ISSN 1066-3622, Radiochemistry, 2017, Vol. 59, No. 6, pp. 579–586. © Pleiades Publishing, Inc., 2017.
Published in Russian in Radiokhimiya, 2017, Vol. 59, No. 6, pp. 510–516.

Coextraction and Successive Selective Stripping of Cerium(IV) and Thorium from Monazite Leach Solutions with Tributyl Phosphate (TBP)[1]

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Received February 15, 2017

Abstract—Solvent extraction of Ce(IV) and Th(IV) from 8 M HNO_3 with TBP in kerosene was studied. The dependence of the extraction on the TBP concentration, kind of diluent, O/A phase ratio, and contact time was examined. The extraction efficiency reaches 99.3 and 97.7% for Ce and Th, respectively, at 5% TBP in kerosene, 1/1 O/A phase ratio, and 5 min contact time. Successive selective stripping of Ce(III) and Th(IV) from the loaded organic phase was reached at 1.1 M H_2O_2 concentration, 10 min contact time, 3/1 O/A phase ratio, and room temperature (25 ± 2°C). After stripping of cerium, the remaining organic was contacted with H_2O for thorium stripping at 5 min contact time, 2/1 O/A phase ratio, and room temperature (25 ± 2°C).

Keywords: coextraction, thorium, cerium, stripping, tributyl phosphate

DOI: 10.1134/S10663622170600054

Monazite consists of phosphates and silicates of thorium, uranium, and rare earths with atomic numbers between 57 and 71 [1]. There are two methods for processing monazite: alkali method [2] and acid method using sulfuric acid [3]. In the first case, uranium, thorium, and rare earths are obtained in the form of insoluble hydroxides, which have to be dissolved in hydrochloric acid for further processing. The acid process uses concentrated sulfuric acid and yields liquor containing rare earths, U, and Th [4–9]. Cerium is the most abundant element among lanthanides, and it can be readily extracted from its ores (monazite and bastnäsite). It differs from other lanthanides in its unique ability to be oxidized to the +4 state.

Hughes and Singh [10] studied the extraction of thorium from sulfate leach solution with 0.1 M Adogen-383 (secondary amine) diluted in kerosene. Their countercurrent extraction system consisted of four stages of extraction (0.1 M H_2SO_4), four scrub stages (0.1 M H_2SO_4), and three strip stages (1.0 M ammonium carbonate). Dong and Jinwen [11] started with a monazite–U–Th–alkaline cake leached with HCl. They used diethylheptyl methylphosphonate (DEHMP) and tributyl phosphate (TBP) as extractants diluted in kerosene to separate and purify the above elements.

Jun Lu et al. [12] studied the separation of Ce(IV) and Th(IV) from rare earths(III) by extraction with Cyanex 923 in *n*-hexane from sulfuric acid medium. They examined the dependence of the extraction on the acidity and temperature. Cyanex 923 proved to be suitable for extracting Ce(IV) and Th(IV) from REE(III) in sulfuric acid solutions. The aqueous phase acidity does not affect the extraction of Ce(IV) but increases the degree of the Th(IV) extraction.

Nikolaev et al. [13] studied the extraction of thorium and cerium(IV) nitrates with TBP in the presence of 1.5 M HNO_3 in the equilibrium aqueous phase. The presence of a small amount of cerium in the aqueous phase resulted in a sharp decrease in the distribution coefficient of thorium. This effect was more pronounced at low concentrations of thorium. Cerium replaces thorium in the organic phase. These data are valid not only for 100% TBP, but also for 20 and 50 vol % TBP in CCl_4. The addition of hydrogen peroxide to reduce Ce(IV) to Ce(III) improved the extraction of thorium from monazite sands.

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[1] The text was submitted by the author in English.

Junmei Zhao et al. [14] studied the extraction behavior of Ce(IV), Th(IV), and REE(III) with di(2-ethylhexyl) 2-ethylhexylphosphonate (DEHEHP) in heptane from a solution obtained after roasting bastnäsite with Na_2CO_3 and subsequent leaching with HNO_3. The extractability varies in the order Ce(IV) > Th(IV) >> REE(III). Therefore, it is possible to find the appropriate conditions under which Ce(IV) can be efficiently separated from Th(IV) and REE(III). Furthermore, Ce(IV) can be stripped from the loaded organic phase with dilute H_2SO_4 containing H_2O_2. Hammad et al. [15] separated uranium from Egyptian monazite alkaline hydrous oxide cake concentrate dissolved in concentrated HCl solution using an anion-exchange column containing Amberlite IRA-400. Thorium in the effluent solution was purified by extraction from nitric acid medium with TBP diluted in kerosene.

Thorium is widely used as an alloying element (1–2% mix with tungsten) in TIG welding electrodes [16], and also as a material in high-end optics and scientific instrumentation. Thorium and uranium are the only significantly radioactive elements with major commercial applications that are not based on their radioactivity. Thorium is predicted to be able to replace uranium as nuclear fuel in nuclear reactors, but only a few thorium reactors have been yet completed.

Ali et al. [17] studied the extraction and stripping of thorium from the hydrous oxide after alkaline dissolution followed by leaching with alkaline carbonate solution. This cake was dissolved in 4 M HNO_3, and thorium was extracted selectively with a countercurrent extraction system using a mixer–settler contactor and Aliquat-336 in kerosene as extractant. The results show that 2 h of continuous operation is necessary to reach the steady-state condition for the process. The extraction efficiency was found to be 80%, and the stripping efficiency was 82%. Zhao et al. [18–21] indicated that DEHEHP may also act as an extractant for the recovery of cerium and fluorine from bastnäsite nitric acid leachate.

Helaly et al. [22] used TBP for impregnation into Amberlite XAD-16 nonionic polymeric resin beads for extracting cerium from nitric acid media. The cerium extraction efficiency was greatly enhanced when nitrate salt was added as a salting-out agent with a total free nitrate concentration of 5 M. The extraction efficiency without salting-out agent addition was 67.8% at the free HNO_3 concentration of 5 M. This efficiency increased to 72.4 and 80.6% when 1.0 and 3.0 M NH_4NO_3, respectively, was added.

Jingui He et al. [23] studied the extraction of Ce(IV) from sulfuric acid solution with an emulsion liquid membrane (ELM) comprised of HDEHP dissolved in sulfonated kerosene containing Span80 as an emulsifier. The results show that the ELM method is a clean and cost-effective process for the extraction of Ce(IV) from sulfuric acid solution. The optimum conditions for Ce(IV) extraction can be summarized as follows: HDEHP concentration 12 vol %, Span80 concentration 2–3 vol %, liquid paraffin concentration 2–4 vol %, HCl concentration in the internal phase 4–5 M, H_2O_2 concentration 0.02 M, volume ratio of the membrane phase to the internal phase 1.5, acidity of the external phase 0.4–0.5 M, volume ratio of the external phase to the membrane phase 2, extraction time 15 min, and stirring rate 250 rpm. The results demonstrate that, among the parameters studied, the HDEHP and H_2O_2 concentrations, acidity of the external phase, and the stirring rate play a vital role in the Ce(IV) extraction.

This paper deals with the evaluation of TBP as extractant at low concentrations for coextracting Th and Ce from monazite leach solution (HNO_3 medium) to obtain highly purified products from cerium and thorium hydroxides. In addition, the successive selective stripping of Ce and Th from loaded TBP was studied. A tentative separation flowsheet for the whole process is suggested.

EXPERIMENTAL

Chemicals and reagents. The chemicals and reagents used were of analytical reagent grade. All solutions were prepared in calibrated flasks using distilled water. Tributyl phosphate (Merck, Germany), sulfuric acid (Riedel–de Haen, Germany, 96%), nitric acid (Riedel–de Haen, Germany, 69%), oxalic acid (Adwic, Egypt), and H_2O_2 (Adwic, Egypt, 50%) were used. The kerosene used was specially prepared for hydrometallurgical specifications. Its characteristics are shown below.

Odor	T_{flash}, °C	ρ, g cm^{-3}	η at 25°C, MPa s
Odorless	62	0.789	1.31

Preparation of nitrate stock solution. About 100 g of monazite (69% purity) was digested in 300 mL of 80% sulfuric acid at 180°C for approximately 3 h. The resulting slurry was dissolved in 2 L of

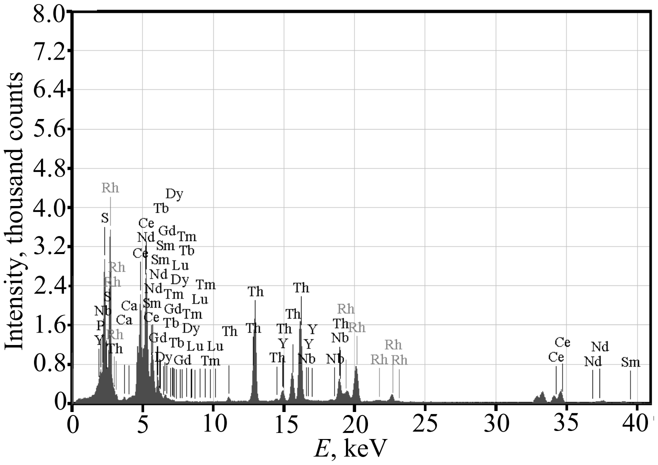

Fig. 1. XRF chart for oxide cake.

cold water with stirring for 1 h, and the solution was filtered. The filtrate was adjusted to pH 1.1–1.5, and a 10% oxalic acid solution was added to precipitate both thorium and total rare earths as oxalate cake. The precipitate was allowed to settle for 6 h, filtered off, and washed with a 2% oxalic acid solution. The oxalate precipitate was calcined at 500°C for 2 h. The oxide cake was subjected to XRF and ICP analysis before dissolution in nitric acid. The results are shown in Fig. 1 and tabulated in Tables 1 and 2. A portion of the calcined cake was dissolved in 1 L of 8 M HNO_3. The nitrate stock solution contained 0.2588% Th and 0.19% Ce(IV).

Batch extraction experiments were carried out as follows. Equal volumes (10 mL) of the organic and aqueous solutions were placed in a conical flask and shaken for 10 min at room temperature (25 ± 2°C). This time was found to be sufficient to attain the equilibrium. The organic and aqueous phases were allowed to settle down and were separated using separating funnels. The concentrations of Th(IV) and Ce(IV) in the aqueous phases were determined by suitable techniques. The concentrations of these metal ions in the organic phase were calculated as the differences of their respective concentrations in the aqueous phase before and after the extraction. The extraction efficiency (E, %) was evaluated as the percent fraction of the extracted metal. The distribution ratio (D_M) of metal ions (M = Th and Ce) was calculated as the ratio of the equilibrium metal concentration in the organic and aqueous phases.

Table 1. XRF data for oxide cake and final products (Th and Ce hydroxides), %

Element	Oxide cake	Th hydroxide	Ce hydroxide
Th	3.4	88.9	0.82
V	–	0.1	–
Sc	–	0.04	–
K	–	1.43	–
Ca	1.0	0.82	0.75
P	1.80	–	–
Nb	0.21	–	–
La	–	0.34	–
Ce	24.14	0.54	95.5
Pr	–	–	0.17
Gd	3.80	1.41	0.34
Dy	0.82	0.43	0.03
Ho	–	–	0.14
Yb	–	0.08	0.03
Eu	–	–	0.42
Mn	–	0.11	–
Tm	0.24	0.10	0.10
Y	3.5	2.43	0.17
Nd	7.5	0.24	0.60
Sm	1.90	0.024	0.20
Lu	0.011	–	–
Tb	0.70	–	0.09
Er	–	0.33	0.06
S	16.54	–	–
Si	–	0.016	–

Table 2. ICP-OES data for the oxide precipitate and final products (Th and Ce hydroxides), g/100 g

Element	Th–REE precipitate	Th hydroxide	Ce hydroxide
Al	0.21	0.007	0.02
Ca	0.6	–	–
Pb	0.12	0.01	0.03
Na	4.12	0.09	3.9
Ni	0.002	0.001	0.001
La	12.5	–	–
Ce	20.3	0.061	95.1
Pr	6.56	0.02	0.26
Nd	7.86	0.023	0.30
Sm	1.99	0.03	0.23
Eu	0.09	–	0.008
Gd	0.72	0.015	0.16
Tb	0.11	0.01	0.04
Dy	0.18	0.008	0.084
Ho	0.005	0.001	0.01
Er	0.25	0.008	0.05
Yb	0.005	0.0004	0.006
Lu	0.0005	0.0003	0.001
Sc	0.008	0.002	0.007
Y	–	0.007	0.15
Th	3.9	86.9	1.9

Batch stripping experiments were carried out as follows. Equal volumes (10 mL) of the loaded organic

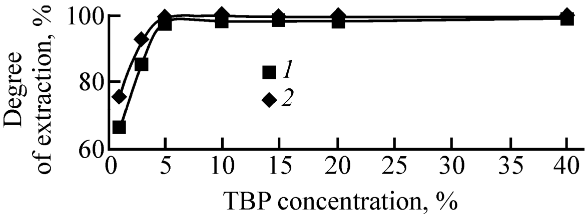

Fig. 2. Effect of TBP concentration on the efficiency of the joint extraction of (*1*) Th and (*2*) Ce.

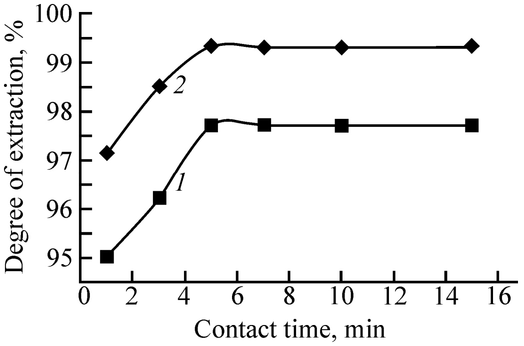

Fig. 3. Effect of contact time on the efficiency of the joint extraction of (*1*) Th and (*2*) Ce.

phase and aqueous stripping solution were placed in a conical flask and shaken for 10 min at room temperature (25 ± 2°C). This time was found to be sufficient to attain the equilibrium. The organic and aqueous phases were allowed to settle and were separated using separating funnels. The stripping efficiency (S, %) was calculated as the percent fraction of the metal that passed from the organic phase to the aqueous phase. The distribution ratio (D_M) of the metal ions (M = Th and Ce) in stripping was calculated as the ratio of the equilibrium metal concentration in the aqueous phase to that in the organic phase.

The separation factor (β) of Th and Ce in stripping was determined as the ratio of the Th and Ce distribution ratios: $\beta_{Th} = D_{Th}/D_{Ce}$ and $\beta_{Ce} = D_{Ce}/D_{Th}$.

The Th(IV) concentration in the aqueous phase was determined spectrophotometrically using Thoron. The absorption was measured at 540 nm [24].

The Ce(IV) concentration in the aqueous phase was determined by titration with a standard $(NH_4)_2Fe\cdot(SO_4)_2$ solution using *o*-phenanthroline as indicator.

The Ce(III) concentration in the aqueous phase after stripping was determined spectrophotometrically with Arsenazo III. The absorption was measured at 650 nm [24].

Analytical techniques. X-ray fluorescence (XRF) analysis was performed with a Philips Unique II spectrometer equipped with a PW 1510 automatic sample changer (30 positions) (Netherlands), connected to a computer system (X-40 software). High-resolution UV-visible spectra were recorded with a Lambda-3 double-beam spectrometer device (Perkin Elmer, the United States). These methods were used for determining relatively high concentrations of Th and Ce in solutions. Inductively coupled plasma optical emission spectrometry (ICP-OES) was used for determining trace concentrations of metal ions (Prodigy Axial high dispersion ICP-OES-USA model) at the Instrumental Analysis Department, Atomic Energy Authority. The pH values of aqueous solutions were measured with a DN-21 digital pH meter.

RESULTS AND DISCUSSION

Coextraction of Th and Ce

Effect of TBP concentration. The TBP concentration in kerosene was varied from 1 to 40%, with the other extraction conditions fixed (O/A ratio 1/1, shaking time 10 min, 25 ± 2°C, working Th and Ce concentrations 2.588 and 1.9 g L^{-1}, respectively). As seen from Fig. 2, the maximal extraction efficiency is reached at 5% TBP in kerosene.

Effect of contact time. The contact time was varied from 1 to 15 min with the other conditions fixed: 10 mL of the pregnant solution was contacted with 10 mL of 5 vol % TBP in kerosene at 25 ± 2°C. As seen from Fig. 3, 5 min is quite sufficient for the efficient coextraction of Th and Ce (extraction efficiency 97.7 and 98.5%, respectively). Longer contact does not lead to noticeable changes in the extraction efficiency.

Effect of diluent. The effect of diluents on extraction of metal ions from different aqueous media was extensively studied previously [21–23]. The diluent polarity is usually considered to be the most significant parameter. In our study, we tested as diluents hexane, benzene, toluene, butanol, and kerosene. The other conditions were kept constant (1/1 O/A phase ratio, TBP concentration 5 vol %, 5 min contact time, 25 ± 2°C. The results are shown in Fig. 4. As can be seen, kerosene is the optimum diluent (coextraction efficiency 97.7 and 99.3% for Th and Ce, respectively). This may be due to high hydrophobicity of kerosene, favoring displacement of water molecules from the metal complexes by TBP molecules in the organic phase. With neat TBP, the extraction efficiency was about 99.6%.

Effect of volume phase ratio (O/A). The effect of the O/A ratio was examined in the range from 2/1 to 1/5 at the other operating conditions kept constant: 5 vol % TBP in kerosene, 5 min contact time, 25 ± 2°C. The results are shown in Fig. 5. As can be seen, the coextraction efficiency increases as the relative amount of the aqueous phase is increased. However, in going from O/A = 2/1 to O/A = 1/1 the decrease is insignificant, and the extraction efficiency at O/A = 1/1 is close to the maximal value.

Successive Stripping of Ce and Th

The loaded organic solvent (TBP) was first contacted with an aqueous solutions of a reducing agent to back-extract Ce(III) selectively. After the back-extraction of Ce in the trivalent state, the remaining TBP solution was contacted with water to back-extract Th. The stripping percentage was calculated relative to the initial amount of M(IV) in the loaded organic solvent. The effect of various factors on stripping was studied.

Cerium stripping. *Effect of stripping agent type.* Two reductants were used for selective stripping of Ce from the loaded organic phase: H_2O_2 and $NaNO_2$. The operating conditions were as follows: 1.1 M reducing agent concentration, O/A volume ratio 1/1, contact time 5 min, initial concentration of Th(IV) and Ce(IV) in the organic phase about 2.528 and 1.88 g L^{-1}, respectively, 25 ± 2°C. The stripping efficiency reached 46.8% with $NaNO_2$ and 40.11% with H_2O_2. However, we preferred H_2O_2 because of higher separation factor β_{Ce}: 0.8 against 0.21 in the case of $NaNO_2$; i.e., with $NaNO_2$ the Th stripping occurs to a greater extent.

Effect of H_2O_2 concentration. The effect of the H_2O_2 concentration on cerium stripping from the loaded TBP phase was studied in the concentration range 0.01–16.17 M, keeping the other conditions constant: contact time 5 min, O/A ratio 1/1, initial Th(IV) and Ce(IV) concentrations in the loaded organic phase about 2.528 and 1.88 g L^{-1}, respectively, 25 ± 2°C. The results are shown in Fig. 6. As can be seen, the highest Ce stripping efficiency and the highest β_{Ce} are reached at 1.1 M H_2O_2. As the H_2O_2 concentration is increased further, the stripping efficiency and β_{Ce} decrease. The results obtained agree with the data of [28].

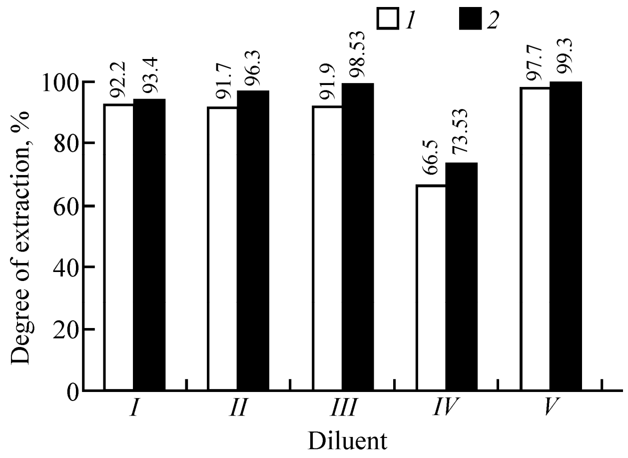

Fig. 4. Effect of diluent on the efficiency of the joint extraction of (*1*) Th and (*2*) Ce.

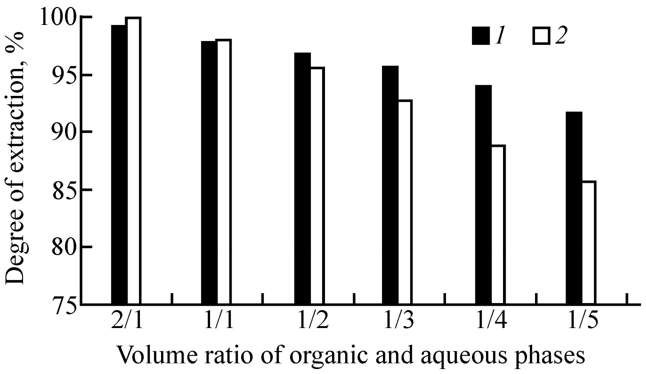

Fig. 5. Effect of the O/A ratio on the efficiency of the joint extraction of (*1*) Th and (*2*) Ce.

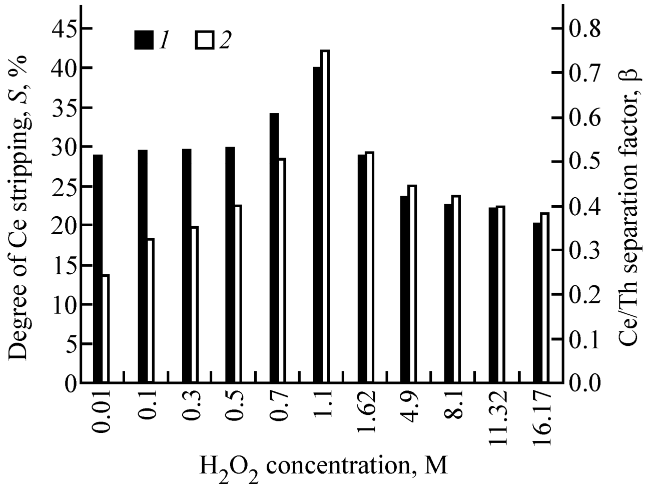

Fig. 6. Effect of stripping agent concentration on the (*1*) efficiency of cerium stripping from loaded TBP and (*2*) Ce/Th separation factor.

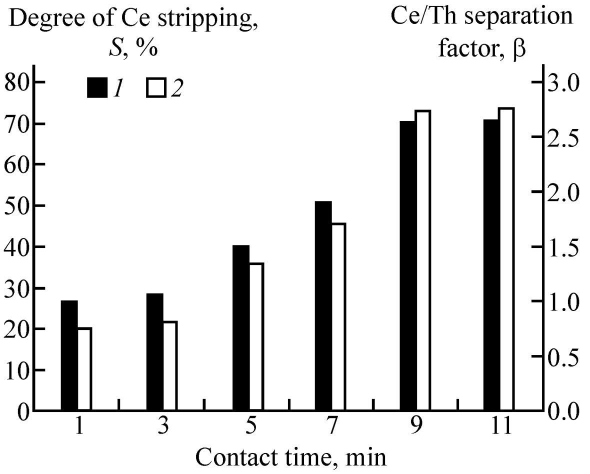

Fig. 7. Effect of contact time on the (*1*) efficiency of cerium stripping from loaded TBP and (*2*) Ce/Th separation factor.

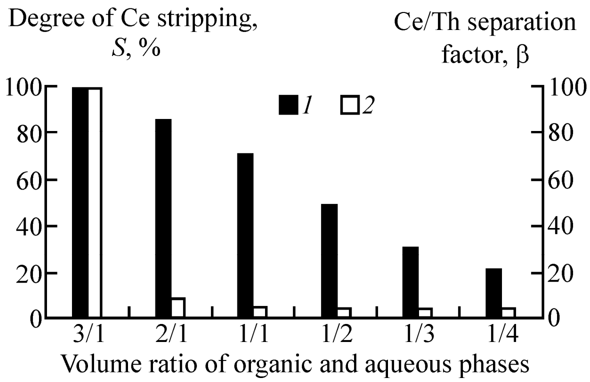

Fig. 8. Effect of the phase volume ratio on the (*1*) efficiency of cerium stripping from loaded TBP and (*2*) Ce/Th separation factor.

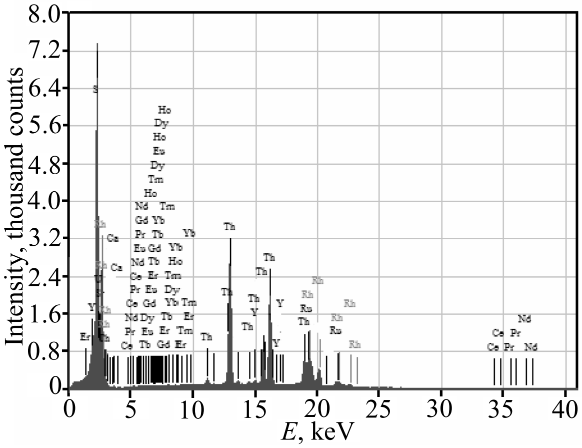

Fig. 9. XRF chart for cerium hydroxide precipitate.

Effect of contact time. The effect of the contact time on cerium stripping from the loaded TBP phase was studied in the range 1–15 min, keeping the other conditions constant: O/A ratio 1/1, initial Th(IV) and Ce(IV) concentrations in the loaded organic phase about 2.528 and 1.88 g L^{-1}, respectively, 1.1 M H_2O_2, 25 ± 2°C. As seen from Fig. 7, the cerium stripping efficiency and β_{Ce} reach a maximum at 10 min and then do not change significantly.

Effect of phase volume ratio (O/A). The effect of the O/A ratio was studied in the range from 3/1 to 1/4, keeping the other conditions constant: 10 min contact time, initial Th(IV) and Ce(IV) concentrations in the loaded organic phase about 2.528 and 1.88 g L^{-1}, respectively, 1.1 M H_2O_2, 25 ± 2°C. As seen from Fig. 8, the cerium stripping efficiency reaches a maximum value at O/A = 3/1.

Thorium stripping. After stripping cerium from the loaded TBP phase under the optimum conditions (10 min contact time, O/A = 3/1, H_2O_2 concentration 1.1 M, 25 ± 2°C), the organic phase was brought in contact with water for Th stripping. The optimum conditions for thorium stripping were as follows: 5 min contact time, O/A = 2/1, 25 ± 2°C.

Precipitation of Ce and Th

Cerium precipitation. After the stripping, the aqueous phase was boiled to remove H_2O_2. The resulting solution was adjusted to pH 1, 5 g L^{-1} EDTA was added to prevent the Th precipitation, and cerium hydroxide was precipitated with aqueous ammonia, filtered off, and washed with hot water until pH became 7. The precipitate was dried in an oven at 100°C for 1 h.

Thorium precipitation. Thorium was precipitated from aqueous phase after stripping with ammonium hydroxide at pH 3.5–4. The precipitate was filtered off, washed with hot water until pH became 7, and dried in an oven at 100°C for 1 h. The final products, Ce and Th hydroxides, were analyzed by XRF and ICP-OES. The results as given in Figs. 9 and 10 and in Tables 1 and 2.

Suggested Process Flowsheet

Monazite is initially digested with 300 mL of 80% sulfuric acid at 180°C for approximately 3 h. The resulting slurry is dissolved in 2 L of cold water with stirring for 1 h and then is filtered. The filtrate is ad-

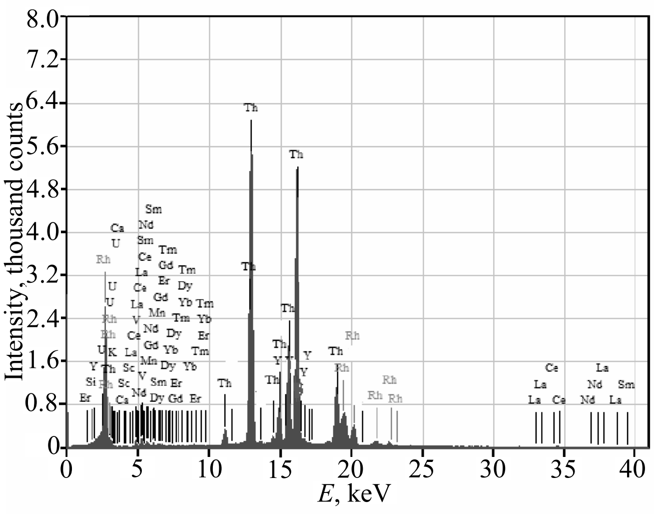

Fig. 10. XRF chart for thorium hydroxide precipitate.

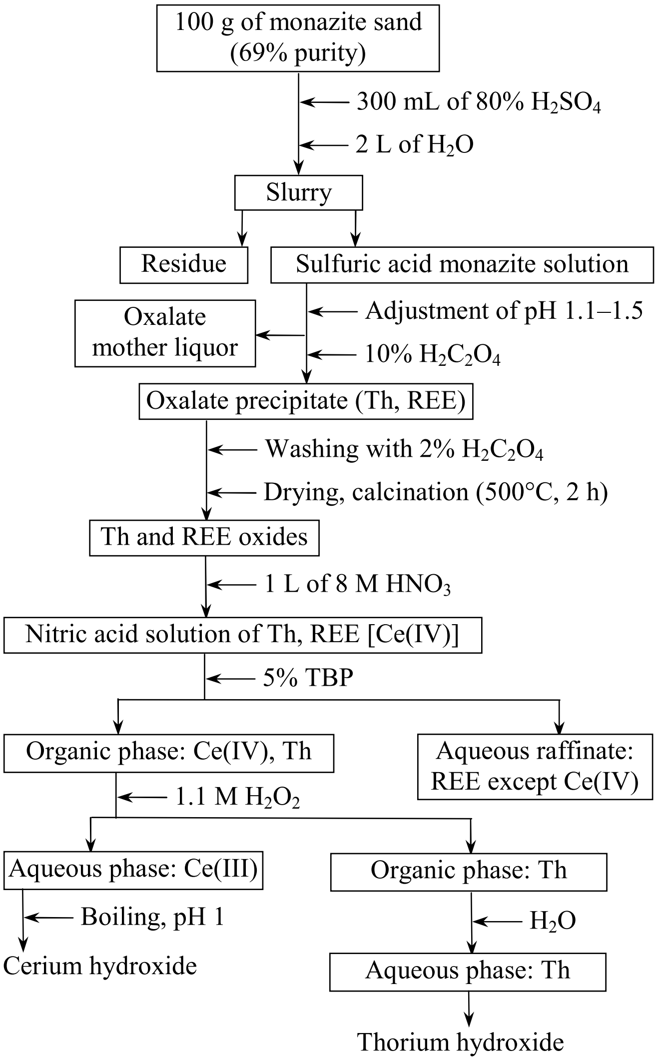

Fig. 11. Suggested flowsheet for the whole process.

justed to pH 1.1–1.5, and a 10% oxalic acid solution is added to precipitate both thorium and total rare earths as oxalate cake. After settling for 6 h, the precipitate is filtered off, washed with a 2% oxalic acid solution, and calcined at 500°C for 2 h. A portion of the calcined cake is dissolved in 1 L of 8 M HNO_3. A typical feed nitrate stock solution contains 0.2588% Th and 0.19% Ce(IV). Coextraction of Th and Ce(IV) is performed under optimum conditions: 5% TBP in kerosene, 5 min contact time, O/A = 1/1, 25 ± 2°C. Then, selective stripping of Ce is performed with a 1.1 M H_2O_2 solution (10 min contact time, O/A = 3/1, 25 ± 2°C). After that, Th is stripped with H_2O (5 min contact time, O/A = 2/1, 25 ± 2°C). The suggested flowsheet is shown in Fig. 11. The product purity is 87% for Th and 95% for Ce.

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