## Recovery of neodymium and dysprosium from NdFeB magnet swarf

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### ABSTRACT

The rare earth elements neodymium and dysprosium were separated and recovered from NdFeB permanent magnet swarf by hydrometallurgical route. The magnet swarf was leached with HCl after crushing and grinding, and from the leach liquor, Nd and Dy were separated by solvent extraction using NaCyanex 302. The separation factor ( $\beta$ ) of Dy/Nd with NaCyanex 302 was high at low equilibrium pH. So Dy was first separated from the leach liquor at pH 0.5 (equilibrium pH 1.2). After separation of Dy, the separation of Nd was also carried out with the same extractant. The effect of equilibrium pH and extractant concentration on the extraction of Nd and Dy with NaCyanex 302 was studied. The McCabe-Thiele plots for extraction of both Dy and Nd with NaCyanex 302 were constructed and the number of stages for quantitative extraction was determined. The stripping of loaded organic was carried out with three different mineral acids and it was observed that the stripping efficiency followed the order HCl < HNO<sub>3</sub> < H<sub>2</sub>SO<sub>4</sub>. The McCabe-Thiele plots for stripping of loaded organic were also constructed. The reaction mechanism of both Nd and Dy was determined from the slope analysis method and the extracted species was found to be MA<sub>3</sub>.5HA.

#### 1. Introduction

Neodymium-Iron-Boron magnets are the strongest permanent magnets known, and its application in modern technology, especially in electronic devices increased many folds in last few years. It is used in computer hard disk, wind turbine, hybrid electric vehicles, hydroelectric turbine generators, missiles, tanks, warplanes and submarines (Coey, 2002). The global demand of permanent rare earth magnets may increase to 13.2% (worth of \$41.41 billion) by 2022 (Research and Markets Dublin, 2016). The recycling of REE is within 1%, but the current situation demands more recycling of REE from pre-consumer scrap, industrial residue and REE containing the end of life products (Binnemans et al., 2013). It will help to save the natural resources and prevent environmental pollution.

A significant amount of rare earth element is wasted as magnet swarf during the manufacturing process of Nd-based magnets. Hence, researchers have been working on the recycling process of RE from magnet swarf. Several ways of potential recycling process have been described in the literature, but none of them has been developed commercially due to low productivity and high cost (Panayotova and Panayotov, 2012). The processes include the use of hydrometallurgical processes, molten salts, extraction with silver and magnesium, melt spinning and the formation of slags (Kara et al., 2010). In the hydrometallurgical process, different unit operations such as leaching, solvent extraction and precipitation are adopted for recovery of REE from the magnet swarf. The solvent extraction attracts great interest due to its high selectivity and significant capital and operating cost savings. Also the process is fast, continuous and works on more concentrated solution (Xie et al., 2014). In general, the purity of REE produced by using solvent extraction is more than that by using precipitation.

Different extractants such as D2EHPA (Radhika et al., 2011; Morais and Ciminelli, 2004; Sanchez et al., 1999), PC88A ((Morais and Ciminelli, 2004, Thakur et al., 1993, Hsiang et al., 2006, Lee et al., 2005), Cyanex 923 (Panda et al., 2012), Cyanex 921 (El-Hefny, 2007), Cyanex 272 (Banda et al., 2012), Aliquat (Lu et al., 1989; Preston, 1996), TBP (Esmaeil and Malek, 2012), sec-octylphenoxyacetic acid (CA12, H2A2) and 8-hydroxyquinoline (HQ) (Miaomiao et al., 2012), dinonyl phenyl phosphoric acid (Anitha et al., 2014) have been used by various authors for solvent extraction of REEs.

Di-(2-ethylhexyl) phosphoric acid (D2EHPA), 2-ethylhexylphosphonic acid mono-2-ethyl hexyl ester (EHEHPA) were used for the separation of Nd(III), Dy(III) and Y(III) from hydrochloric acid solution (Mohammadi et al., 2015) and it was reported that pure D2EHPA (0.06 mol/L and 0.09 mol/L) is most efficient for separating Nd(III) from Y(III) and Dy(III), respectively. Jia et al. (2004) studied the extraction of Nd, Dy and La by using 1-phenyl-3-methyl-4-benzoyl-pyrazalone-5 (HPMBP) and triisobutyl phosphinesulfide (TIBPS) from a

nitrate medium. When HPMBP was used alone, the extraction of metals followed the order as  $Dy^{3+} > Nd^{3+} > La^{3+}$ . However, the separation was much better when the mixture of the extractants was used.

Yoon et al. (2016) reviewed the recycling strategies for permanent magnet for recovery of Nd and Dy using organophosphoric acid extractant. Two extractants D2EHPA and PC88A were compared for extraction of Nd and Dy in a case study. Apart from conventional commercial extractants, ionic liquids (IL) had also been used for the separation of Nd and Dy in solvent extraction process (Riaño and Binnemans, 2015). From a nitric acid leached solution, cobalt was extracted by an IL trihexyl(tetradecyl)phosphonium nitrate. Then separation of Nd, and Dy was carried out by selective complexation with EDTA during solvent extraction with the same IL. Different solvent extraction parameters were optimised, and finally, the oxides of rare earths, and Co were prepared. The purity of Nd<sub>2</sub>O<sub>3</sub>, Dy<sub>2</sub>O<sub>3</sub> and CoO was 99.6%, 99.8% and 99.8%, respectively. Matsumiya et al. (2014) investigated the recovery of rare earths from NdFeB magnets by using triethyl-pentyl-Phosphoniumbis (trifluoromethyl-sulfonyl)amide ([P2225][TFSA]) as ILs. It was observed that the exaction efficiency of rare earths increased significantly in TBP with [P2225][TFSA]. Similarly, Kikuchi et al. (2014) removed REEs from spent NdFeB magnets by TBP with an IL tricaprylmethyl ammonium nitrate [A336][NO<sub>3</sub>].

Cyanex 302 (bis(2,4,4-trimethylpentyl)monothiophosphinic acid) had been used for the selective separation of many rare earth metals like scandium(III) and yttrium(III) (Karve and Vaidya, 2008). It was also mixed with other extractants for synergistic solvent extraction (Kumar et al., 2014; Miaomiao et al., 2013; Belova et al., 2009; Wang et al., 2006) of rare earth elements. A comparative study between various extractants such as Cyanex 302, PC88A and Cyanex 272 for extraction of Nd from a synthetic chloride solution containing 1.0 g/L Nd only has been carried out earlier (Padhan and Sarangi, 2015). The extraction efficiency of the neutral form of these extractants followed the order NaPC88A < NaCvanex 272 < NaCvanex 302. As the extraction efficiency of NaCyanex 302 was better for Nd extraction, it was used in the present work for separation of Nd and Dy from the leached solution of NdFeB magnet swarf. A detailed study has been carried out for separation of Nd and Dy. The parameters were optimised for extraction and stripping of Nd and Dy and McCabe-Thiele diagrams were constructed.

#### 2. Experimental

Chemicals and reagents

The commercial extractant Cyanex 302 which is bis(2,4,4-trimethylpentyl) monothiophosphinic acid was received from Cytec Inc. USA and was used as such without any purification. Distilled kerosene (b.p. 180–240 °C) was used as the diluent. All other chemicals such as H<sub>2</sub>SO<sub>4</sub>, HCl, NaOH used in this study were of AR grade and obtained from Qualigens, India.

#### Procedure

The extractant (Cyanex 302) was neutralized up to 70% by adding the stoichiometric amount of concentrated NaOH followed by vigorous shaking until the two phases were completely miscible. The NaCyanex 302 thus obtained was used as the extractant and 5 vol% nonylphenol was added as phase modifier. The liquid- liquid extraction was carried out by shaking aqueous feed solution and extractant solutions in separating funnels using a mechanical shaker. According to a preliminary study 3-4 min shaking time was sufficient to achieve the equilibrium, hence in all the experiments aqueous and organic phases were equilibrated for 5 min. The digital pH meter (HI 2215 pH/ORP Meter) provided with a combined glass electrode was used to measure the pH of aqueous solutions. The metal ion concentration in aqueous solution was measured by ICP – OES (Inductively coupled plasma – Optical emission

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spectroscopy), model number Optima-2100DV, Perkin Elmer. The metal concentration in the organic phase was calculated from the difference between the metal concentration in aqueous phase before and after extraction.

The distribution ratio D was calculated as the ratio of metal concentration in the organic phase to that of the aqueous phase as given in Eq. (1).

$$D = \frac{Total \ concentration \ of \ solute \ in \ organic \ phase}{Total \ concentration \ of \ solute \ in \ aqueous \ phase}$$
(1)

The distribution ratio was used to calculate the separation factor ( $\beta$ ) of two metal ions under identical condition. If A (major extracting species) and B (minor extracting species) represents the solutes present in a solution, then the separation factor ( $\beta$ ) is related to individual distribution ratios as follows:

$$\frac{Separation\,factor\,\,(\beta)}{D_B} = \frac{D_A}{D_B} \tag{2}$$

where:  $D_{\text{A}}$  is the distribution ratio of A and  $D_{\text{B}}$  is the distribution ratio of B.

#### 3. Results and discussion

A typical NdFeB magnet swarf containing 31% Nd, 1.1% Dy, 65% Fe and 0.71% B was used for the study. The NdFeB swarf was subjected to leaching with 0.5 M HCl after crushing and grinding. The particle size used for leaching was within 75–106  $\mu$ m. Other conditions for leaching were 3% pulp density, 90 °C and 2 h leaching time. The leaching efficiency of Nd, Dy, Fe and B were 98.92%, 99.99%, 80.0% and 93.5%, respectively and the leach liquor contained 9.2 g/L Nd, 0.329 g/L Dy, 15.6 g/L Fe and 0.19 g/L B. From this leach liquor, Fe was first removed by precipitation with lime at pH 3.5. After precipitation, the leach liquor was filtered and the filtrate was analysed. The concentration of leach liquor after removal of Fe was 9.0 g/L Nd, 0.3 g/L Dy, 0.006 g/L Fe and 0.18 g/L boron.

#### Extraction studies

*Effect of equilibrium pH on extraction of Nd and Dy* 

The equilibrium pH of the solution has the major influence on the extraction of metal ions. Hence, the effect of equilibrium pH on the extraction of neodymium and dysprosium was studied by varying the initial pH of the solution from 0.5 to 3. A comparative study was carried out, to observe the effect of both acidic and neutral form of extractant (Cyanex 302 and NaCyanex 302) on the extraction of Nd and Dy. The concentration of extractants and aqueous to organic phase ratio were kept constant at 0.2 M and 1:1, respectively. The corresponding equilibrium pH was varied from 1.2 to 4.66 for NaCyanex 302 and from 0.98 to 2.22 for Cyanex 302. The plot of equilibrium pH against percentage extraction was shown in Fig. 1, which showed an increase in percentage extraction of Nd and Dy from 18.19 to 89.03% and from 92.33 to 98.0%, respectively with NaCyanex 302. However, the percentage extraction of Nd and Dy increased from 6.61 to 13.07 and from 10.33 to 18.33, respectively with Cyanex 302. From this plot, it was clear that NaCyanex 302 is a better extractant for the extraction of Nd and Dy. The graph of equilibrium pH versus log  $D_{Nd}$  (Fig. 2) for Nd with NaCyanex 302 was a straight line with the slope value of 1.09.

The separation factor ( $\beta$ ) value more than one indicates that the two metals can be separated. Higher the value of  $\beta$ , the lesser is the number of stages (and vice versa) required to achieve given metal ratio in the loaded organic. Some authors have determined the separation factor for Nd and Dy with various extractants. With the mixture of extractants, sec-octylphenoXyacetic acid and 8-hydroXyquinoline, the  $\beta_{Nd/dy}$  value was 2.0 (Miaomiao et al., 2012). The value was 3.17 for 1-phenyl-3-methyl-4-benzoylpyrazalone- 5 extractant (HPMBP), and 1.16 for the mixture of HPMBP and sec-octylphenoXyacetic acid (Tong et al., 2009).



Fig. 1. Effect of equilibrium pH on extraction of Nd and Dy. Experimental conditions: [Extractant] = 0.2 M, A:O = 1:1.



Fig. 2. Plot of logD $_{\rm Nd}$  vs. equilibrium pH. Experimental conditions:0.2 M NaCyanex 302, A:O = 1:1.

# The value was calculated to be 4.29 with Cyanex 925 in nitrate medium (Li et al., 2007).

In this experiment  $\beta$  value of Nd and Dy was determined with NaCyanex 302. The separation factor of Dy/Nd at different equilibrium pH was given in Table 1. The table indicated higher separation factor at lower equilibrium pH. The maximum separation factor of 53.65 was obtained at equilibrium pH of 1.2. So Dy can be easily separated from Nd at equilibrium pH 1.2.

#### Extraction of Dy

To optimise the parameters for Dy extraction and to minimise the coextraction of Nd, the concentration of the NaCyanex 302 was varied from 0.05 to 0.15 M at initial pH 0.5. The percentage extraction of Nd and Dy as a function of extractant concentration was shown in Fig. 3. It was noted that the extraction of Nd and Dy increased from 2.53 to 8.83% and 27.83 to 91.92%, respectively with increasing the extractant

Table 1
Separation factor ( $\beta$ ) of Dy/Nd at different equilibrium pH.

Eq.pH	1.26	3.65	4.22	4.35	4.47	4.58	4.62	4.63	4.66
Separation factor	53.65	43.37	15.34	9.87	7.88	6.88	7.89	7.06	5.61
(β)									

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Fig. 3. Effect of [Extractant] on extraction of Nd and Dy. Experimental conditions:Initial pH 0.5, A:O = 1:1.

concentration from 0.05 to 0.15 M. As the Dy extraction and Nd coextraction were 86.5% and 6.25%, respectively with 0.125 M NaCyanex 302, the McCabe-Thiele plot for dysprosium was constructed with this concentration of NaCyanex 302. The phase ratio of aqueous and organic was varied from 1:5 to 5:1, while keeping the total volume of phases constant. As shown in Fig. 4, the McCabe-Thiele diagram of Dy indicated the requirement of two counter current extractions stages at A:O = 1:1 phase ratio for quantitative extraction of Dy from the solution. The two-stage counter current simulation study for Dy extraction at above conditions showed 0.006 g/L Dy in 2nd stage raffinate indicating 98% extraction efficiency. The co-extraction of neodymium was 0.65 g/L (7.22%). The raffinate after Dy extraction contained 0.006 g/L Dy and 8.35 g/L Nd from which Nd was extracted with the same extractant.

#### Extraction of Nd

*Effect of extractant concentration.* At equilibrium pH 4.62 for which the initial pH of the solution was 2.0, neodymium was extracted up to 84.14%. So, the effect of extractant concentration was studied at initial pH of 2.0. The extractant concentration was varied within the range of 0.05 to 0.3 M at A:O phase ratio of 1:1. The percentage extraction was plotted against extractant concentration in Fig. 5. Nd



Fig. 4. McCabe-Thiele plot for extraction of Dy. Experimental conditions:Initial pH 0.5, 0.125 M [NaCyanex302].



Fig. 5. Effect of [Extractant] on extraction of neodymium. Experimental conditions:Initial pH 2.0, A:O = 1:1.



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Fig. 7. McCabe-Thiele plot for extraction of neodymium. Experimental conditions:Initial pH 2.0, 0.2 M NaCyanex 302.



Fig. 6. Plot of log  $D_{Nd}\,$  vs. Log[Extractant]. Experimental conditions:Initial pH 2.0,  $A{:}O=1{:}1.$ 

extraction increased from 24.46 to 99.61% with an increase in the concentration of NaCyanex 302 from 0.05 to 0.3 M. The plot of log  $D_{Nd}$  versus log [Extractant] was shown in Fig. 6. The plot was a straight line with the slope value of 2.8.

*McCabe-Thiele plot for extraction.* The extraction of neodymium with 0.2 M NaCyanex 302 was 88.17%. Hence, data were generated for the construction of McCabe-Thiele diagram with 0.2 M NaCyanex 302 at different A:0 ratio with in 1:5 to 5:1. The initial pH was kept constantat 2.0. The McCabe-Thiele diagram for Nd was shown in Fig. 7 which indicated two extraction stages at an A:0 ratio of 1:1 for quantitative extraction. To confirm the prediction, a 2 stage counter current simulation study was carried out in similar conditions. The raffinate of the second stage contained 0.02 g/L Nd. So the extraction efficiency of Nd was calculated to be 99.79%. The final loaded organic was analysed to contain 8.33 g/L Nd.

#### Stripping studies

#### Stripping of Nd

The stripping study of neodymium loaded organic phase was carried out with different mineral acids ( $H_2SO_4$ , HCl and HNO<sub>3</sub>) at unit phase

Fig. 8. Effect of [Acid] on stripping of neodymium. Experimental conditions:[L0] = 8.33 g/L Nd, A:O = 1:1.

ratio. The concentration of the stripping reagents varied from 0.5 to 3 vol% in all experiments. The percentage stripping with all acids were calculated and plotted in Fig. 8. With increasing the acid concentration from 0.5 to 3 vol%, the percentage of stripping increased from 41.55 to 85.71% and 37.31 to 81.14% for HCl and HNO<sub>3</sub>, respectively. But in the case of sulphuric acid, the stripping percentage increased from 76.84 to 94.79 with increasing acid concentration from 0.5 to 1 vol%, and then decreased. Hence, 1 vol% H<sub>2</sub>SO<sub>4</sub> was chosen for the stripping study. The stripping efficiency of different acids follows the trends as  $H_2SO_4 > HNO_3 > HCl$ . The stripping efficiency with  $H_2SO_4$  was more which may be due to the weaker bond between sulphate and Nd. A study on extraction of Sm(III) and Ce(III) with PC88A from sulphate, chloride and nitrate media showed the extraction efficiency in the order  $HCl \ge HNO_3 > H_2SO_4$  (Agarwal and Safarzadeh, 2017). The low extraction rate in sulphate media leads to high stripping rate. Other authors have also found the similar trends (Panda et al., 2012; El-Hefny, 2007).

The experiments were carried out to construct a McCabe-Thiele diagram for neodymium stripping with 1 vol% sulphuric acid. The graph was generated by plotting the Nd concentration in strip solution and spent organic phase as shown in Fig. 9. The figure indicated quantitative stripping of Nd in three counter-current stages at



Fig. 9. McCabe-Thiele plot for stripping of neodymium. Experimental conditions: [L0] = 8.33 g/L Nd, 1%  $\rm H_2SO_4$ 

A:O = 1:2. The prediction was confirmed by performing a three stage counter current simulation study at the optimised condition. The strip solution was analysed to contain 16.6 g/L Nd with 99.64% stripping efficiency.

#### Stripping of Dy

As the stripping efficiency of H<sub>2</sub>SO<sub>4</sub> was highest as compared to other acids used for stripping of Nd loaded organic, it was also used to strip the Dy-Nd loaded organic obtained during Dy extraction. The loaded organic contained 0.294 g/L Dy and 0.65 g/L Nd. For the stripping study, the concentration of the sulphuric acid was varied from 0.01 to 0.1 vol%, and A:O ratio was kept constant at 1:1. After stripping, the strip solution was analysed for the concentration of metal ions, and the percentage stripping was plotted against H2SO4 concentration in Fig. 10. It was observed that both Nd and Dy were stripped out from the loaded organic and the stripping percentage were nearly same for both the rare earths. With 0.08% H<sub>2</sub>SO<sub>4</sub> and A:O = 1:1, the stripping of Nd was 84.61% and that for Dy was 85.71%. So, a two stage counter-current simulation study for stripping was carried out with above condition as done earlier (Padhan and Sarangi, 2015) and the strip solution was analysed. The 2nd stage strip solution contained 0.62 g/L Nd and 0.282 g/L Dy indicating 95.38% Nd and 95.92% Dy stripping.



Fig. 11. Effect of equilibrium pH on extraction of Dy. Experimental conditions:0.01

Fig. 10. Effect of  $[H_2SO_4]$  on stripping of Nd and Dy. Experimental conditions:[L0] = 0.65 g/L Nd and 0.294 g/L Dy, A:O = 1:1.

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Reaction mechanism

The plot of  $log D_{Nd}$  vs. equilibrium pH as shown in Fig. 2 was a straight line with a slope value of 1.09. This indicated that 1 mol of hydrogen ion was released to the aqueous solution for extraction of 1 mol of the Nd ion. The slope value of log D<sub>Nd</sub> vs. log [extractant] (Fig. 6) is 2.8 which suggested that 3 mol of extractant were required to extract 1 mol of neodymium. The metal to extractant ratio of 1:3 for Nd and Cyanex 302 was also found by other authors (Jensen et al., 2001). In Fig. 1, the extraction of Dy was varied from 92.33 to 98% due to the use of higher concentration of NaCyanex 302, so it was not possible to know the no. of moles of H+ ions participated in the extraction. So another extraction experiment was carried out, from a solution containing 0.3 g/L Dy only with 0.01 M NaCyanex 302. The percentage extraction of Dy and logD<sub>Dy</sub> vs. Equilibrium pH were plotted in Fig. 11. The slope of  $log D_{Dy}$  vs. equilibrium pH plot was found to be 0.92 which indicated the release of 1 mol of H+ ion during extraction as with Nd. The data of Fig. 3 was used to plot logD<sub>Dy</sub> vs. log [extractant] in Fig. 12, and the slope value of the straight line was 3.2. The logD vs. equilibrium pH and logD vs. log [extractant] plots for both Nd and Dy had same slope values, so same extraction mechanism can be written for both rare earth elements.

For extraction of Nd and Dy, 70% of Cyanex 302 was converted to NaCyanex 302 with the addition of stoichiometric amount NaOH. The neutralisation reaction can be written as Eq. (3), where 'HA' represents Cyanex 302.

$$2Na^{+}_{aq} + (HA)_{2(org)} \leftrightarrow 2NaA_{(org)} + 2H^{+}_{aq}$$
(3)

As 70% of Cyanex 302 was neutralized, both the forms of Cyanex 302 took part in extraction and the extraction mechanism of both Nd and Dy can be expressed as follows:



Fig. 12. Plot of logD $_{\rm Dy}$  vs. log[extractant]. Experimental conditions:Initial pH 0.5,  $A{:}O=1{:}1.$ 

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 $M^{3+} + 3(HA)_2 + 2A^- \leftrightarrow MA_3.5HA + H^+$ 

Where M represents for RE element (Nd or Dy).

The equilibrium constant can be written as given in Eq. (5)

$$K = \frac{[MA_3.5HA][H^+]}{[M^{3+}][(HA)_2]^3[A^-]^2}$$
(5)

$$K = D \frac{[H^{+}]}{[(HA)_{2}]^{3} [A^{-}]^{2}}$$
(6)

where,  $D = \frac{[MA_3.5HA]}{[^{3}t]}$ 

Taking logarithm of Eq. (6) and rearranging Eq. (7) was obtained

$$\log D = \log K + pH + 3\log[(HA)_2] + 2\log[A]$$
(7)

The slope values of plots of log D vs. Equilibrium pH (Figs. 2, 11) and log D vs. log[Cyanex 302] (Fig. 6, 12) were in good agreement with Eq. (7). So the extraction mechanism of both Nd and Dy with Cyanex 302 can be explained by Eq. (4).

#### 4. Conclusion

The separation of neodymium and dysprosium from leached liquor of NdFeB magnet was achieved by solvent extraction using NaCyanex 302 as the extractant. Maximum separation factor  $(D_{Dy}/D_{Nd})$  of 53.65 was observed at low equilibrium pH(1.2). So Dy was separated from Nd at this pH with 0.125 M NaCyanex 302. The extraction of Dy was 98% and co-extraction of Nd was 7.22% in two stages of counter current extraction with 0.125 M NaCyanex 302 at A:0 = 1:1. After Dy separation, Nd was extracted, and the extraction of Nd was 99.79% with 0.2 M NaCyanex 302 in two counter current extraction stages at 1:1 phase ratio. The stripping of Nd loaded organic was carried out with HCl, HNO<sub>3</sub>, and H<sub>2</sub>SO<sub>4</sub>. The stripping efficiency followed the trend  $HCl < HNO_3 < H_2SO_4$ . With 1% sulphuric acid the stripping of Nd from loaded organic was 99.64% in three counter current stages at A:O ratio of 1:2. The loaded organic after Dy extraction contained both Dy and Nd and stripping was done with 0.08% H<sub>2</sub>SO<sub>4</sub>. The stripping of Nd and Dy was 84.61% and 85.71%, respectively.

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