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RESEARCH ARTICLE

Solvent extraction of europium(III) from technogenic solutions with the use of surfactants

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Objectives. *The extraction and separation of rare-earth metals is a complicated process that requires a multidisciplinary and detailed investigation. Liquid-liquid extraction with the use of surfactant, along with the thermodynamic analysis of the parameters is considered a promising approach. The extraction and separation of rare-earth metals from low-concentration solutions represents an attractive research opportunity. The extraction of europium(III) from nitric acid solutions in the form of dodecyl sulfates has been experimentally studied. This work focuses on the study of fundamental and alternative sources of rare-earth metals and their extraction and separation.*

Methods. *The extraction was performed on a top drive ES-8300 D equipment for 30 min at about 700 rpm. Infrared spectroscopy (Nicolet 6700 spectrometer) was used to determine the type of salts extracted into the organic phase. Extraction was studied in solutions with single cations and with a combination of the target element and interfering cations. For the latter, the concentrations of extracted elements in the aqueous phase were determined by optical emission spectroscopy with inductively coupled plasma on an ICPE-9000 (Shimadzu) spectrometer. The spectrometer was calibrated using standard samples for ICP CertiPUR (Merck).*

Results. *The dependence of the distribution and separation coefficients of rare-earth metals during extraction on the pH value of the aqueous phase at equilibrium was investigated. Moreover, the form in which the elements are extracted was analyzed based on thermodynamic parameters. The minimum concentration of the target component in the aqueous phase was observed at pH 4.0. In general, the dependence of the distribution coefficient on the pH value of the medium is poorly expressed over the entire range of the pH range of the water phase. Based on the spectra of spent and pure isooctyl alcohol, it was concluded that europium dodecyl sulfates are extracted into the organic phase as* $Eu(C_{12}H_{25}OSO_3)_3$ *solvates.*

Conclusions. *The extraction of europium(III) from nitric acid solutions in the form of dodecyl sulfates was demonstrated. The advantages of the proposed method are the possibility of selective extraction of the target component from dilute solutions and the use of an easily available surfactant (sodium dodecyl sulfate). The efficiency of extraction of europium dodecyl sulfates was maximal in the pH range from 2.0 to 7.5, which reflects a weak dependence on the acidity of the aqueous phase. In addition, in the highly alkaline pH region, the extraction efficiency is reduced.*

Keywords: *extraction, rare-earth elements, extraction efficiency, distribution and separation coefficients, sodium dodecyl sulfate.*

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ОРИГИНАЛЬНАЯ СТАТЬЯ

Жидкостная экстракция европия(III) из техногенных растворов с использованием поверхностно-активного вещества

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Цели. *Литературные данные свидетельствуют о сложности извлечения и разделения редкоземельных металлов экстракционными методами и доказывают необходимость многостороннего и детального изучения данных процессов. Перспективным является осуществление экстракции с применением поверхностно-активного вещества и термодинамическим обоснованием полученных технологических величин. Инновационной идеей научного исследования является применение экстракционного метода для извлечения и разделения редкоземельных металлов из низкоконцентрированных растворов. Экспериментально изучен процесс извлечения европия(III) из азотнокислых растворов в форме додецилсульфатов экстракцией. Исследования ориентированы на изучение основных и альтернативных источников редкоземельных металлов, способов их извлечения и разделения.*
Методы. *Процесс проводили на верхнеприводном экстракторе ES-8300 D в течение 30 мин со скоростью около 700 об/мин. Для определения формы экстрагируемых солей в органической фазе применяли метод инфракрасной спектроскопии (спектрометр Nicolet 6700). Экстракцию изучали в растворах с единичными катионами и с комбинацией целевого компонента и интерферирующих катионов. Для последних растворов концентрации экстрагируемых элементов в совместном присутствии в водной фазе определяли методом оптической эмиссионной спектроскопии с индуктивно-связанной плазмой на спектрометре ICPE-9000 (Shimadzu). Калибровка спектрометра проводилась по стандартным образцам для ICP CertiPUR (Merck).*
Результаты. *Получены зависимости коэффициентов распределения и разделения редкоземельных металлов в процессе экстракционных процессов от величины pH равновесной водной фазы с установлением и термодинамическим обоснованием формы извлекаемых соединений. Установлено, что в процессе экстракции минимальная концентрация целевого компонента в водной фазе наблюдается при pH = 4.0. В целом на всем интервале исследуемого диапазона pH водной фазы зависимость коэффициента распределения от величины кислотности среды выражена слабо. По результатам анализа спектров отработанного и чистого изооктилового спирта сделано заключение, что додецилсульфаты европия извлекаются в органическую фазу в виде сольватов* $Eu(C_{12}H_{25}OSO_3)_3$.
Выводы. *Экспериментально показана возможность извлечения европия(III) из азотнокислых растворов в форме додецилсульфатов экстракцией. Преимуществами предлагаемого метода являются возможность избирательного извлечения целевого компонента из разбавленных техногенных растворов и использование поверхностно-активного вещества (додецилсульфата натрия). Эффективность извлечения додецилсульфатов европия при экстракции максимальна в исследуемом диапазоне pH от 2.0 до 7.5, что отражает слабую зависимость от кислотности водной фазы. Кроме того, в сильнощелочной области pH эффективность извлечения экстракцией понижается.*

Ключевые слова: *экстракция, редкоземельные элементы, эффективность извлечения, коэффициенты распределения и разделения, додецилсульфат натрия.*

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INTRODUCTION

The use of metal extraction processes has increased in various sectors of the national economy [1, 2]. This is due to the selectivity and simplicity of such processes, their speed, and high efficiency at low initial concentrations of metal cations.

The extraction of rare-earth metals by carboxylic acids has been widely studied because their use in sulfuric and hydrochloric acid industries for processing rare-metal raw materials is more effective than, for example, organophosphates [1, 2]. The degree of extraction and separation of rare-earth elements (REE) and aluminum with octyl-phenoxy isopropionic acid (OPIPA) presented in [3] reaches 86.43% with a purity of 99.69%. Cerium(III) extraction with a solution of oxine and 2-methyloxine in various organic solvents under conditions similar to industrial scenarios (a strongly acidic solution with a 0.3–4.4 mol/kg content of sulfuric acid) is reported in [4].

The chemistry of the extraction of lanthanide(III) nitrates with trialkylbenzylammonium naphthenate has also been investigated [5]. With a phase ratio of 2 : 1, rare-earth metals were extracted into the organic phase as $(R_4N)_2[Ln(NO_3)_3(RCOO)_2]$. In the lanthanum–lutetium series, extraction is reduced, while the degree of yttrium extraction is significantly lower than for lanthanides. Trialkylbenzylammonium naphthenate can be used to separate lanthanides with lower ordinal numbers from other lanthanides and also in systems for removing lanthanides from yttrium compounds.

The extraction of lanthanum, thorium, and yttrium with composite materials using porous carriers and liquid extractants is a promising method. When thorium(IV), lanthanum(III), and yttrium(III) are extracted together from water-salt solutions by a composite material based on super-crosslinked polystyrene with trialkylamine (TAA), lanthanum(III) and yttrium(III) nitrates are practically not absorbed; when the concentration of lanthanum(III) and yttrium(III) increases in the aqueous phase, the content of thorium(IV) nitrate in the composite material phase increases [6]. This system can be used for removing thorium(IV) impurities from concentrated solutions of rare-earth metal nitrates.

In addition, the extraction of rare-earth metals from nitric acid solutions with fluoride ions has been reported [7, 8]. Moreover, the extraction of zirconium(IV) cations from hydrochloric acid solutions in the presence of potassium fluoride with 1-octanol was investigated [9]. Extraction methods can be thus used to obtain metal cations from industrial waste dilute solutions (processing of molybdenum, copper, copper nickel ores, apatite-nepheline, eudialite ores, and red slurries).

It is clear that the extraction and separation of rare-earth metals is a complex process that must be investigated from different perspectives in a detailed manner. Using a surfactant for the extraction, along with the thermodynamic analysis of the process' parameters, seems like a promising approach.

The primary objectives of this work are:

- developing a method for the extraction and separation of rare-earth metals using surfactants and for the experimental assessment of distribution and separation coefficients and degree of extraction, followed by their implementation in the industrial processing of low-concentrated mineral raw materials;
- studying the dependence of the distribution and separation coefficients of rare-earth metals on the pH of the aqueous phase during extraction by determining the form in which the target elements are extracted, based on thermodynamic parameters.

During the extraction of rare-earth metals from aqueous solutions, sodium dodecyl sulfate (NaDS), known as a foamer and collector, is used as surfactant because of its many advantages in comparison with other surfactants. NaDS is non-toxic (hazard class IV) and cheap and can be regenerated from the obtained products. In addition, extraction processes require amounts of NaDS, depending on stoichiometry of the chemical reaction. NaDS can be used in a wide range of acidity of the liquid phase. Compared with similar surfactants, the degree of recovery of rare-earth metals by NaDS has the maximum values [10–12].

This work focuses on the study of fundamental alternative sources of rare-earth metals, and their extraction and separation. According to the literature [13–18], cost-effective sources of rare-metal raw materials are the minerals bastnesite $Ln(CO_3)F$ (70–75%), monazite $LnPO_4$ (55–60%), laparite $NaCaLn(TiO_3)_2(NbO_3)_2$ (30–35%), and xenotima (Y, Eu, Gd)PO_4 (55–60%). Alternative sources of rare-earth metals are waste products of industrial processes with a poor rare-earth metals content, converter dusts, slags, red slurries of aluminum production, and mineral raw materials with low concentrations of the desired elements.

Modern technologies to obtain rare-metal raw materials focus on minerals of different types of deposits as sources. Russia does not have reserves of pure rare-metal raw materials, such as bastnesite, monazite, and xenotima, which are used by the rare-earth industry abroad. In the Russian Federation, the most accessible and efficient sources for processing are the Lovozero loparite, eudialite, and Khibiny apatite-nepheline ores from the Kola Peninsula. These sources have low contents of rare-earth metals. The Russian rare-earth industry is currently working in the development of new deposits, which is far from sufficient to meet the growing demand for rare metals and their derivatives. For the progress of the Russian rare-metal industry, special attention should be paid to the associated extraction of individual rare-earth metals in the processing of multicomponent ores, and not only to the development of new deposits. Complex use of mineral raw materials is of the utmost importance in efficient resource usage. Since pure rare-earth metals are of great value, it is crucial to increase the efficiency of extraction and separation of rare-earth metals with similar physical and chemical properties, which will reduce the cost of these elements and their compounds, and expand the possibilities of their use [13–18].

This work proposes a method for the extraction and separation of rare-earth metals from low-concentration solutions. The advantages of the method over existing processes are the possibility of selective extraction of the target component from dilute solutions and the use of easily accessible surfactants.

MATERIALS AND METHODS

All reagents were chemically pure. A 0.01 M europium nitrate solution was prepared by dissolving $Eu(NO_3)_3 \cdot 5H_2O$ in 99.9% pure distilled water. All subsequent working solutions (0.001 M) were prepared from this stock. The exact concentration was confirmed by titration with Triton B and xylenol orange. For the total extraction of REE, solutions of $Er(NO_3)_3 \cdot 5H_2O$, $Ho(NO_3)_3 \cdot 5H_2O$, and $Sm(NO_3)_3 \cdot 6H_2O$ were prepared and titrated, similar to $Eu(NO_3)_3 \cdot 5H_2O$. Sodium dodecyl sulfate (NaDS) for biochemistry (*Acros Organics*, USA, 99%, $C_{12}H_{25}OSO_3Na$) was used as surfactant. For the experiments, a dry NaDS suspension was added to the working solution. Nitric acid and sodium hydroxide, added before setting the desired pH value, were freshly prepared before use. Isooctyl alcohol (*Aldrich*, USA, 96%, 2-ethyl-1-hexanol) was used as extractant.

Liquid-liquid extraction was performed to separate europium dodecyl sulfate using isooctyl alcohol as the extractant. NaDS was used as the transport agent of europium cations from the aqueous to the organic phase. The surfactant (0.003 M) was added according to the stoichiometry of the following reaction:

$$Eu^{3+} + 3C_{12}H_{25}OSO_3^{-} = Eu(C_{12}H_{25}OSO_3)_3$$

The pH value was adjusted to a specific value by adding a 1 N NaOH solution or diluted HNO_3. The pH value of the aqueous solutions was determined using a pH-150 MA pHmeter (*AQUA-LAB*, Russia).

The volumes of the aqueous and organic phases were 200 and 5 mL, respectively. The process was performed on a top drive ES-8300 D extractor (*ECROSKHIM*, Russia) for 30 min at about 700 rpm. The parameters of the extraction were experimentally set to achieve optimum results. The type of salts extracted into the organic phase was determined using infrared spectroscopy using a Nicolet 6700 (*Thermo Fisher Scientific*, USA) spectrometer.

Extraction of europium(III) cations in the presence of holmium(III), erbium(III), and samarium(III) cations was performed using the method described above. Working solutions containing Sm^{3+}, Eu^{3+}, Ho^{3+}, and Er^{3+} were prepared by mixing 50 mL of 0.001 M solutions of each element. The volume of the solution with a final total concentration of 0.001 M was 200 mL. The concentration of the extractable elements in the aqueous phase was determined by optical emission spectroscopy with inductively coupled plasma (ICP) on an ICPE-9000 spectrometer (*Shimadzu*, Japan), which was calibrated using CertiPUR ICP standard samples (*Merck*, Germany).

The concentration of europium cations extracted into the organic phase was determined by the difference in concentration in the initial and equilibrium aqueous phases, considering the volume ratio of the phases:

$$C_{org} = \frac{(C_0 - C_{aq}) \cdot V_{aq}}{V_{org}}, \qquad (1)$$

where C_0 is the initial europium concentration in the aqueous phase, mol/L; C_{org} and C_{aq} correspond to the concentration of europium cations in the organic and aqueous phases at equilibrium, mol/L; V_{aq} and V_{org} are the volume of the organic and aqueous phases, mL.

The distribution coefficient of Eu^{3+} in individual solutions (K_{distr}) and in the presence of Sm^{3+}, Ho^{3+}, and Er^{3+} ($K_{distr\Sigma}$) was calculated from the ratio of molar concentrations of the target component in the organic and aqueous phases according to [16].

The degree of extraction was calculated using the following equation:

$$\alpha = \frac{C_{org} \cdot V_{org}}{C_{aq} \cdot V_{aq} + C_{org} \cdot V_{org}} \cdot 100\% \qquad (2)$$

RESULTS AND DISCUSSION

Table 1 shows the results of extraction of europium dodecyl sulfates from nitrate solutions in the pH range 2.0–11.0. As seen from the data, the maximum distribution coefficient during extraction was obtained at pH = 4.0.

According to the results reported in references [11, 12], the Gibbs energy of formation of $Eu(OH)^{2+}$ monohydroxocomplexes from its elements, $\Delta_f G^0_{298}$, and from ions, $\Delta_{compl} G^0_{298}$, is 782.60 and 47.42 kJ/mol, respectively; that of $Eu(OH)_3$ hydroxide is 1199.11 and 149.23 kJ/mol, respectively. In addition, pH of complexation, pH_{compl}, is 5.80, and pH of hydrate formation, pH_{hydr}, is 6.52; i.e. these are the pH values where the formation $Eu(OH)^{2+}$ and $Eu(OH)_3$ hydroxides begins.

During extraction, the minimum concentration of the target component in the aqueous phase is observed at pH = 4.0. In general, the dependence of the distribution coefficient of rare-earth metals during extraction on the pH value of the medium is poorly expressed over the entire pH range. Based on the spectra of spent and pure isooctyl alcohol, europium dodecyl sulfates are extracted into the organic phase as $Eu(C_{12}H_{25}OSO_3)_3$ solvates (Figs. 1 and 2). The interaction between alcohol molecules and europium cations is confirmed by a shift in the frequency of $\nu_{O–H}$ valence vibrations. However, in nitrate solutions that do not contain NaDS, the extraction of europium cations does not occur due to the lack of shielding of the target component by non-polar hydrocarbon radicals.

The efficiency of extraction of europium dodecyl sulfates is maximal from pH 2.0 to 7.5, which reflects a weak dependence on the acidity of the aqueous phase. In the highly alkaline pH region, the extraction efficiency is reduced.

Table 2 shows the results of extraction of europium dodecyl sulfates with isooctyl alcohol as an extractant in the presence of erbium(III), samarium(III), and holmium(III).

When extracting REE from individual solutions, the maximum distribution coefficients are found at pH 4.0–4.5, where REE is extracted into the organic

Table 1. Europium(III) cation extraction

pH	$[Eu^{3+}]_{aq}$ ×10^4 mol/kg	$[Eu^{3+}]_{org}$ ×10^2 mol/kg	K_{distr}
3.0	1.93	3.88	200.5
3.5	1.82	3.93	216.1
4.0	1.70	3.99	233.9
4.5	1.84	3.92	213.8
5.0	1.97	3.86	196.3
5.5	1.94	3.87	199.4
6.0	1.93	3.88	201.5

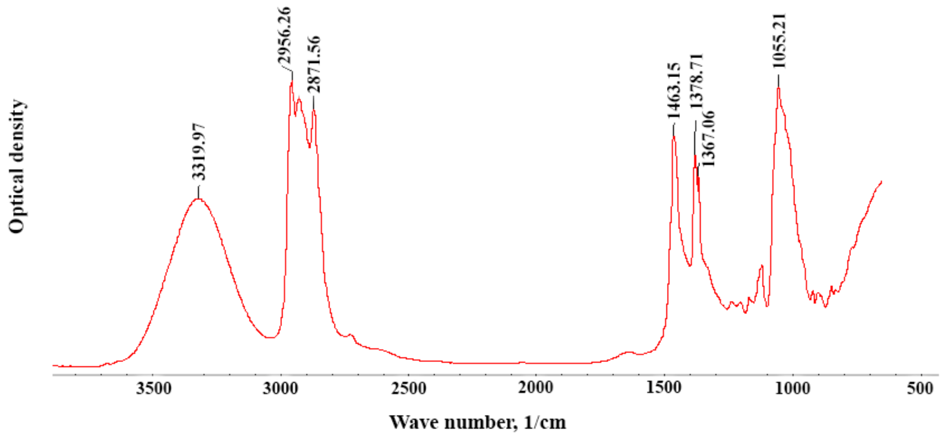

Fig. 1. Absorption spectrum of isooctyl alcohol.

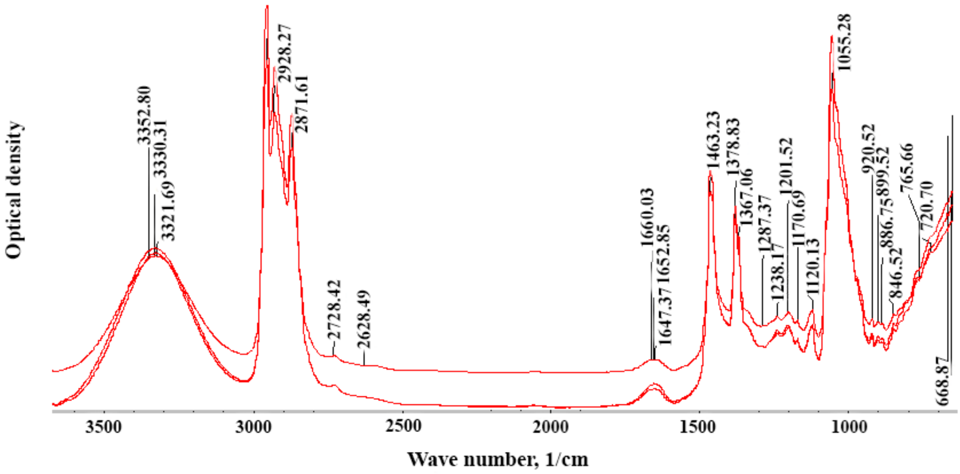

Fig. 2. Absorption spectrum of Eu extract.

Table 2. Coefficients of distribution of rare-earth elements (III) cations depending on the pH for extraction in the presence of interfering cations ($K_{distr\Sigma}$) and individually (K_{distr})

pH	Sm		Eu		Ho		Er	
	$K_{distr\Sigma}$	K_{distr}	$K_{distr\Sigma}$	K_{distr}	$K_{distr\Sigma}$	K_{distr}	$K_{distr\Sigma}$	K_{distr}
3.0	1437.3	306.9	1355.6	200.5	1282.5	443.1	1355.6	122.9
4.0	1752.4	326.2	1690.9	233.9	1481.9	481.4	1752.4	163.0
5.0	1752.4	426.2	1690.9	196.3	1529.2	404.9	1818.6	147.7
6.0	3091.7	395.6	3300.0	201.5	3300.0	359.6	4512.2	132.1

phase as alcohol solvates of medium dodecyl sulfates. As a result, the concentration of dodecyl sulfate in the aqueous phase at equilibrium is negligible. In the presence of interfering cations, the REE distribution coefficients increase by an order of magnitude and are maximum at pH ~ 6. At this value, REE in the aqueous phase are mostly found in the form of $Ln(OH)^{2+}$ monohydroxocomplexes and are extracted as solvates of the main salts of $Ln(OH)(DS)_2 \cdot nROH$. Thus, the consumption of dodecyl sulfate decreases, and the balance shifts toward extraction.

CONCLUSIONS

The extraction of europium(III) cations from aqueous solutions with and without the presence of interfering REE cations was studied. The extraction was successful at low concentrations, i.e., for concentrating components wastewater treatment.

The maximum extraction of europium(III) cations was at pH 3.0–4.5, 92%, using isooctyl alcohol as an extractant. Moreover, compared to extraction from individual solutions, the distribution coefficient of europium(III) cations significantly increased during extraction in the presence of interfering ions, and the pH of maximum extraction shifted to higher values (pH ~6).

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The authors declare no conflicts of interest.

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