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Separation and Recovery of Dysprosium and Neodymium from Waste Neodymium Magnet Using Solvent-Impregnated Resin

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The separation and recovery of dysprosium (Dy) and neodymium (Nd) from waste Nd magnet was investigated using a solvent-impregnated resin loaded with 2-ethylhexyl phosphonic acid mono-2-ethylhexyl ester. The leaching solution obtained from the magnet contained Dy and Nd, together with a large amount of iron (Fe). Since Fe suppressed the adsorption of Dy and Nd, ascorbic acid was added to the leaching solution to reduce Fe(III) to Fe(II), enabling their selective adsorption. Although the separation of Dy and Nd was difficult using the conventional frontal separation mode in column operation, the separation was achieved in the gradient elution mode. The proposed separation and recovery process, based on a connected column system, yielded Dy with a purity of 98.9 wt% and a recovery yield of 96.9%, and Nd with a purity of 96.2 wt% and a recovery yield of 95.6%.

1. Introduction

The 17 rare earth metals (REMs), comprising the lanthanides together with scandium and yttrium, are essential for high-tech industries. China produced about 69% of the world's REMs in 2023 [1], and almost all heavy REMs are produced in China [2]. Therefore, the separation and recovery of heavy REMs from waste products is an important issue. The separation and recovery of dysprosium (Dy) and neodymium (Nd) from waste Nd magnets is currently receiving much attention because of the widespread use of these magnets in motors of electric or hybrid vehicles [3-5].

Liquid-liquid extraction is one of the major hydrometallurgical methods for the separation and recovery of REMs. It has high selectivity and is applicable to large-scale operations, making it suitable for industrial use. Acidic organophosphorus extractants, such as 2-ethylhexylphosphonic acid mono-2-ethylhexyl ester (PC-88A), have been widely used to separate REMs in liquid-liquid extraction processes [6,7]. However, this method requires a large amount of organic solvent to dissolve the extractant and extracted species, resulting in a high environmental burden. In addition, leaching solutions obtained from the waste materials are often dilute, which makes liquid-liquid extraction less effective. Ion exchange processes are feasible for such dilute solutions. However, few commercial ion exchange resins have high selectivity for REMs. As a result, solvent-impregnated resins (SIRs) containing extractants with selectivity for REMs have attracted attention as alternative separation materials [8].

We previously investigated the separation of REMs from the leaching solution of Nd magnet using a SIR [8], where Fe was first removed from the leaching solution by adding oxalic acid, since a large amount

of Fe suppresses the adsorption of REMs. Dy and Nd were then recovered using the SIR in a column operation. However, this process had several drawbacks, particularly the method of removing Fe from the leaching solution. When oxalic acid was added, the REMs were precipitated while the Fe remained in the leaching solution. The precipitates were then calcined to remove oxalic acid, followed by re-leaching of the REMs with dilute nitric acid. The Fe removal process was therefore complicated, and an alternative process is required for the continuous operation. Nishihama *et al.* reported that the extraction ability of Fe with bis(2-ethylhexyl)phosphoric acid was greatly reduced by reducing Fe(III) to Fe(II) [9]. The adsorption of REMs from leaching solution is therefore expected to improve by reducing Fe. Another drawback of the previous method was the need for scrubbing to separate Dy and Nd during column operation. While scrubbing is often used in liquid-liquid extraction [10,11], it requires a large amount of solution in column operation. Gradient elution is an alternative method that improves the separation efficiency in column operation [12]. In this method, adsorption is stopped before saturation, and the elution is conducted while increasing the eluent concentration. For PC-88A-impregnated resin, the separation performance is expected to improve when the concentration of the acid employed as the eluent is gradually increased.

In the present work, the separation and recovery of Dy and Nd from waste Nd magnet was investigated using PC-88A-impregnated resin. The reduction of Fe(III) in the leaching solution with ascorbic acid [13-15] and gradient elution for the column operation were used to improve the separation performance for the REMs. The batchwise adsorption of Dy and Nd with the SIR was first carried out. The effect of pH on the adsorption of Dy and Nd in the binary system, and of Dy, Nd, and Fe in the ternary system, was then examined. In addition, adsorption behavior in the ternary system in the presence of ascorbic acid was investigated. The SIR was then applied to a column adsorption system, and the separation of Dy and Nd from the ternary solution and leaching solution was investigated. Finally, the separation and recovery process of Dy and Nd from the leaching solution using a connected column system was demonstrated.

2. Experimental

2.1 Reagents

The commercial extractant PC-88A was obtained from Daihachi Chemical Co. (Osaka, Japan) and used as received. HP2MG was supplied by Mitsubishi Chemical Co. (Tokyo, Japan). All other organic and inorganic reagents were purchased from FUJIFILM Wako Pure Chemical Co. (Osaka, Japan) as analytical-grade reagents.

Binary (Dy and Nd) and ternary (Dy, Nd, and Fe(III)) solutions were prepared by dissolving $DyCl_3 \cdot 6H_2O$, $NdCl_3 \cdot 6H_2O$, and $FeCl_3 \cdot 6H_2O$ in deionized water, and pH values were adjusted by adding appropriate concentrations of HCl or NaOH solution. The concentrations of Dy and Nd in the binary solution were fixed at ca. 1.0 mmol/L, while the ternary solution contained [Dy] = ca. 1.00 mmol/L, [Nd] = ca. 6.00 mmol/L, and [Fe(III)] = ca. 10.0 mmol/L. In cases with ascorbic acid, two equivalents of ascorbic acid were added to Fe in the aqueous solution.

The leaching of actual waste Nd magnet was carried out. Preliminary experiments revealed that the waste Nd magnet dissolved in 5.0 mol/L HCl solution with a small amount of blackish residue, but a large amount of residue remained with 1.0 mol/L HCl solution. A piece of waste Nd magnet (21 g) was immersed

in 300 mL of 5.0 mol/L HCl solution and stirred for 24 h. The leachate was then filtered and analyzed using inductively coupled plasma atomic emission spectroscopy (ICP-AES; ICPE-9000, Shimadzu, Kyoto, Japan). The composition of the Nd magnet, calculated from the ICP-AES measurement of the leachate, is shown in Table 1. The leachate was used as a feed solution for the column experiments, following the pH was adjusted to 2.00, and two equivalents of ascorbic acid to Fe were added [16]. The typical composition of the leachate after pH adjustment is also shown in Table 1.

Table 1. Composition of Nd magnet and leachate solution after pH adjustment.

	pH	[Dy]	[Nd]	[Fe]	[Co]	[B]
Nd magnet†	-	39.5	235	644	9.53	11.9
Leachate after pH adjustment‡	2.00	5.54	44.4	278	3.80	21.6

Unit: † mg/g; ‡ mmol/L

2.2 Preparation of solvent-impregnated resin (SIR)

A previously described procedure was used to prepare the SIR [12]. HP2MG (40 g) was washed with methanol (250 mL), and the resin was dried *in vacuo*. The washed HP2MG (2.5 g) was then immersed in 50 mL of 0.10 mol/L PC-88A-toluene solution overnight. After impregnation, the toluene was evaporated using an evaporator (EYELA OSB-2200), and the resin was dried overnight to obtain SIR. The amount of impregnated PC-88A was 2.06 mmol/g. The resultant SIR (3.0 g) was immersed in an aqueous solution (50 mL) containing 1.0 g of acetamide and 3.0 wt% polyvinyl alcohol, and the mixture was shaken for 17 h. Then, 1.0 mol/L potassium chloride (10 mL) was added to the solution, which was shaken for 24 h. The resultant SIR was filtered and resuspended in 0.32 mol/L sulfuric acid solution (10 mL) for 1 h. Finally, 0.32 mol/L glutaraldehyde solution (10 mL) was added, followed by shaking for 24 h, to crosslink the polyvinyl alcohol. The coated SIR was vacuum-filtered and washed with deionized water.

2.3 Batchwise adsorption

Batchwise adsorption was conducted by shaking 10 mL of the aqueous solution and 20 mg of SIR for 24 h at 25 °C using a thermostatic mechanical shaker. The binary or ternary solution was used as the feed solution. The aqueous solution was then filtered, and the concentrations of metal ions were determined using ICP-AES. The pH was measured by a pH meter (Horiba F-72). The adsorption of each metal (q_M) was calculated as follows:

$$q_M = \frac{([M]_{feed} - [M]_{eq}) \cdot V}{w} \quad (1)$$

where $[M]_{feed}$ and $[M]_{eq}$ are the initial and equilibrium concentrations of the metal (mmol/L), respectively, V is the volume of aqueous solution (L), and w is the mass of SIR (g).

2.4 Column adsorption of Dy and Nd from ternary solution

The separation and recovery of Dy and Nd from the ternary solution was conducted by column adsorption. For the separation of Dy from the ternary solution, SIR (wet volume: 2.4 mL) was packed in a column (length: 20 cm, inner diameter: 10 mm), and the ternary solution (pH_{feed} = 2.00) was fed upward at a flow rate of 0.20 mL/min (space velocity = 5.0 /h) using a dual-plunger pump (Flom KP-21). When frontal separation mode was applied, the adsorption was continued until saturation, and then deionized water was fed into the column to wash out any excess feed solution remaining in the column. Elution was

carried out with 2.0 mol/L HCl solution. When gradient elution mode was applied, the adsorption was stopped before saturation, and then the column was washed with deionized water. The loaded metals were then eluted by HCl with the concentration gradually changed from 0.050 mol/L to 0.10 mol/L for Nd and 1.0 mol/L for Dy. The concentrations of components collected by the fraction collector (EYELA DC-1500) were determined by ICP-AES. The bed volume of the effluent was calculated as follows:

$$\text{Bed volume} = \frac{v \cdot t}{V_{\text{adsorbent}}} \tag{2}$$

where v is flow rate of the solution (mL/min), t is the time over which the solution is supplied (min), and $V_{\text{adsorbent}}$ is the wet volume of adsorbent in the column (mL). The purity was determined based on the weight of the individual metal (wt%).

2.5 Separation and recovery of Dy and Nd from leachate of waste Nd magnet

Dy and Nd were separated and recovered from the actual leaching solution by column adsorption. In this case, about 40 mL of leachate with pH adjusted to 2.00 was fed upward at a flow rate of 0.25 mL/min (space velocity = 5.4 /h) to the column (length: 20 cm, inner diameter: 10 mm) packed with SIR (wet volume: 2.8 mL) using a dual-plunger pump. After adsorption, deionized water was fed into the column for 12 h. Elution was then conducted by gradient elution with the concentration gradually increased from 0.050, 0.10, and 2.0 mol/L.

During the adsorption of Dy, a large amount of Nd was not adsorbed and remained in the effluent. The adsorption of Nd from the effluent was thus carried out. The pH-adjusted effluent ([Dy] = 0.117 mmol/L, [Nd] = 26.4 mmol/L, [Fe] = 171 mmol/L, [Co] = 2.29 mmol/L, [B] = 12.6 mmol/L, pH = 2.00) was fed to a column (length: 50 cm, inner diameter: 15 mm) packed with SIR (wet volume: 29 mL) at a flow rate of 0.50 mL/min (space velocity = 1.0 /h). After adsorption, the column was washed with deionized water, followed by elution with 2.0 mol/L HCl solution. The concentrations of components collected by the fraction collector were determined by ICP-AES.

Based on the column studies, the separation and recovery of Dy and Nd from the leachate of the waste Nd magnet was investigated using connected columns, as shown in Figure 1. In each operation, the concentrations of components collected by the fraction collector were determined by ICP-AES.

(1) Operation #1 [(a) closed, (b) open.]

In Operation #1, columns 1 and 2 were connected in series, and the leaching solution was fed to the columns. Dy was recovered using column 1, while the Nd remaining in the effluent from column 1 was recovered using column 2. About 40 mL of actual leachate with pH adjusted to 2.00 was fed upward at a flow rate of 0.24 mL/min (space velocity = 5.1 /h to column 1) using a

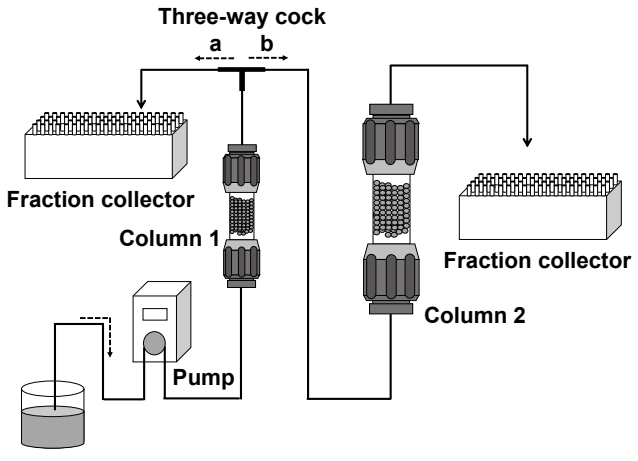

Figure 1. Schematic diagram of designed processes. Columns 1 and 2 were connected with a three-way cock, enabling separate operation of the elution.

dual-plunger pump.

(2) Operation #2 [(a) open, (b) closed]

In Operation #2, Dy and Nd adsorbed in column 1 were separated by gradient elution. The concentration of HCl solution in the gradient elution was gradually changed from 0.050, 0.10, and 2.0 mol/L at a flow rate of 0.12 mL/min (space velocity = 2.6 /h).

(3) Operation #3 [(a) closed, (b) open]

In Operation #3, the columns were connected in series again, and the elution of Nd adsorbed in both columns was conducted with 2.0 mol/L HCl solution at a flow rate of 0.48 mL/min (space velocity = 0.90 /h).

3. Results and Discussion

3.1 Batch adsorption behavior of Dy and Nd

The effect of pH on the adsorption of Dy and Nd from the binary solution is shown in Figure 2. The adsorption of both REMs onto the PC-88A-impregnated SIR increased with pH, as observed in the conventional liquid-liquid extraction system [17]. Dy was selectively adsorbed over Nd at pH_{eq} = 1.6 – 2.5. The adsorption of Dy, Nd, and Fe from the ternary solution was then investigated, since Fe is the main impurity in the leachate from the waste Nd magnet. Figure 3a shows the effect of pH on the adsorption of the three metals from the ternary solution. Fe(III) was selectively adsorbed than the two REMs. The adsorption was therefore conducted with ascorbic acid added to the feed solution to reduce Fe(III) to Fe(II) (Figure 3b). The adsorption of Fe markedly decreased due to the reduction of Fe(III) to Fe(II) in the aqueous solution. In addition, the adsorption of Dy and Nd increased with the addition of ascorbic acid. Therefore, the reduction of Fe(III) with ascorbic acid can prevent the suppression of REM adsorption by the large amount of Fe in the leachate.

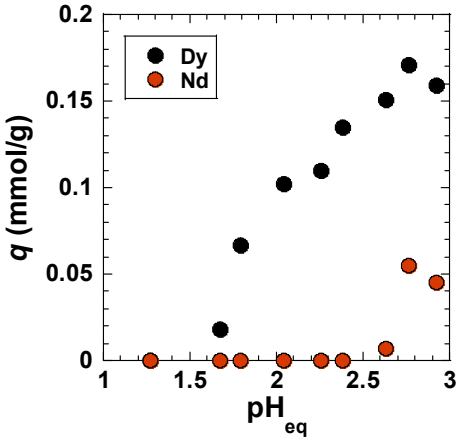

Figure 2. Effect of pH on the adsorption of Dy and Nd in the binary system. $[Dy]_{feed}$ = 1.02 mmol/L and $[Nd]_{feed}$ = 1.02 mmol/L.

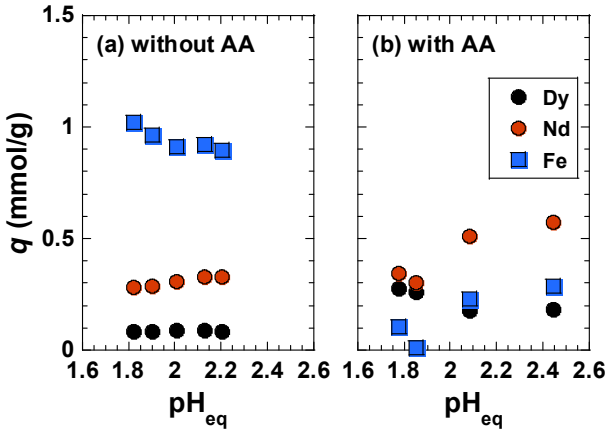

Figure 3. Effect of pH on the adsorption of Dy, Nd, and Fe in the ternary system (a) in the absence and (b) in the presence of ascorbic acid (AA). $[Dy]_{feed}$ = 1.19 mmol/L, $[Nd]_{feed}$ = 5.58 mmol/L, and $[Fe]_{feed}$ = 10.4 mmol/L.

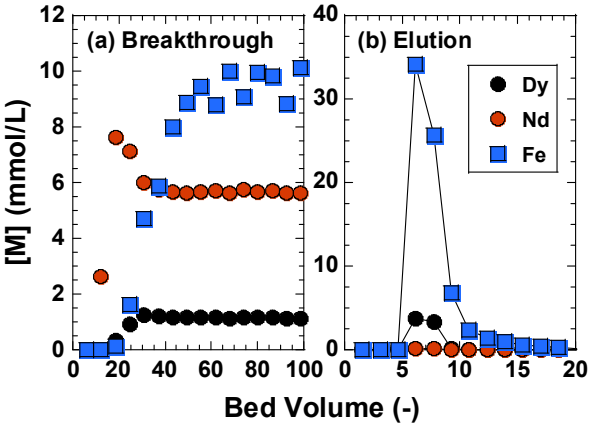

Figure 4. (a) Breakthrough and (b) elution curves of Dy, Nd, and Fe from the ternary solution in the absence of ascorbic acid. $[Dy]_{feed}$ = 1.06 mmol/L, $[Nd]_{feed}$ = 6.10 mmol/L, $[Fe]_{feed}$ = 9.79 mmol/L, and pH_{feed} = 2.00.

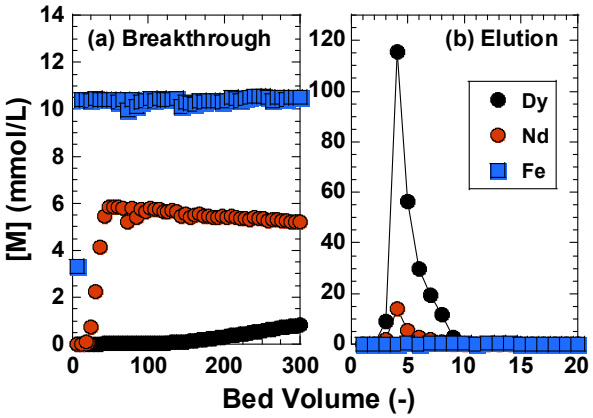

Figure 5. (a) Breakthrough and (b) elution curves of Dy, Nd, and Fe from the ternary solution in the presence of ascorbic acid. $[Dy]_{feed}$ = 1.04 mmol/L, $[Nd]_{feed}$ = 5.04 mmol/L, $[Fe]_{feed}$ = 10.2 mmol/L, and pH_{feed} = 2.00.

3.2 Separation of Dy and Nd from ternary solution with column operation

The separation of Dy and Nd from the ternary solution by column adsorption was investigated. Figure 4 shows the breakthrough and elution curves of Dy, Nd, and Fe without ascorbic acid. Dy and Fe were almost completely adsorbed until bed volume = ca. 20, while Nd broke through at bed volume = ca. 10. The concentration of Nd in the effluent after breakthrough increased compared with that in the feed solution. This increase was due to displacement between the adsorbed Nd and the Dy and Fe in the feed, resulting in minimal adsorption of Nd on the adsorbent. The adsorbed Dy was quantitatively eluted with 2.0 mol/L HCl. However, the elution yield of Fe was relatively low (39.6%) because of the strong adsorption of Fe(III). Despite its low elution yield, Fe was the main component in the eluent, indicating that Fe was predominantly and strongly adsorbed without ascorbic acid, whereas the adsorption of Dy and Nd was suppressed.

Column adsorption was therefore investigated by adding ascorbic acid to the feed solution. Figure 5 shows the breakthrough and elution curves of the three metals in the presence of ascorbic acid. In this case, Fe immediately broke through, indicating that its adsorption is suppressed by reduction with ascorbic acid, even in column operation. The complete adsorption of Dy continued until bed volume = ca. 120, six times larger than in the absence of ascorbic acid. Nd was completely adsorbed until bed volume = ca. 12. The adsorption of REMs in the column system was therefore improved by the reduction of Fe. Quantitative elution of the loaded metals was also possible. However, the elution curves of Dy and Nd overlapped, and thus the purity of Dy in the eluent was 90.4 wt%. The separation of the adsorbed metals was not possible using the conventional frontal separation mode.

Gradient elution is an operation mode of column adsorption and is effective for improving separation performance [12]. Adsorption was stopped prior to saturation of the metals, followed by elution with dilute acid of gradually increasing concentration. The adsorption was therefore stopped at bed volume = 40, and

then elution was conducted using 0.050, 0.10, and 1.0 mol/L HCl solution. Figure 6 shows the breakthrough and elution curves. The two REMs were separated using gradient elution. Nd was eluted with 0.050 – 0.10 mol/L HCl solution, while Dy remained on the adsorbent. Dy was then eluted with 1.0 mol/L HCl, together with a small amount of Fe. The purity and recovery yield of Dy were 98.3 wt% and 100%, and those of Nd were 99.5 wt% and 54.0%, respectively. The low recovery yield of Nd was because the feed solution was supplied after Nd had broken through (bed volume = 20 – 40).

3.3 Separation of Dy and Nd from leaching solution by column adsorption

The separation and recovery of Dy and Nd from the leaching solution of Nd magnet was investigated. The pH-adjusted leaching solution including ascorbic acid was fed to the column. The adsorption was stopped at bed volume = 15, then gradient elution was carried out. Figure 7 shows the breakthrough and elution curves of the metals. The Fe in the leaching solution was reduced, and thus the selective adsorption of Dy proceeded. In the elution, Nd was eluted with 0.050 – 0.10 mol/L HCl solution, and Dy was eluted with 2.0 mol/L HCl solution. Almost all the Dy was recovered, with a purity of 99.1 wt%.

The recovery of Nd from the effluent solution of Figure 7a was investigated. In this case, the pH of the collected effluent was adjusted to 2.00, and then the effluent was fed to the column. Figure 8 shows the breakthrough and elution curves of the metals. Nd in the effluent was effectively adsorbed and eluted with a purity of 99.0 wt%. The concentrations of Fe and B in the effluent from the adsorption gradually decreased at bed volume > 1.5, as shown in Figure 8a. This might be because the void volume in the column was large due to the use of a larger column, leading to unstable flow of the solution in the column. However, almost no Fe and B were adsorbed, and these metals were scarcely observed during the elution, as shown in

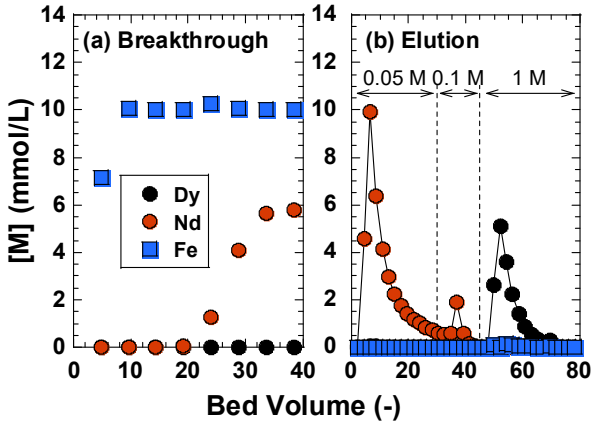

Figure 6. (a) Breakthrough and (b) elution curves of Dy, Nd, and Fe from the ternary solution. Gradient elution was applied with 0.050, 0.10, and 1.0 mol/L HCl. $[Dy]_{feed}$ = 1.11 mmol/L, $[Nd]_{feed}$ = 5.04 mmol/L, $[Fe]_{feed}$ = 10.1 mmol/L, and pH_{feed} = 2.00.

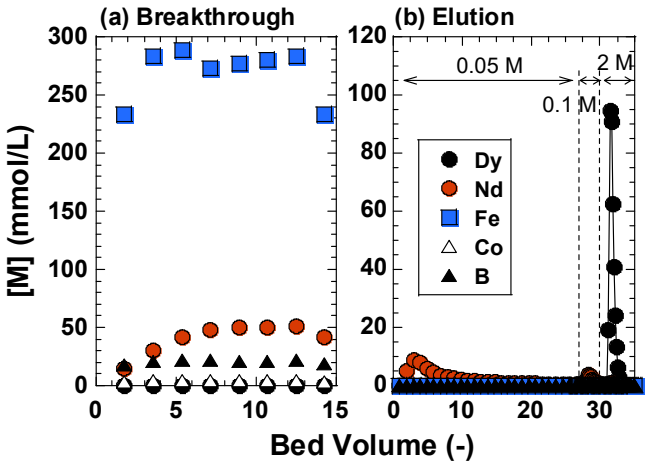

Figure 7. (a) Breakthrough and (b) elution curves of Dy, Nd, Fe, Co, and B from leaching solution of waste Nd magnet. Gradient elution was applied with 0.050, 0.10, and 2.0 mol/L HCl. $[Dy]_{feed}$ = 5.63 mmol/L, $[Nd]_{feed}$ = 45.1 mmol/L, $[Fe]_{feed}$ = 273 mmol/L, $[Co]_{feed}$ = 3.82 mmol/L, $[B]_{feed}$ = 22.8 mmol/L, and pH_{feed} = 2.00.

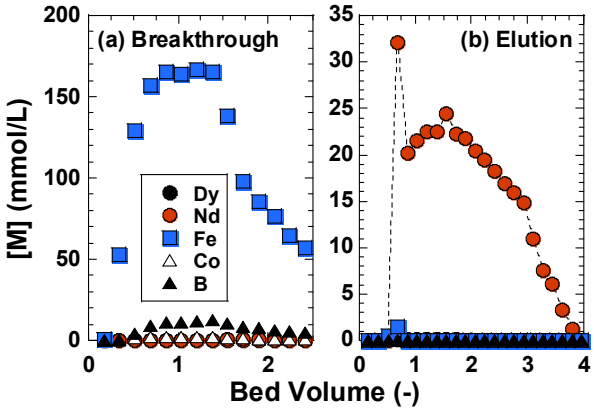

Figure 8. (a) Breakthrough and (b) elution curves of Dy, Nd, Fe, Co, and B from the effluent solution of Figure 7a. $[Dy]_{feed}$ = 0.117 mmol/L, $[Nd]_{feed}$ = 26.4 mmol/L, $[Fe]_{feed}$ = 171 mmol/L, $[Co]_{feed}$ = 2.29 mmol/L, $[B]_{feed}$ = 12.6 mmol/L, and pH_{feed} = 2.00.

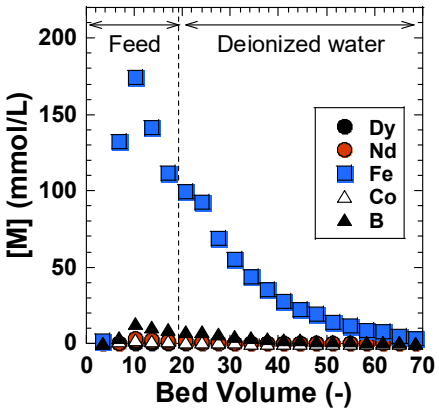

Figure 9. Breakthrough curves and successive concentration profiles of Dy, Nd, Fe, Co, and B during washing with deionized water of Operation #1 from actual leaching solution of waste Nd magnet. $[Dy]_{feed}$ = 5.54 mmol/L, $[Nd]_{feed}$ = 44.4 mmol/L, $[Fe]_{feed}$ = 278 mmol/L, $[Co]_{feed}$ = 3.80 mmol/L, $[B]_{feed}$ = 21.6 mmol/L, and pH_{feed} = 2.00.

Figure 8b.

Based on the results obtained, a successive separation and recovery process for Dy and Nd from the leaching solution was investigated using the column system shown in Figure 1. Firstly, Operation #1 was conducted to adsorb Dy and Nd (Figure 9). Dy was adsorbed in column 1, and Nd remaining in the effluent was adsorbed in column 2. Figure 9 shows the breakthrough curves and successive concentration profiles of Dy, Nd, Fe, Co, and B during column washing with deionized water. Almost all Dy and Nd were adsorbed, while most Fe and other impurities remained in the effluent. Operation #2 was then carried out. Column 1 is considered to adsorb mainly Dy, with a small amount of Nd also adsorbed. Therefore, gradient elution was applied to only column 1 in Operation #2. Figure 10 shows the elution curves of the metals from the first column. Nd was eluted with dilute HCl, while Dy remained adsorbed and then was eluted with 2.0 mol/L HCl. Dy and Nd were thus separated by gradient elution. The recovery yield of Dy was 96.9% with a purity of 98.9 wt%. Finally, Operation #3 was carried out. Since most of the metal adsorbed on both columns is expected to be Nd, the elution was conducted with 2.0 mol/L HCl solution. As shown in Figure 11, Nd was quantitatively eluted with a purity of 96.2 wt%. The recovery yield of Nd was 95.6%, as approximately 5% of Nd was not adsorbed in Operation #1. One advantage of the connected column system is that gradient elution could be limited to column 1 because most of the metal adsorbed on column 2 was Nd. A high concentration of HCl could therefore be applied for elution in column 2, decreasing the volume of eluent required. The separation and recovery process of Dy and Nd from the leaching solution of waste Nd magnet, including the reduction of Fe with ascorbic acid and gradient elution, is therefore effective. In the future, the operational conditions of the gradient elution mode should be optimized to improve the purities of Dy and Nd.

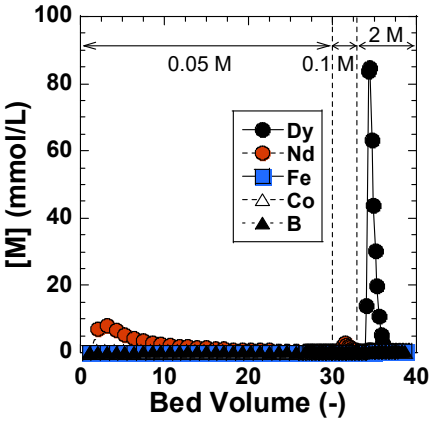

Figure 10. Elution curves of Dy, Nd, Fe, Co, and B from column 1 with gradient elution with 0.050, 0.10, and 2.0 mol/L HCl in Operation #2.

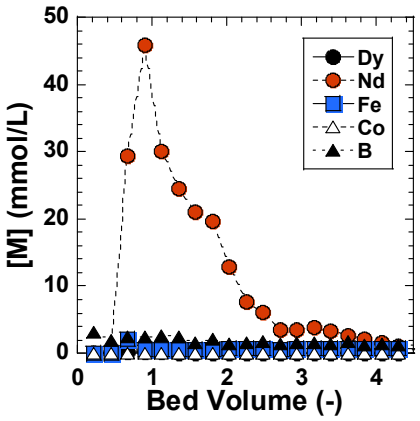

Figure 11. Elution curves of Dy, Nd, Fe, Co, and B from column 2 in Operation #3.

4. Conclusion

The separation and recovery of Dy and Nd from a leaching solution of waste Nd magnet was investigated in the present work, with the following results.

(1) Effective adsorption of Dy and Nd was difficult due to the existence of Fe(III) in the aqueous solution. REM adsorption was improved by reducing Fe(III) to Fe(II) with ascorbic acid.

(2) Separation of Dy and Nd was difficult in the conventional frontal separation mode but was improved using the gradient elution mode.

(3) The separation and recovery of Dy and Nd from the leaching solution of waste Nd magnet was achieved by Fe(III) reduction and gradient elution.

(4) A separation and recovery process for Dy and Nd was proposed, which involves two connected columns.

By separating the columns after adsorption, the volume of eluents required could be decreased.

Declaration

The authors declare that they have no conflict of interest.

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