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CERIUM EXTRACTION FROM RARE EARTH CONCENTRATES BY ELECTROCHEMICAL AND EXTRACTION METHODS FOR PREPARATION OF POLISHING MATERIALS

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Abstract: *The Russian rare earth raw metals (REM) are complexmaterials to contain up to 57% of ceriumin the total amount of lanthanides. Authors recommended to separate cerium at the first stages of rare earth concentrate treatment by combined methods of electrochemical oxidation and liquid extraction. The process has been suggested to optimize by using up-to-date equipment, including a diaphragm-type electrolyzer of OXITRON-58L-O2 type and centrifugal type extractor EC-10FA.*

To organize continuous automated process at chosen equipment, various operation modes of electrochemical oxidation Ce (III) to Ce (IV) and extraction of tetravalent cerium out of mixture of trivalent rare earth materials have been tested. Investigations have been performed over process solutions prepared out of Solikamsk Magnesium Plant concentrates extracted out of loparite. Conditions have been created to achieve oxidation level $Ce^{+3} \geq 99$ % in terms of electric power consumption less than 0.8 kW/h per 1 kg of CeO_2 with subsequent cerium extraction out of rare earth concentrate at a cascade of centrifugal extractors to obtain 99.6% of cerium dioxide (from ∑REM). Polishing powders have been synthesized on the basis of nanosized cerium oxide to comply with appropriate composition of cerite and polirite.

Keywords: *rare-earth materials; cerium (III) and (IV); oxidation; electrolysis; electrolyzer; extraction; centrifugal extractor; polishing powders*

1. Introduction

Materials based on rare earth elements (REE) are widely used in modern areas of science and engineering. Today, it is difficult to find innovation technology that does not use REE as components of application, e.g. development of superconducting materials, special alloys, super magnets, accumulators, catalysts and etc. After the USSR dissolution, Russia lost leadership in the production of rare earth metals (REM), and majority of plants and feedstock sources of REM appeared to beoutside Russia. Today, the world REM marketis dominated by China which possesses sufficiently rich and easy transformable feedstock of REM. This enables China to fix metal prices and terms of sale.

Nowadays in Russia, under support of the Russian Government the industrial production of REM is revived. Note that mineral feedstock and technogenic waste has been reputed to be a source of REM. Approximate compositions of rare earth concentrates (REC)) and ores for industrial processing are presented in Table 1.

Table 1. Approximate compositions of REC and ores which are promising for development of industrial processing in Russia

REE composition in ore,% mass.	Loparite	Apatite	Phosphogypsum	Eudialyte	Tomtor ore
	32.4	0.98	0.52	2.0	13.67
	Ln_2O_3		$\sum Ln_2O_3 - 100\%$ in concentrate		
La_2O_3	21.11	27.2	20.86	9.8	23.31
CeO_2	57.72	43.55	46.75	26.0	42.68
Pr_6O_{11}	5.36	5.8	5.15	4.0	4.14
Nd_2O_3	14.4	14.3	17.31	12.0	16.72
Sm_2O_3	0.89	1.9	2.38	4.2	2.46
Eu_2O_3	0.18	0.5	0.63	0.6	0.79
Gd_2O_3	0.15	1.8	1.80	4.2	1.67
Tb_4O_7	0.02	0.2	0.07	0.6	–
Dy_2O_3	0,11	0.7	0.96	3.3	0.83
Ho_2O_3	0.02	–	0,14	0.6	0.15

Table 1. Approximate compositions of REC and ores perspective for industrial processing in Russia.

REE composition in ore,% mass.	Loparite	Apatite	Phosphogypsum	Eudialyte	Tomtor ore
	Ln_2O_3		$\sum Ln_2O_3 - 100\%$ in concentrate		
Er_2O_3	–	0.15	0.27	2.5	0.57
Tm_2O_3	–	–	–	0.4	0.06
Yb_2O_3	–	–	0.10	1.8	0.24
Lu_2O_3	–	–	–	0.3	–
Y_2O_3	0.023	3.9	3.56	30.0	6.37

Each source of REM is unique by its composition (REM and impurities content) and structure. Specific feature of domestic feedstock of REM is the predominant content of cerium in it - up to 57% of total amount of rare earth metals. Therefore, in the traditional technological scheme of rare-earth metals production the extraction of cerium from other lanthanides makes up the first stage. The process is based on cerium oxidation in the tetravalent state by chemical (oxidation by O_2, ozone, potassium permanganate or hydrogen peroxide) or electrochemical method with subsequent cerium separation from rare earth metals by precipitation, ion exchange or extraction method.

In their previous works, authors [1-3] proved the efficiency of combination of electrochemical oxidation and liquid extraction for cerium extraction. Benefits of this technology include high productivity, continuous operation, easy automation and achievement of high rates of rare earth metal extraction in the end-product. This method of production was developed by scientific-research institute "Giredmet" in the 1980’s of the 20th century and put into practice at industrial enterprises manufacturing rare earth metals. However, due to the collapse of enterprises after the dissolution of the Soviet Union this method has no longer been used in Russia. Nowadays, technological schemes of rather complicated, laborious and high reagent inputs are used in existing domestic processes for cerium extraction from lanthanide mixture [1,2,4,5]. These include:

- cerium oxidation using expensive chemical reagents;

- separation of tetravalent cerium from lanthanides due to pH difference in Ce^{4+} and REM^{3+} hydrating;
- cerium purification from REM by multiple reprecipitation or its hydroxide or by extraction.

Extraction purification is carried out in mixing-settling extractors which are traditional in the technologies of REM processing. Such type of apparatus is easy controllable, however, cascade-linked apparatuses require high consumption of technological solutions for charging, including toxic and inflammable organic solvents that affect process cost and environmental conditions [1,2,6].

Since industry of REM is currently revived in Russia, it is important to renew electrochemical technology of cerium extraction and improve it with advantages of modern equipment. The work analyzes factors capable of optimizing the process of cerium extraction and obtaining cerium oxide with purity of no less than 99.5% through the use of diaphragm-type electrolyzer and centrifugal extractor. Technological parameters and optimal conditions of process have been studied.

2. Experimental

Studies have been pursued on a model and afterwards in technological nitrate solutions prepared out of rare earth concentrate of Solikamsk Magnesium Plant. Rare earth concentrate was prepared out of loparite concentrate containing 293.6 to 345.5 g/l of rare earth oxide (REO). The composition of the prepared rare earth concentrate is shown in Table 2.

Table 2. Composition of prepared rare earth concentrate

Compound	Composition, mass.%
La_2O_3	26.1
CeO_2	54.2
Pr_6O_{11}	5.0
Nd_2O_3	13.0
Sm_2O_3	0.97
CaO	0.06
SrO	0.04
Fe_2O_3	0.001
SiO_2	0.02
Cl	0.05

Model solutions were prepared by dissolution of CeO_2 in the nitric acidto contain 50-100 g/l of HNO_3 and 140-160 g/l of Ce_2O_3. In turn, rare earth element solutions were prepared by dissolution of carbonates in the nitric acid. Note that REE ions in solutions were determined by titration method with complexing agent EDTA in the presence of indicator. Cerium ions were detected by titration using mohr's salt and potassium permanganate solution.

Composition study of aqueous and solid phases has been carried out by inductively coupled plasma atomic emission spectrometry with the help of ICAP 6300 JY-38 spectrometer (Thermo Fisher Scientific) and roentgen-fluorescent method by means of ARL OPTIM'Xspectrometer (Thermo Fisher

Scientific) according to standardized procedures as set forth in "Giredmet".

Required concentration of acid in aqueous phase was maintained by adding HNO_3. Acid concentration was measured by volume or potentiometric titration.

Coulomb efficiency BT was calculated according to the following equation:

$$BT = \left(\frac{Q_{exp}}{Q_t}\right) * 100\% \qquad (1)$$

where Q_{exp} and Q_t are electrical charges (A∗ h) consumed in oxidation process, defined experimentally and calculated by Faraday's laws of electrolysis, correspondingly. Values of electrical charges were determined according to equations below:

$$Q_t = C_{Ce^{4+}} * \frac{V}{q} \qquad (2)$$

where $C_{Ce^{4+}}$ is current concentration of Ce^{+4} (g/l); V is a volume of treated solution (l); q = 5.224 (g –eqv/ A∗h) is electrochemical equivalent; t – time (h).

$$Q_{exp} = I * t \qquad (3)$$

where I is current supplied to electrochemical reactor (A), t – time (h).

Degree of cerium oxidation was calculated according to the following equation:

$$\alpha = \left(\frac{C_{Ce^{4+}}}{C_{Ce_0}}\right) * 100\% \qquad (4)$$

where $C_{Ce_0}$is total concentration of cerium in the processedsolution (g/l).

Studies were conducted in two-ways:

- electrochemical oxidation of cerium;
-extraction and purification of oxidized cerium by liquid extraction method.

3. Results and discussion

3.1. Electrochemical cerium oxidation

Membrane and diaphragm-type electrolyzers have been considered [7–10] to conduct experiments on electrochemical oxidation of cerium. It should be noted that the diaphragm material is resistant to high temperatures, easily regenerated, adjustable and has no limitations on current density, a diaphragm-type electrolyzer OXITRON-58L-O2 was chosen[11]. The diaphragm-type electrolyzer is manufactured in LLC "Delfin Aqua" and applied for electrochemical synthesis of strong oxidizers - hydrogen peroxide, hypochlorous acid, peroxydisulphiric acid, etc. Electrochemical setup is shown in Fig. 1. A main part of laboratory setup is an electrochemical reactor MB-26-21-15К shown in Fig. 2.

Note that a reactor vessel consists of 4 platinizing titanic anodes and 1 titanic cathode of cylindrical shape while electrode spaces are divided by ceramic diaphragm made of aluminum oxide and zirconium. Two types of circulation are provided in the reactor vessel: external/ internal circulation for anolyte and external one for catholyte.

External circulation rate is 30 l/h and the internal one is 100 l/h. Hydrodynamics in reactor provides the mixing of electrolyte in the whole volume of anode space of reactor and gives equiprobable access of electro-oxidized reagent to the total anode surface. Therefore, in the whole volume of anode space of reactor the same constant concentration of oxidizable cerium is settled. A pressure regulator mounted on the reactor vessel makes it possible to regulate pressures in electrode chambers. Technical features of setup are shown in Table 3.

Technological process of electrochemical cerium oxidation initially has been analyzed in model solutions which contained 136 to 150 g/l of Ce^{+3} and 50 to 120 g/l of HNO_3. As a catholyte, aqueous solutions of nitric acid and cerium nitrate are considered. After determination of optimal modes of electrolyzer operation on model solutions the further investigations were conducted in technological solutions of REM concentrates. For this purpose, cathode and anode chambers of reactor were pumped with technological solutions. Then, electrolyte external circulation and anolyte internal circulation preheated up to 50°C was switched on in such a way that circulation rate of anolyte proved to be higher 3 times as compared tothe circulation rate of catholyte.

Fig. 1. Laboratory electrochemical setup

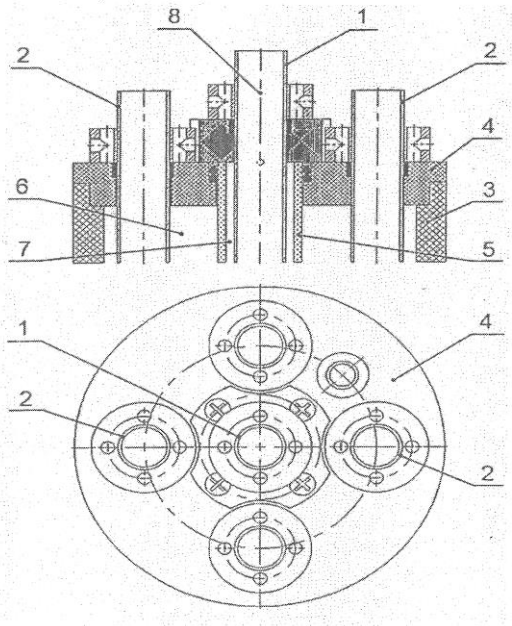

Fig. 2. Electrochemical reactor MB-26-21-15K: 1 – cathode, 2 – anode, 3 – reactor vessel, 4 – plug, 5 – diaphragm, 6 – anode chamber, 7 – cathode chamber, 8 – perfs.

Table 3. Technical features of OXITRON-58L-O2 setup

Parameter	Units of measurement	Value
Feed rate of solution	l/h	
- in cathode chamber		10–40
- in anode chamber		10–40
Electrode potential in reactor	V	3–30
Strength of current in reactor	A	3–30
Pressure	MPa	
- in cathode chamber		0–1.0
- in anode chamber		0–1.0
Mass of setup	Kg	50
Dimensions (height×width×depth)	Mm	890×480×500
Maximal time of operating mode stabilization	Min	15

The use of pressure regulator made it possible to create a positive pressure of 0.3 atm in the cathode chamber. After that, current was supplied and electrolysis conducted at the constant current density. While in catholyte, a certain quantity of nitric acid was added at regular intervals (~15 min) to maintain its concentration at 50-70 g/l. After completion of operating cycle, the current supply was suspended, pumps switched off and drain valves open to sample catholyte and anolyte for further analytical control.

Studies have been carried out intechnological solutions to show the possibility of achieving high degree of oxidation Ce^{+3} (≥ 99 %) at power consumption less than 0.8 kW·h per 1 kg of CeO_2. Kinetics of oxidation process in salt concentrates with ∑REM and Ce_2O_3 ~ 53 % content is given in Table 4.

Table 4. CeO_2 concentration vs. electrolysis time

Time, hours	Concentration, g/l	
	CeO_2	HNO_3
1	35.5	66.88
2	63.6	69.98
3	95.1	63.6
4	123.1	56.4
5	142.3	59.9
6	151.1	62.1
7	155.1	66.8
8	155.9	62.4

Note: average indicator BT=76.4%, current density is 2.3 A/dm^2, electrolyze time – 8 hours.

Mass balance of Ce^{+3} oxidation process at certain operating mode of electrolyzer is shown in Table 5.

Table 5 shows that the level of Ce^{+3}oxidation consists of 99.68%. Further extraction and purification of oxidized cerium from REM solutions were conducted by means of extraction techniques.

Table 5. Mass balance of electrolysis process

Anolyte	∑REE, g/l	Ce^{+3} concentration, g/l	Ce^{+4} concentration, g/l	Cerium mass, g
Before electrolysis	300	159.0	0	477
After electrolysis	300	0.5	158.5	477

3.2. Cerium extraction from total amount of REM

Usually, extraction of oxidized cerium from ∑REM is carried out by means of extraction method in box-type mixing-settling extractor [1,2] which is characterized by long-term conduction process and use of expensive and inflammable reagents. There are no such drawbacks in case of using centrifugal extractors that makesit possible to increase productivity repeatedly and decrease essentially the consumption of very expensive extractants [6]. The use of centrifugal extractors in REM technology has limitations in the equipment which operateswithin the framework of multistage processes. However, modern hardware allows automation control and eliminating these limitations. In this work the application of centrifugal extractors for obtaining cerium oxide with purity > 99.5 % from solution of REM was considered. As extractant, a well-known reagent in REM processing - tri-n-butylphosphine (TBP) was used. Extraction cascade was formed by series of centrifugal extractors (model EC-10FA, Scygrad Group) [12]. The main technical features of laboratory extractor are shown in Table 6. For tubing connections in cascade REM distribution coefficients D and cerium separation coefficients($\beta_{\frac{Ce^{4+}}{REE^{3+}}}$) in the technological solutions of REM were determined. Composition of solutions is shown in Table 7.

Table 6. Technical features of laboratory extractor EC-10FA

Parameter	Units of measurement	Value
Capacity*	l/h	up to 10
Ratio between densities of initial solutions		up to 0.95
Ratio between flow rates of initial solutions		any
Working volume of mixing chamber	ml	22
Working volume of separation chamber	ml	32
Radius of separation chamber	mm	17
Radius of overflow chamber for heavy phase	mm	9
Radius of overflow chamber for light phase	mm	adjustable
Gear– electric motor ДАТ75-40-3.0-У3	rpm	2750
Installed power	W	40
Three-phase motor voltage	V	380
Power network frequency	Hz	50
Rotary rotations per minute	rpm	2620
Dimensions (length×width×height)	mm	180×120×240
Apparatus mass	kg	3.5
Apparatus material		polytetrafluorethylene (GOST 10007-80)

**Solution system: 30%TBP in kerosene and 2M HNO_3 at the ratio between flow rates of aqueous and organic phases A:O=1:1*

Table 7. Composition of technological solutions of REM

Compound	Composition, mas.%
CeO_2	54.5
La_2O_3	26.1
Pr_6O_{11}	4.1
Nd_2O_3	14.9
Sm_2O_3	0.86
Gd_2O_3	0.33
Eu_2O_3	0.2
Tb_4O_7	0.08
Dy_2O_3	0.04
CaO	0.06
Fe_2O_3	0.002
HNO_3	70–80 g/l

Table 8. Distribution coefficients and REM separation coefficients

REE	D_{REE}	$(\beta_{\frac{Ce^{4+}}{REE^{3+}}})$
Ce	10.56	-
La	0.029	364.1
Pr	0.087	121.4
Nd	0.099	106.7
Sm	0.148	71.3
Gd	0.201	52.5
Eu	0.415	25.4

To decrease viscosity of TBP, the latter was diluted by kerosene. Extractant was processed by nitric acid solution which came into equilibrium with initial solution at the ratio of aqueous and organic phases A:O=1:1. Laboratory experiments on extraction of REE were conducted in separating funnels. Phase contact time consisted of 5 minutes, which was sufficient for equilibration. Values of D and $(\beta_{\frac{Ce^{4+}}{REE^{3+}}})$ are shown in Table 8.

Saturation of cerium extractant was detected by subsequent contacting of organic phase with aqueous solution at the ratio A:O=1:1. Cerium content in extractant after the second contacting made up 157 g/l. Extractant composition corresponded to 100 % Ce^{4+}. However, there was an inversion (turnover) of aqueous and organic phases due to the similarity of their density values - 1.222 and 1.225 g/cm^3 respectively which is not desirable for extraction process. To prevent inversion, an optimal content of REM no more 300 g/l in the technological solution was selected.

Given the data obtained, the countercurrent mode of extraction for oxidized cerium separation was estimated [13,14], as well as the number of transfer units and ratio of phases in extraction and rinsing parts of cascade calculated. The following parameters were used for calculation: $\beta_{Ce/Sm} = 71$; a degree of cerium extraction in the product – 99% with content of impurities (La, Pr, Nd, Sm) less than 0.5%.

Pilot study was performed in the laboratory setup and represented a cascade of 18 centrifugal extractors which isidentical to the scheme as described in the work [1,15,16].

Centrifugal extractors were connected with each other by means of tubing that allowed to operate with process solutions at any step of cascade. Trials were carried out to reproduce the process as described in the work [1]; however, the scheme of reactors showed unstable work of cascade and formation of emulsion in its rinsing part, and as a consequence, low quality of cerium in re-extractant.

Therefore, some modifications were done to improve the cascade performance - the number of units was increased up to 20 pieces. Photo of cascade is shown in Fig.3.

Fig.3. Cascade with centrifugal extractors

After the equilibrium set in a cascade, the test sample of cerium oxide was obtained by oxalate precipitation from cerium re-extractant with subsequent filtration, drying and calcination at a temperature 800–850 °C of the formed sediment in a muffle furnace. Analyses showed that CeO_2 content in a sample obtained made up was 99.6%.

Note that a pilot batch of nanosized powders with composition in line with cerite and polirite has been producedout of extracted cerium. The composition of obtained samples has been determined in an accredited laboratory.

4. Conclusion

Analysis has been carried out to obtain cerium oxide with purity 99.5 % and higher by using combination of two methods – electrochemical and extraction in electrochemical reactor MB-26-21-15K and a cascade of centrifugal extractors EC-10FA. The proposed technology is rather promising from industrial application standpoint.

Despite the complicated multicomponent composition of initial raw materials, the proposed technological scheme and related equipment provide benefits as follows:

- ensuring a degree of cerium extraction at $\geq$99% and power consumption at 0.8 kW·h/kg CeO_2;
- reducing multiple consumption of reagents, including those damaging the environment, and inflammable extractants;
- achieving the purity of CeO_2 higher than 99.5% from (∑REM);
- obtaining polishing cerium-containing nanosized powders.

The main result is that the proposed technology of cerium extraction based on electrochemical and extraction methods can be applied in REM processing out of various cerium-containing raw materials. It is easy to automatically control the proposed technological process and thus allow extracting cerium up to 95% out of REM concentrate that sufficiently reduce processing time and consumption of expensive reagents.

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ИЗВЛЕЧЕНИЕ ЦЕРИЯ ИЗ КОНЦЕНТРАТОВ РЗМ МЕТОДАМИ ЭЛЕКТРОХИМИИ И ЭКСТРАКЦИИ ДЛЯ ИЗГОТОВЛЕНИЯ ПОЛИРУЮЩИХ МАТЕРИАЛОВ НА ЕГО ОСНОВЕ

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Акционерное общество "Государственный научно-исследовательский и проектный институт редких металлов "Гиредмет" (АО "Гиредмет"), Москва, Россия;
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Российское редкоземельное (РЗМ) сырье является комплексным и содержит до 57 % церия на фоне суммы лантаноидов. Авторами рекомендовано отделять церий на первых стадиях переработки РЗМ последовательным сочетанием методов электрохимического окисления и жидкостной экстракции. Процесс предложено оптимизировать применением преимуществ современного российского оборудования: электролизера диафрагменного типа ОКСИТРОН-58Л-О и экстракторов центробежного типа модели ЭЦ-10ФА. Для организации непрерывного автоматизированного процесса на выбранном оборудовании разработаны режимы электрохимического окисления Ce (III) в Ce (IV) и извлечения четырехвалентного церия из смеси трехвалентных РЗМ экстракцией. Исследования выполнены на технологических растворах, приготовленных из концентрата Соликамского магниевого завода, выделенного из лопарита. Разработаны условия для достижения степени окисления $Ce^{+3} \geq 99$ % при расходе электроэнергии не выше 0.8 кВт·ч на 1 кг CeO_2 с последующим выделением церия из концентрата РЗМ на каскаде центробежных экстракторов с получением 99.6 % диоксида церия (от ∑РЗМ). Синтезированы полирующие порошки на основе наноразмерного оксида церия состава соответствующего маркам церит и полирит.

Ключевые слова: *редкоземельные металлы, церий (III) и (IV), окисление, электролиз, электролизер, экстракция, центробежные экстракторы, полировальные порошки.*

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