

Research article

Kaolinite clay as a raw material for erbium extraction



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ABSTRACT

Currently, due to the high rate of development of the rare-earth industry, new sources of raw materials are being mastered and new technologies for obtaining rare-earth metals (REM) are being developed. Studies have shown that REM in kaolinite clays of Alexeevskoe deposit in Kazakhstan and Egypt deposits in Sinai Peninsula (K-Watt, K-Tech) and in Aswan region (KB,KPL) are mainly represented by erbium (Er). Production of Er concentrate is considered as a by-product in a comprehensive middlings processing of kaolinite clays to produce alumina and building materials. The possibility of obtaining Er concentrate by sulfuric acid leaching and sorption concentration methods has been determined. Optimal technological conditions of kaolinite clays leaching is the use of 5% solution of H₂SO₄, at temperature 50 °C, duration 60 min and L:S ratio = 5. Under these conditions the separation of REM from the main components Fe₂O₃, Al₂O₃, SiO₂ is achieved. Concentrates were obtained with the content of the sum of REM oxides from 91.3 to 93.4%, in which the relative content of Er was from 64.89 to 90.82%. The results showed that the developed technology can be used for processing of erbium-containing kaolinite clays of various deposits.

1. Introduction

The rare-earth industry is developing at a fast pace - consumption in various sectors is expanding, consequently, new sources of raw materials are being mastered and new technologies for obtaining rare-earth metals (REM) are being developed.

The main sources of REM are the minerals bastnaesite, monazite, loparite and ion-absorption ores. The largest part of the world's economic rare-earth metal resources is concentrated in the bastnaesite deposits of the PRC, with a global share of 44%. Large REM resources are also associated with monazite deposits in Australia, Brazil, China, India, Malaysia, South Africa, Sri Lanka, Thailand and the USA [1,2].

The possibility of using middlings products from bauxite, nepheline, apatite, phosphate rock, kaolinite clays, etc. as additional REM raw materials is being studied [3–5]. According to statistics published by the U.S. Geological Survey (USGS) in 2022, global kaolin production in 2020 was estimated at about 46 million tons. The main kaolin-producing countries are the United States, Uzbekistan, China, India, Germany, the Czech Republic and others. Among these countries, India was the leader with a total estimated production of about 7.6 million tons in 2020 [6].

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Kaolin and kaolinite clays of various grades are mainly used in the production of ceramic products, porcelain, special cements, paper, alum and aluminum sulphate. The annual consumption of kaolin for these needs is more than 1 million tons and 75% is provided by the own mining industry, but for the production of high-quality ceramic and paper products kaolin is enriched using flotation [7].

The chemical and mineralogical composition of kaolin is usually influenced by its genesis, weathering of source rock, sedimentary transport to precipitation and diagenetic conditions, which strongly influence its physical properties, such as color, opacity and whiteness, compactness, plasticity and rheology, or its physical and chemical properties, including sorption or cation-exchange capacity [8].

Our studies of kaolinite clays of Kazakhstan and Egypt showed that they can also be used as REM raw materials. The advantage of these kaolinite clays is the fact that REM composition consists of 70–90% of one element of the heavy yttrium group of erbium (Er); this creates prerequisites for obtaining its selective concentrate.

The most important use of Er is in the nuclear power industry. A mixture of erbium oxide and uranium oxide can dramatically improve the operation of a high-power reactor, increasing their energy distribution and improving the safety of reactors [9].

Erbium oxide nanocomposite materials are widely investigated for the development of optical amplifiers in telecommunications systems operating at light wavelengths of 1.54 μm [10].

In modern optoelectronics, the introduction of erbium into amorphous silicon improves its photoelectric properties [11,12]. The properties of erbium and its compounds are being actively studied and the field of application is expanding, and because of this, the extraction of erbium from various sources is being actively explored [13,14].

Er is contained mainly in the minerals euxenite, yttrialite, fergusonite, gadolinite, samarskite, and xenotime, with an Er content of 0.21–6.5% in these minerals (Table 1).

It follows from the given data that the relative content of erbium from $\sum\text{REM}$ in minerals is 0.32–12.67%, and to obtain a selective erbium concentrate requires a complex multi-step processing of raw materials. The use of kaolinite clays as a raw material to produce Er concentrate compares favorably with other raw material sources. To create a profitable production, it is necessary to consider the possibility of complex processing of clays.

In this paper, the production of Er concentrate is considered as a by-product in a comprehensive waste-free processing of kaolinite clays by sintering, with the production of alumina and building materials (Fig. 1).

2. Materials and method

X-ray fluorescence analysis was performed with a Venus 200 PANalytical B.V. spectrometer with wave dispersion (Malvern PANalytical B.V., Almelo, The Netherlands). Chemical analysis of the samples was performed on an optical emission spectrometer with inductively coupled plasma Optima 2000 DV (PerkinElmer, Waltham, MA, USA). X-ray experimental data were obtained with a Bruker D8 ADVANCE (Bruker Corporation, Billerica, MA, USA) apparatus using copper radiation at an accelerating voltage of 36 kW and a current of 25 mA and JEOL JXA-8230 Electron Probe Micro Analyzer.

Acid leaching of kaolinite was carried out under mechanical agitation with an agitator speed of 400 rpm.

The sorption of REM from the leaching solutions was performed statically by dipping the sorbent into the sample solution. The solution was stirred mechanically to accelerate the equilibrium. For the final determination of sorbed REM the sorbent was separated by filtration and desorbed after washing.

The desorption was performed in sulphate-sulphate solution containing 30 g/dm³ H₂SO₄ + 300 g/dm³ (NH₄)₂SO₄; at temperature 25 °C; ratio L:S = (0.8 ÷ 1.1):1; and duration 1.0–1.5 h.

REM was precipitated from desorbate by sodium carbonate solution with concentration of 80 g/dm³ Na₂CO₃. For the purpose of effective precipitation desorbate solution was mixed with water in a ratio of 1:5 in an amount sufficient to obtain a clear solution. The precipitates were in contact with the mother liquor for 3 h.

3. Results and discussion

Physical and chemical studies of kaolinite clays of Alexeevskoe deposit of the Republic of Kazakhstan (RK), deposits KB, KPL of Aswan region and K-Tech, K-Watt of the Sinai Peninsula of the Arabian Republic of Egypt (ARE) (Table 2) were carried out.

The phase compositions of kaolinite clays are shown in Table 3.

Table 1
Characteristics of minerals containing erbium.

Mineral	Chemical formula	Content of $\sum\text{REM}$ in the mineral [15]	Content of Er in the mineral [16]	Relative content of Er from $\sum\text{REM}$
Rinkolite	$\text{Na}_2\text{Ca}_4\text{CeTiOF}_2(\text{Si}_2\text{O}_7)_2$	18,55	0,06	0,32
Orthite	$(\text{Ca}, \text{Ce}, \text{La}, \text{Y})_2(\text{Al}, \text{Fe})_3(\text{SiO}_4)_3(\text{OH})$	27,43	0,21	0,76
Gadolinite	$(\text{Y}, \text{Ce})_2\text{FeBe}_2(\text{Si}_2\text{O}_{10})$	55,40	0,66	1,19
Euxenite	$(\text{Y}, \text{Er}, \text{Ce}, \text{U}, \text{Pb}, \text{Ca})(\text{Nb}, \text{Ta}, \text{Ti})_2(\text{O}, \text{OH})_6$	40,00	1,7	4,25
Yttrialite	$(\text{Y}, \text{Th})_2(\text{Si}_2\text{O}_7)$	51,70	3,1	5,99
Xenotime	YPO_4	61,40	3,4	5,54
Fergusonite	$(\text{Y}, \text{Er}, \text{Ce})(\text{Nb}, \text{Ta}, \text{Ti})\text{O}_4$	51.28	6.5	12,67

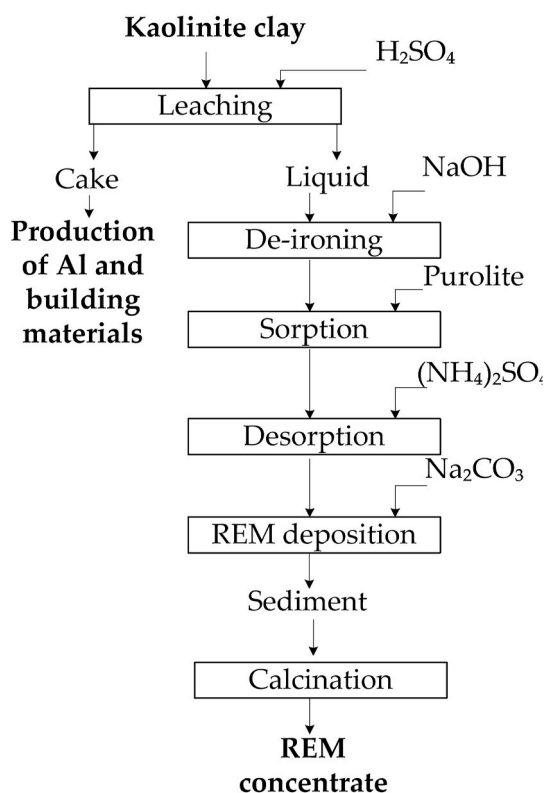


Fig. 1. Obtaining Er concentrate by complex kaolinite clay processing technology.

Er content in kaolinite clays is shown in Tables 4–5.

Analysis of the forms of Er in kaolinite clays showed that it is mainly bound to Fe minerals (Fig. 2).

To determine the optimal technological conditions for REM extraction from clays, we investigated the dependence of the degree of extraction in the leaching solution by mineral acids on the type of acid, concentration, ratio L:S, temperature and duration. At this stage, the raw material was the clay of the Alexeevskoe deposit.

Table 2

Chemical composition of kaolinite clays.

Name	Fields				
	KB	KPL	K-Tech	K-Watt	Alexeevskoe
	Content, %				
Al_2O_3	39,09	37,96	34,12	34,72	17,21
SiO_2	43,16	45,3	49,75	48,48	66,27
Fe_2O_3	0,56	0,718	1,68	1,269	0,51
TiO_2	3,39	2,695	1,82	2,103	0,574
Na_2O	–	0,047	0,05	0,041	0,119
K_2O	–	0,042	0,042	0,083	0,9
P_2O_5	0,037	0,057	0,033	0,0901	0,011
CaO	0,093	0,192	0,116	0,1540	0,166
Cr_2O_3	0,052	0,039	0,032	0,0373	0,015
NiO	–	0,005	–	0,008	–
ZnO	–	0,006	0,005	0,035	0,004
Ga_2O_3	0,009	0,007	0,008	0,007	0,004
SrO	0,003	0,01	0,009	0,019	–
SO_3	0,011	0,008	0,0393	0,116	0,006
Y_2O_3	0,005	0,011	0,0062	0,0115	–
ZrO_2	0,222	0,161	0,087	0,121	0,003
Nb_2O_5	0,015	0,021	0,014	0,0161	–
Cl	0,006	0,009	0,027	0,0265	0,053
K_2O	–	0,042	0,042	0,083	0,9
$\sum \text{REM}$	0,45902	0,44835	0,4059	0,39703	0,08521
others	12,89	12,22	11,64	12,17	13,748

Table 3

The phase composition of kaolinite clays.

Name		Fields				
		KB	KPL	K-Tech	K-Watt	Alexeevskoe
		Content,%				
Kaolinite-1A	Al ₂ (Si ₂ O ₅)(OH) ₄	31,4	85,1	68,6	62,0	53,5
Anatase	TiO ₂	–	5,3	18,6	3,5	3,3
Silica	SiO ₂	–	5,1	7,5	–	–
Quartz	SiO ₂	67,5	4,5	5,2	34,6	43,2
Muscovite	KAl ₂ (AlSi ₃)O ₁₀ (OH,F) ₂	1,1	–	–	–	–

Table 4Relative Er content of $\sum\text{REM}$ in kaolinite clays.

Fields	Content of $\sum\text{REM}$ in the mineral	Content of Er in the mineral	Relative content of Er from $\sum\text{REM}$
Alexeevskoe	0,085	0,0759	89,29
KB	0,45902	0,4398	95,813
KPL	0,44835	0,41882	93,414
K-Tech	0,4059	0,39559	97,460
K-Watt	0,39703	0,28863	72,697

Table 5

Content of REM elements in kaolinite clays.

Clay name	Content, %										
	Ce	Er	Gd	Ho	La	Lu	Nd	Sc	Sm	Y	Yb
Alexeevskoe	0,003	0,0759	0,0005	0,00003	0,0014	0,00002	0,0012	0,0009	0,00048	0,0006	0,00006
KB	0,0053	0,4398	0,0072	0,00002	0,0011	0,00003	0,0017	0,00153	0,00049	0,00139	0,00016
KPL	0,01305	0,41882	0,00112	0,00006	0,00628	0,00003	0,00377	0,00114	0,00068	0,00271	0,00021
K-Tech	0,0037	0,39559	0,00062	0,00003	0,0016	0,00004	0,00095	0,00117	0,00017	0,00168	0,00014
K-Watt	0,0952	0,28863	0,00097	0,00004	0,00455	0,00002	0,00325	0,00129	0,00073	0,00178	0,00016

H_2SO_4 , HNO_3 and HCl solutions with concentrations of 5–20% were used to determine the dependence of the degree of extraction on the type of mineral acid. Leaching was carried out at temperature 20 °C, ratio L:S = 3 and duration of 40 min (Fig. 3a–c).

Behavior of individual REM elements during leaching in mineral acid solutions is presented in Fig. 4(a–c).

It follows from the results that the highest extraction of $\sum\text{REM}$ and low Fe_2O_3 , Al_2O_3 , SiO_2 , which further deteriorate the quality of the REM concentrate, were obtained in 5% solutions of mineral acids. Under these conditions, Er recovery was 60.3% in H_2SO_4 solution, 49.98% in HNO_3 and 36.59% in HCl .

With increasing concentrations of H_2SO_4 solutions of Er extraction was significantly lower. Extraction of the elements Ho, Ce, Nd, La, Y, Yb, Gd increases with increasing solution concentration. The extraction degree of Tm, Sc, Lu does not depend on the acid's concentration and remains constant at about 100%.

The behavior of REM elements during leaching in HNO_3 solutions is similar. In HCl solutions with the increase of acid concentration the extraction of all REM elements decreases except Ho, Tm, Sc, Lu the extraction of which remains constant. Solutions of 5% H_2SO_4 were used for further studies when selecting the optimal technological conditions.

Influence of leaching duration on recovery of major elements and REM was carried out at temperature 20 °C, ratio L:S = 3 and duration of 20–90 min (Fig. 5 a,b).

A study of the dependence of REM extraction during acid leaching on the duration showed that the optimum duration is 60 min. With further increase in the duration the degree of extraction changes insignificantly. Extraction of Fe_2O_3 , Al_2O_3 , SiO_2 depends little on the leaching duration.

Influence of leaching temperature on recovery of major elements and REM was carried out at temperatures of 20–70 °C, ratio L:S = 3 and duration of 60 min (Fig. 6 a,b).

The optimum leaching temperature is 50 °C. Further increase in temperature has little effect on the degree of REM extraction, at the same time, the extraction of undesirable impurities Fe_2O_3 , Al_2O_3 , SiO_2 into the solution increases significantly.

Influence of L:S ratio on the extraction of major elements and REM was carried out at temperature 50 °C, L:S ratio = 3 ÷ 7.5:1 and duration of 60 min (Fig. 7 a,b).

The optimum ratio L:S during leaching can be considered equal to 5. Further increase of L:S ratio has little effect on REM extraction degree, but leads to increase of Fe_2O_3 , Al_2O_3 , SiO_2 extraction into solution.

Based on the research we can conclude that the optimal technological conditions of clay leaching Alexeevskoe deposit is the use of 5% solution of H_2SO_4 , at a temperature of 50 °C, duration of 60 min and the ratio L:S = 5. After leaching a sulfuric acid solution with pH 3.3 was obtained. Extraction into solution was, %: 18.98 Al_2O_3 ; 16.19 SiO_2 ; 10.39 Fe_2O_3 and $\sum\text{REM}$ 55.92 (Er 62.1%).

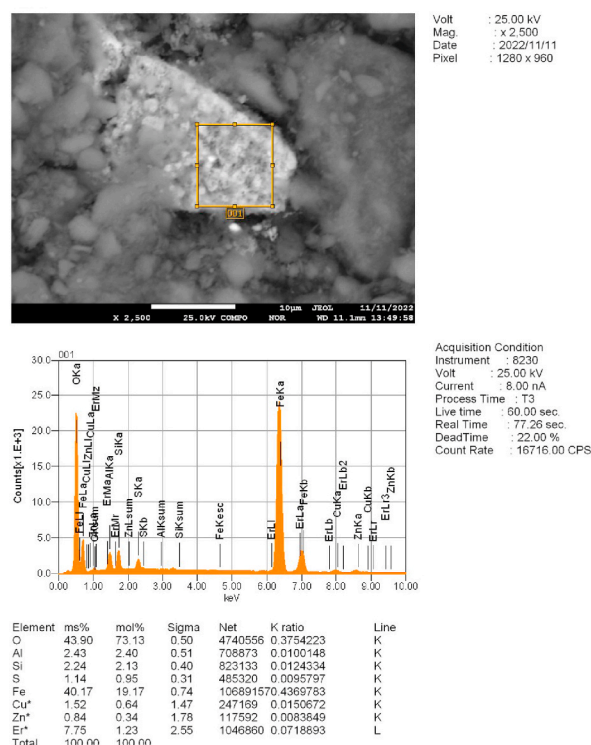


Fig. 2. The presence of erbium in the mineral formation of kaolinite.

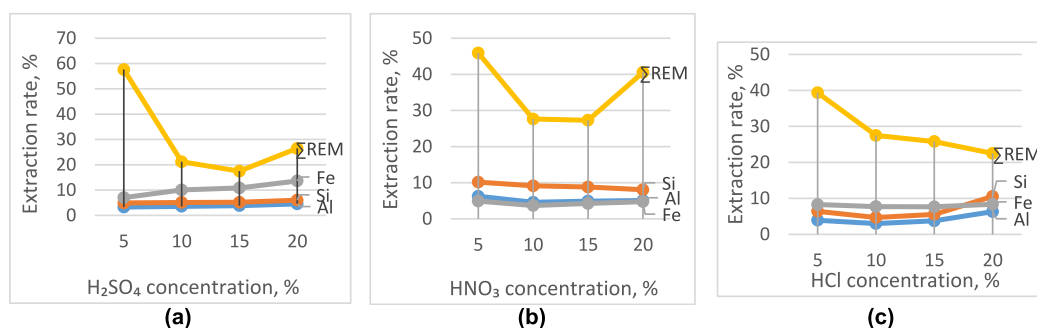


Fig. 3. Extraction of major elements and Σ REM by leaching in mineral acid solutions: (a) H_2SO_4 ; (b) HNO_3 ; (c) HCl .

REM extraction from ARE kaolinite clays (KB, KPL, K-Watt, and K-Tech) was performed under optimum technological conditions of acid leaching defined for kaolinite clay of Alexeevskoe deposit with 5% H_2SO_4 solution (Table 6).

The results of studies on acid leaching under certain optimal technological conditions using 5% H_2SO_4 solution showed that under these conditions the separation of Σ REM from the main components Fe_2O_3 , Al_2O_3 , SiO_2 present in ARE clays is achieved, which is similar to the data obtained for Alexeevskoe clay deposit.

To extract REM from sulfuric acid leaching solutions we performed sorption on strongly acidic cationite Purolite C100 [17–19]. For sorption we used the clay leaching solution (Table 7).

Before sorption, the initial solutions were purified of iron by alkaline precipitation at pH 5.5 (Table 8).

As a result of REM sorption on cationite Purolite C100 the recovery was 83.1–85.0%. The composition of the desorption solutions is given in Table 9.

The precipitate formed as a result of desorbate treatment with sodium carbonate solution was calcined at 950 °C for 2 h. The composition of the obtained concentrates and the relative content of REM elements are presented in Tables 10 and 11.

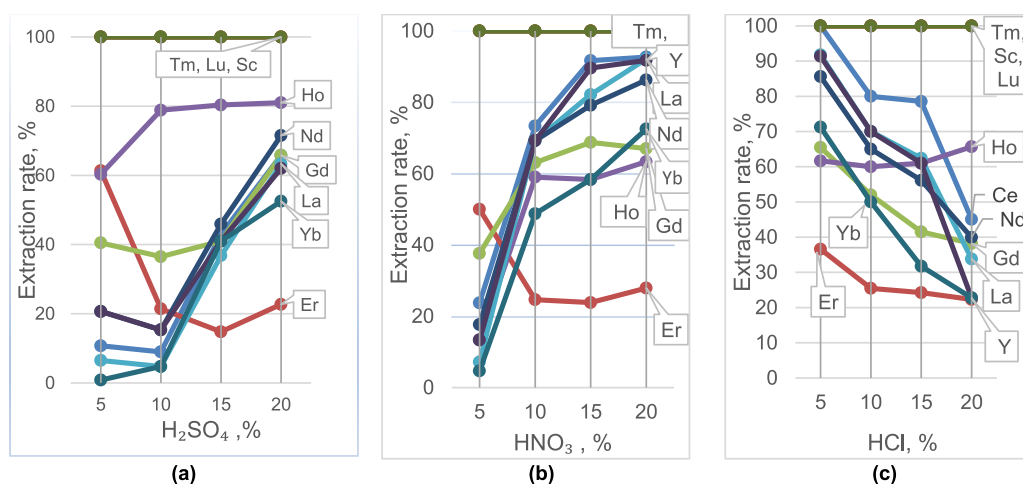


Fig. 4. Extraction of REM elements in mineral acid solutions: (a) H₂SO₄; (b) HNO₃; (c) HCl.

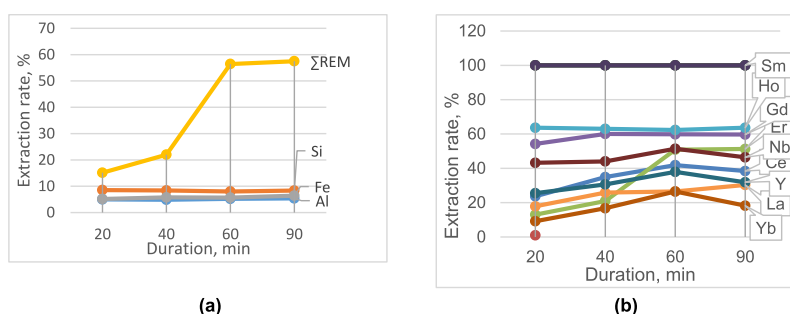


Fig. 5. Effect of leaching duration on recovery of major elements and ΣREM (a); REM elements (b).

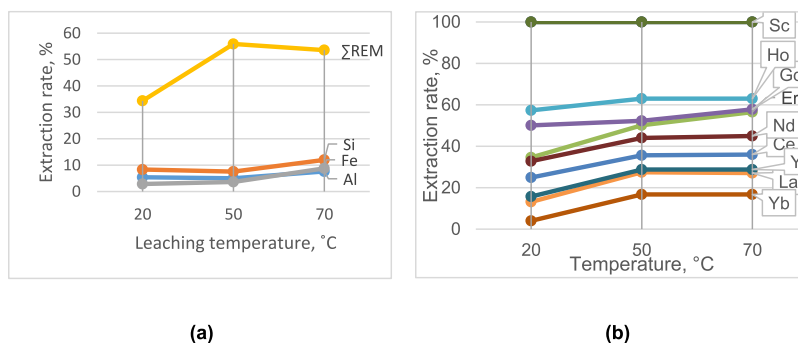


Fig. 6. Effect of leaching temperature on recovery of major elements and ΣREM (a); REM elements (b).

4. Conclusions

The high relative content of erbium in REM elements of kaolinite clays makes them a promising raw material for obtaining its selective concentrate.

According to the results of electron microscopy analysis of kaolinite clay composition, the presence of Er was found to be associated with mineral formations containing iron.

REM extraction as a by-product of complex processing of kaolinite clay using mineral acids H₂SO₄, HCl and HNO₃ for leaching has been investigated.

Optimum technological conditions of kaolinite clay leaching of Alexeevskoe deposit and ARE are the use of 5% solution of H₂SO₄, at temperature 50 °C, duration 60 min and L:S ratio = 5. Under these conditions, separation of REM from the main components Fe₂O₃, Al₂O₃, SiO₂ is achieved.

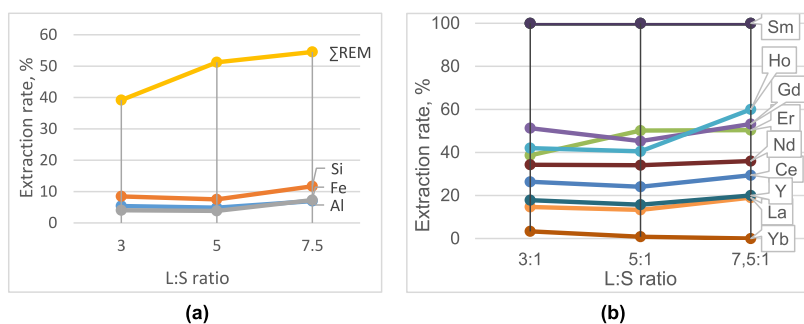


Fig. 7. Effect of L:S ratio on recovery of major elements and Σ REM (a); REM elements (b).

Table 6

Extraction of major elements and Σ REM by leaching of ARE clays in 5% H_2SO_4 .

Name	KB	KPL	K-Tech	K-Watt	KB	KPL	K-Tech	K-Watt
	content, %;				extraction rate, %			
Al_2O_3	31,181	30,834	26,944	28,249	1,77	1,4	1,97	1,409
SiO_2	43,272	45,23	43,206	48,917	2,45	2,34	1,54	2,22
Fe_2O_3	0,446	0,612	1,307	1,145	1,07	1,53	1,41	0,93
Σ REM	0,332	0,3041	0,328	0,303	28,653	32,467	20,479	24,313
Ce	0,004	0,0108	0,00315	0,078	25,66	17,66	16,14	18,89
Dy	0,00031	0,00041	0,0002	0,00042	18,06	31,24	29,43	16,31
Er	0,004	0,0108	0,00315	0,078	25,66	17,66	16,14	18,89
Gd	0,00031	0,00041	0,0002	0,00042	18,06	31,24	29,43	16,31
Ho	0,00002	0,00005	0,00002	0,00003	20	16,67	33,33	25
La	0,00086	0,0051	0,0014	0,0033	21,82	18,79	12,5	27,47
Lu	0,00002	0,00002	0,00003	0,00001	33,33	19,67	22,5	25
Nd	0,0011	0,00198	0,00084	0,00237	35,88	47,48	11,58	27,08
Sc	0,00146	0,00076	0,00088	0,00125	4,5752	33,333	24,786	3,1008
Sm	0,00011	0,00021	0,00013	0,00049	77,551	69,1176	23,5294	32,8767
Y	0,00127	0,0017	0,0011	0,0018	8,6331	37,269	34,524	33,708
Yb	0,00012	0,00017	0,0001	0,00018	25	19,05	18,57	17,5

Table 7

Composition of leaching solutions for sorption, g/L.

Fields	Al_2O_3	SiO_2	Fe_2O_3	Σ REM
Alexeevskoe	13,25	18,73	0,018	0,05
KB	13,86	0,89	0,20	0,22
KPL	12,13	0,49	0,18	0,24
K-Tech	12,63	11,98	0,65	0,14
K-Watt	11,25	0,08	0,22	0,16

Table 8

Composition of solution after de-ironing, g/L.

Fields	Al_2O_3	SiO_2	Fe_2O_3	Σ REM
Alexeevskoe	13,25	18,73	0,001	0,05
KB	13,86	0,89	0,002	0,22
KPL	12,13	0,49	0,002	0,24
K-Tech	12,63	11,98	0,003	0,14
K-Watt	11,25	0,086	0,002	0,16

The degree of extraction of the main components and separate REM elements during leaching was determined.

To purify the sulfuric acid leaching solution from iron before sorption, a precipitation method was used by neutralising the solution with alkali to pH 5.5.

A sorption method on a strongly acidic Purolite C100 cationite was used to obtain REM concentrate from the leaching solution.

Concentrates with the content of the sum of REM oxides for clays of Alexeevskoe deposit (Republic of Kazakhstan) 91.25%, for deposits KB 93.4% KPL 91.3%, K-Tech 93.2% and K-Watt 91.3% (Arab Republic of Egypt) in which the relative content of Er was, %

Table 9

Composition of desorption solution, g/L.

Fields	Fe ₂ O ₃	ΣREM
Alexeevskoe	0,031	1,57
KB	0,056	6,49
KPL	0,04	7,08
K-Tech	0,042	4,13
K-Watt	0,04	4,72

Table 10

Composition of REM concentrates, %.

Fields	Fe ₂ O ₃	ΣREM oxides
Alexeevskoe	1,8	91,25
KB	0,8	93,4
KPL	0,51	91,3
K-Tech	0,94	93,2
K-Watt	0,77	91,3

Table 11

Relative content of REM elements in concentrates, %.

Clay name	Content, %										
	Ce	Er	Gd	Ho	La	Lu	Nd	Sc	Sm	Y	Yb
Alexeevskoe	3,56	81,56	0,43	0,03	1,89	0,01	1,24	1,34	0,32	0,79	0,09
KB	1,12	89,17	1,63	0,007	0,24	0,01	0,31	0,41	0,03	0,36	0,03
KPL	3,24	84,67	0,27	0,02	1,53	0,01	0,59	0,23	0,06	0,51	0,05
K-Tech	0,90	90,82	0,15	0,01	0,40	0,01	0,24	0,25	0,04	0,31	0,03
K-Watt	23,49	64,89	0,24	0,01	0,99	0,007	0,71	0,38	0,15	0,36	0,05

were obtained: 81.56; 89.17; 84.67; 90.82 and 64.89, respectively.

The determined technological regimes of REM concentrate production by acid leaching and sorption methods can be used for erbium-containing kaolinite clays of different deposits.

This technology is the simplest solution, which does not destroy the structure of the basic minerals of the raw material. And this raw material can be used to produce alumina, ceramics, building materials or pharmaceuticals.

Author contribution statement

Nazym Akhmediyeva, Yerkezhan Abikak: Performed the experiments; Wrote the paper. Rinat Abdulvailiev, Sergey Gladyshev: Conceived and designed the experiments.

Alfiyam Manapova: Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

Galiya Ruzakhunova: Analyzed and interpreted the data.

Bulat Sukurov: Contributed reagents, materials, analysis tools or data.

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Data availability statement

Data included in article/supp. material/referenced in article.

Declaration of interest's statement

The authors declare no conflict of interest.

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