

Article

Recovering and Purifying Neodymium and Dysprosium from Simulated Leaching Solution of Spent NdFeB Magnets via Ion Exchange Processes

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Abstract

As critical rare earth elements (REEs), the industrial demand for neodymium (Nd) and dysprosium (Dy) increases rapidly due to their specific physical and chemical properties. Recycling these REEs from secondary resources such as spent NdFeB magnetic materials is an efficient approach for sustainable production. However, the separation of neodymium and dysprosium in aqueous solutions is an arduous task because of their close chemical properties. Recovering and purifying neodymium and dysprosium from a simulated leaching solution of spent NdFeB magnets were conducted by employing selective ion exchange resins. It was found that Purolite S950 PLUS resin functionalized with aminophosphonic groups demonstrated selective adsorption toward Nd^{3+} and Dy^{3+} while maintaining low affinity for Fe(II) at low pH (i.e., 0.65), which could realize efficient iron removal from the solution. Purolite MTX7010 resin impregnated with di-(2-ethylhexyl) phosphoric acid (D2EHPA) had a strong adsorption preference for Dy^{3+} over Nd^{3+} , which is highly suitable for Dy separation from their mixed solutions under optimized conditions. By employing a multistage adsorption–elution process analogous to distillation, a prospective purity of 98.51% for Dy and a purity over 99.90% for Nd were realized with high metal recoveries from the synthetic leaching solution of spent NdFeB magnets. This research demonstrates that recovery and purification of single REEs from leaching solutions containing mixed REEs and other metals can be achieved with selective resin adsorption processes analogous to distillation despite large concentration differences in the metals in the solutions, which presents a new approach.

Keywords: neodymium (Nd); dysprosium (Dy); spent NdFeB magnets; recovering and purifying; selective ion exchange



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1. Introduction

As critical rare earth elements (REEs), neodymium (Nd) and dysprosium (Dy) are experiencing a rapidly increasing demand due to their specific physical and chemical properties, e.g., significant magnetic properties and thermal performance for producing neodymium permanent magnets (NdFeB) [1], which are the most important magnets with widespread applications such as wind turbines [2,3], electric vehicles [4], and other green energy technologies [5]. However, the supply and economically viable resources of REEs are limited only in a few countries, with China holding a dominant position in the entire value chain, particularly in refining and processing technologies [6,7]. Recycling the REEs

from secondary resources is a promising and efficient approach for sustainable production, particularly due to the high contents of REEs in various spent electrical and magnetic products such as spent NdFeB magnetic materials.

Selective separation and recovery of rare earth elements are challenging due to their similar chemical properties as exhibited in the periodic table. Thus, it is crucial to develop efficient and selective processes for recovering and separating these critical metals.

Different methods such as precipitation, electrochemical method, membrane technology, biosorption process, conventional solvent extraction, and ion exchange have been explored [8,9]. The precipitation processes used to separate the REEs involve direct precipitation with hydrogen fluoride or oxalic acid [10,11], or double salt precipitation at varying pH values [12,13]. The main restrictions of the precipitation approach are attributed to the large consumption of chemicals and high losses in REEs [14,15]. Conventional solvent extraction and the ionic liquid solvent (ILS) extraction are commonly used methods [16], despite their disadvantages of high solvent consumption [17], high volume of waste generated, limited selectivity under acidic conditions, and hazardous environmental impact [18–21].

Resin adsorption, including ion exchange technologies, is a solid–liquid separation method that usually utilizes polymeric resins containing active functional groups, which can be designed to enhance adsorption selectivity for specific ion species [22]. Compared to conventional solvent extraction, ion exchange offers several key advantages, including higher selectivity and adsorption efficiency reaching more than 99.999%, which makes it the best-introduced method for yielding ultra-pure REEs [23], with simpler operation and handling [24], greater control over process parameters, smaller environmental footprint, and suitability for small-scale processes [25]. As summarized in Table 1, researchers have investigated the equilibrium state and adsorption efficiency of various types of resins for different REE recovery and separation [14,26,27]. The SQS-6 resin with sulfonic functional groups was used by Elgoud and co-workers [28] to study the adsorption mechanism of La (III) and Nd (III) from phosphoric acid media with the concentration range from 4 to 10 M. They found that the equilibrium state was reached within 10 min at the optimum phosphoric acid concentration of 8 M, and the resin loading capacity was 13.8 mg/g and 12.7 mg/g for La and Nd, respectively. In another study, the SQS-6 resin was used to investigate its adsorption efficiency for Ce (IV), Pr (III), Er (III), and Y (III) from phosphoric acid solution [29], where the experimental results indicated that the adsorption efficiency decreased with rising acid concentration and the adsorption capacity reached 5.2, 12.6, 8.8, and 3.8 mg/g at 8 M phosphoric acid concentration for Ce (IV), Pr (III), Y (III), and Er (III), respectively. Masry and colleagues [30] employed Dowex 50WX8 resin functionalized with sulfonic acid groups to investigate its uptake behavior for individual Dy (III), Pr (III), and Y (III) ions dissolved in nitrate solution. The results revealed that increasing HNO₃ concentration led to a decrease in the resin's uptake capacity, where the maximum adsorption capacity at the optimum adsorption condition reached 50, 30, and 60 mg/g for dysprosium, praseodymium, and yttrium ions, respectively. A weak acrylic resin known as ACRI-BOND 110 resin was examined by Xiong et al. [31] to remove Nd (III) from aqueous solution. A series of experiments was conducted to optimize the adsorption factors, including temperature, solution pH, and contact time at equilibrium, where the highest adsorption capacity was estimated to be 308 mg/g at 298 K after 60 h adsorption.

Ion exchange resins offer remarkable flexibility, such as allowing the modification or replacement of their active functional sites, which reinforces their adaptability for various applications. Improvement on the resins' selectivity could also be achieved through the introduction of the solvent-impregnated resins (SIR), which integrates the superior features of solvent extraction and ion exchange resin and avoids the emulsion and potential fire hazard issues in solvent extraction processes. Mondal et al. [32] studied the uptake

behavior of neodymium (Nd), lanthanum (La), yttrium (Y), and erbium (Er) from a synthetic solution at concentrations comparable to those found in coal fly ash leachate. First, the N,N,N',N'-tetra(2-ethylhexyl) diglycolamide (TEHDGA)-impregnated Amberlite XAD-7 resin was prepared by loading 50% (w/w) TEHDGA onto the XAD-7 resin matrix. Then, the adsorption of Nd, La, Y, and Er in 4 mol/L HNO₃ was conducted in a column. The uptake behavior indicated a stronger affinity for heavier rare earth elements Y and Er than the lighter rare earth elements Nd and La. In a study conducted by Zhang and coworkers [33], a trialkylphosphine oxide (TRPO)-impregnated Levextrel resin was synthesized and used to adsorb neodymium and zirconium from aqueous solution. The TRPO resin exhibited a significant adsorption ability, reaching 48 mg/g for Nd (III) and 47 mg/g for Zr (IV). Inan et al. [34] prepared a solvent-impregnated resin, i.e., the Cyanex 272-impregnated XAD-7 resin, to study its adsorption behavior for six rare earth elements, i.e., La, Pr, Nd, Sm, Eu, and Gd, from nitric acid solution. They discovered that the tested SIR had a stronger affinity toward the heavier elements Sm, Eu, and Gd than the lighter ones at pH 2.45 under temperature 289 K. A novel solvent-impregnated resin was prepared by Kovalenko et al. [35] to investigate the recovery efficiency of neodymium from nitric acid solution. The study reported the impact of the ionic liquid ratio in the SIR preparation on Nd adsorption. The results indicated that the SIR containing Cyanex 923 in the 2:1 ratio was the most effective, showing the strong potential of ionic liquid-based SIR for recovering REEs. Oye and co-workers [24] conducted a study in which they synthesized Resorcinol-TPA (terephthalaldehyde) resins and evaluated their adsorption performance for La (III), Nd (III), Dy (III), Eu (III), and Yb (III) ions from nitrate solutions. The results demonstrated that the resorcinol-TPA resin possessed excellent chemical stability and achieved nearly 100% adsorption efficiency for all the REEs at an optimal pH of 4.75.

In principle, selective uptake of rare earth elements is recognized as selective extraction or targeted adsorption of individual REEs or specific REE groups, which is considered crucial in both research and industrial applications. However, achieving selective separation and purification of a specific REE remains a major challenge due to the remarkable similarity in the chemical and physical properties of the REEs, despite small differences in ionic radii and oxidation states, especially between the light and heavy REEs [3,36]. Moreover, the uneven distribution and varying concentrations of REEs in leach solutions further complicate the selective separation process, necessitating optimized operating conditions and highly selective functional materials, such as ion-exchange resins bearing specific groups. Reported studies have demonstrated the limited effectiveness of conventional methods, such as solvent extraction and coprecipitation, in selectively separating individual rare earth elements (REEs), while ion exchange is considered a promising approach.

Yamada et al. [37] studied the separation efficiency of three REEs, i.e., Nd, Dy, and Pr from the leaching solution of waste NdFeB magnets using the solvent-impregnated resin functionalized with 2-ethylhexylphosphonic acid mono-2-ethylhexyl ester and coated with polyvinyl alcohol crosslinked by glutaraldehyde. Initially, Fe (III) was precipitated out using oxalic acid from the acid leaching solution of the waste NdFeB magnets. The rare earth oxalates from the left solution were then crystallized and calcined to produce the corresponding oxides, which were subsequently dissolved in 0.1 M nitric acid to prepare the feed solution for recovering the REEs. The separation process of Nd (III)/Pr (III) and Dy (III) was conducted via a chromatographic method. The results indicated that the Dy (III) was totally separated from the mixture, while the effluent containing Nd (III) and Pr (III) was further processed using the same chromatographic system, achieving a Nd purity of approximately 91.0%. The process they developed is complicated and requires large consumption of oxalic acid and energy, which demonstrates the difficulties of recovering and separating the REEs.

In this work, we aim to selectively separate and recover the REEs from the leaching solution of a spent NdFeB magnet, which only contains the REEs neodymium (Nd) and dysprosium (Dy), using a simpler and more practical approach than those previously reported, that is, direct resin adsorption. Up to now, there has been limited research in the literature on the separation and recovery of Nd and Dy directly from the leaching solutions of spent NdFeB magnets using ion-exchange techniques. The NdFeB magnet leaching solution and corresponding synthetic solution used contain Fe, Nd, and Dy with a large concentration difference, where the Fe concentration is over 2.16 times higher than that of Nd, and the Nd concentration is 30 times that of Dy in the solution (see Section 2.3), which increases the challenges of our work and could promote scientific understanding that facilitates the use of ion exchange technology to improve the purity of REEs, increase productivity, lower operation costs, and minimize environmental footprints. The commercial ion exchange resins explored, including Purolite S950 PLUS resin bearing aminophosphonic groups and Purolite MTX7010 resin impregnated with di-(2-ethylhexyl)phosphoric acid (D2EHPA), are listed in Table 2.

Table 1. Summary of reported studies on rare earth element recovery using various resins.

Used Resin	Functional Group	Target Elements	Elution Process	Adsorption Conditions			Resin Adsorption Efficiency	Ref.
				pH	T (K)	Time		
Strongly cation resin (SQS-6)	Sulfonic groups	La and Nd	HF + HCl	4.0	298	10 min	13.8 mg/g for La (III) 12.7 mg/g for Nd (III).	[28]
Weak acrylic resin (110 resin)	Carboxylic acid	Nd	3.0 M HCl	6.0	298	60 h	308 mg/g the maximum adsorption capacity	[38]
TRPO-impregnated Levextrel resin	Trialkylphosphine oxide	Nd, Zr	-	5.7	298	2 h	47 mg/g for Zr (IV) and 48 mg/g for Nd (III)	[33]
Cyanex 272 impregnated XAD-7	Phosphinic acid	La, Pr, Nd, Sm, Eu, Gd	0.01 M HNO ₃ or 0.01 M HCl	2.4	298	3 h	Gd > Eu > Sm > Nd > Pr > La	[34]
TEHDGA impregnated XAD-7	Diglycolamide	La, Nd, Y, and Er	0.01 M HNO ₃	-	-	40 min	Total load capacity of the La, Nd, Y, and Er 7.07 mg/g	[32]
Amberlite IRC-747 and Lewatit TP-260	Aminophosphonic and Aminomethylphosphonic	La, Nd, Gd, Dy, Er, Yb, Sc, and Y	H ₂ SO ₄ (9 M & 18 M) or Na ₂ CO ₃ 1 M	-	323	3 h	Maximum 1.8 meq/g	[14]
Purolite S957	Phosphonic & Sulphonic acid	La, Ce, Nd, Fe, Ni, Co, Cu and Zn	2.0 M HNO ₃ or 2.0 M HCl	-	293	1 h	50% Ni (II) And 100% La (III)	[26]
Dowex 50WX8	Sulphonic acid	Dy, Pr, and Y	1.0 M HNO ₃	-	298	15 min	30 mg/g Pr, 50 mg/g of Dy, 60 mg/g of Y	[31]
Resorcinol-terephthalaldehyde (RTPA)	Carboxylic acid	La ³⁺ , Nd ³⁺ , Eu ³⁺ , Dy ³⁺ , Yb ³⁺	2.0 M HNO ₃	4.7	298	60 min	Higher than 50 mg/g	[24]
Coated solvent-impregnated resin (SIR)	2-ethylhexyl-phosphonic acid mono-2-ethylhexyl ester	Nd ³⁺ , Pr ³⁺ , Dy ³⁺	1.0 M HNO ₃	2.0 and <2.0	298	24 h	0.230 mmol/g for Nd ³⁺ and 0.182 mmol/g for Dy ³⁺	[37]

Table 2. The ion-exchange resins used in this work Adapted from Ref. [22].

Ion Exchange Resin	Polymer Structure	Functional Groups	Delivery Form	Bed Size (mm)	Total Capacity	Maximum Operating Temperature	pH Range
Puromet MTS 9100	Polyacrylic crosslinked with divinylbenzene	Amidoxime	-	<0.3 max	40 g Cu/L	-	-
ResinTech SIR 500	Styrene/DVB Macroporous	Aminophosphonic	Na ⁺	0.297–1.19	>1.4	85 °C	2–10
Purolite S950plus	Macroporous polystyrene crosslinked with divinylbenzene		Na ⁺	0.425–0.850	24 g Ca/L	90 °C	0–14
Purolite S940	Macroporous polystyrene crosslinked with divinylbenzene		Na ⁺	0.425–0.850	20 g Ca/L	90 °C	0–14

Table 2. Cont.

Ion Exchange Resin	Polymer Structure	Functional Groups	Delivery Form	Bed Size (mm)	Total Capacity	Maximum Operating Temperature	pH Range
Puromet MTS9500	Macroporous Styrene-divinylbenzene	Aminophosphonic	Na ⁺	0.3–1.2	26 g/L	80 °C	0–14
Purolite MTS 9570	Macroporous polystyrene crosslinked with divinylbenzene	Phosphonic and Sulfonic acid	H ⁺	0.315–0.850	18 g Fe/L		0–14
Dowex G-26	Gel/Styrene-DVB	Sulfonic acid	H ⁺	0.650 ± 50	2	134 °C	0–14
Amberlite IRC-120	Gel/Styrene divinylbenzene copolymer		H ⁺	0.620–0.830	≥1.80 (H ⁺ form)	135 °C	0–14
Lewatit vp oc 1026	Macroporous/crosslinked polystyrene	Di-(2-ethylhexyl) phosphoric acid (D2EHPA)	H ⁺	0.31–1.65	13 g Zn/L	−20–40 °C	<4
Purolite MTX7010	Macroporous polystyrene crosslinked with divinylbenzene		H ⁺	0.3–1.66	13 g Zn/L	80 °C (max)	4 (max)
Lewatit MDS TP 260	Macroporous/Styrenic	Aminomethyl-phosphonic acid	Na ⁺	0.40 (+/− 0.04)	3 (H ⁺ form)	−20–40 °C	0–14
Amberlite IRN-150	Gel	Sulfonic acid (strong acid cation)/Trimethyl-ammonium(strong base anion)	H ⁺ /OH [−]	≥1.20 eq/L (OH [−] form) ≥1.90 eq/L (H ⁺ form)	≥1.90 eq/L (H ⁺ form) ≥1.20 eq/L (OH [−] form)	5–100 °C	0–14
Lewatit® TP 272	Macroporous/crosslinked polystyrene	Bis-(2,4,4-trimethylpentyl-) phosphinic acid		>90%	12.5 g/L	−20–40 °C	0–14
Purolite MTS9300	Macroporous polystyrene crosslinked with divinylbenzene	Iminodiacetic	Na ⁺	0.425–1.0	50 g/L	80 °C	0–14
Purolite S930 Plus	Macroporous crosslinked polymer		Na ⁺	0.425–1.0	2.9 eq/L	80 °C	0–14
Lewatit monoplus TP207	Macroporous crosslinked polymer		Na ⁺	0.61 (+/− 0.05)	2.0 eq/L	−20–40 °C	0–14
Lewatit monoplus TP208	Macroporous crosslinked polymer		Na ⁺	0.65 (+/− 0.05)	2.5 eq/L	−20–40 °C	0–14
Purolite S930	Macroporous crosslinked polymer		Na ⁺	0.60–0.85	30 g/L	70 °C	2–6 (H Form)/6–11 (Na Form)
PuroliteMTS9140	Macroporous polystyrene crosslinked with divinylbenzene	Thiourea	-	0.30–1.2	1 eq/L	100 °C	0–14
Lewatit monoplusTP214	Macroporous polystyrene crosslinked with divinylbenzene	Thiourea	-	0.55 (+/− 0.05)	1.1 eq/L	20–40 °C	0–14
ResinTech SIR-600	Zeolite Crystalline	Aluminosilicate	Na ⁺ /K	0.297–1.19	0.6	100 °C	6–10

Table 2. Cont.

Ion Exchange Resin	Polymer Structure	Functional Groups	Delivery Form	Bed Size (mm)	Total Capacity	Maximum Operating Temperature	pH Range
Puromet MTS9200	Microporous polystyrene crosslinked with divinylbenzene	Isothiouonium	H ⁺	0.63 (+/− 0.05)	1.7 eq/L	80 °C	0–7
Puromet MTS9240	Microporous polystyrene crosslinked with divinylbenzene	Thiol Chelating resins	H ⁺	0.3–1.0	200 g Hg/L	60 °C	-
DOWEX™ MAC-3	Polyacrylic, microporous	Carboxylic acid	H ⁺	0.30–1.2	3.8 eq/L	120 °C	5–14

2. Experimental

To achieve effective separation of iron, neodymium, and dysprosium, experiments were designed (as shown in Figure 1) to test and evaluate the adsorption efficiency and selectivity of 24 resins, which contain 14 different functional groups from various suppliers (Table 2).

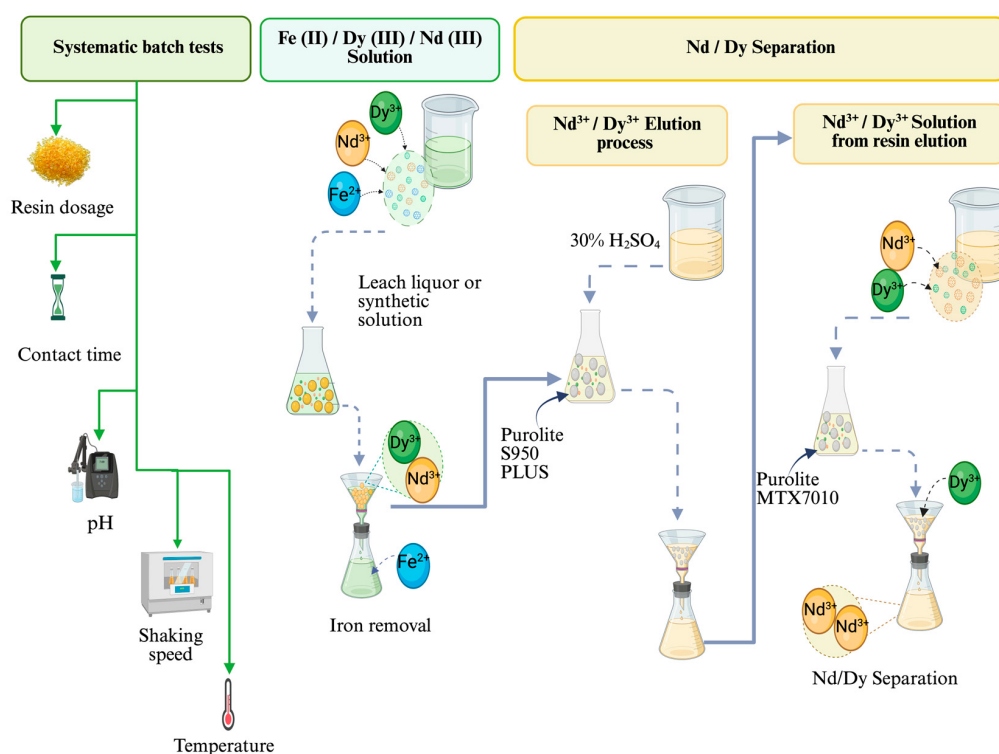


Figure 1. Experimental flowchart for recovering and separating Nd and Dy from leaching solution of spent NdFeB magnet using resin adsorption.

2.1. Chemicals and Instruments

For preparing the synthetic solutions, the chemicals used are $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ of ACS reagent grade, 99.9% pure neodymium (III) sulfate hydrate (Sigma-Aldrich, St. Louis, MO, USA, Cat. #325813-50G), and dysprosium (III) sulfate (ProChem Inc., Rockford, IL, USA), where the pH of the solution was adjusted with 1 M hydrochloric acid, freshly prepared by diluting concentrated 37% HCl (Fisher Scientific, Pittsburgh, PA, USA, Cat. #A144S-212). Twenty-four resins containing 14 different functional groups were used in this study (see Table 2). Throughout the experimental procedures, deionized (DI) water purified via a

Millipore Milli-Q water purification system was used. The collected solution samples were analyzed using Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES). We measured the pH using a Thermo Scientific Orion Star A111 pH meter (Waltham, MA, USA). A platform shaker (Promax 2020, Heidolph Instruments GmbH & Co. KG, Schwabach, Germany) and an incubator shaker (InnovaR43, New Brunswick Scientific, Edison, NJ, USA) were used for adsorption tests.

2.2. Preparing and Activation of the Resins

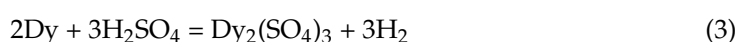
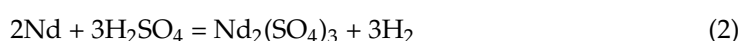
Initially, all the resins used in the experiments were activated to achieve homogeneity, remove impurities or preservatives, initiate active sites, ensure consistent swelling and performance of the resin. This pre-conditioning involved treating the appropriate amount of resin (e.g., 1 or 10 g of resin) in 20 mL of 30% (v/v) HCl solution and shaking it on a platform shaker (Promax 2020, Heidolph Instruments GmbH & Co. KG, Schwabach, Germany) or incubator shaker (InnovaR 43, New Brunswick Scientific, Edison, NJ, USA) at 150 rpm for 24 h, then the resin–liquid mixture was filtered using large pore size filter paper (e.g., Whatman® qualitative filter paper) on a funnel. The resin was washed with 50 mL deionized water, then being continuously shaken in 20 mL deionized water at 150 rpm for 1 h and filtered. Finally, the resin was dried at room temperature for 12 h before the adsorption test. All solid–liquid adsorption experiments were carefully designed based on the chemical solutions intended for resin testing (see Table 3).

Table 3. The composition of the prepared synthetic solutions.

Solutions	Conc. (mg/L)	Chemicals	pH
$\text{Fe}^{2+} + \text{Nd}^{3+} + \text{Dy}^{3+}$	13,000 of Fe, 6000 of Nd and 200 of Dy	$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ $\text{Nd}_2(\text{SO}_4)_3 \cdot x\text{H}_2\text{O}$ and $\text{Dy}_2(\text{SO}_4)_3 \cdot 8\text{H}_2\text{O}$	0.65
Nd^{3+}	6000 of Nd	$\text{Nd}_2(\text{SO}_4)_3 \cdot x\text{H}_2\text{O}$	0.65
Dy^{3+}	200 of Dy	$\text{Dy}_2(\text{SO}_4)_3 \cdot 8\text{H}_2\text{O}$	0.65
$\text{Nd}^{3+} + \text{Dy}^{3+}$	6000 of Nd and 200 of Dy	$\text{Nd}_2(\text{SO}_4)_3 \cdot x\text{H}_2\text{O}$ and $\text{Dy}_2(\text{SO}_4)_3 \cdot 8\text{H}_2\text{O}$	0.65

2.3. Leaching of NdFeB Magnet Powder and Preparing Synthetic Solutions for the Adsorption Experiments

Spent NdFeB magnet powder with an average particle size of 50 to 60 μm was supplied by Intelligent Materials Private Limited. The composition of the magnet powder was 64–68 wt.% Fe, approximately 30 wt.% Nd, and 0.8–1.2 wt.% Dy. 20 g of NdFeB magnet powder was leached in 1000 mL of 30% (v/v) H_2SO_4 (5.63 M) solution at 50 °C. Complete dissolution of the magnet powder was achieved within 30 min. The resulting clear leachate contained 12,995 ppm Fe, 5990 ppm Nd, and 203 ppm Dy via ICP analysis. The leaching process can be represented as below:



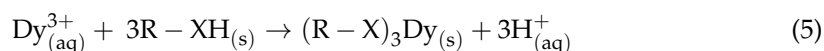
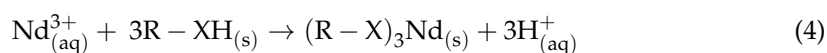
The ferrous ions (Fe^{2+}) produced could be oxidized to ferric ions (Fe^{3+}) within several hours in open air. To obtain stable and consistent results, synthetic solutions prepared freshly were used for adsorption tests unless otherwise specified.

For the adsorption experiments, four individual solutions were prepared (see Table 3). The first one is the ternary solution containing Fe^{2+} , Nd^{3+} , and Dy^{3+} ions, prepared by dissolving the required amounts of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, $\text{Nd}_2(\text{SO}_4)_3 \cdot x\text{H}_2\text{O}$, and $\text{Dy}_2(\text{SO}_4)_3 \cdot 8\text{H}_2\text{O}$ in 1000 mL deionized water, yielding concentrations of 13,000 mg/L Fe, 6000 mg/L Nd, and 200 mg/L Dy. The second and third solutions were single metal solutions containing 6000 mg/L Nd and 200 mg/L Dy, respectively. The fourth solution was a binary solution containing 6000 mg/L Nd and 200 mg/L Dy (see Table 3). The pH of the synthetic solutions was adjusted using 1M hydrochloric acid.

2.4. Resin Adsorption Tests

The adsorption experiments were conducted using 24 resins comprising 14 different functional groups (listed in Table 2). The initial pH of the solutions was adjusted to 0.65 using 1 M HCl. The tests were performed with different resin dosages (i.e., 0.05, 0.1, 0.15, 0.2, and 0.3 g/mL), each soaked in 20 mL of solution in 50 mL Erlenmeyer flasks in batch tests. These samples were agitated on a shaker at a fixed speed of 150 rpm for 60 min at room temperature unless otherwise specified.

Since the resin (represented here as $\text{R}-\text{XH}$) provides protonated functional groups, the uptake of the rare earth ions could occur via ion exchange with these protons. The adsorption (ion-exchange) of Nd^{3+} and Dy^{3+} ions onto cation-exchange resins can be represented by the general chemical reactions shown in Equations (4) and (5), respectively, where the protons in the resin's functional groups are replaced by the trivalent rare-earth cations.



The adsorption efficiency (i.e., metal recovery on the resin in adsorption process), $\text{R}\%$, of the Nd (III) and Dy (III) ions and the ion exchange capacity of the resin, q_e , in (mg/g) at equilibrium, were calculated using the following relationships represented in Equations (6) and (7):

$$\text{R}\% = \left(\frac{C_0 - C_e}{C_0} \right) \times 100\% \quad (6)$$

$$q_e = (C_0 - C_e) \times V/m \quad (7)$$

where q_e refers to the amount of Nd^{3+} and Dy^{3+} ions adsorbed on the polymeric resin. C_0 and C_e represent the Nd^{3+} and Dy^{3+} ion concentrations in the solution (mg/L) at the initial time and the equilibrium, respectively. V is the volume of solution (0.020 L here), and m is the mass of polymeric resin (g). The filtered solution samples were analyzed using Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) to determine metal concentrations.

The elution rate, i.e., metal recovery in the eluted (or washing) solution was also calculated from the metal concentration analyzed following similar physical significance to $\text{R}\%$. Elution rates of metals refer to the eluted metal percentages for the current elution process.

The prospective purity (pp%) of Dy or Nd was calculated with Equation (8):

$$\text{pp}\% = \left(\frac{C_1}{C_1 + C_2} \right) \times 100\% \quad (8)$$

where C_1 and C_2 refer to the concentrations of metal 1 (e.g., Dy or Nd) and metal 2 (e.g., Nd or Dy) in a purified solution with certain volume V_p (0.020 L here). Purified metal Dy or Nd in the solution could be produced via concentration by resin adsorption–elution followed by electrowinning.

Metal recovery in a purified solution with certain volume V_p was calculated with Equation (9):

$$\text{recovery}\% = \left(\frac{C}{C_{\text{start}}} \right) \times 100\% \quad (9)$$

where C and C_{start} refer to the metal concentration in the purified solution and metal concentration before purification with certain volume V_p , respectively.

3. Results and Discussion

3.1. Iron Removal

To obtain purified Nd and Dy, iron removal from the (simulated) leaching solution is the first step, which was systematically explored using 24 ion exchange resins (Table 2). Among the resins, those functionalized with aminophosphonic groups demonstrated favorable selectivity, i.e., exhibiting strong adsorption toward Nd (III) and Dy (III) while having low adsorption efficiency for Fe (II). Based on this screening, Purolite S950 PLUS bearing aminophosphonic groups, was selected for further investigation.

As shown in Figure 2, at a contact time of 60 min and pH 0.65, the adsorption efficiency of Fe (II) increased gradually from 5.13% at a resin dosage of 0.05 g/mL to 16.90% at 0.30 g/mL. Despite the increased resin dosage, Fe (II) adsorption remained relatively low, indicating a weak interaction between Fe (II) ions and the aminophosphonic functional groups under given conditions. Such selective behavior is highly desirable in hydrometallurgical processing and demonstrates the potential of this resin for effective Fe/REEs separation.

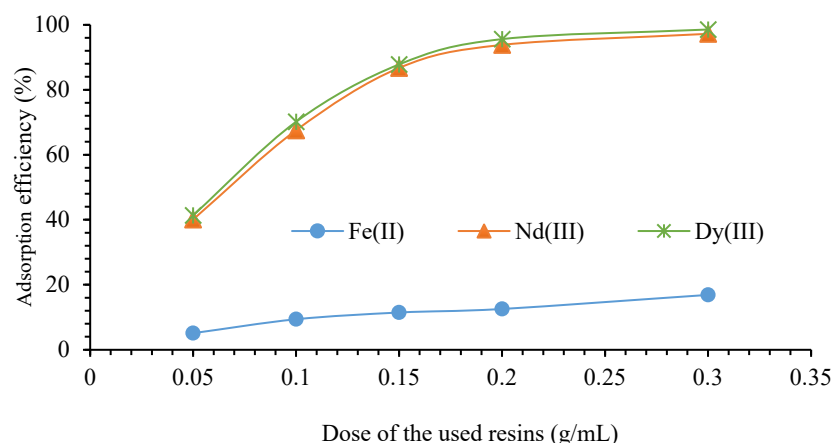


Figure 2. Effect of resin dosage on the adsorption of Fe^{2+} , Nd^{3+} , and Dy^{3+} ions at pH 0.65, 150 rpm, room temperature, and 60 min adsorption time.

In contrast, Nd (III) and Dy (III) exhibited a pronounced increase in adsorption efficiency with increasing resin dosage. The adsorption efficiency improved from 40.1% and 41.4% at 0.05 g/mL to 97.3% and 98.6% at 0.30 g/mL for Nd and Dy, respectively. This substantial enhancement indicates a strong affinity between the resin and the trivalent rare earth ions. Their preferential adsorption is likely attributed to their higher charge density and favorable coordination interactions with aminophosphonic functional groups.

Further investigation was conducted to evaluate the effect of contact time on adsorption performance using Purolite S950 PLUS at pH 0.65 and a resin dosage of 0.1 g/mL. As illustrated in Figure 3, the three metal ions displayed distinct adsorption behaviors. Nd (III) and Dy (III) achieved consistently high adsorption efficiencies, exceeding 96% at the shortest contact time (10 min), with only a slight decline upon prolonged contact. This slight decrease may be associated with the limited resin dosage and possible equilibrium

redistribution, as higher resin amounts and optimized contact time have previously shown improved stability in removal efficiencies. Conversely, Fe (II) exhibited a very low adsorption rate, reaching 5.11% at the shortest contact time, with small improvement over extended durations. This behavior further confirms the relatively weak interaction between Fe (II) and the resin at the test conditions. The differences in adsorption performance can be attributed to variations in ionic charge, ionic radius, and coordination chemistry between divalent Fe (II) and trivalent rare earth ions.

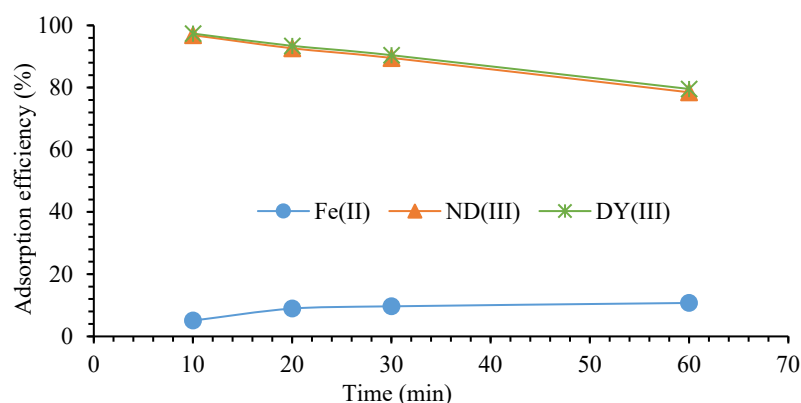


Figure 3. Impact of contact time on the adsorption efficiency of Fe²⁺, Nd³⁺, and Dy³⁺ ions at pH 0.65, 150 rpm, room temperature, and 0.1 g/mL resin dosage.

From a separation standpoint, the aminophosphonic-functionalized resin demonstrates clear advantages due to the persistent and significant adsorption efficiency gap between Fe (II) and Nd (III)/Dy (III) under the test conditions. This behavior directly supports the proposed process strategy, wherein most of the Fe (II) is selectively retained in solution during the first separation step while Nd (III) and Dy (III) are effectively captured on the resin. Consequently, improved separation efficiency and enhanced downstream purification of neodymium and dysprosium can be achieved.

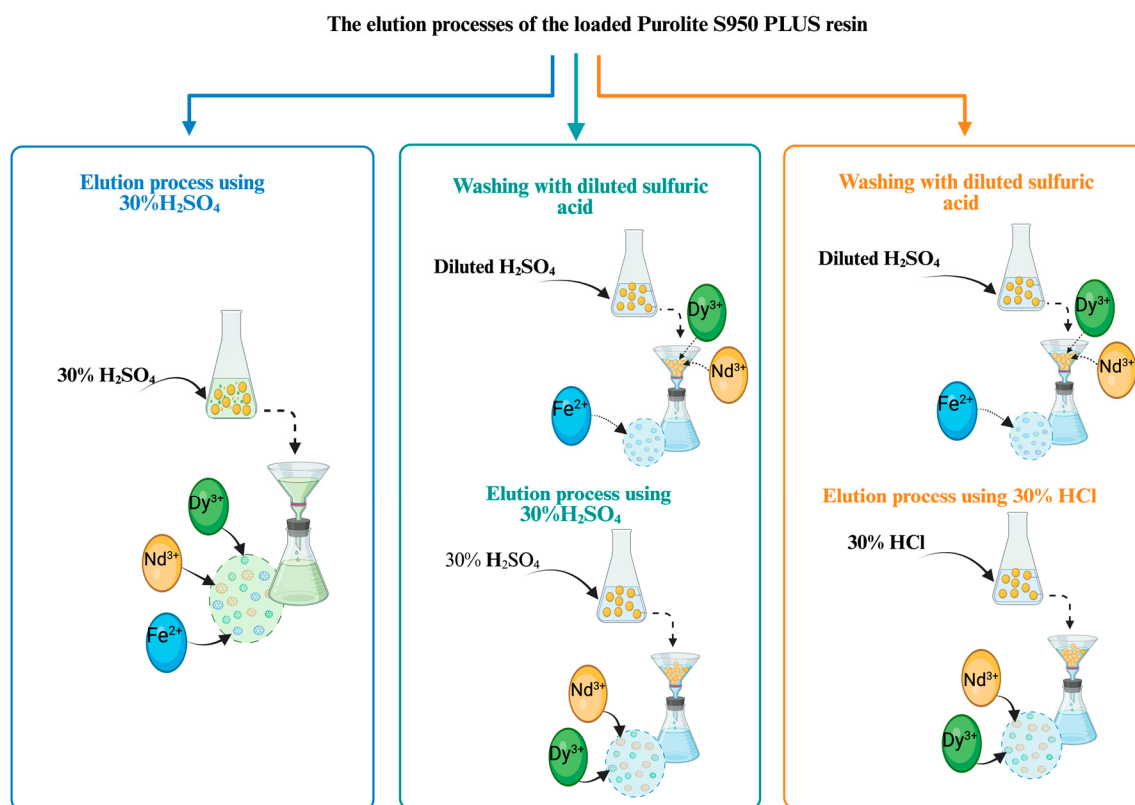
As shown in Figure 3, less than 700 mg/L equivalent of Fe (II) remained associated with the resin after 10 min adsorption. This corresponds to less than one-eighth of the adsorbed neodymium and about three times the amount of dysprosium retained on the resin. Comparing with the composition of the synthetic solutions (Table 3), efficient and selective iron removal was obtained.

3.2. Fe, Nd and Dy Elution from Loaded Purolite S950 PLUS Resin

As illustrated in Figure 4 and Table 4, three different elution processes were investigated. In the first approach, 30% (v/v) H₂SO₄ was applied for 30 min elution immediately following the filtration after adsorption to assess its stripping effect without a preceding rinsing step. As summarized in Table 4, samples 1, 2, and 3 present the corresponding adsorption and elution performance data. The use of 30% (v/v) H₂SO₄ resulted in substantial desorption of Fe, Nd, and Dy, with elution rates ranging from approximately 92% to over 98%, demonstrating the strong stripping capability of the 30% (v/v) H₂SO₄ solution. However, the residual concentration of Fe ions in eluted solutions remained relatively high. This is because even Fe (II) adsorption efficiency during the loading stage was very limited, its amount on the resin was still relatively large due to its high concentration in the initial leaching solution, which will adversely affect downstream separation efficiency and product purity. Therefore, a washing step with dilute H₂SO₄ solution of pH 0.65 was introduced to selectively rinse residual Fe (II) off prior to elution, thereby improving the overall separation performance and reducing iron contamination in the recovered rare earth fraction.

Table 4. Adsorption and elution (including washing) efficiency for iron removal.

Sample	Fe ²⁺		Nd ³⁺		Dy ³⁺		Washing Process				Elution Process			
	Final Conc. in Solution (mg/L)	Resin Ad-sorption Efficiency %	Final Conc. in Solution (mg/L)	Resin Ad-sorption Efficiency %	Final Conc. in Solution (mg/L)	Resin Ad-sorption Efficiency %	Washing Time (min.) Diluted H ₂ SO ₄ (pH 0.65)	Elution Rate			Elution	Elution Rate		
								Fe ²⁺ %	Nd ³⁺ %	Dy ³⁺ %		Fe ²⁺ %	Nd ³⁺ %	Dy ³⁺ %
1	11,747.78	5.39	284.34	94.10	5.81	97.35	---	---	---	---	30% H ₂ SO ₄	91.71	97.53	98.29
2	11,795.85	5.00	268.67	94.42	6.10	97.22	---	---	---	---	30% H ₂ SO ₄	95.04	98.13	97.33
3	11,806.58	4.92	256.00	94.69	5.36	97.55	---	---	---	---	30% H ₂ SO ₄	95.27	98.56	98.43
4	11,787.82	5.07	214.46	95.55	6.10	97.22	5 min	81.7	0.038	0.327	30% H ₂ SO ₄	97.9	96.0	99.1
5	11,798.27	4.98	206.27	95.72	6.61	96.98	10 min	83.1	0.046	0.329	30% H ₂ SO ₄	96.0	95.8	99.4
6	11,728.97	5.54	275.94	94.27	7.08	96.77	20 min	74.7	0.037	0.330	30% H ₂ SO ₄	96.3	97.2	99.1
7	11,698.95	5.78	229.78	95.23	6.98	96.81	5 min	82.2	0.043	0.250	30% HCl	97.9	96.0	99.1
8	11,741.72	5.44	219.00	95.45	7.85	96.42	10 min	83.5	0.049	0.336	30% HCl	96.0	95.8	99.4
9	11,697.23	5.80	226.45	95.30	8.03	96.33	20 min	82.5	0.057	0.303	30% HCl	96.3	97.2	99.1

**Figure 4.** Flow sheet of three different elution processes.

After adsorption and filtration, washing times of 5, 10, and 20 min were tested to remove weakly bound Fe (II) from the resin. From the data of samples 4 to 9 presented in Table 4, Fe removal during the washing step was significant; more than 80% of the loaded Fe was rinsed off the resin. This indicates that most of the loaded Fe (II) was weakly associated with the resin and could be selectively desorbed. In contrast, Nd³⁺ and Dy³⁺ losses during washing were negligible, with elution rates remaining extremely low ($\leq 0.06\%$), confirming their strong coordination with the aminophosphonic functional groups.

Increasing washing time from 5 to 20 min did not result in substantial additional benefit, suggesting that most loosely bound Fe (II) is removed within the first 5 min. These findings confirm that the washing stage effectively enhances selectivity by rinsing most of the attached Fe (II) while preserving nearly all adsorbed Nd (III) and Dy (III).

Following the washing stage, the elution process was evaluated using 30% (v/v) H_2SO_4 or 30% (v/v) HCl for a contact time of 30 min. Both acids demonstrated high elution efficiencies. Neodymium elution rates ranged from 95.8% to 97.2%, while dysprosium exhibited the highest elution rates, consistently exceeding 99%. The remaining Fe (II) was also effectively eluted, with efficiencies between 96.0% and 97.9%. These high elution efficiencies confirm that the strongly adsorbed Nd (III) and Dy (III) ions can be effectively stripped from the resin using concentrated acid solutions. This ensures efficient resin regeneration and recycling in the process, which is critical for commercial production.

The rare earth elements left in the solution after resin adsorption can be further recovered. And purified Fe containing solution and Nd-Dy mixed solution can be obtained by applying a distillation-analogous mechanism with optimized multiple adsorption–elution processes as discussed in Section 5.

3.3. Separation of Dy and Nd Using Resin Impregnated with di-(2-Ethylhexyl) Phosphoric Acid (D2EHPA)

Studies have been carried out to investigate the removal and recovery of neodymium (Nd) and dysprosium (Dy) from various aqueous media using different methods, including solvent extraction, precipitation, and ion-exchange [39,40]. Despite these developments, the selective separation of Nd from Dy still represents a challenging task. These two metals possess surprisingly similar chemical and physical properties as trivalent cations of rare earth elements [17]. They, however, belong to different categories of the lanthanide series: Nd is classified as a light rare earth element (LREE) and Dy as a heavy rare earth element (HREE). This categorization reveals subtle but distinctive differences in ionic size, wherein the Nd^{3+} value (1.109 Å) shows a larger size as compared to Dy^{3+} (1.027 Å) [36,41]. While these small differences in size and bonding behaviors could be exploited for the purposes of separation. Overall, their high resemblance demands the utilization of highly specific ligand-functionalized ion-exchange materials, or multi-step procedures to realize efficacious discrimination between Nd (III) and Dy (III).

A comprehensive investigation was conducted on 14 different functional groups (Table 2) to identify the most effective resins for adsorbing Nd or Dy. Through explored experiments, it was found that SIR impregnated with di-(2-ethylhexyl) phosphoric acid (D2EHPA), e.g., Purolite MTX7010 resin, had an adsorption preference for Dy^{3+} over Nd^{3+} , which is highly suitable for Nd separation from mixed Nd and Dy solutions under certain conditions. The binary solution of Nd and Dy was prepared according to the procedure outlined in Section 2.3.

3.3.1. Effect of Resin Dosage

Batch adsorption experiments using the Purolite MTX7010 resin were performed to evaluate the competitive uptake of Nd and Dy from binary solutions. The experiments were conducted under the conditions of pH 0.65, agitation speed 150 rpm, and various resin dosages (i.e., 0.05, 0.1, 0.15, 0.2, and 3.0 g/mL) to study the impact of resin dosage on the adsorption efficiency of Dy^{3+} and Nd^{3+} at room temperature. As shown in Figure 5, the Dy adsorption recovery rises rapidly, reaching over 76% at the lowest dosage of 0.05 g/mL and 90% at 0.3 g/mL. While Nd^{3+} exhibits a much lower adsorption recovery, beginning from 4.7% at 0.05 g/mL and peaking at about 25% at 0.3 g/mL as resin dosage increases. This pattern indicates that the resin greatly favors Dy^{3+} adsorption, which demonstrates

Dy^{3+} has a higher affinity to the active sites on the resin despite the higher concentration of Nd^{3+} in the solution.

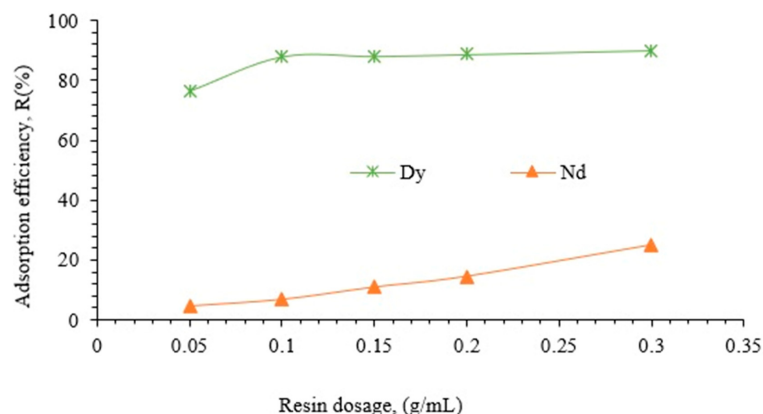


Figure 5. Effect of resin dosage on Nd^{3+} and Dy^{3+} adsorption.

3.3.2. Effect of Adsorption Time

The adsorption of Nd^{3+} and Dy^{3+} was tested over 5–150 min at pH 0.65, 0.1 g/mL resin dosage, 150 rpm agitation, and room temperature. As shown in Figure 6, Dy exhibits rapid uptake, with adsorption efficiency reaching over 87% within the first 5 min, then levelling off around 88% with longer adsorption times. In contrast, Nd^{3+} shows a lower adsorption efficiency of around 22% after 150 min. This demonstrates the stronger affinity of ligands on the resin for Dy^{3+} ions. This preferential sorption is probably because the smaller ionic radius ($\text{Dy}^{3+} \approx 0.91 \text{ \AA}$ vs. $\text{Nd}^{3+} \approx 0.983 \text{ \AA}$) and higher charge density of Dy^{3+} [36] enhance its electrostatic interaction and coordination stability with the di-(2-ethylhexyl)phosphoric acid (D2EHPA) on the resin.

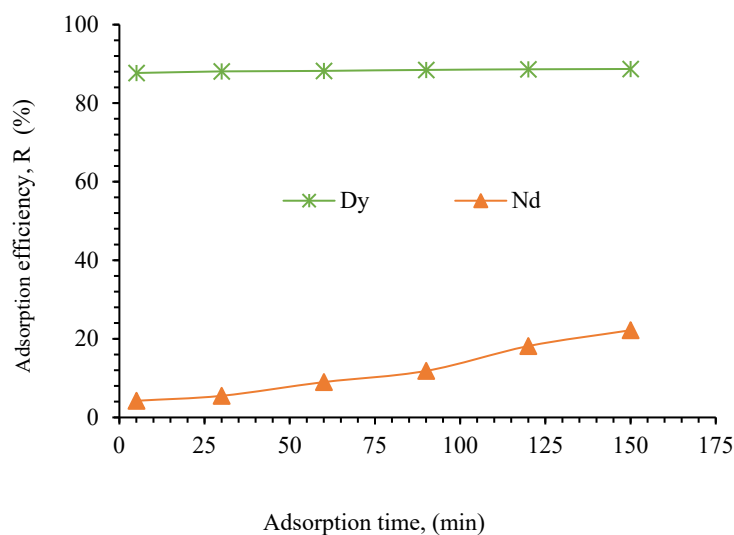


Figure 6. Effect of adsorption time on Nd^{3+} and Dy^{3+} adsorption.

3.3.3. Effect of pH on Metal Adsorption

The effect of pH on adsorption efficiency was examined from pH 0.25 to 2.5 using 0.1 g/mL Purolite MTX7010 resin for a contact time of 5 min at room temperature. Figure 7 shows that pH has remarkable influences on the adsorption efficiency (R%) of Dy (III) and Nd (III) ions, highlighting a distinct difference between them. Dy (III) adsorption efficiency increases sharply from 63.64% at pH 0.25 to over 87% at pH 0.6. Beyond this point, the efficiency stabilizes, remaining around 88% despite further pH increases. This indicates

that, even in highly acidic conditions, Dy (III) maintains strong interactions with the resin's functional groups. In contrast, Nd³⁺ exhibits a slower rise in adsorption efficiency as pH increases. Notably, Nd adsorption efficiency was below 1% at the lower pH values and grew to over 35% at the highest pH (2.5). This suggests that Purolite MTX7010 resin has a stronger affinity for Dy within this pH range than for Nd. The neodymium's adsorption efficiency, although limited, improves with rising pH. This may be due to more dissociation of H⁺ from di-(2-ethylhexyl) phosphoric acid on the resin at higher pH, which benefits the ion exchange process for Nd³⁺.

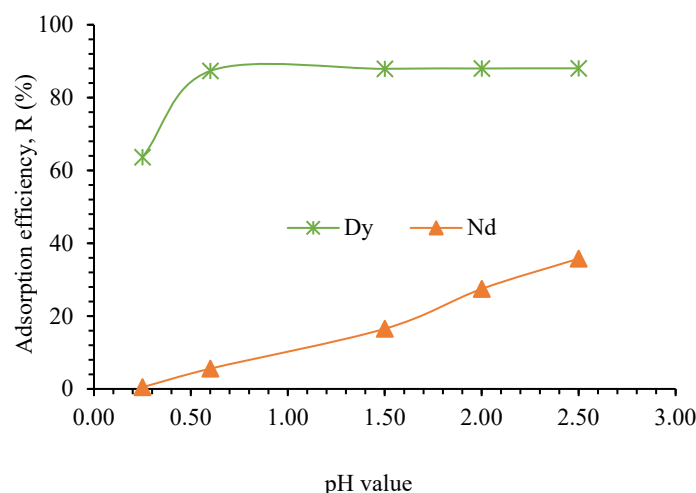
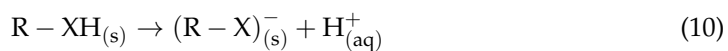


Figure 7. Effect of pH on Nd³⁺ and Dy³⁺ adsorption.

3.3.4. Effect of Shaking Speed (rpm)

The influence of shaking speed on the adsorption performance of Nd and Dy ions was studied at a contact time of 5 min, a resin dosage of 0.1 g/mL, and a pH of 0.65 under room temperature. As shown in Figure 8, the adsorption efficiency (R%) of Dy rises sharply with increasing shaking speed, exceeding 88% at 150 rpm, and then shows minimal improvement at higher speeds. In contrast, Nd adsorption remains low across all rpm levels. This indicates that the resin preferentially adsorbs Dy; higher agitation speed enhances mass transfer, improves access of metal ions to active sites, and further boosts their absorption.

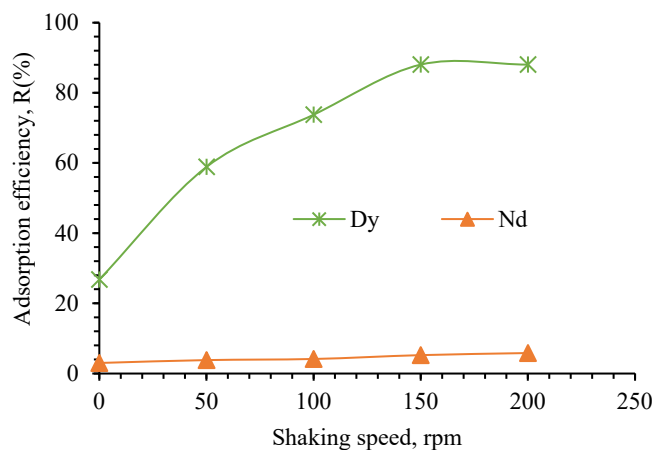


Figure 8. Effect of agitation speed on Nd³⁺ and Dy³⁺ adsorption.

3.3.5. Impact of Temperature on Metal Adsorption

Experiments were carried out to investigate how temperature affects the adsorption efficiency of Nd and Dy using Purolite MTX7010 resin at an adsorption time of 5 min, a resin dosage of 0.1 g/mL, an agitation speed of 150 rpm, and a pH of 0.65. As shown in Figure 9, the resin exhibited a strong preference for adsorbing Dy (III) ions across the tested temperature range, with the adsorption efficiency slightly increasing from 88.08% at room temperature (22 °C) to 90.48% at the highest temperature tested (70 °C). For Nd, the adsorption efficiency was below 6% at 22 °C but increased to 21% at 70 °C, which exhibits the endothermic character of this adsorption process as temperature increment favors an endothermic reaction thermodynamically.

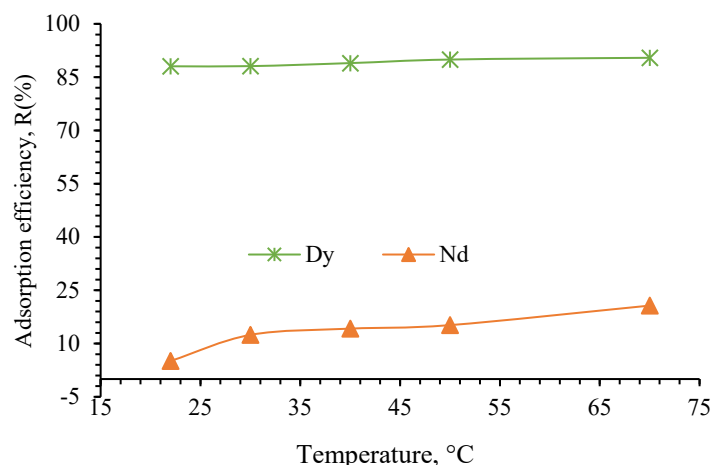


Figure 9. Impact of temperature on Nd³⁺ and Dy³⁺ adsorption.

4. Nd and Dy Elution from Loaded Purolite MTX7010 Resin

The recovery of adsorbed metal ions from ion exchange resins is known as the stripping or elution process, which utilizes different eluting reagents such as acids under various operating conditions [18,42,43]. Additionally, this process is considered significant for regenerating and reusing the resin, thereby enhancing the overall sustainability and cost-effectiveness of the ion-exchange system.

The adsorption tests using Purolite MTX7010 resin were conducted at an adsorption time of 5 min, a resin dosage of 0.1 g/mL, an agitation speed of 150 rpm, and a pH of 0.65 under room temperature. The loaded resin was obtained after filtration. The elution of Nd and Dy from the loaded resin was examined using 20 mL H₂SO₄ and HCl with three concentrations (i.e., 10% v/v, 20% v/v, and 30% v/v) in a two-stage process, with each stage lasting 30 min.

Data from Table 5 confirmed that both acids showed strong elution efficiency for Nd and Dy, with elution rate increasing as acid concentration rose. In the first stage, the highest elution efficiency with 30% v/v H₂SO₄ exceeded 91% for Nd and 95% for Dy; similarly, 30% v/v HCl achieved over 91% elution rate for Nd and over 94% elution rate for Dy. To recover the remaining Nd and Dy retained on Purolite MTX7010 resin, a second elution stage was performed using the same method as the first one. Obviously, overall stripping efficiency could be improved with second elution. Both acids demonstrated high elution efficiency, which is similar to other works [13].

Table 5. Summary of the elution processes for loaded Purolite MTX7010 resin using H₂SO₄ and HCl at three different concentrations in two separate stages.

Adsorption				First Elution Stage for 30 min.						Second Elution Stage for 30 min.					
Nd ³⁺		Dy ³⁺		Loaded Nd ³⁺ (mg/L)	Loaded Dy ³⁺ (mg/L)	H ₂ SO ₄					H ₂ SO ₄				
Conc. (mg/L)	R%	Conc. (mg/L)	R%			Acid Conc.	Eluted Nd (mg/L)	Eluted Dy (mg/L)	Nd ³⁺ (%)	Dy ³⁺ (%)	Acid Conc.	Eluted Nd ³⁺ (mg/L)	Eluted Dy ³⁺ (mg/L)	Nd ³⁺ (%)	Dy ³⁺ (%)
5137.23	5.62	24.90	89.10	306.05	203.49	10%	236.87	178.93	75.83	87.50	10%	64.04	21.35	84.84	83.50
5130.37	5.75	24.11	89.44	312.91	204.28	20%	253.06	189.00	80.24	92.55	20%	56.70	13.15	86.18	86.42
5132.13	5.72	24.70	89.19	311.15	203.69	30%	287.88	195.78	91.15	95.72	30%	26.27	8.41	93.92	96.06

2 g resin dosage and 5 min contact time						First elution stage for 30 min.					Second elution stage for 30 min.				
Nd ³⁺		Dy ³⁺		loaded Nd ³⁺ (mg/L)	loaded Dy ³⁺ (mg/L)	HCl					HCl				
conc. (mg/L)	R%	conc. (mg/L)	R%			acid conc.	eluted Nd (mg/L)	eluted Dy (mg/L)	Nd ³⁺ (%)	Dy ³⁺ (%)	acid conc.	eluted Nd ³⁺ (mg/L)	eluted Dy ³⁺ (mg/L)	Nd ³⁺ (%)	Dy ³⁺ (%)
5130.92	5.74	23.90	89.54	312.36	204.49	10%	230.72	174.34	75.39	85.68	10%	60.72	24.17	80.61	82.93
5127.89	5.79	24.17	89.42	315.38	204.22	20%	259.67	182.74	82.99	89.46	20%	45.58	19.65	85.62	86.61
5127.42	5.80	23.86	89.55	315.85	204.53	30%	283.89	191.62	91.24	94.07	30%	25.86	11.57	94.86	95.78

5. Multistage Adsorption–Elution Process for Nd and Dy Purification

As discussed in Section 3.3, the Dy adsorption efficiency could reach over 88%. Conversely, Nd recovery remained below 6%, but due to its higher concentration in the Nd-Dy solution, it still accounted for a larger concentration than Dy in the elution solution. To address this main challenge, a multistage adsorption–elution process analogous to distillation (i.e., the metal loaded on resin analogous to vapor and the left solution analogous to heavy oils) will be employed to achieve high separation and purification efficiency by taking advantage of the strong resin affinity for Dy, given the substantial concentration difference. Experiments were carried out to evaluate a multistage adsorption–elution process for separating neodymium and dysprosium using Purolite MTX7010 resin under optimized conditions (pH 0.65, agitation 150 rpm, room temperature, contact time 5 min) with varying resin dosages. Figure 10 illustrates the entire sequence of three multi-adsorption periods and two elution phases with the initial solution containing 266.34 mg/L Dy and 6049.87 mg/L Nd. The results of Nd and Dy separation via multistage adsorption–elution processes using Purolite MTX7010 are shown in Table 6.

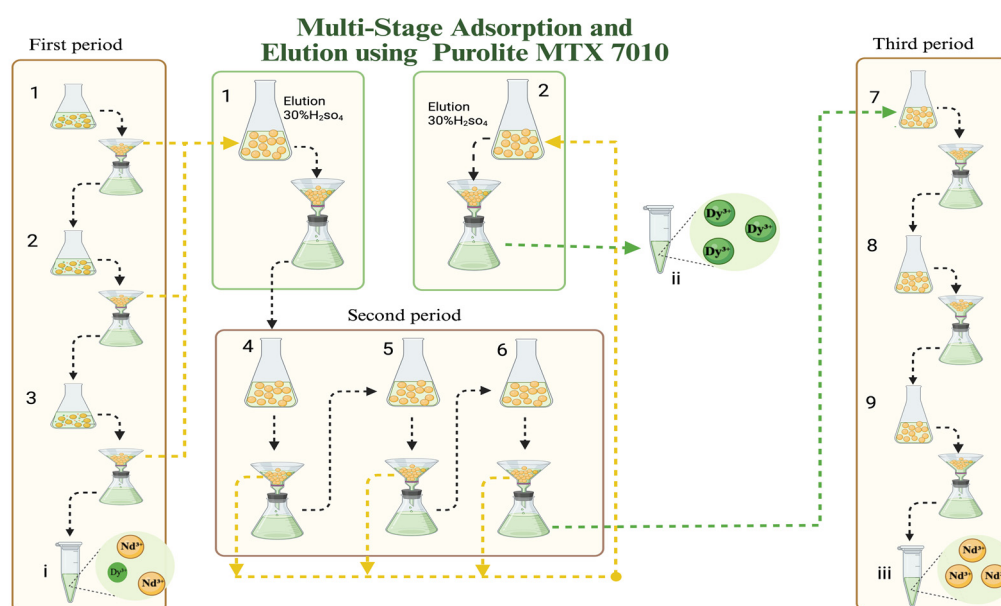
**Figure 10.** Nd and Dy separation via multistage adsorption–elution process with Purolite MTX7010.

Table 6. Results of Nd and Dy separation via multistage adsorption–elution.

Solution	Dy Conc. (mg/L)	Nd Conc. (mg/L)	Dy Recovery %	Nd Recovery %	Dy Purity	Nd Purity
Start solution	266.34	6049.87			4.22%	95.78%
Solution-i	5.59	5660.82	2.10	93.57	0.10%	99.90%
Solution-ii	258.48	3.90	97.05	0.06	98.51%	1.49%
Solution-iii	0.02	377.04	0.0075	6.23	0.01%	99.99%

During the first adsorption period for Nd purification, a resin dosage of 0.1 g/mL (i.e., 2 g resin) was used in adsorption stage 1, followed by 0.05 g/mL in adsorption stage 2 and 0.025 g/mL in adsorption stage 3. The ending solution-i from the first adsorption period was analyzed, which contained 5660.82 mg/L Nd and 5.59 mg/L Dy, representing a prospective purity of 99.90% (calculated with Equation (8)) and a recovery of 93.57% (calculated with Equation (9)) for Nd in the solution, as listed in Table 6, where Dy adsorption recovery was 97.90%, while Nd adsorption recovery was 6.43%, confirming that multi-stage adsorption effectively facilitated the separation of Nd and Dy.

The first elution phase was performed on the resins collected from the adsorption stages 1, 2, and 3 in the first adsorption period (3.5 g in total) using 20 mL of 30% (*v/v*) H₂SO₄ for 30 min. Then followed by the second adsorption period for Dy purification, which included adsorption stages 4, 5, and 6, with resin dosages of 0.05 g/mL, 0.015 g/mL, and 0.010 g/mL, respectively. The final solution served as input for the third adsorption period, including stages 7, 8, and 9 for further Nd recovery. The total resin collected (1.5 g) from the second adsorption period was used in the second elution phase for purified Dy recovery, with the same volume and concentration of H₂SO₄ as in the first elution. The obtained solution-ii was analyzed with 258.48 mg/L Dy and 3.90 mg/L Nd representing a prospective purity of 98.51% and a recovery of 97.05% (calculated with Equations (8) and (9)) for Dy, which confirmed the expectations for Dy recovery and purification. The third adsorption period consisted of three adsorption stages: 7, 8, and 9, with resin doses of 0.5 g, 0.2 g, and 0.1 g, respectively. The analysis of the final solution-iii with a Nd concentration of 377.04 mg/L and a Dy concentration of 0.02 mg/L indicated the prospective purity for Nd above 99.99%.

The above process can be employed for the further processing of solution-i obtained from the first adsorption period for Nd purification to produce Nd with prospective purity above 99.90% Nd. Solution-ii can also be further processed to produce purer Dy. Overall, the multistage adsorption–elution process, analogous to distillation, successfully enhanced separation efficiency and obtained high-purity neodymium and dysprosium.

6. Conclusions

Due to limited supply channels, recycling REEs from secondary resources with high REE contents such as spent NdFeB magnetic materials would be an efficient approach for sustainable production.

Spent NdFeB magnet powder containing 60% Fe, 30% Nd, and 0.15% Dy was totally dissolved/leached in 30% *v/v* H₂SO₄ solution at 50 °C within 30 min.

To obtain stable and consistent results, freshly prepared synthetic leaching solution of spent NdFeB magnets was used for adsorption tests. Selective resins containing various functional groups (ligands) were explored for recovering and purifying neodymium and dysprosium via ion exchange processes.

Resin functionalized with aminophosphonic groups (Purolite S950 PLUS) demonstrated selective adsorption toward Nd (III) and Dy (III) over Fe (II) under adopted conditions. Nd (III) and Dy (III) achieved consistently high adsorption efficiencies (above 96%) within short contact time (10 min). Conversely, Fe (II) exhibited a very low adsorption rate, reaching only 5.11% at 10 min contact time, with negligible improvement over extended durations. Efficient iron removal could be realized.

Resin impregnated with Di-(2-ethylhexyl) phosphoric acid (D2EHPA) (Purolite MTX7010) was used to realize the separation of Nd and Dy due to its stronger adsorption preference for Dy ions over Nd ions. At a resin dosage of 0.1 g/mL, a pH of 0.65, a contact time of 5 min, an agitation speed of 150 rpm, and a temperature of 22 °C, there was over 88% adsorption efficiency for Dy(III) whereas below 6% for Nd(III).

Nd and Dy could be efficiently eluted from the loaded resins using both HCl and H₂SO₄ solutions. Efficient elution ensures resin regeneration and recycling in the process, which is critical for commercial production.

By employing a multistage adsorption–elution process analogous to distillation, a prospective purity of 98.51% for Dy and above 99.90% purity for Nd could be realized from the synthetic leaching solution of spent NdFeB magnets via direct resin adsorption processes without conventional metal precipitation.

The research results demonstrate that recovery and purification of single REEs from leaching solutions containing mixed REEs and other metals can be achieved using selective resin adsorption processes analogous to distillation despite large concentration differences in the metals in the solutions, which presents a novel and promising approach for metal recovery and purification.

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