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Separation of yttrium from rare earth using hollow fiber-supported liquid membrane: factorial design analysis

Naphaphan Kunthakudee[a], Niti Sunsandee[b], Ura Pancharoen[c], Prakorn Ramakul[a,*]

[a]*Faculty of Engineering and Industrial Technology, Department of Chemical Engineering, Silpakorn University, Nakhon Pathom 73000, Thailand, Tel. +66 844595229; email: phan_6_2@hotmail.com (N. Kunthakudee), Tel. +66 816822105; Fax: +66 34219368; email: prakorn@su.ac.th (P. Ramakul)*
[b]*Government Pharmaceutical Organization, Ratchathewi, Bangkok 10400, Thailand, Tel. +66 898953231; email: n_rx@hotmail.com*
[c]*Faculty of Engineering, Department of Chemical Engineering, Chulalongkorn University, Patumwan, Bangkok 10330, Thailand, Tel. +66 22186891; email: ura.p@chula.ac.th*

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ABSTRACT

The selective separation of yttrium ions from other rare earth elements using a hollow fiber-supported liquid membrane was performed. The extractant was Cyanex272 and the stripping solution was nitric acid. Yttrium was extracted preferentially in comparison to other rare earth elements. The pH and flow rate of the feed solution, as well as the concentration of Cyanex272, were regarded as factors in the experiments. A 2^3 factorial design was used to determine the significant factor, their interaction, and optimized condition. Linear regression model and surface response were applied to predict the percentages of extraction of yttrium at different values of significant factors. The validity of linear regression model was evaluated through a comparison with experimental data, with good agreement achieved.

Keywords: Yttrium; Rare earth; Hollow fiber; Liquid membrane; Factorial design

1. Introduction

Yttrium is found in most monazite ore, as well as some rare earth elements. It is used in super alloys of nickel and cobalt as well as solid oxide fuel cells. Yttrium oxide has a high melting point, with its greatest use for advanced ceramics, such as yttrium-stabilized zirconium. It is also used in electronic ceramics as an oxygen sensor. Compounds of yttrium are used as catalysts and electrolytes in solid oxide fuel cells [1,2]. Unfortunately, yttrium and rare earth elements co-exist with very low concentrations. For this reason, a number of hydrometallurgical processes for the separation of yttrium from rare earth have been investigated during recent decades. Liquid–liquid extraction is an effective method to extract and separate rare earth on an industrial scale, but there are disadvantages because a large number of stages are required [3]. Thus, the development of a more efficient separation process is necessary.

In recent years, the separation of ions with very low concentration has focused on liquid membrane (LM) techniques. Particularly, hollow fiber-supported liquid membrane (HFSLM) has proven to be excellent for extracting metal ions from a dilute solution, even at ppm levels. A HFSLM can carry out simultaneous extraction and stripping processes in one stage. This

*Corresponding author.

benefits a non-equilibrium mass transfer and up-hill effect, where the solute can move from low to high concentration solutions [4]. The most interesting case arises when this solubility is controlled by a chemical reaction between the transported solute and the extractant-carrier molecule to form a solute-carrier complex. It is possible to attain high selectivity and high efficiency for low metal concentration [5,6]. HFSLM has a high surface area per unit volume for mass transfer [7]. Therefore, these advantages of a hollow fiber contactor are suitable for application in the separation of yttrium from rare earth elements.

This study was developed from several previous researches in yttrium separation. Zhang et al. [8] studied the extraction of yttrium from chloride medium with synergistic extraction of HEHEHP and Cyanex272. The extraction mechanism was determined and a high yield of yttrium was obtained. However, yttrium was the only element in the feed solution. Therefore, the selectivity was not present. Moreover, the extraction mechanism can only be utilized for laboratory purposes and cannot be scaled up to industrial level. Yanliang et al. [9] studied the separation of yttrium from other rare earth by solvent extraction using a mixture of sec-octylphenoxy acetic acid and Cyanex272. High selectivity of yttrium was obtained. However, the solvent extraction consumed a high level of extractant and yttrium could not be recycled into strip phase. Ramakul et al. [10] studied the separation of yttrium from mixed rare earth by hollow fiber-supported LM. However, selectivity was not high enough because a one-factor-at-a-time (OFAT) experiment was performed. The factorial design technique [11,12], which is widely used in the statistical planning of experiments to obtain the main factor and optimized condition, was applied for this study. It can investigate the influence of factors in experiments, assess its individual effect as an isolated factor and its interaction with other factors, and determine which variables could be neglected. Kavak et al. [13] used full factorial design experiments to screen the factors affecting lead removal efficiency using cation-exchange resin. The percentage removal of lead was examined by varying the experimental conditions with center points. The best condition, in terms of maximizing the removal of lead (II) ions, was achieved with an optimization procedure. Emídio et al. [14] used a 2^{5-1} fractional factorial design for screening and a central composite design for optimization of significant variables in hollow fiber-based liquid-phase microextraction and gas chromatography–tandem mass spectrometry for the determination of Δ^3 tetrahydrocannabinol, cannabidiol, and cannabinol in samples of human hair. Chang et al. [15] used a 2^{6-1} fractional factorial design to identify the factors that provide significant effects on the efficiency of Cu(II) extraction from aqueous solutions. The results showed that a concentration of D2EHPA, pH, and their interaction influenced the percentage of Cu(II) extraction. In previous work by Leepipatpaiboon et al. [16], a 3^2 factorial design was applied for the optimization method in the extraction and stripping of uranium (VI) from other impure elements. The concentration of nitric acid in the feed solution and the concentration of TBP in the LM were regarded as factors for optimization. Kavak [17], Öztürk and Kavak [18,19] applied a 2^3 full factorial design to batch adsorption and process removal of boron from aqueous solutions. The results showed that the temperature, pH, and mass of adsorbent affected boron removal by adsorption and optimum conditions were found.

In this work, a 2^3 full factorial design was applied to the separation of yttrium and rare earth from aqueous solutions by hollow fiber-supported LM. A two-level full factorial design was used to determine the prediction of %Ex of yttrium ion and to examine the effects of main parameters and their interactions on the extraction of yttrium. In the factorial design technique, the output parameter or response is %Ex of yttrium and the input is the process parameters, namely the pH of feed solution, concentration of Cyanex272, and the flow rate of feed solution. Consequently, the response surface and linear regression models correlate with the significant factors that were developed. The equation was used to predict the percentages of yttrium extraction at different values of significant factors and its validity was examined.

2. Background theory

2.1. Hollow fiber-supported LM

A hollow fiber-supported LM is composed of a module, which contains many hollow microporous polyethylene fibers, as shown in Fig. 1. The fibers are woven into fabric and wrapped around a central tube feeder that supplies the shell-side fluid. The woven fabric allows more uniform fiber spacing, which in turn leads to a higher rate of mass transfer than that obtained with individual fibers. Inside the module, the organic LM phase is located between the aqueous feed solution, which contains metal ions and the stripping phase.

In a HFSLM, the Cyanex272 extractant was dissolved in kerosene to generate an organic solution, which was embedded in the hydrophobic micropores

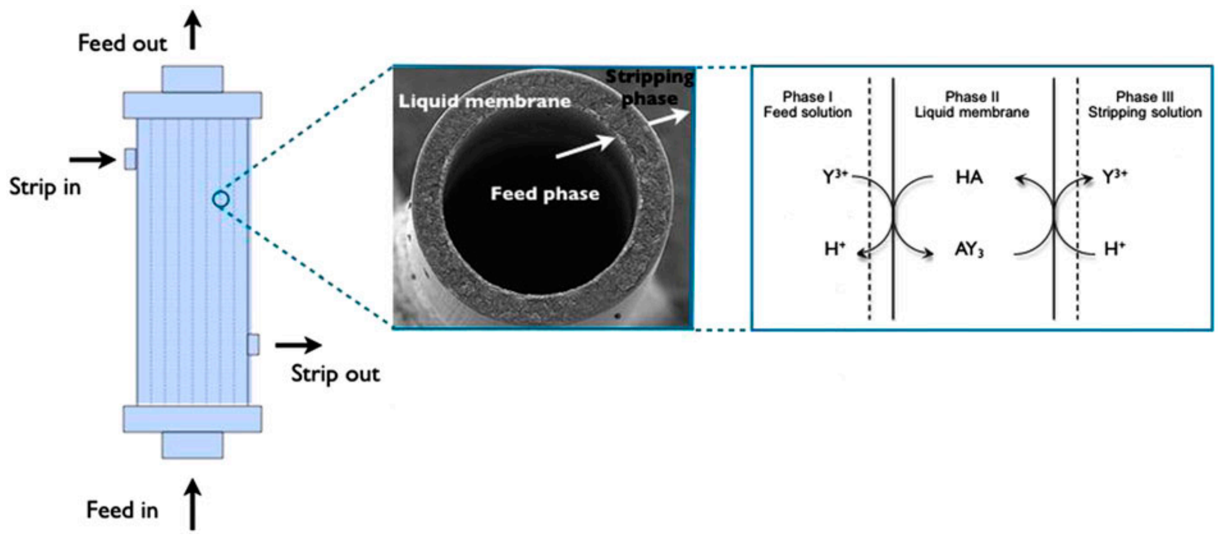

Fig. 1. Transport scheme of extraction and stripping of yttrium in a LM process using Cyanex272.

of the hollow fiber module. The feed solution contained rare earth (yttrium, cerium, lanthanum, neodymium, and praseodymium). The stripping solution was 0.5 M nitric acid solution. The feed solution was pumped into the inner side, while the stripping solution was simultaneously pumped into the outer side of each hollow fiber in the system. The transport mechanism of yttrium ions in the microporous hollow fiber is presented in Fig. 1. Yttrium ions in the feed solution were in the form of Y^{3+} and reacted with Cyanex272 (HA), occurring in complex species form following Eq. (1) [10,20].

$$Y^{3+} + \overline{3HA} \leftrightarrow \overline{AY_3} + 3H^+ \quad (1)$$

The complex species of yttrium AY_3 in the LM phase permeated from the feed solution–LM interface through the LM-stripping solution interface, with the concentration gradient as a driving force. At the LM-stripping interface, AY_3 complex species reacted with H^+ ions, as in Eq. (2), and transferred to stripping phase.

$$3H^+ + AY_3 \leftrightarrow Y^{3+} + \overline{3HA} \quad (2)$$

2.2. Factorial design study

The determination of significant factors and the interaction between factors studied are very important for the separation of yttrium from rare earth solutions by hollow fiber-supported LM. In this work, a 2^3 factorial design was used by varying three important factors: the pH of feed solution, concentration of Cyanex272, and the flow rate of feed solution. It should be noted that the low and high level of these factors were designated as minus (−1) and plus (+1), respectively [21]. The symbols of categorical factor levels and the responses were tabulated in Table 1. The response was the %Ex of yttrium. Minitab 16.0 software was used to calculate statistical data, analysis of variance and graphs of the 2^3 factorial design.

3. Experimental

3.1. Reagents

Rare earth solution, from the Rare Earth Research and Development Center, Office of Atoms for Peace, Bangkok, Thailand, was supplied as a raw material for the experiments. The concentrations of yttrium and other rare earth are shown in Table 2. The pH of feed

Table 1
Factors and factorial design levels

Factor	Low level (−1)	High level (+1)	Unit
pH of feed solution (A)	1	5	–
Conc. of Cyanex272 (B)	0.2	0.5	molar
Flow rate of feed solution (C)	100	400	mL/min

Table 2
The concentration of rare earth elements in feed solution

Elements	Y	La	Ce	Pr	Nd
Concentration (ppm)	156	350	150	250	410

solutions was adjusted using nitric acid and sodium hydroxide. Cyanex272 was supplied by Cytec Industries Inc. The organic diluent was kerosene and the stripping solution was nitric acid. All chemicals, except the feed solution, were of AR grade and purchased from Merck Co. Ltd.

3.2. Apparatus

(1) A Liqui-Cel® Laboratory Liquid/Liquid Extraction System (composed of two gear pumps, two variable speed controllers, two rotameters, and two pressure gages) was used.
(2) A Liqui-Cel® Extra-Flow module, offered by Celgard (Charlotte, NC; formerly Hoechst Celanese), was used as a support material.
(3) Concentrations of yttrium and other rare earth elements were measured by Inductivity Coupled Plasma-Atomic Emission Spectrometry, ICP-AES (Optima 4300 DV, Perkin-Elmer).

3.3. Procedures

The LM was prepared by dissolving Cyanex272 in kerosene. The stripping solution was nitric acid. The organic LM contained Cyanex272 circulated in a tube and shell sides for 20 min. Subsequently, the experiment began by pouring the feed solution into the tube side of the hollow fiber module. Simultaneously, 0.5 M of nitric acid, as the stripping solution, was counter-currently pumped into the shell side of the hollow fiber module. The experiments were performed in a once-through mode. A schematic diagram of the actual process and a photograph of the instrument used are shown in Fig. 2. Samples (10 mL) were taken out at final times from the raffinate and stripping tanks; the concentrations of yttrium and rare earth ions were measured using the ICP-AES. Three important factors, pH in the feed solution, concentration of

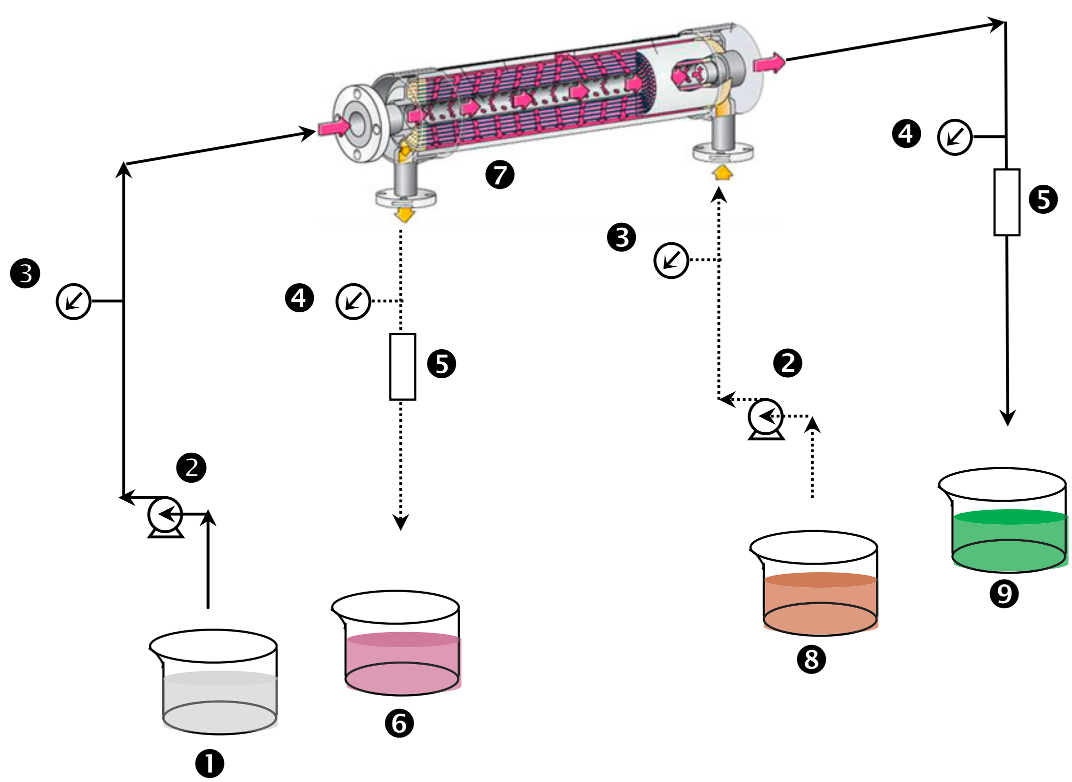

Fig. 2. Schematic counter-current flow diagram for one-through-mode operation in a HFSLM. 1. inlet feed solution reservoir, 2. gear pump, 3. inlet pressure gages, 4. outlet pressure gages, 5. flow meters, 6. outlet stripping solution reservoir, 7. hollow fiber module, 8. inlet stripping solution reservoir, and 9. outlet feed solution.

Cyanex272 in the LM, and flow rate of feed solution were examined and analyzed according to a 2^3 full factorial design. All experiments were done in triplicate.

4. Results and discussion

The extractabilities of rare earth ions by Cyanex272 with a pH feed solution of 5.0 are shown in Fig. 3. When Cyanex272 concentration was increased from 0.2 to 0.6 M, the extractability of yttrium and other rare earth ions also increased. This can be explained by Le Chatelier's Principle. When the concentration of Cyanex272 is increased, yttrium ion reaction increased and extracted into LM phase by Eq. (2). However, when the Cyanex272 concentration exceeded 0.6 M, extractability decreased because of the increase in LM viscosity as the extractant concentration increases. Higher viscosity generates a lower diffusion speed of the species and affects the mass transfer process [22,23].

According to selectivity, it is evident that yttrium ions were extracted more by Cyanex272 than other rare earth ions because yttrium has the smallest ionic radius and a very different atomic number of 39 compared to other rare earth elements of 57–63. The equilibrium constant of the extraction reaction of rare earth elements decreases as the ionic radius increases [24]. The ionic numbers of the lanthanide series are shown in Table 3. All of these results also occur in the stripping phase and other unextracted rare earth ions (La, Ce, Nd, Pr, Sm, Gd, and Dy) were rejected into the raffinate. Because high selectivity of yttrium occurred, further investigations, in Sections 4.1 and 4.2, were performed by focusing on the %Ex of yttrium ions.

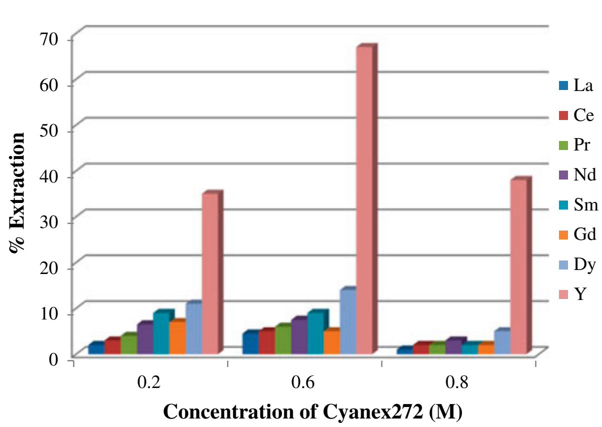

Fig. 3. Extractabilities of rare earth ions by Cyanex272.

Table 3
Atomic number and ionic radius of some rare earths in lanthanide series [17]

Elements	Y	La	Ce	Pr	Nd	Sm	Eu
Atomic number	39	57	58	59	60	62	63
Ionic radius (Å)	104	117	115	113	112.3	110	108

4.1. Factorial design analysis

Based on previous researches [10,25], the pH of feed solution, concentration of Cyanex272 in LM, and flow rate of feed solution were chosen as the main factors because they affect the extraction reaction in a hollow fiber module.

The results of three individual effects are shown in Fig. 4(a). It is evident that increasing pH in feed solution (A) or concentration of Cyanex272 in LM (B) results in positive effect on the %Ex of yttrium. Conversely, a negative effect on the %Ex of yttrium was obtained when the flow rate of feed solution (C) increases. The interactions between the two factors are shown in Fig. 4(b). The data of full 2^3 factorial design and their responses are tabulated in Table 4.

According to the Pareto Chart in Fig. 5, it is apparent that the effect of factors on the %Ex of yttrium was in the following order: B > C > A > BC > AB > AC > ABC. Where A, B, and C are concentrations of Cyanex272 in the LM, pH in the feed solution, and flow rate of the feed solution, respectively, AB, AC, and ABC are the interactions between them. Minitab 16 software also calculated the coefficients that were used in the regression equation with the following expression:

$$\begin{aligned}\%\text{Y Ex} = {} & 33.2 + 6.33\text{A} + 10.6\text{B} - 9.54\text{C} + 1.67\text{AB} \\ & - 0.792\text{AC} - 3.08\text{BC} - 0.333\text{ABC}\end{aligned} \tag{3}$$

The magnitude of coefficients represents the strength of the effect and the sign represents the way that it responds to the %Ex of yttrium. From the analysis of variance (ANOVA) in Table 5, the p-value of AC and ABC are higher than 0.05. This means that these interactions are not significant with 95% confidence. Thus, AC and ABC terms can be removed from the regression model, and the regression model that predicts % Ex of yttrium was changed as follows:

$$\begin{aligned}\%\text{Y Ex} = {} & 33.2 + 6.33\text{A} + 10.6\text{B} - 9.54\text{C} + 1.67\text{AB} \\ & - 3.08\text{BC}\end{aligned} \tag{4}$$

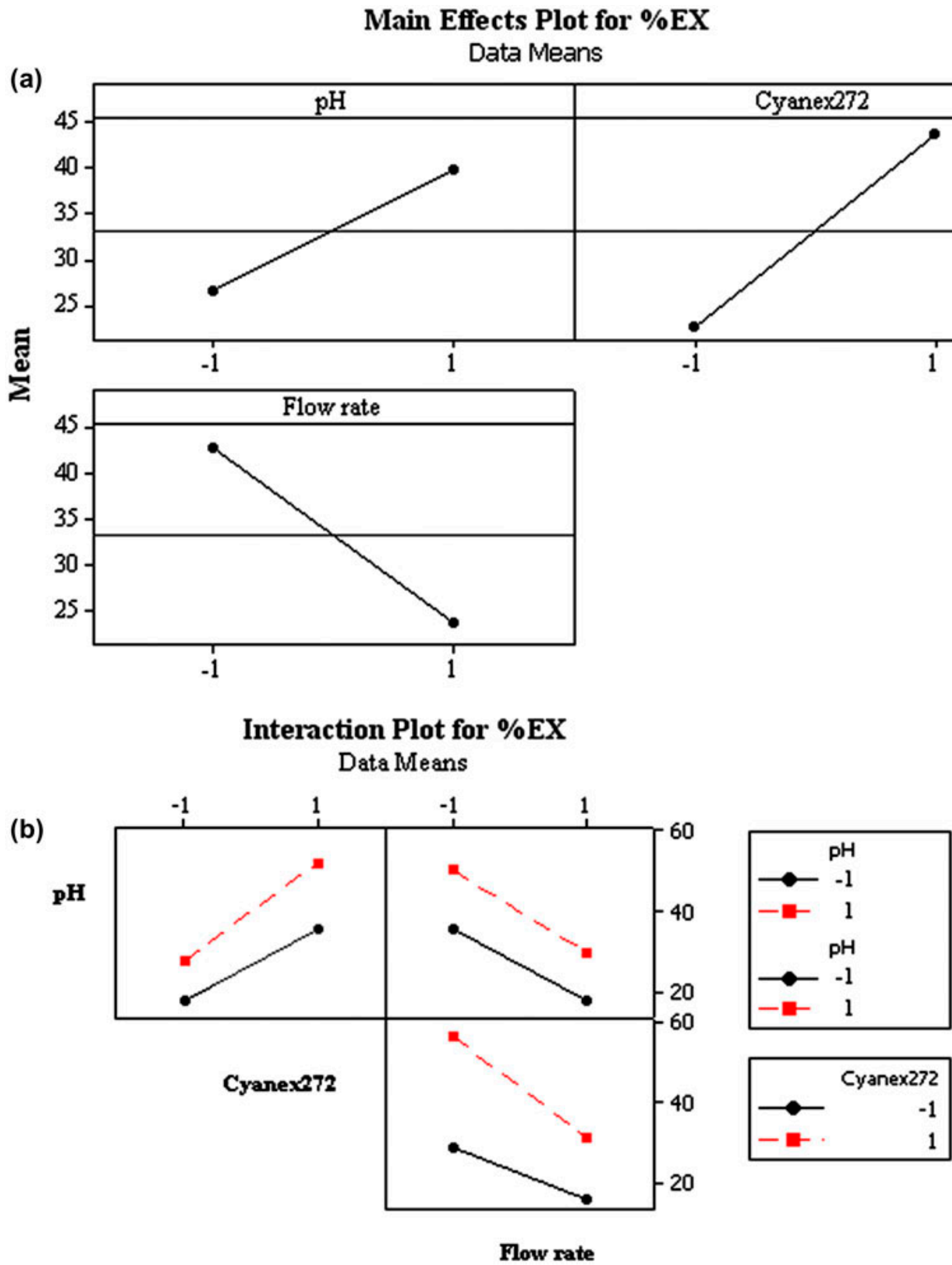

Fig. 4. Plot of the effects of pH, concentration of Cyanex272, and flow rate on the extraction of yttrium by 2^3 factorial designs. (a) The main effect plots and (b) the interaction plots.

4.2. Results of experiments, response surface, and regression model verification

This section describes the effects of (i) pH in the feed solution, (ii) concentration of Cyanex272 in the LM, and (iii) flow rate of feed solution on %Ex of yttrium ion through LM. The response surfaces were performed and the results of experiments were compared with predicted values from the regression model in Eq. (4).

4.2.1. Effect of pH of the feed solution

Before the pH of the feed solution was examined, the response surface was generated by regression model in Eq. (4). It was plotted by varying Cyanex272 and flow rate, while the pH of the feed solution was fixed at zero, as shown in Fig. 6. The optimized condition is a flow rate of −1 (100 mL/min) and a concentration of Cyanex272 of 1 (0.5 M). Consequently, the optimized condition was fixed and the experiments

Table 4
2^3 Factorial design factors and the responses

	Factors			Responses		
Condition no.	A	B	C	Rep. 1	Rep. 2	Rep. 3
1	−1	−1	−1	23	23.5	24.5
2	+1	−1	−1	35	35	33.5
3	−1	+1	−1	50	45	46
4	+1	+1	−1	66	64.5	67
5	−1	−1	+1	12	12	11
6	+1	−1	+1	20	22	20
7	−1	+1	+1	24	24	24
8	+1	+1	+1	38	37	40

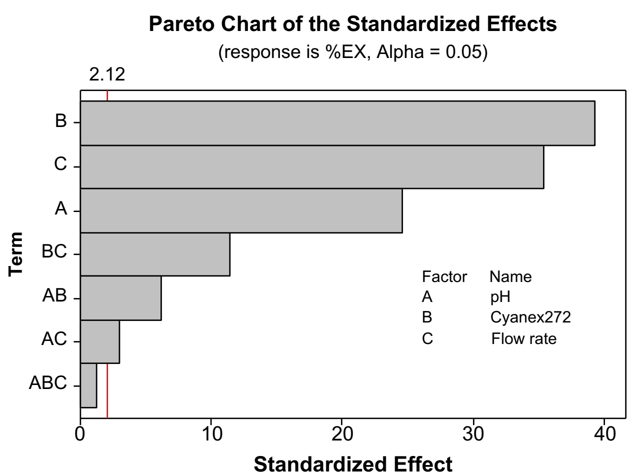

Fig. 5. Pareto chart of the factors that effect on the %Ex of yttrium.

were conducted by varying the pH of feed solution from 1.0 to 5.0 or from −1 to 1 in factorial design code. From the extraction reaction expressed in Eq. (1) by Le Chatelier's Principle, a separation with enhanced reaction occurred. When the pH of feed solution increased or the concentration of hydrogen ions decreased, the concentration gradient of hydrogen ions between the feed and stripping solutions increased [20]. Therefore, more yttrium ions were extracted into the LM and stripped into the stripping solution. However, it should be noted that a low value of hydrogen concentration caused rare earth ions to precipitate, which would hinder extraction. The results of experiments were compared with the regression model in Eq. (4), as shown in Fig. 7, with good agreement achieved.

4.2.2. Effect of Cyanex272 concentration in LM

The variation of pH and flow rate was plotted in response surface, while Cyanex272 concentration was fixed at zero, as shown in Fig. 8. It was found that a flow rate of −1 (100 mL/min) and pH of 1 (5.0) were the optimized conditions and they were fixed. The concentration of Cyanex272 in LM was varied from 0.2 to 0.6 M, or from −1 to 1 in factorial design code. The dependence of the %Ex of yttrium and rare earth with Cyanex272 concentration is shown in Fig. 9 and the results were compared with a regression model. According to the results, %Ex of yttrium increased when the concentration of Cyanex272 increased.

4.2.3. Effect of flow rate of feed solution

The variation of pH and concentration of Cyanex272 were also plotted in response surface, while the flow rate of feed solution were fixed at zero, as shown in Fig. 10. A concentration of Cyanex272 of 1 (0.5 mol/L) and pH of 1 (5.0) were the optimized conditions. The flow rates were examined at 100, 200, 300, and 400 mL/min, while the concentrations of Cyanex272 and pH were fixed at the optimized conditions. According to Fig. 11, the results indicate that %Ex of yttrium and rare earth decreased with increased flow rate of feed solution. Therefore, the best flow rate was 100 mL/min and the highest %Ex of yttrium

Table 5
Analysis of variance: (ANOVA)

Source of variance	SS	df	MS	*F*	*p*-value
A	1,053.38	1	1,053.38	605.53	0.002
B	2,688.17	1	2,688.17	1,545.29	0.001
C	2,185.04	1	2,185.04	1,256.07	0.001
AB	66.67	1	66.67	38.32	0.025
AC	15.04	1	15.04	8.65	0.099
BC	228.17	1	228.17	131.16	0.008
ABC	2.67	1	2.67	1.53	0.341
Error	27.83	16	1.74		

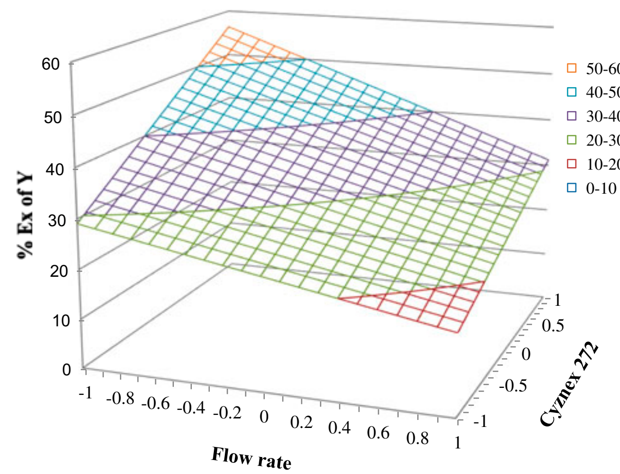

Fig. 6. Surface plot of concentration of Cyanex272 and flow rate vs. %Ex of yttrium from Eq. (4) by fixing pH of feed solution as zero.

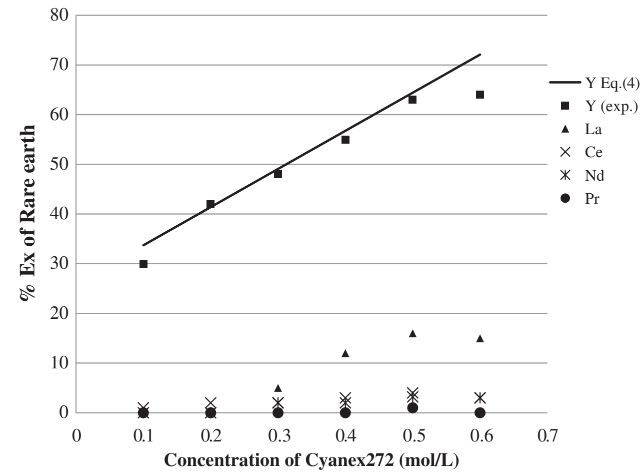

Fig. 9. Effect of concentration of Cyanex272 on the %Ex of yttrium and rare earth elements compared with regression model in Eq. (4).

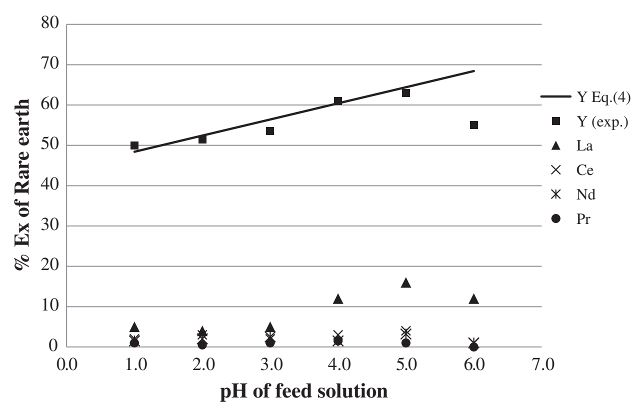

Fig. 7. Effect of pH of feed solution on the %Ex of yttrium and rare earth elements compared with regression model in Eq. (4).

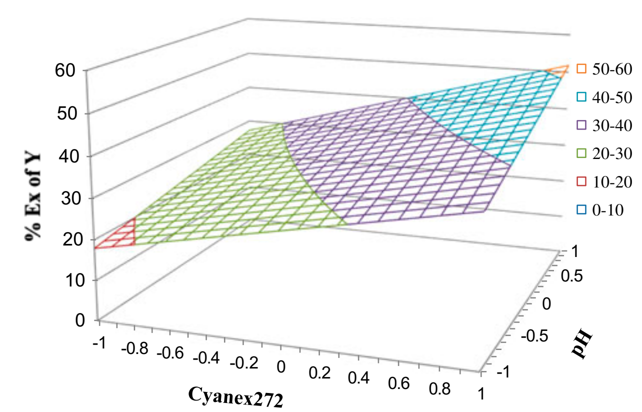

Fig. 10. Surface plot of concentration Cyanex272 and pH vs. %Ex of yttrium from Eq. (4) by fixing flow rate as zero.

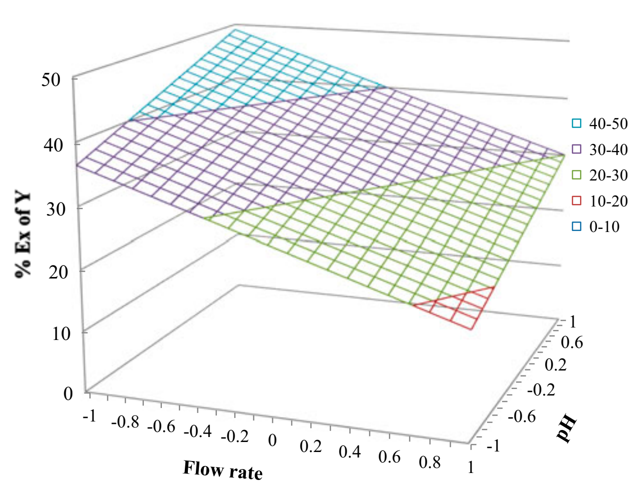

Fig. 8. Surface plot of pH and flow rate vs. %Ex of yttrium from Eq. (4) by fixing concentration of Cyanex272 as zero.

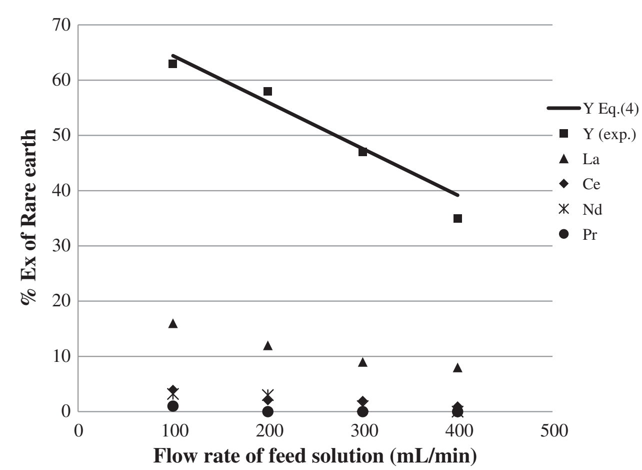

Fig. 11. Effect of flow rate of feed solution on the %Ex of yttrium and rare earth elements compared with regression model in Eq. (4).

obtained was 63%. As shown, the flow rates of feed solution are highly effective on the extractability of hollow fiber-supported LM. When flow rate increased, %Ex of yttrium and other rare earth decreased because the LM at the pore mouth deteriorated when the flow rate was higher [26].

The experimental data of yttrium and rare earth separation are shown in Sections 4.2.1–4.2.3. Clearly, the optimized condition was obtained using the factorial design technique. It brings significantly greater yttrium extraction and its selectivity is greater than previous works [9,10]. Synergistic extraction with two extractants is not necessary.

5. Conclusions

The selective separation of yttrium from solutions containing rare earth by hollow fiber-supported LM was achieved. Yttrium ions were selectively extracted using Cyanex272 as the extractant, while other rare earth elements (lanthanum, cerium, neodymium, and praseodymium) were rejected in the raffinate. The concentration of Cyanex272 is the most significant factor analyzed by a 2^3 factorial design. A pH feed solution of 5.0, a Cyanex272 concentration of 0.5 M, and a flow rate of 100 mL/min were found to be the optimum conditions. A linear regression model can predict the percentages of extraction of yttrium at different values of the significant factors, and good agreement with the experimental values occurs.

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