Study of the Decomposition of Eudialyte Concentrate by Means of Sorption Conversion in Sulphuric Medium

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Abstract

The decomposition of eudialyte concentrate was studied by means of sorption conversion in the 2 mass % solution of sulphuric acid. It was shown that eudialyte decomposes at a temperature of 80 °C with the formation of silica gel, while silica sol is formed at 20 °C. The efficiency of the sorption of refractory rare elements by the sorbent in the mode of silica sol formation increased in comparison with the mode of silica gel formation from 52.5 to 89.4 for zirconium, from 60.1 to 89.2 for hafnium, from 7.4 to 85.1 for niobium, from 0 to 67.1 for tantalum, from 27.6 to 80.0 for titanium, respectively, with the provision of a high degree of extraction of rare earth elements (REE) – 82.9 %, including REE of yttrium and medium groups up to 86–89 %. The major part of the losses of niobium, tantalum, titanium and REE is determined by their inclusion in loparite and other accessory minerals that are untreatable under the studied conditions. It is assumed that a substantial increase in the sorption of rare elements of IV and V groups at 20 °C (in comparison with the results obtained at 80 °C) is due not only to the formation of silica sol but also to a decrease in the hydrolysis of the cations of these metals with the formation of their forms that are not sorbed by the sulphocationite. The features of decomposition of the eudialyte concentrate by the low-concentrated solutions of nitric and sulphuric acids were compared. The reasons providing the formation of silica sol during the low-temperature sulphuric decomposition of eudialyte are discussed. It is stressed that the studied method may be the basis of an efficient technology of eudialyte concentrate processing because the application of this method results in a decrease in acid consumption by a factor of 3–5 in comparison with traditional acid procedures. As a consequence, the amount of liquid wastes subjected to utilization is reduced, the effective specific radioactivity of the silica residue and the consumption of energy resources decrease.

Keywords: eudialyte concentrate, acid processing, rare elements, sorption
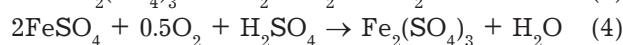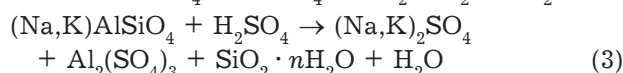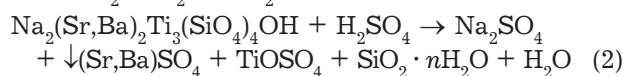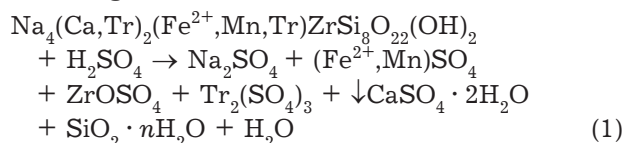
INTRODUCTION

A promising raw material source for obtaining zirconium products is eudialyte concentrate containing, in addition to zirconium and hafnium, also rare-earth elements (REE), niobium and tantalum. The REE sum of eudialyte concentrate is enriched with yttrium and lanthanides of the medium and yttrium groups.

The major problem in the development of hydrochemical technologies for processing eudialyte concentrate is its acid decomposition with the formation of a solution containing rare elements, and a silica residue. Decomposition with sulphuric acid is of the greatest interest [1–8], because sulphuric acid is a cheap low-volatile compound, and environmental regulations admit rather high concentration of sulphate ions in waste water.

Lamprophyllite, nepheline, aegirine, loparite, feldspar are the major accessory minerals of eudialyte concentrate. Decomposition of eudialyte,

lamprophyllite and nepheline is described by the following schemes:

$$Na_4(Ca,Tr)_2(Fe^{2+},Mn,Tr)ZrSi_8O_{22}(OH)_2$$
$$+ H_2SO_4 \rightarrow Na_2SO_4 + (Fe^{2+},Mn)SO_4$$
$$+ ZrOSO_4 + Tr_2(SO_4)_3 + \downarrow CaSO_4 \cdot 2H_2O$$
$$+ SiO_2 \cdot nH_2O + H_2O \qquad (1)$$

$$Na_2(Sr,Ba)_2Ti_3(SiO_4)_4OH + H_2SO_4 \rightarrow Na_2SO_4$$
$$+ \downarrow(Sr,Ba)SO_4 + TiOSO_4 + SiO_2 \cdot nH_2O + H_2O \quad (2)$$

$$(Na,K)AlSiO_4 + H_2SO_4 \rightarrow (Na,K)_2SO_4$$
$$+ Al_2(SO_4)_3 + SiO_2 \cdot nH_2O + H_2O \qquad (3)$$

$$2FeSO_4 + 0.5O_2 + H_2SO_4 \rightarrow Fe_2(SO_4)_3 + H_2O \quad (4)$$

where the formulas of minerals are presented according to the data reported in [9].

In the recommended modes, aegirine, loparite, feldspar do not interact with sulphuric acid and pass into the solid residue based on hydrated silica. In addition, the residue contains calcium, strontium and barium sulphates, and low-soluble double sulphates $NaTr(SO_4)_2 \cdot H_2O$ formed in the interaction of the sulphates of cerium-group REE with sodium sulphate. The major part of thorium and a part of yttrium-group REE are crystallized isomorphically with these double sulphates. Effective specific radioactivity (A_{eff}) of the silica residue may exceed 0.74 kBq/kg. Only ≤40 % of A_{eff} is determined by thorium of loparite that has passed into the residue.

In early studies, silica was obtained in the form of gel, and zirconium recovery did not exceed 78 % [1]. Later on, methods were developed that allowed obtaining low-watered and rather easily filtered silica [2−8]. The consumption of H_2SO_4 was 120−200 % of the stoichiometric amount, while up to 84 % of zirconium passed into the sulphate solution, but REE remained mainly in the silica residue in the form of low-soluble double sulphates with sodium and co-crystallized sulphates of the medium and yttrium groups, as well as non-decomposed loparite [6, 7].

The recovery of zirconium into solution increased to 94 % after the introduction of NaF into sulphuric solution in the amount providing the ratio F/Zr ≥ 3 [8].

The known methods of sulphuric decomposition of eudialyte concentrate have the following main disadvantages:

− great consumption of sulphuric acid and, as a consequence, the formation of an increased amount of salt-containing liquid wastes for which no utilization methods had been developed;

− REE spreading between the solution and silica residue, which hinders the isolation of these elements;

− absence or distortion of the information about the amount and composition of solid and liquid wastes, and on radionuclide distribution. For example, it is stated that the developed technology provides the formation of 9.1 kg of wastes (calculated for solid products) per 1 t of eudialyte concentrate, though one can see in the material balance data that the amount of wastes is larger by a factor of 67 [8]; the declared possibility to use the silica product is not validated, though its amount is almost a half of initial concentrate; its contamination with thorium, calcium and strontium sulphates is not taken into account;

− the addition of fluoride ions providing the high degree of zirconium recovery, while a noticeable part of these ions would get into discharge solutions, which would bring even more complications into utilization of waste waters.

Because of this, the studies aimed at a decrease in the consumption of sulphuric acid and the amount of wastes requiring utilization, an increase in the degree of rare element recovery and integrated use of raw material are highly relevant.

The goal of the present work is to study the regularities of the separation of rare metals and silica during sulphuric decomposition of the eudialyte concentrate by means of sorption conversion.

EXPERIMENTAL

The physicochemical foundations of sorption conversion are described in [10]. The chemical composition of eudialyte concentrate used in the work is reported in [11]. Experimental procedure and analytical techniques, as well as the procedures used to process experimental results, are described in [12]. The size of particles comprising the concentrate did not exceed 0.2 mm.

To investigate the efficiency of sorption conversion of eudialyte concentrate using sulphuric acid, we chose the following experimental conditions. The consumption of KU-2-8chS sorbent (GOST 20298−74) in H^+-form is equal to 70 % of the stoichiometric amount necessary to absorb all cations of the concentrate (6 cm³ of swollen sorbent per 1 g of the concentrate). The ratio of the volume (in mL) of a 2 mass % H_2SO_4 solution (Kh. Ch. reagent grade, GOST 4204−77) and concentrate mass (g) was 15. Process temperature (T) in experiment 1 was 80 °C, in experiment 2 it was 20 °C. The time of interaction was 4 h.

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RESULTS AND DISCUSSION

The major results of the studies are presented in Tables 1−3. The products formed in experiment 1 were saturated sorbent, silica gel and the residue of untreated minerals, while in experiment 2 the products included saturated sorbent, the residue of untreated minerals and silica sol which passed into silica gel only after long-term exposure. In experiment 1, the mass of silica gel and the residue of untreated minerals dried at 80 °C was 50.4 rel. %, while in experiment 2 (T = 20 °C) the mass of the residue of untreated minerals was 56.0 rel. %.

Table 4 shows the coefficients (K_d) of component distribution between the sorbent and the solution that were calculated from experimental data using equation

$$K_d = \frac{m_{sorb} \cdot V_{sol}}{m_{sol} \cdot V_{sorb}} \tag{5}$$

where m_{sorb} and m_{sol} are the masses of components in the sorbent and in solution, respectively; V_{sol}, V_{sorb} are the volumes of the solution and the sorbent, respectively.

Initial [H$^+$] concentration is equal to 0.306 mol/L. At the end of the process, [H$^+$] decreased to ~0.15 mol/L.

According to the data of X-ray phase analysis, the major part of solid residues is an X-ray amorphous substance. Loparite, microcline (possibly orthoclase) and aegirine were also detected

TABLE 1

Components of the aydialyte concentrate absorbed by the sorbent, rel. %

Experiment	Na_2O	K_2O	MgO	CaO	SrO	MnO	Al_2O_3	Fe_2O_3
1	47.6	82.6	86.7	72.2	88.7	82.4	72.5	64.8
2	77.3	89.0	97.0	84.5	84.3	87.9	92.7	88.9
Experiment	SiO_2	TiO_2	ZrO_2	HfO_2	Nb_2O_5	Ta_2O_5	ThO_2	UO_2
1	0	27.6	52.5	60.1	7.4	0	66.8	87.4
2	0	80.0	89.4	89.2	85.1	67.1	65.2	91.4
Experiment	Y_2O_3	La_2O_3	Ce_2O_3	Pr_2O_3	Nd_2O_3	Sm_2O_3	Eu_2O_3	Gd_2O_3
1	92.0	79.1	79.8	84.2	86.5	88.0	90.1	90.3
2	89.6	77.6	77.0	77.0	83.6	87.3	87.5	85.7
Experiment	Tb_2O_3	Dy_2O_3	Ho_2O_3	Er_2O_3	Tm_2O_3	Yb_2O_3	Lu_2O_3	ΣTr_2O_3
1	90.8	91.0	91.0	91.3	91.1	90.5	91.6	85.7
2	86.1	87.0	85.6	88.8	83.5	88.5	86.0	82.9

TABLE 2

Metal oxide content in the products dried at 80 °C, mass %

Experiment	Product	Na_2O	K_2O	MgO	CaO	SrO	MnO	Al_2O_3	Fe_2O_3
1	1-1	2.99	0.156	0.095	0.73	0.39	0.23	0.48	1.33
	1-2	7.90	2.56	0.384	3.19	1.50	1.26	4.29	4.54
2	2	2.23	0.145	0.042	0.67	0.54	0.34	0.41	0.67
Experiment	Product	SiO_2	TiO_2	ZrO_2	HfO_2	Nb_2O_5	Ta_2O_5	ThO_2	UO_2
1	1-1	55.3	1.10	4.48	0.133	0.64	0.046	0.0034	0.0012
	1-2	39.8	5.30	3.96	0.084	0.49	N.a.*	0.0114	0.003
2	2	40.7	0.63	1.42	0.034	0.16	0.012	0.0060	0.0008
Experiment	Product	Y_2O_3	La_2O_3	Ce_2O_3	Pr_2O_3	Nd_2O_3	Sm_2O_3	Eu_2O_3	Gd_2O_3
1	1-1	0.0324	0.0418	0.0816	0.0084	0.0304	0.0076	0.002	0.007
	1-2	0.119	0.212	0.402	0.033	0.112	0.028	0.007	0.023
2	2	0.0769	0.0975	0.200	0.0233	0.0672	0.0146	0.0043	0.0175
Experiment	Product	Tb_2O_3	Dy_2O_3	Ho_2O_3	Er_2O_3	Tm_2O_3	Yb_2O_3	Lu_2O_3	ΣTr_2O_3
1	1-1	0.001	0.006	0.001	0.004	0.0005	0.004	0.0004	0.227
	1-2	0.004	0.023	0.004	0.014	0.002	0.013	0.002	0.997
2	2	0.0031	0.0165	0.0037	0.0088	0.0017	0.0079	0.0012	0.544

Note. Products: 1-1 − silica gel; 1-2, 2 − residue of untreated minerals.
* Not analysed.

in the residue with the help of crystal optical analysis.

When estimating the losses of REE and niobium, it is necessary to take into account the fact that a part of these elements enters the composition of loparite which is not decomposed during the treatment. The studies showed that the concentration of loparite in the concentrate is slightly more than 1 mass % [11]. About 12 % of REE, mainly of cerium group, and up to 20 % of niobium and tantalum are confined to this mineral.

The acid decomposition of the eudialyte concentrate usually resulted in the formation of silica gel with which a substantial fraction of rare elements was lost. It was assumed that these losses were due to the high content of dispersion solution, concentrated with respect to the rare elements, in silica gel. In addition, the formation of silica gel determined the low rate of filtration during the separation of the solid and liquid phases.

Several approaches were proposed to prevent the formation of silica gel. Within the best studied method, the eudialyte concentrate is treated with a 30−40 % sulphuric acid at a temperature of 80−120 °C, with the consumption of the acid at a level of 125−200 % of the stoichiometric amount [8]. The recovery of zirconium and REE into solution after the separation of sulphuric solution and two-stage washing with water increased to 84.3 and 56.2 %, respectively. The recovery of niobium, the radioactivity of the formed products, and the ratios between REE in the resulting concentrate were not investigated.

During sorption conversion, multiply charged metals possessing higher ion potentials than those of alkaline metals are efficiently sorbed by the sulphocationite even in the case of low concentrations in solution. Because of this, the content of rare elements in the dispersion solutions of sorption conversion is not high. However, with this method of concentrate decomposition, the losses of rare metals are substantial due to the formation of hydrate polymers of these metals with silica gel. It was assumed that if we exclude the formation of silica gel, the recovery of rare elements into the sorbent would increase [13].

A 2 % H_2SO_4 solution was used for the decomposition of eudialyte concentrate by means of sorption conversion. The nature of interaction was strongly dependent on process temperature:

TABLE 3

Concentration of components in mother solutions, mg/L

Experiment	Na_2O	K_2O	MgO	CaO	SrO	MnO	Al_2O_3	Fe_2O_3
1	2074	45.8	2.9	60.9	18.0	0.024	6.3	36.9
2	1128	42.5	3.7	158	11.5	29.14	27.2	42.4

Experiment	SiO_2	TiO_2	ZrO_2	HfO_2	Nb_2O_5	Ta_2O_5	ThO_2	UO_2
1	6460	101	1138	124	3.74	26.8	0.079	4.09
2	12220	41.2	128	5.21	0.75	2.22	0.077	0.22

Experiment	Y_2O_3	La_2O_3	Ce_2O_3	Pr_2O_3	Nd_2O_3	Sm_2O_3	Eu_2O_3	Gd_2O_3
1	0.26	0.057	0.13	0.016	0.074	0.024	0.0065	0.023
2	1.50	2.72	1.88	0.21	0.75	0.20	0.068	0.22

Experiment	Tb_2O_3	Dy_2O_3	Ho_2O_3	Er_2O_3	Tm_2O_3	Yb_2O_3	Lu_2O_3	ΣTr_2O_3
1	0.004	0.028	0.0057	0.018	0.0026	0.0035	0.033	1.39
2	0.050	0.25	0.062	0.15	0.026	0.13	0.020	8.24

Note. Experiment 1 − acid solution; experiment 2 − sol.

TABLE 4

Coefficients (K_d) of component distribution in the sorbent

Experiment	Na	K	Mg	Ca	Sr	Mn	Th	U
1	4.2	39.7	505	82.5	114	100	429	161
2	14.8	46.1	425	35.2	248	98	135	67

Experiment	Al	Fe	ΣTr	Ti	Zr	Hf	Nb	Ta
1	755	116	1404	9.42	7.17	20.1	0.60	0
2	209	138	309	66.6	108	71.5	1064	14.7

at 80 °C, in addition to the sorbent and the solid residue, a solution and silica gel were obtained, while at 20 °C − only silica sol. A decrease in decomposition temperature promoted deceleration of silica coagulation in solution. The concentration of SiO_2 in the resulting sulphuric solutions was 2.2 g/L 80 °C and 12.2 g/L at 20 °C. The sorbent was rather easily separated from reaction products on a mesh filter. The most complete separation was achieved in the case of silica sol formation.

As expected [13], elimination of silica gel formation promoted the sorption of multi-charged metals by the sorbent (see Table 1), especially zirconium, hafnium, niobium, tantalum. With an increase in temperature, the sorption of the majority of metals (except REE and thorium) by the sorbent decreased.

In the concentrate, yttrium-group REE are almost completely included in the composition of eudialyte [11]. Judging from the total recovery into the sorbent and in solution estimated on the basis of results presented in Tables 1 and 3, eudialyte decomposition at 80 °C exceeded 92 %, while at 20 °C it was higher than 89 %. The recovery of REE (including cerium group) into the sorbent was substantially higher than the recovery of these elements into the sulphate solution during usual leaching with sulphuric solutions [5]. REE content in mother solutions is low and increases only insignificantly with a decrease in process temperature (see Table 3). An exception is sodium cation: its sorption is complicated by the low consumption of the sorbent.

An insignificant decrease in the amount of recovered REE with a decrease in temperature is likely to be determined by hindered decomposition of accessory minerals containing these elements, for example lamprophyllite.

At a temperature of 20 °C, the recovery of the elements of groups IV and V (zirconium, hafnium, niobium, tantalum) correlated with the degree of eudialyte decomposition, but at 80 °C their sorption by the sorbent was substantially lower than decomposition degree. The content of these metals in silica gel and in the residue of untreated minerals obtained at 80 °C was much higher than in he same residue obtained at 20 °C (see Table 2). An increase in the losses is determined by the enhancement of the formation of hydrate polymers with silicic acid by silica gel and the residue of untreated minerals, which is promoted by an increase in the hydrolysis of their cations with an increase in temperature. The formation of hard-to-sorb complexes in solution is

also connected with hydrolysis. This is confirmed by a substantial increase in the concentration of these elements not only in the solid residues (see Table 2) but also in mother solutions (see Table 3). A substantial change n the nature of sorption of the elements of IV and V groups is illustrated by a multiple decrease in their K_d values with an increase in temperature, while for other elements the corresponding K_d values either remain practically unchanged or increase (see Table 4).

At 20 °C, high recovery into the sorbent was achieved both for REE and for other rare elements: zirconium, hafnium, niobium, tantalum, titanium. At the same time, the major part of divalent elements including alkaline earths did not get into the silica residue. With the traditional methods of sulphuric decomposition of eudialyte concentrate, the content of the sulphates of alkaline earth metals in the silica residue exceeds 23 mass %, which may be a substantial obstacle for its use.

The major part of thorium and uranium is also sorbed by the sorbent. This defines a sharp decrease in A_{eff} of silica products, which is 0.33−0.37 kBq/kg without the extraction of loparite, while after its removal it becomes negligibly small (0.04−0.08 kBq/kg).

The consumption of H_2SO_4 was 40 % of the stoichiometric amount. So, the consumption of H_2SO_4 for the sorption of the eudialyte concentrate and, as a consequence, the amount of liquid wastes subject to utilization decrease by a factor of 3−5 in comparison with the traditional methods. The implementation of decomposition at room temperature determines a substantial decrease in energy consumption.

The components that are present in 1 L of the sorbent obtained at 20 °C are, g: Na_2O 25.6, K_2O 3.00, MgO 2.43, CaO 8.53, SrO 4.35, MnO 4.39, Al_2O_3 8.74, ΣTr_2O_3 3.92, TiO_2 4.21, ZrO_2 21.3, HfO_2 0.57, Nb_2O_5 1.32, Ta_2O_5 0.05, ThO_2 0.0167, UO_2 0.021. Desorption methods were found that provided the transition of alkaline, alkaline earth, rare earth elements, manganese, aluminium, iron and titanium into the eluate, while zirconium, hafnium, niobium and tantalum remained in the sorbent. Subsequent processing of the eluate provides consecutive isolation of hydroxyl cake containing manganese, aluminium, iron and titanium, then carbonate concentrate of REE, calcium carbonate, and strontium carbonate concentrate.

So, sorption conversion is a promising method for the integrated processing of eudialyte concentrate. The methods of acid sol utilization, desorp-

tion of heavy rare elements (especially niobium and tantalum) and their recovery from eluates are still to be developed.

It should be stressed that only silica gel was formed as a result of sorption conversion using a solution of HNO_3 (2 mass %) either at 20 °C and at 80 °C. This is likely to be determined by a higher rate of eudialyte decomposition by the nitric solution, while poorly soluble calcium and strontium sulphates partially block up eudialyte particles decelerating its decomposition. Deceleration of the acid decomposition of eudialyte leads to a decrease in the rate of heat evolution in this exothermal reaction and, as a consequence, to weakening of local overheating at the interface between the interacting phases, while overheating promotes polymerization of hydrated silica and an increase in hydrolysis extent.

CONCLUSION

The decomposition of eudialyte concentrate by means of sorption conversion in the 2 mass % solution of sulphuric acid was investigated. It was established that the nature of decomposition process is strongly dependent on temperature: silica gel is formed at 80 °C, while at 20 °C silica sol is formed.

Process mode providing the formation of silica sol promoted an increase in the sorption of zirconium from 52.5 to 89.4, hafnium from 60.1 to 89.2, niobium from 7.4 to 85.1, tantalum from 0 to 67.1, titanium from 27.6 to 80 % with the provision of REE recovery at a level of 82.9 %, in particular REE of yttrium and medium groups up to 86–89 %. A substantial increase in the absorption of rare elements of IV and V groups by the sorbent at 20 °C in comparison with the results obtained at 80 °C may be due to the formation of silica sol during eudialyte decomposition, with the simultaneous decrease in the hydrolysis of the cations of indicated elements. A decrease in the hydrolysis of the cations of these metals promotes an increase in the efficiency of their leaching, a decrease in their capture by silica gel, exclusion of their transition into non-sorbed forms. As a con-

sequence, their content in the residue of untreated minerals and in silica gel decreases, so do the losses with mother solution.

For the sorption conversion of eudialyte concentrate, the consumption of the acid decreases by a factor of 3–5 in comparison with traditional approaches; as a consequence, the amount of liquid wastes for utilization decreases, the efficient specific radioactivity of silica residue and the consumption of energy decrease too.

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