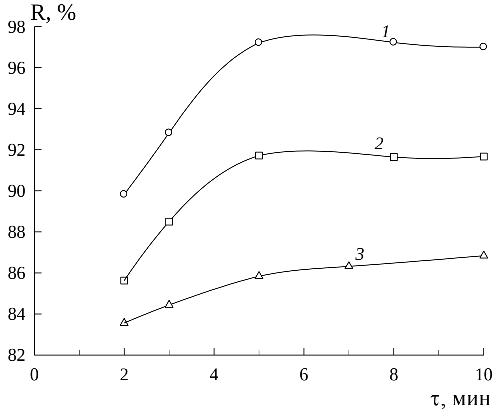

Fig. 2. Dependence of REE (III) ions extraction degree on a flotation time: 1 – La(III), 2 – Tb(III), 3 – Sm(III). La : ABSA = 1:3.5; (Sm,Tb) : ABSA = 1:3, $C_{Ln} = 1 \cdot 10^{-3}$ mol/L

Рис. 2. Зависимость степени извлечения ионов РЗЭ (III) от времени флотирования: 1 – La(III), 2 – Tb(III), 3 – Sm(III). La : АБСК = 1:3.5; (Sm,Tb) : АБСК = 1:3, $C_{Ln} = 1 \cdot 10^{-3}$ моль/л

Thus, for flotation with alkylbenzene sulfonic acid it is optimal to use concentrations close to 0.001 mol/L. The solutions give moderately stable volumetric foams, convenient for collecting.

Ionic flotation. It was previously found that it is enough 3 min for the complete precipitation maturation of the selected lanthanides ions with ABSA [25]. Therefore, after injecting the REE ion and ABSA solutions into the flotation chamber, the mixture was kept under constant stirring for this time.

The recovery of lanthanum (III), samarium (III) and terbium (III) ions with ABSA depending on the amount of collector and flotation time was studied.

Table 1

Dependence of Sm(III), Tb(III) and La(III) ions recovery degree on the amount of ABSA
($C_{Ln} = 1 \cdot 10^{-3}$ mol/L, $\tau_{fl} = 5$ min)

Таблица 1. **Зависимость степени извлечения ионов Sm(III), Tb(III) и La(III) от количества АБСК**
($C_{Ln} = 1 \cdot 10^{-3}$ моль/л, $\tau_{фл} = 5$ мин)

REE:ABSA	Sm		Tb		La	
	$pH_{равн}$	R, %	$pH_{равн}$	R, %	$pH_{равн}$	R, %
1:1	3.57	68.25	3.38	53.14	3.14	47.54
1:1.5	3.35	74.56	3.15	62.85	3.03	55.19
1:2	3.03	77.06	3.07	71.32	2.94	71.46
1:2.5	2.97	81.94	3.05	79.20	2.90	81.11
1:3	2.91	85.84	2.93	91.72	2.88	89.82
1:3.5	excess foaming*		excess foaming		2.72	97.21
1:4	–	–	–	–	excess foaming	

Note: *More than a third of the solution is carried away with the foam

Примечание: *с пеной уносится более трети раствора

As can be seen from Table 1, when the amount of ABSA in the flotation mixture increases to a threefold molar excess with respect to samarium and terbium, there is an increase in the degree of metal ions extraction. The optimum molar ratio for lanthanum, was 1:3.5. It should be noted that the main part of alkylbenzenesulfonate ions is involved in the precipitation of REE ions, and the remaining part is responsible for the foaming process. Lanthanum precipitate is less soluble than terbium and samarium precipitates (solubility product, respectively, $2.08 \cdot 10^{-11}$, $6.90 \cdot 10^{-10}$ and $2.87 \cdot 10^{-10}$ [25]). Therefore, the precipitation of Tb(III) and Sm(III) leaves more anions in solution, sufficient to form stable foams. In the case of La(III) ions, a small excess of ABSA has a positive effect on the extraction process. Further increase in surfactant concentration leads to abundant foaming and formation of emulsions, which has a negative effect on the flotation quality.

Probably, the process of solubilization of REE precipitate with ABSA begins to exceed the adsorption process at a ratio of more than 1:3.5. The formed particles of lanthanide alkylbenzenesulfonates dissolve in the hydrophobic part of ABSA micelles and remain in solution. To study the effect of flotation time on the extraction of ions, the La : ABSA ratio was maintained equal to 1:3.5, (Sm or Tb) : ABSA – 1:3.

According to Fig. 2, 80% of the ions of each metal flotate during the first 2-3 min. It takes 5 min to achieve maximum extraction of lanthanum and terbium (97.21 and 91.72%, respectively), after which the foam almost completely disappears. Flotation of samarium grows up to 10 min, but it was not possible to extract it more than 87%.

Table 2

Optimal conditions for flotation of REE ions with ABSA (C_{Ln} = 0.001 mol/L)

***Таблица 2.* Оптимальные условия проведения флотации ионов РЗЭ с АБСК (C_{Ln} = 0,001 моль/л)**

РЗЭ	Ln: АБСК	$\tau_{фл}$, мин	$pH_{равн}$	R_{max}, %
La(III)	1:3.5	5	2.72	97.21
Sm(III)	1:3	10	3.04	86.84
Tb(III)	1:3	5	2.93	91.72

Increasing the airflow rate from 40 to 60 mL/min has no effect on the extraction quality. According to the results of the studies, the optimal conditions for ionic flotation of the considered rare-earth element ions with alkylbenzenesulfonic acid were established (Table 2).

CONCLUSIONS

The possibility of using the anionic surfactant alkylbenzenesulfonic acid as a reagent for flotation of rare-earth element ions was shown on the example of lanthanum, samarium and terbium. The conditions for the most effective extraction of lanthanum and terbium from their 0.001 mol/L aqueous solutions have been established.

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