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Separation and Recovery of Rare Earths in Reciprocating Extraction Columns

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Separation and Recovery of Rare Earths in Reciprocating Extraction Columns

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Abstract: Multi-component rare earth (RE) separation (Nd group/Sm group) and recovery (Nd group) in sulfuric acid solution by di(2-ethyl-hexyl) phosphoric acid (D_2EHPA) dissolved in kerosene were investigated using two 3.7 cm inner diameter reciprocating extraction columns (RPCs) (height: 5.0–5.6 m) connected in sequence. The height equivalent to a theoretical stage (HETS) for Nd group/Sm group separation was in the range of 0.80–0.90 m and 0.66 m for extraction and scrubbing process, respectively. Under testing conditions, the concentration of Sm_2O_3 left in the raffinate (Nd group) was less than 0.1 wt% and the concentration of Eu_2O_3 in concentrated Sm Eu Gd oxides in the scrubbed organic phase was larger than 8.0 wt% (commercial grade). In the concentrating process of Nd group elements, the total RE concentration left in the raffinate was less than 0.1 g/L with a recovering efficiency more than 99%. The concentrated RE concentration was more than 250 g/L in the stripping raffinate and more than 14 times the feed concentration (17 g/L). The results show that the RPC has a great promise as an alternative of mixer-settlers for saving space, solvent input, and operating (energy) cost in rare earth extraction and separation using the $D_2EHPA/RE_2(SO_4)_3$ system.

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1. INTRODUCTION

Solvent extraction is one of the most effective separation methods for producing individual rare earth (RE) in high purity. In recent years, mono (2-ethyl-hexyl) 2-ethyl-hexyl phosphate (HEH[EHP]), di (2-ethyl-hexyl) phosphoric acid (D_2EHPA), and naphthenic acid (HA) have been widely used as extractants in industry for rare earth separations (1–3). Various processes have been developed based on these extractants, such as group element separations (Nd group/Sm group) and individual rare earth separation in high purity (Nd, Eu, Y) (1–3).

Compared to advanced process development, mixer-settlers are nearly the only type of extractors used for rare earth separation in industry (4). Easy to startup, stable operation, and less sensitive to the disturbance in operating and environmental conditions are the main advantages of mixer-settlers. However, a large solvent inventory, large space requirement, and slow response to the change in feed conditions are the main drawbacks of mixer-settlers. Considering the rapid development in process control to produce individual rare earth in high purity, it is a challenge for mixer-settlers to satisfy the requirement of fast response for process control. In order to overcome the disadvantages of mixer-settlers, extraction columns are considered to be potential alternatives. Space requirements are lower and the inventory of extractant per unit of throughput (an important contribution to capital cost) is also much less than in a mixer-settler. These advantages make extraction columns quite suitable for process control, which require a quick response to changes in operating and environmental conditions. Among the various types of extraction columns, reciprocating extraction column (RPC) is one type of continuous, differential extractor that has been widely used for extraction operations in different braches of industry (5–9). Until now, this type of extractor has been successfully used in hydrometallurgical processes for metal ion separations and nuclear spent-fuel reprocessing (10–13). Excellent performance was observed in these industries. Our previous studies also indicate that the RPC is a potential alternative for mixer-settlers for rare earth separations in the hydrochloric acid solution using HEH[EHP] as extractant (14).

The purpose of this study was to investigate the feasibility of RPCs for rare earth separation in the sulfuric acid solution using D_2EHPA as extractant and to obtain information of mass transfer characteristics. A typical characteristic of rare earths in the sulfuric acid solution is the relatively diluted concentration in feed (36–38 g/L), which will require much larger-scale extractors and solvent inventory, compared to the higher feed concentration (>200 g/L) in the HEH[EHP]/$RECl_3$ system. It is anticipated that the RPC would show significant advantages over mixer-settlers for rare earth separation in the sulfuric acid solution. This communication reports the results of the D_2EHPA (kerosene)/$RE_2(SO_4)_3$ system for Nd group/Sm group separation and Nd group elements extraction and concentration from the sulfuric acid solution into the hydrochloric acid solution in RPCs.

2. EXPERIMENT

Three experiments were conducted in this study: 1) single column extraction and scrubbing experiment to obtain the design parameter of the height equivalent to a theoretical stage (HETS) for Nd group/Sm group separation, 2) continuous Nd group/Sm group separation in two columns connected in sequence (one for extraction and the other for scrubbing), and 3) conversion and concentration of Nd group elements (diluted raffinate of Nd group/Sm group separation) from sulfuric solution to hydrochloric solution in two columns connected in sequence (one for extraction and the other for stripping).

In the present study with RPCs, the middle and heavier rare earths (Sm group: Sm, Eu, Gd, Y, and other heavier rare earths) were extracted from the sulfuric acid solution of rare earths mixture into the D_2EHPA dissolved in kerosene, and the light rare earths (Nd group: La, Ce, Pr, Nd) were left in the aqueous raffinate (15, 16). Single column extraction and scrubbing experiments were conducted to estimate the height equivalent to a theoretical stage (HETS) prior to the continuous extraction and scrubbing experiments using two columns connected in sequence. Mass transfer (extraction and scrubbing) experiments with RPCs were conducted under different operating conditions.

After Nd group/Sm group separation, the concentration of the light rare earths (Nd group) in the aqueous raffinate (after pH adjustment) is very low (usually 17–20 g/L) (15, 16), a very large-scale solvent extraction plant is needed for further separation of the light rare earths (La, Ce, Pr, Nd) into individual rare earth in high purity with this diluted feed. Consequently, a concentrating step is highly desirable to increase the feed concentration and thus reduce the scale of extractors. A conversion of Nd group elements in the aqueous raffinate from the sulfuric acid solution into the hydrochloric acid solution can reach this goal with D_2EHPA as extractant (15, 16). As the required theoretical stage number for this conversion process is small (four to five stages) (15, 16), continuous extraction and stripping experiments were directly conducted in two RPCs connected in sequence without single column experiments in advance.

The feed for experiments was from Baotou Rare Earths and Iron & Steel Company, P.R. China. Solids rare earths (mixed oxide compounds) were dissolved into distilled water with the addition of concentrated sulfuric acid to adjust pH ($[H^+] = 0.2\,N$) and to make a feed concentration of rare earth oxides (REO) 36–38 g/L prior to extraction/separation experiments.

Table 1. The relative amount of each rare earth element in feed solutions

	Feed for Nd group/Sm group separation	Feed for the conversion and concentration of Nd group element[a]
Total concentration of ΣREO (g/L)	36–38	17–20
Concentration of H^+ [N]	0.20	10^{-4}
REO wt%		
La_2O_3	28.38	28.24
CeO_2	49.28	50.68
Pr_6O_{11}	5.26	5.28
Nd_2O_3	15.03	15.80
Sm_2O_3	1.15	<0.1
Eu_2O_3	0.2	<0.1
Gd_2O_3	0.27	<0.1
Y_2O_3 + the others	0.43	<0.1

[a] The raffinate from Nd group/Sm group separation was diluted, after pH adjustment, as the feed for Nd group concentration from sulfuric rare earth to chloric rare earth.

Table 1 provides the relative concentration of each rare earth element in the feed determined by an atom scan 2000 inductively coupled plasma emission spectroscopy (ICPES) for RPC extractors.

Tables 2 and 3 list the process and operating conditions of Nd group/Sm group separation and the conversion and concentration of Nd group elements in two columns connected in sequence.

All reagents were technical grade. The total concentration of rare earths oxides (REO) was analyzed by complex titration with ethylenediaminetetraacetate (EDTA) standard solution. The concentration of each REO and element was determined by an X-ray fluorescence spectroscopy (XFS) and ICPES.

Figure 1 shows the flow sheet of the two RPCs connected in sequence for rare earths separation and concentration. The two columns are made of glass tubes with an inner diameter of 3.7 cm. The plate stack height of the two columns was 500 cm and 560 cm, respectively, and the plate stack contained the polyvinyl chloride (PVC) plates spaced at intervals of 5.0 cm. The hole diameter is 12 mm and the opening area is 58%. The design of the plate stack allowed the mounting of plates of various configurations on the central shaft and the variation of plate spacing. The continuous countercurrent separation experiments of extraction, scrubbing (Nd group/Sm group separation), or stripping (Nd group concentration) were carried out in the two RPCs connected in sequence with various experimental and operating conditions.

Organic phase (initially rare earth-free) was 33% v/v solution of D_2EHPA in kerosene for Nd group/Sm group separation, and 50% v/v solution of D_2EHPA in kerosene for the concentration of Nd group

Table 2. Process and operating conditions of Nd group/Sm group separation in two RPCs connected in sequence

	Feed		Scrubbing solution H_2SO_4	Ratio of flow rates O:F:S[a] (ml/min)	Operating conditions		
No. of run	REO (g/L)	H_2SO_4 (N)			Amplitude (cm)	Frequency (Hz)	Temperature (°C)
A	36.00	0.10	0.50	34 : 182 : 11.8	1.28	390	21 ± 1
B	37.00	0.10	0.49	32 : 189 : 12.0			19 ± 1
C	36.64	0.10	0.49	31 : 198 : 12.4			19 ± 1

[a] O:F:S = organic phase flow rate:feed flow rate:scrubbing solution flow rate.

Table 3. Process and operating conditions of Nd group elements concentration in two RPCs connected in sequence

Feed		Stripping solution	Ratio of flow rates	Operating conditions		
REO (g/L)	H^+ (N)	HCl (N)	O : F : S[a] (L/hr)	Amplitude (cm)	Frequency (Hz)	Temperature (°C)
16.9	10^{-4}	6.0	5 : 4.35 : 0.288	1.28	370	18 ± 1

[a] O:F:S = organic phase flow rate:feed flow rate:scrubbing solution flow rate. $[D_2EHPA] = 1.5M$.

elements from the raffinate of Nd group/Sm group separation (15, 16). In the beginning, the organic phase was fed into the bottom of the extraction column as a dispersed phase by the dosing pump. The dispersed phase was allowed to coalesce at an interface held about 20 cm above the plate stack, then the organic phase overflowed from the extraction column was transferred into the bottom of the scrubbing or stripping column as a dispersed phase by a centrifugal pump. The organic phase coalesced at the interface held above the plate stack of the scrubbing or stripping column and overflowed by gravity to the tank.

The aqueous feed and the scrubbing solution or the stripping solution were fed into an interface controller and then to the top of the columns. The flow rates of aqueous raffinate from extraction, scrubbing, or stripping column were controlled by dosing pumps. Experiments showed that the flow rate of organic phase from one column to the other column by the centrifugal pump was controlled steadily by the different pressure of the two sides of the centrifugal pump, when the two RPCs were operated under steady conditions.

During an operation, the organic phase was chosen as the dispersed phase, because it is desirable to have a low organic/aqueous flow ratio and a low inventory of expensive extractants in the column. The frequency and amplitude of operation were 5–7.3 Hz and 1.0–2.0 cm, respectively.

3. RESULTS AND DISCUSSION

3.1. Height Equivalent to a Theoretical Stage (HETS) for Nd Group/Sm Group Separation

During the operation of single column for extraction or scrubbing of Nd group/Sm group elements, the steady-state concentration profiles of rare earths were measured for both the aqueous phase (X_{Nd}, X_{Sm} vs. column height) and the organic phase (Y_{Nd}, Y_{Sm} vs. column height). Former studies

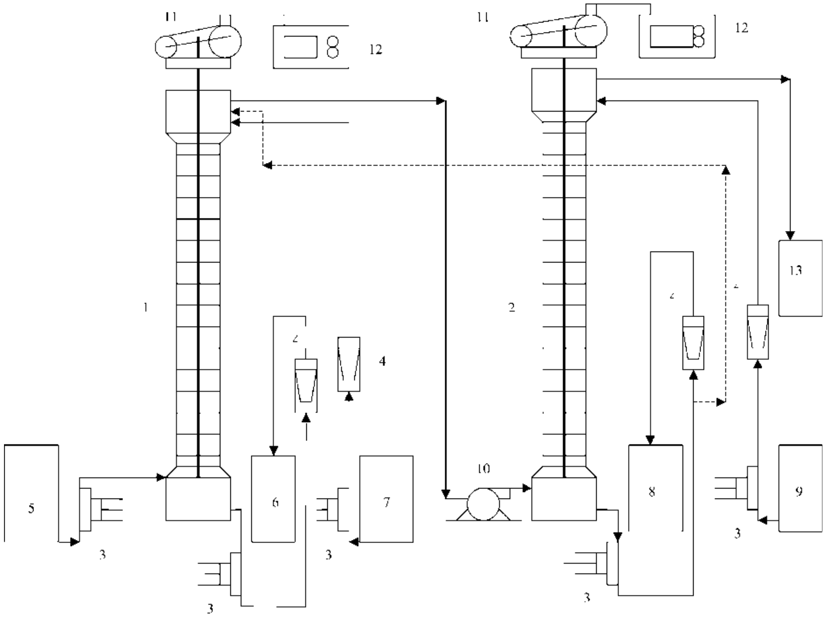

Figure 1. Schematic diagram of two RPCs connected in sequence for rare earth separation and concentration (dash line-scrubbing solution goes to the separation RPC 1 for Nd/Sm separation rather than raffinate tank 8). 1, extraction or separation RPC; 2, scrubbing or stripping RPC; 3, metering pump; 4, rotary flowmeter; 5, extractant tank; 6, raffinate tank of the extraction or separation RPC; 7, feed (rare earth aqueous solution) tank; 8, raffinate tank of the stripping RPC; 9, scrubbing (H_2SO_4) or stripping (HCl) solution; 10, centrifugal pump; 11, motor; 12, speed controller; 13, tank for outlet of the scrubbed organic phase.

(15, 16) indicated that Nd group/Sm group could be effectively separated using D_2EHPA as the extractant in sulfuric acid solution. The target products (Nd_2O_3 is less 0.1 wt% and Eu_2O_3 is larger than 8.0 wt% in the Sm group; Sm_2O_3 is less than 0.1 wt% in the Nd group) could be obtained using seven theoretical stages for extraction and nine theoretical stages for scrubbing. The separation factor ($\beta_{Sm/Nd}$) of Nd from Sm obtained from laboratory- and pilot-scale counter-current extraction and scrubbing experiments (15) was relatively stable, ranging from nine to 12 in the extraction section (seven stages) and from eight to 10 in the scrubbing section (nine stages) (15, 16). The average $\beta_{Sm/Nd}$ of Nd from Sm calculated from the average of individual $\beta_{Sm/Nd}$ in each stage was 10 in the extraction section and 9 in the scrubbing section (15, 16). According to the method of estimating the HETS for multi-component rare earth separations (14) (a plot of Y_{Sm}/Y_{Nd} and $\beta_{Sm/Nd}(X_{Sm}/X_{Nd})$ vs. column height as shown in Figs. 2 to 4), the equivalent theoretical stage number is 6.3 (Fig. 2), 7.0 (Fig. 3), and 8.5 (Fig. 4),

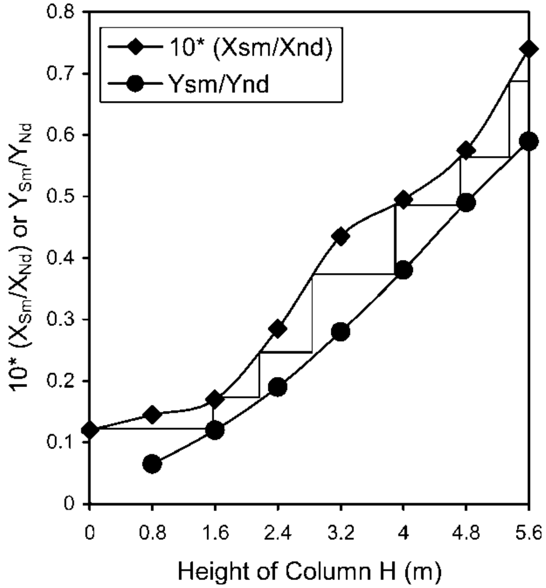

Figure 2. A plot of Y_{Sm}/Y_{Nd} and $\beta_{Sm/Nd}$ (X_{Sm}/X_{Nd}) vs. the height of extraction column for the estimation of HETS for Sm group/Nd group separation (extraction-1, $\beta_{Sm/Nd} = 10$).

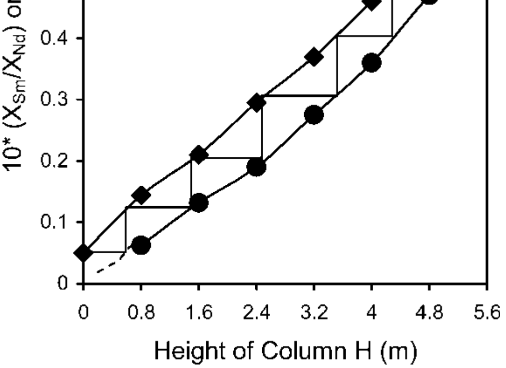

Figure 3. A plot of Y_{Sm}/Y_{Nd} and $\beta_{Sm/Nd}$ (X_{Sm}/X_{Nd}) vs. the height of extraction column for the estimation of HETS for Sm group/Nd group separation (extraction-2, $\beta_{Sm/Nd} = 10$).

respectively, which corresponds to an HETS of 0.90 m (extraction), 0.80 m (extraction), and 0.66 m (scrubbing) for critical component (Nd and Sm) separation. These HETS values were obtained at one column height of 5.6 m under different testing conditions and the concentrations (X_{Nd}, X_{Sm}, Y_{Nd}, Y_{Sm}) were measured simultaneously at different axial locations in the column.

The reported range of HETS (0.66–0.90 m) for Nd group/Sm group separation is consistent with these found in previous studies for rare earth separation (14), metal ion separation (10–12), and nuclear fuel reprocessing (13), implying similar mass transfer characteristics of the RPC for rare earth separation in the sulfuric acid solution and other systems (10–14) could be obtained. These obtained HETS values could be used to scale-up RPCs in pilot- and full-scale plants for rare earth extraction and separation.

3.2. Experimental Results of Continuous Experiments Using Two Connected Columns

Based on the experimental results of a single column for extraction and scrubbing, a continuous extraction unit incorporates two RPCs for both extraction/scrubbing, and extraction/stripping was constructed as shown in Fig. 1. Continuous extraction and scrubbing (Nd group/Sm group separation) or extraction and stripping (Nd group recovery) experiments were conducted using this unit with two RPCs connected in sequence. A series of studies were carried out at constant amplitude, frequency, and feed conditions being varied for Nd group/Sm group separation. As shown in Table 4, the relative amount of Sm_2O_3 left in raffinate was less than 0.1 wt%, and the relative amount of Eu_2O_3 in concentrated SmEuGd oxides (REO) was more than 8.0 wt% (runs B and C). These results indicate that a commercial grade of SmEuGd oxides with $Eu_2O_3 > 8.0$ wt% could be obtained under testing conditions. However, the Nd_2O_3 concentration in SmEuGd oxides was still high (about 7.8 wt%) and did not achieve at less than 0.1 wt%. This was mainly because the quantity of scrubbing solution used was not enough. A laboratory study (15) indicated that the purity of SmEuGd oxides is highly sensitive to the quantity of scrubbing solution used. A reduction of 10% of the desired quantity of scrubbing solution resulted in an increase in the Nd_2O_3 concentration in SmEuGd oxides from less than 0.1 wt% to 8.3 wt% (15). As shown in Table 4, an increase in the flow rate of scrubbing solution (Table 2) from run A to run C resulted in a decrease in Nd_2O_3 concentration in SmEuGd oxides from 14.3 wt% (run A) to 7.8 wt% (run C). It is anticipated that a further increase in the flow rate of scrubbing solution would produce a product of SmEuGd oxides with $Eu_2O_3 > 8.0$ wt% and $Nd_2O_3 < 0.1$ wt%. Unfortunately, no further experiment was conducted due to the limitation of feed solution. These results indicate that RPC is a potential alternative for mixer-settlers for Nd

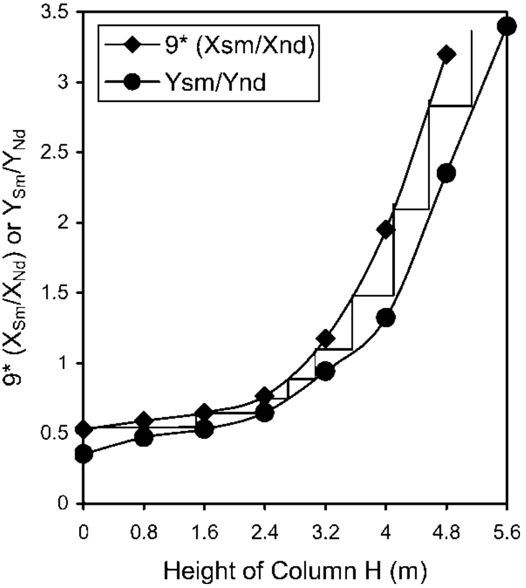

Figure 4. A plot of Y_{Sm}/Y_{Nd} and $\beta_{Sm/Nd}$ (X_{Sm}/X_{Nd}) vs. the height of scrubbing column for the estimation of HETS for Sm group/Nd group separation (scrubbing, $\beta_{Sm/Nd} = 9$).

group/Sm group separation in the sulfuric acid solution to produce commercial grade SmEuGd oxides.

Continuous extraction and stripping experiments of Nd group elements were conducted in the same unit for Nd group/Sm group separation. As shown in Table 5, the start-up process took about 24 hr to reach a steady-state operation. Under steady-state operation, the total concentration of RE left in raffinate was less than 0.1 g/L, which indicated a recovery efficiency of >99%. The stripping raffinate contained more 254 g/L, which was more than 14 times concentrated than the feed (16.9 g/L). The steady concentration profiles of rare earths and hydrogen ion are shown in Fig. 5. A significant amount of REO in organic phase was stripped off in the top section of the stripping column, due to the fact that the highest concentration of stripping solution [(HCl) = 6 N] on the top of the stripping column.

3.3. Scale-Up and Comparison Between RPCs and Multistage Mixer-Settlers for Rare Earths Separation and Concentration

Based on the process conditions given in former studies (15, 16) and the HETS data obtained from this study, a preliminary design of RPCs for rare earth separation and concentration was conducted.

Table 4. Experimental results of reciprocating plate extraction columns for Nd group/Sm group separation

		Run A			Run B			Run C		
		Feed	Raffinate	Outlet of scrubbed organic phase	Feed	Raffinate	Outlet of scrubbed organic phase	Feed	Raffinate	Outlet of scrubbed organic phase
3REO (g/L)		36.00	33.10	5.80	37.00	34.84	5.65	36.00	34.56	5.32
H^+ (N)		0.20	0.275	—	0.20	0.265	—	0.20	0.260	—
	La_2O_3	28.38	29.45	0.71	28.45	28.24	0.42	28.45	28.81	0.22
	CeO_2	49.28	50.00	7.37	49.38	50.68	5.39	49.38	50.18	4.02
	Pr_6O_{11}	5.26	5.19	1.88	5.20	5.28	1.37	5.20	5.19	3.37
	Nd_2O_3	15.03	15.34	14.57	14.89	15.80	9.86	14.89	15.81	7.82
	Sm_2O_3	1.13	<0.10	44.21	1.07	<0.10	42.71	1.07	<0.1	41.52
	Eu_2O_3	0.20	<0.10	7.35	0.20	<0.10	8.46	0.20	<0.10	8.87
	Gd_2O_3	0.27	<0.10	14.63	0.27	<0.10	15.30	0.27	<0.10	16.29
REO wt%	Tb_2O_3	<0.10	<0.10	0.90	<0.10	<0.10	1.11	<0.10	<0.10	1.20
	Dy_2O_3	#0.10	<0.10	2.06	#0.10	<0.10	3.31	#0.10	<0.10	3.48
	Ho_2O_3	<0.10	<0.10	0.15	<0.10	<0.10	0.34	<0.10	<0.10	0.38
	Er_2O_3	#0.10	<0.1	<0.1	#0.10	<0.10	0.24	#0.10	<0.10	0.30
	Tm_2O_3	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10
	Yb_2O_3	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10
	Lu_2O_3	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10
	Y_2O_3	0.23	<0.10	6.17	0.23	<0.10	11.43	0.23	<0.10	12.46
Total rare earths 3REO %		96.56	96.80	96.87	96.93	97.50	99.80	96.93	92.20	99.28

Table 5. Experimental results of Nd group elements conversion and concentration from sulfuric acid solution into hydrochloric cid solution (two RPCs connected in sequence)

	Extraction column		Stripping column	
Experimental time (hr)	Total REO in extraction raffinate (g/L)	$[H^+]$ in extraction raffinate (N)	Total REO in stripping raffinate (g/L)	$[H^+]$ in stripping raffinate (N)
0.5	7.70	0.20	N/A	N/A
1.5	0.78	0.26	146.0	2.57
5	0.35	0.29	173.0	1.94
8	0.15	0.30	227.7	0.73
14	0.1	0.31	231.6	0.35
19	<0.1	0.31	249.0	0.32
24	<0.1	0.31	251.0	0.31
26	<0.1	0.31	253.0	0.30
28	<0.1	0.31	254.0	0.30

For a plant with a treatment capacity of 800 T REO/year (operation: 300 days) using a $D_2EHPA/RE_2(SO_4)_3$ system, based on the design flow-velocities as shown in Table 6, the diameters of RPCs required to handle the flow rates of aqueous solution and organic phase for an 800 T REO/year

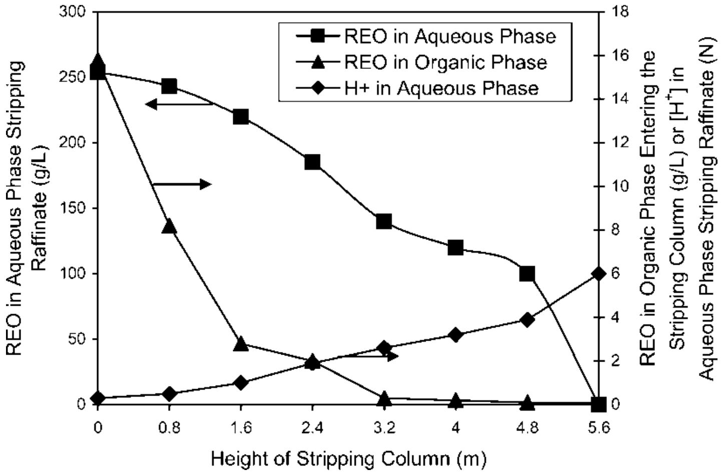

Figure 5. Steady concentration profiles of rare earths in both organic and aqueous phase and hydrogen ion in aqueous phase during the stripping process for Nd concentration operation.

Table 6. Design parameters of RPCs for Nd group/Sm group separation and Nd group concentration for an 800T REO/year plant

Item	Column diameter[a] (cm)	HETS[b] (m)	Required Theoretical Stages[c]	Column height[d] (m)
Nd group/Sm group separation				
Extraction	57	2.54	7	17.8
Scrubbing	40	1.63	9	14.7
Stripping[e]	40	2.47	9	22.2
Nd group concentration				
Extraction[e]	104	2.49	6	14.9
Stripping[e]	100	3.50	4	14.0

[a] The total flow velocities of both phases used for column diameter design were 0.4 cm/s, 0.16 cm/s, and 0.12 cm/s for extraction, scrubbing, and stripping, respectively, for Nd group/Sm group separation and 0.4 cm/s and 0.25 cm/s for extraction and stripping, respectively, for Nd group concentration (the total flow velocities used for column diameter design were determined by hydrodynamic experiments in a separated study).

[b] Scale-up of HETS in terms of column diameter was based on Eq. (1).

[c] The numbers of required theoretical stages were from References 15 and 16.

[d] Column height = HETS × The number of required theoretical stage.

[e] HETS estimated from the 3.7 cm diameter RPCs experiments was 1.0 m for stripping in Nd group/Sm group separation, 0.7 m and 1.0 m for extraction and stripping, respectively in Nd group concentration.

plant are 57 cm, 40 cm, and 40 cm for extraction, scrubbing, and stripping, respectively, for Nd group/Sm group separation, and 104 cm and 100 cm for extraction and stripping, respectively, for Nd group concentration and conversion.

According to Karr's empirical Equation (9), the relationship between the HETS and column diameter (D_i) can be expressed as:

$$(\mathrm{HETS})_{D2}/(\mathrm{HETS})_{D1} = (D_2/D_1)^{0.38} \tag{1}$$

where D_i is the diameter of column i (i = 1,2) and $(\mathrm{HETS})_{Di}$ is the HETS of column at a diameter of D_i.

The HETS values for larger columns calculated from Eq. (1) are 2.54 m (D = 50 cm), 1.63 m (D = 40 cm), and 2.47 m (D = 40 cm) for extraction, scrubbing, and stripping, respectively, for Nd group/Sm group separation, and 2.49 m (D = 104 cm) and 3.50 m (D = 100 cm) for extraction and stripping, respectively, for Nd group concentration and conversion. Consequently, based on the required number of theoretical stages for desired separation obtained from previous studies (15, 16), the required column heights for an 800T REO/year plant are 17.8 m, 14.7 m, and 22.2 m for

Table 7. Comparison of solvent input between RPCs and mixer-settlers for an 800 T REO/year plant

	RPCs[a]		Mixer-settlers (MS)[b]		Compared to MS, the quantity saved by RPCs	
Item	Total RPC volume (m^3)	Total solvent input (m^3)	Total MS volume (m^3)	Total solvent input (m^3)	Total volume	Total solvent input
Nd group/Sm group separation	9.18	6.18	27.2	40.8	66%	85%
Nd group concentration	22.8	17.1	45.6	68.4	50%	75%

[a] The hold-up of dispersed (organic) phase was assumed at 25% in RPCs. The total solvent input was assumed at three times of the solvent in RPCs.

[b] The total volumes of mixer-settlers were from an 800 T REO/year plant in operation. The hold-up of dispersed (organic) phase was assumed at 50% in mixer-settlers. The total solvent input was assumed at three times of the solvent in mixer-settlers.

extraction, scrubbing, and stripping, respectively, for Nd group/Sm group separation, and 14.9 m and 14.0 m for extraction and stripping, respectively, for Nd group concentration and conversion.

Based on the design parameters shown in Table 6 and operating data of an 800T REO/year full-scale plant using multistage mixer-settlers, a comparison in the total solvent input for Nd group/Sm group separation and Nd group concentration and conversion between RPCs and multistage mixer-settlers is shown in Table 7. The results indicate that the use of RPCs to replace mixer-settlers can save 85% and 75% solvent input in the Nd group/Sm group separation and Nd group concentration and conversion section, respectively, for an 800T REO/year plant.

4. CONCLUSIONS

Separation (extraction and scrubbing) and recovery/concentration (extraction and stripping) of rare earths were performed for the $D_2EHPA/RE_2(SO_4)_3$ system in single or two RPCs connected in sequence. The results indicate that the HETS was about 0.80–0.90 m and 0.66 m for Nd group/Sm group extraction and scrubbing, respectively.

Under tested conditions, the relative amount of Eu_2O_3 in the concentrated Sm-Eu-Gd oxide compounds was more than 8.0 wt% (commercial grade) from Nd group/Sm group separation. The relative amount of Sm_2O_3 in raffinate was less than 0.1 wt%. These results indicate that the application of RPC for Nd group/Sm group separation could obtain a commercial product of mixed Sm-Eu-Gd oxides.

The Nd group elements in raffinate of Nd group/Sm group separation could be concentrated from 16.9 g/L (sulfuric form) to more than 250 g/L (chloride form) in stripping raffinate using a RPC. The total concentration was less than 0.1 g/L in the extraction raffinate with a recovery efficiency larger than 99%.

Preliminary design of RPCs for an 800 T REO/year plant indicates that the use of RPCs to replace mixer-settlers can save 50–66% space and 75–85% total solvent input.

NOMENCLATURE

D	Diameter of column (cm)
HETS	Height equivalent to a theoretical stage (m)
X_{Nd}	Nd concentration in the aqueous phase (g/L)
X_{Sm}	Sm concentration in the aqueous phase (g/L)
Y_{Nd}	Nd concentration in the organic phase (g/L)
Y_{Sm}	Sm concentration in the organic phase (g/L)

Greek Letter

$\beta_{Sm/Nd}$	Separation factor of Sm relative to Nd $[= (Y_{Sm}/Y_{Nd})/(X_{Sm}/X_{Nd})]$

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