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Liquid-liquid extraction of Nd(III) using [P66614][Cy272] ionic liquid

Prasanjit DAS[1], Kali SANJAY[2], and Niharbala DEVI[1,*]

[1] *Department of Chemistry, Institute of Technical Education and Research, Siksha 'O' Anusandhan Deemed to be University, Bhubaneswar-751030, Odisha, India*
[2] *Department of Hydro and Electrometallurgy, CSIR-Institute of Minerals and Materials Technology (IMMT), Bhubaneswar-751013, Odisha, India*

***Corresponding author e-mail**: niharbaladevi@soa.ac.in

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Abstract

This study explores the extraction of Nd(III) from synthetic solutions using the ionic liquid [P66614][Cy272]. Key extraction parameters, including the effect of phase contact time, initial pH, salting-out agents, extractant concentration, and temperature, were systematically optimized. Results revealed that a phase contact time of 15 min, an initial pH of 2.14, and 0.2 mol·L^{-1} NaCl as a salting-out agent and extractant concentration of 0.015 mol·L^{-1} [P66614][Cy272], achieved maximum extraction efficiency of 99.7%. Thermodynamic analysis confirmed the process is to be exothermic, spontaneous, and entropy-driven, highlighting the strong complexation between Nd(III) and the ionic liquid [P66614][Cy272]. Stripping tests revealed that using 0.1 mol·L^{-1} H_2SO_4, achieved complete recovery (100%) of Nd(III) from the loaded-organic phase. These findings underscore the potential of [P66614][Cy272] ionic liquid as a green, efficient alternative to conventional extractants, providing high efficiency and reduced environmental impact. This research advances hydrometallurgical recycling of REEs, particularly from end-of-life NdFeB magnets, supporting sustainable resource recovery and addressing global supply challenges for critical materials.

1. Introduction

Rare earth elements (REEs), which include scandium, yttrium, and lanthanides, have gained more attention due to concerns about future supply shortages. REEs are vital components in numerous advanced technologies, playing a crucial role in clean energy production and supporting the transition to green economy. They are extensively utilized in applications such as permanent magnets, ceramics, catalysts, and batteries. Furthermore, REEs are indispensable to key industries, including electronics, automotive manufacturing, and renewable energy, underscoring their strategic importance in modern technological advancements [1,2]. The European Commission has classified that, REEs as the most critical raw materials due to their high supply risk, a concern echoed by the U.S. Department of Energy, which has identified neodymium, europium, terbium, dysprosium, and yttrium are the most critical REEs [3]. Since the 1990s, China has dominated the global supply of REEs, providing over 90% of the global demand. However, in recent years, China increasing domestic demand has led to restrictions on REE exports [4]. The permanent magnet industries are the largest consumers of REEs, particularly the NdFeB magnet, which is heavily reliant on Dy, Pr, and Nd. Due to rising demand, particularly in the production of electric vehicles (EVs), hybrid electric vehicles (HEVs), and wind turbines, projections suggest significant supply challenges for Nd, Dy, and Pr in the coming decade.

NdFeB magnet was first developed in the 1980s, are widely used for their outstanding power-to-size ratio, making them the ideal choice for various applications. They are extensively utilized in small electronic devices like hard disk drives, smartphones, and music players, as well as in large-scale industrial systems such as EVs, HEVs, refrigerators, wind turbines, and MRI machines [5-7]. This versatility underscores their importance in both consumer and industrial technologies. All NdFeB magnet contains neodymium (Nd), with some applications incorporating, terbium (Tb) to enhance performance or dysprosium (Dy) to increase operating temperature. Praseodymium (Pr) is often added to replace Nd for cost-efficiency. The elemental composition of NdFeB magnet consists of 60% to 70% iron, 20% to 30% neodymium, 0.5% to 7% praseodymium, 0.2% to 6% dysprosium, 0.3% to 1% boron, 0.1% to 0.9% aluminum, and 0.4% to 3% cobalt [8,9]. The size, weight, and life cycles of these magnets vary depending on their applications. For example, small magnets used in electronic devices, weighing between 1 g to 30 g, typically reach end-of-life (EOL) after 2 year to 5 year [10,11]. In contrast, magnets in EVs and HEVs, which weight is more than 1 kg have lifespan of around 16 year. Wind turbines utilize large NdFeB magnets, weight ranging from 1,000 kg to 2,000 kg, have operational lifespans of 20 year to 30 year. These varying life cycles of magnets, pose both opportunities and challenges for recycling and resource recovery at the end of their use. Considering the above, EOL NdFeB magnets is a feasible source for recycling of REEs. Several methods have been investigated to recover REEs, but the hydrometallurgical route (i.e. leaching and solvent extraction) is better than other metallurgical processes due to its good extraction efficiency, low operating cost, ability to concentrate the metals, and

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reusability of the chemicals and reduced hazardous gas emissions [12-14]. Hence, the current research has been attracted to the hydrometallurgical process for recycling waste/spent NdFeB magnets.

Solvent extraction (SX) has been extensively investigated as a promising method for the efficient extraction and separation of REEs such as lanthanum (La), neodymium (Nd), and praseodymium (Pr). Various cation exchange extractants, including D2EHPA, PC 88A, Cyanex 301, and Cyanex 272 have been utilized for this purpose [15]. Among these, Cyanex 272 has shown superior extraction efficiency. However, the drawback of using acidic extractants is the release of H^+ ions, which can reduce the overall extraction performance and organic solvents pose environmental and safety concerns due to their toxicity and flammability [16]. To mitigate these risks, room-temperature ionic liquids (RTILs) have gained attention as the green solvents. These ionic liquids exhibit desirable properties such as low vapor pressure, thermal stability, and reduced flammability, making them safer and more environmentally friendly alternatives [17,18].

In this study, the ionic liquid [P66614][Cy272] was employed for the extraction of Nd^{3+} from a synthetic chloride solution. The work systematically investigates the influence of key solvent extraction parameters, including impact of phase contact time, initial pH, salting-out agent concentration, extractant concentration, and temperature, on the extraction efficiency. The objective is to optimize these parameters to achieve maximum recovery of neodymium. After the extraction process, the stripping of the loaded organic phase has also been investigated. The extraction mechanism between the ionic liquid and Nd^{3+} has been discussed using slope analysis methods.

2. Experimental

2.1 Materials and reagents

The ionic liquid [P66614][Cy272] was synthesized using Cyanex 272 (bis(2,4,4-trimethylpentyl)phosphinic acid) and Cyphos IL 101 (trihexyl(tetradecyl)phosphonium chloride) as the extractants. The synthesis method of [P66614][Cy272] ionic liquid was based on our earlier work [19]. For this study both the extractants, Cyanex 272 and Cyphos IL 101 were provided as gift samples by Cytec Solvay, Canada. Neodymium (III) oxide with purity (99.9%) was purchased from Loba Chemicals Pvt. Ltd., Mumbai. All other reagents used in this study are in the analytical grade.

2.2 Solvent Extraction Process

A synthetic solution of Nd was prepared by dissolving its respective oxide in distilled water, maintaining a working concentration of 0.023 $mol{\cdot}L^{-1}$ Nd. Solvent extraction experiments were carried out in a 60 mL glass separating funnel, with the aqueous phase pH adjusted using dilute HCl or NaOH. Each experiment involved mixing of 10 mL of the aqueous solution with 10 mL of the extractant. This mixture was shaken for 15 min at room temperature using a mechanical shaker, except in cases where temperature variation studies were conducted. After mixing, the phases were allowed to separate by leaving the solution undisturbed for a few minutes. Following phase separation, the aqueous phase (raffinate) was collected in a sample tube for further analysis. The concentration of metal ions in the raffinate was measured using ICP-OES (inductively coupled plasma optical emission spectrometry) with an iCAP PRO Thermo Scientific instrument. The metal ion concentrations in the organic phase were determined indirectly by mass balance calculations based on the initial and final concentrations in the aqueous phase depicted in Equation (1).

$$\left[\mathrm{Nd}^{n+}\right]_{org} = \left[\mathrm{Nd}^{n+}\right]_{i} - \left[\mathrm{Nd}^{n+}\right]_{f} \qquad (1)$$

where Nd^{n+}_{org} , Nd^{n+}_{i} and Nd^{n+}_{f} indicates that, the concentration of Nd in the organic phase, as well as the initial and final concentrations of Nd in the aqueous phase, respectively. The distribution coefficient (D), percentage extraction efficiency (% E), and percentage stripping (% S) have been calculated using the following formulas to interpret the data.

$$D = \frac{[\mathrm{Nd}^{n+}]_{org}}{\mathrm{Nd}^{n+}_{f}} \times \frac{V_{aq}}{V_{org}} \qquad (2)$$

$$\%\,E = \frac{D}{D + \frac{V_{aq}}{V_{org}}} \times 100 \qquad (3)$$

$$\%\,S = \frac{[\mathrm{Nd}^{n+}]_{(aq)st.}}{[\mathrm{Nd}^{n+}]_{(loaded)org.}} \times \frac{V_{aq}}{V_{org}} \times 100 \qquad (4)$$

Where, V_{aq} and V_{org} represents volumes of the aqueous and organic phases, respectively. $\left[\mathrm{Nd}^{n+}\right]_{(aq)st.}$ and $\left[\mathrm{Nd}^{n+}\right]_{(loaded)org.}$ refers to $[Nd^{3+}]$ in the stripping aqueous phase and the loaded organic phase, respectively.

3. Result and discussion

3.1 Impact of shaking duration

The extraction of Nd^{3+} at concentration of 0.0023 $mol{\cdot}L^{-1}$ was studied using 0.005 $mol{\cdot}L^{-1}$ [P66614][Cy272] ionic liquid, with 0.02 $mol{\cdot}L^{-1}$ NaCl as an additive, emphasizing the influence of phase contact duration. The shaking time was varied between 1 min to 30 min to determine its influence on extraction efficiency. Results showed that the extraction efficiency increased with increasing shaking times, reaching a maximum of 54.3% at 15 min (Figure 1). Beyond 15 min, no further improvement in extraction efficiency was observed, indicating that equilibrium had been achieved. Therefore, shaking duration of 15 min was identified as optimal for achieving maximum extraction efficiency.

3.2 Impact of initial pH

The initial pH of the aqueous phase is a key parameter that significantly impacts the efficiency of solvent extraction processes. To examine its impact on the extraction of 0.0023 $mol{\cdot}L^{-1}$ Nd^{3+}, the pH was adjusted between 1.03 to 5.08 using 0.005 $mol{\cdot}L^{-1}$ [P66614][Cy272] as the extracting agent. As shown in Figure 2, at low pH of 1.03, the extraction efficiency of Nd^{3+} was 26.1%. As the pH increased, the extraction efficiency also improved, reaching a maximum of 54.3% at pH 2.14. Beyond this point, further increases in the pH had minimal impact on the extraction efficiency, indicating that the extraction of Nd^{3+} stabilizes after pH 2.14, with little to no enhancement observed at higher pH levels. This suggests that pH 2.14 is optimal for achieving maximum extraction efficiency under the studied conditions.

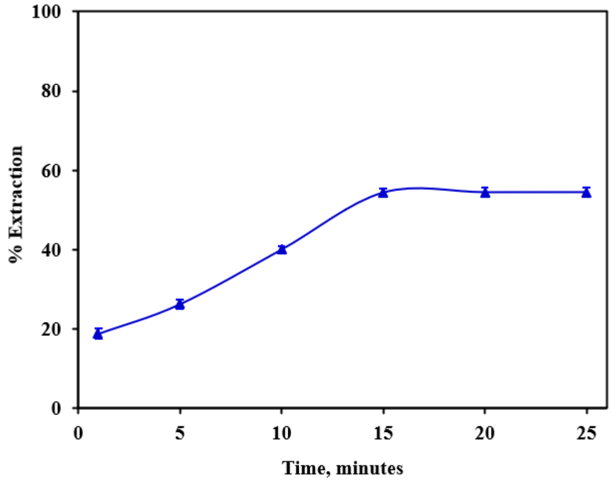

Figure 1. Plot of shaking time versus percentage extraction for the extraction of 0.0023 mol·L^{-1} Nd^{3+} using 0.005 mol·L^{-1} [P66614][Cy272] in the presence of 0.02 mol·L^{-1} NaCl, with an O/A ratio of 1:1 at pH 2.14.

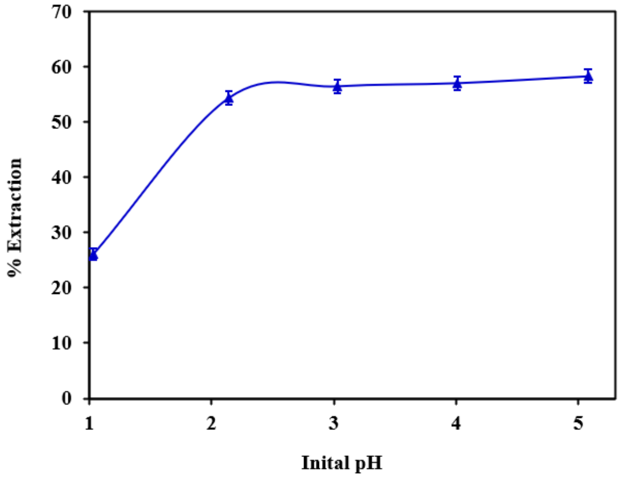

Figure 2. Plot of initial pH versus percentage extraction for the solvent extraction of 0.0023 mol·L^{-1} Nd^{3+} using 0.005 mol·L^{-1} [P66614][Cy272] in the presence of 0.02 mol·L^{-1} NaCl, with an O/A ratio of 1:1.

3.3 Effect of salting out agent

The addition of salting-out agent, such as NaCl, to an aqueous solution, can significantly enhance the efficiency of extraction processes. To study the impact of chloride ions on the extraction process, the chloride ion concentration in the aqueous phase was varied by adding NaCl. The experiments were conducted using an aqueous solution containing 0.0023 mol·L^{-1} Nd^{3+} and 0.005 mol·L^{-1} [P66614][Cy272] as the extractant. The experiments were conducted at a unity organic-to-aqueous (O/A) phase ratio and pH of 2.14. The results, illustrated in Figure 3, demonstrate that increasing the NaCl concentration from 0.02 mol·L^{-1} to 0.2 mol·L^{-1} notably improves the extraction efficiency. At the highest NaCl concentration of 0.2 mol·L^{-1}, a maximum extraction efficiency of 72.4% was achieved. This enhancement in extraction efficiency can be attributed to the elevated ionic strength resulting from the increased chloride ion concentration in the aqueous phase. The rise in ionic strength reduces the number of H_2O molecules surrounding the REE in their hydration spheres, thereby increasing their hydrophobicity. As a result, the REE exhibits a greater tendency to transfer into the organic phase, leading to improved extraction performance.

3.4 Impact of extractant concentration

The influence of [P66614][Cy272] concentration (0.0025 mol·L^{-1} to 0.02 mol·L^{-1}) on Nd^{3+} extraction was examined at a fixed aqueous Nd^{3+} concentration of 0.0023 mol·L^{-1}. The experiments were conducted with 0.2 mol·L^{-1} NaCl at pH 2.14. Figure 4 illustrates that the extraction efficiency improved with increasing [P66614][Cy272] concentration. At a lower concentration of 0.0025 mol·L^{-1} [P66614][Cy272], the extraction efficiency was minimal, indicating insufficient extractant to effectively bind with the Nd^{3+} ions. However, as the concentration of the extractant increased, a significant improvement in extraction efficiency was observed. At 0.01 mol·L^{-1} [P66614][Cy272], the extraction efficiency reached 93.3%, demonstrating a marked enhancement in the extraction

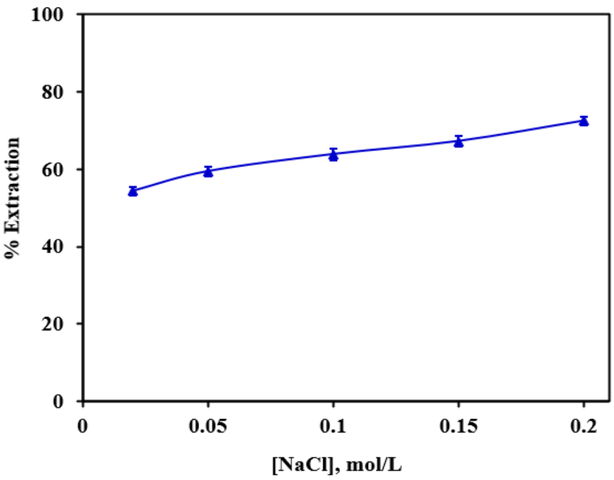

Figure 3. Influence of concentration of NaCl on the extraction of 0.0023 mol·L^{-1} Nd^{3+} using 0.005 mol·L^{-1} [P66614][Cy272] at pH 2.14, O/A = 1:1.

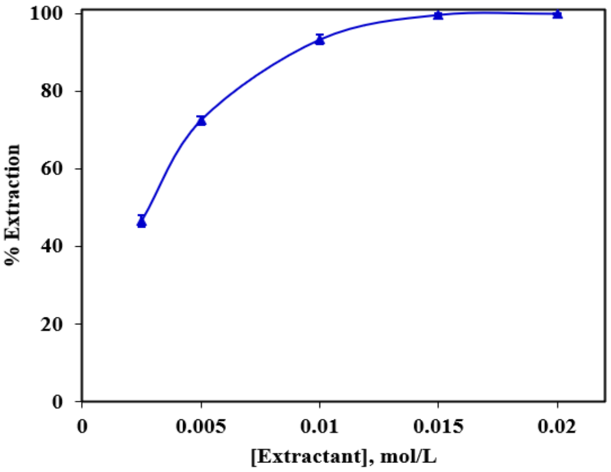

Figure 4. Plot of [Extractant] Vs. percentage of extraction for the extraction of 0.0023 mol·L^{-1} Nd^{3+} in the presence of 0.2 mol·L^{-1} NaCl using [P66614][Cy272], O/A =1:1 at pH 2.34.

process. Further increasing the extractant concentration to 0.015 mol·L^{-1} resulted in a maximum extraction efficiency of 99.7%. This indicates that a higher concentration of [P66614][Cy272] enhances the availability of binding sites, leading to a nearly complete extraction of Nd^{3+} ions. The slope of log D vs. log [Extractant] is depicted in Figure 5, which illustrates the linear slope value close to three, indicating the participation of three moles of the extractant per mole of metal ion in the extraction reaction. The ionic liquid [P66614][Cy272] is an organic salt (R_4PA_{org}) made up of the trihexyl(tetradecyl)phosphonium (R_4P^+) cation and the bis(2,4,4-trimethylpentyl)phosphinate (A^-) anion. The mechanism for the extraction of Nd^{3+} with [P66614][Cy272] ionic liquid may be represented as:

$$\mathrm{Nd^{3+}_{aq}} + 3\mathrm{Cl^{-}_{aq}} + 3\mathrm{R_4PA_{org}} \rightleftharpoons 3\mathrm{R_4Cl_{org}} + \mathrm{NdA_{3org}} \qquad (5)$$

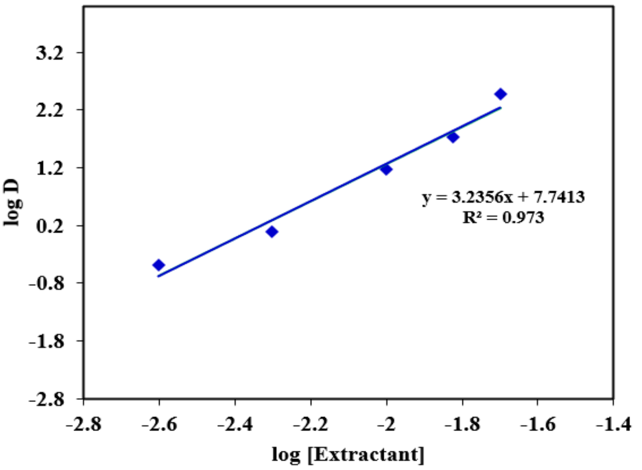

Figure 5. Plot of Log D vs. log [Extractant] for 0.0023 mol·L^{-1} Nd^{3+} extraction with 0.2 mol·L^{-1} NaCl using [P66614][Cy272] at O/A = 1:1 and pH 2.34.

3.5 Effect of temperature

The thermodynamic parameters such as ΔG°, ΔH°, and ΔS° for Nd^{3+} were evaluated from the extraction of 0.0023 mol·L^{-1} Nd^{3+} at temperatures ranging from 298 K to 323 K while holding the other factors constant. With the rise in temperature from 298 K to 323 K, the extraction percentage decreased from 93.3% to 73.5% of Nd. The decrease in extraction percentages for Nd as the temperature rises from 298 K to 323 K suggests that the extraction process is influenced by temperature. This behavior is consistent with thermodynamic principles, where higher temperatures often lead to lower extraction efficiencies due to changes in the equilibrium constants and reaction rates involved in the extraction process. The standard enthalpy change (ΔH°) and entropy change (ΔS°) are derived from the slope and intercept of the plot of 1000/T versus log D (Figure 6) using Equation (6).

$$\log \mathrm{D} = \frac{-\Delta H^\circ}{2.303RT} + \frac{\Delta S^\circ}{2.303R} + 3\log \mathrm{Cl^-} + 3\mathrm{R_4PA_{org}} \qquad (6)$$

The value of standard Gibb's energy is calculated using the equation, ΔG° = ΔH° – TΔS° and the results are shown in Table 1.

From Table 1, the negative signs of ΔH° values showed that the extraction processes are exothermic nature. Positive ΔS° values indicate increased randomness during complex formation, driven by the dehydration of the metal ion's inner core, signifying entropy-driven processes. Negative ΔG° values imply that the extraction is spontaneous and energetically favourable.

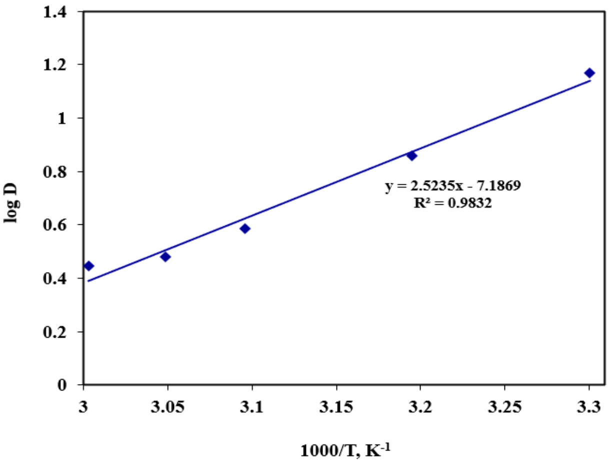

Figure 6. Plot of log D versus 1000/T for the extraction of 0.0023 mol·L^{-1} Nd^{3+} in the presence of 0.2 mol·L^{-1} NaCl using 0.01 mol·L^{-1} [P66614][Cy272] at pH 2.14 and an O/A ratio of 1:1.

Table 1. Thermodynamic parameter values derived from temperature studies for 0.0023 mol·L^{-1} Nd^{3+} extraction in the presence of 0.2 mol·L^{-1} NaCl using 0.01 mol·L^{-1} [P66614][Cy272] at pH 2.14.

Metal ion	ΔH° [kJ·mol^{-1}]	ΔS° [J·k^{-1}mol^{-1}]	ΔG° (303K) [kJ·mol^{-1}]
Nd^{3+}	–49	7.26	–51.2

3.6 Stripping studies

To recover metal ions from the loaded organic phase, different concentrations of HCl and H_2SO_4 were tested as stripping agents. Table 2 show that, the effect of their concentrations on the stripping efficiency. At 0.05 mol·L^{-1} H_2SO_4 achieved 87.9% stripping compared to 79.6% with HCl. Increasing the concentration to 0.1 mol·L^{-1} resulted in 98.3% stripping with HCl and complete recovery (100%) with H_2SO_4. At 0.15 mol·L^{-1}, both reagents achieved 100% stripping efficiency. These results highlight that 0.1 mol·L^{-1} H_2SO_4 is the most effective stripping agent for the complete recovery of the metal from the organic phase.

Table 2. Effect of HCl and H_2SO_4 concentration on stripping efficiency.

Concentration	% Stripping	
	Stripping reagents	
	HCl	H_2SO_4
0.05 mol·L^{-1}	79.6	87.9
0.1 mol·L^{-1}	98.3	100
0.15 mol·L^{-1}	100	100

4. Conclusions

This research systematically explored the extraction of neodymium from synthetic solutions utilizing the ionic liquid [P66614][Cy272] as the extractant. Key parameters such as impact of phase contact time, initial pH, addition of NaCl, concentration of extractant, and temperature were systematically optimized to maximize extraction efficiency. Optimization of key parameters revealed that a phase contact time of 15 min, an initial pH of 2.14, 0.2 $mol\cdot L^{-1}$ NaCl as a salting-out agent, and an extractant concentration of 0.015 $mol\cdot L^{-1}$ [P66614][Cy272] achieved maximum extraction efficiency of 99.7%. Thermodynamic analysis indicated that the process was exothermic, spontaneous, and entropy-driven, confirming the favourable complexation of Nd^{3+} with the ionic liquid. The study also examined the mechanism of extraction, showing that three moles of the extractant interact with one mole of Nd^{3+} to form a stable complex. Stripping experiments using HCl and H_2SO_4 demonstrated that 0.1 $mol\cdot L^{-1}$ H_2SO_4 is the most effective stripping agent, achieving 100% recovery of neodymium ion from the loaded organic phase. These results highlight the potential of [P66614][Cy272] as a green and efficient alternative to conventional extractants, offering high extraction efficiency and reduced environmental impact. This research contributes to advancing hydrometallurgical recycling processes for REEs, particularly from EOL NdFeB magnets, promoting sustainable resource recovery and addressing global supply challenges for critical materials.

Conflict of interest

The authors declare no conflicts of interest for this publication.

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