Effect of Rare Earth Dissolution on Rare Earth Elements by Solvent Extraction from Malaysian Rare Earth Oxalate

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Abstract

Light rare earth elements (LREEs), including Lanthanum (La), Cerium (Ce), Praseodymium (Pr), Neodymium (Nd), and Samarium (Sm), are crucial for modern technologies. Notably, Nd and Pr are core components in permanent magnets for high-efficiency electric motors and generators, essential for low-carbon technologies, and their demand is projected to rise significantly. This study successfully characterized a rare earth oxalate (REOx) sample from undisclosed location in Malaysia, and optimized key conditions for its REE extraction. Initial characterization using X-ray fluorescence (XRF) showed the REOx contained 90.02 wt.% total rare earth elements (TREE), with 9.98 wt.% impurities. Inductively Coupled Plasma Mass Spectrometry (ICP-MS) further quantified the TREE in the REOx at 9656.50 mg/L. The study began by optimizing the dissolution process of REOx using hydrochloric acid (HCl). Parameters investigated included the liquid-to-solid ratio (L/S) (2:1 to 10:1, with 1.0 M HCl for 10 minutes) and HCl concentration (0.2 M to 1.8 M). The optimal dissolution was achieved at an L/S of 10:1, yielding 92.02% TREE extraction, and an optimal HCl concentration of 0.2 M, resulting in 86.98% TREE separation. These optimized conditions facilitated efficient REE extraction while minimizing acid usage. Following dissolution optimization, a 10-stage scrubbing process was conducted using 1.0 M HCl at each stage. The initial impurity concentration (Fe, K, Mg, Mn, Na) after dissolution was 131.98 mg/L, of which 94.50% were successfully removed after the 10th stage. During scrubbing, LREE recovery into the organic phase progressively increased across the stages, achieving over 97% overall recovery. Specifically, La and Ce recoveries reached 100% (from initial 99.84% and 99.83% respectively) by the 5th stage, while Pr and Nd achieved near-complete recovery at the 6th stage (99.97% and 99.99% respectively). These findings confirm the high effectiveness of multi-stage scrubbing for purifying LREEs from REOx.

Keywords: *Di-2-ethylhexyl-phosphoric acid; dissolution, light rare earth elements, rare earth oxalate, scrubbing stages, solvent extraction.*

1. Introduction

A group of 17 chemically related elements, known as Rare Earth Elements (REEs), are indispensable to the production of numerous cutting-edge technologies, including electronic gadgets, batteries, catalysts, and high-performance magnets. Specifically, lanthanum (La) and cerium (Ce) are widely utilized as catalysts in various chemical reactions and for wastewater treatment. In recent decades, the separation and purification of REEs have gained significant importance in response to increasing global demand. Given their typically low natural concentrations in ores, efficient separation and purification techniques are essential components in the overall REE extraction process. Di-(2-ethylhexyl) phosphoric acid (D2EHPA) was selected as an ideal extractant due to its chemical stability, low solubility in the aqueous phase, cost-effectiveness, accessibility, and widespread industrial hydrometallurgical applications, which involve the formation of coordination complexes.

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One of the initial steps in REE processing involves the dissolution of REE-containing minerals using hydrochloric acid (HCl) to convert solid phases into soluble forms suitable for further processing. For instance, a study by Tamayo and colleagues (2024) successfully leached approximately 70% of La and Ce from ferrocarbonatite ore with 1M HCl [1]. Additionally, Kim et al. (2016) introduced a two-stage HCl process for Mongolian apatite ore, utilizing 1 M HCl for Ca removal in the first stage and 2 M HCl for subsequent REE extraction, ultimately achieving over 90% total REE recovery. The efficiency of this dissolution process is influenced by various parameters, including acid molarity, temperature, contact time, and agitation speed. Despite established dissolution methods, the subsequent challenge lies in effectively separating and purifying REEs from complex leachates, which often contain co-extracted impurities. The solvent extraction (SX) method stands out as a highly effective technique for this purpose, particularly for achieving the high purity levels demanded by advanced applications. A critical stage within the SX process is scrubbing, which aims to selectively remove impurities from the loaded organic phase, thereby enhancing the purity of the target REEs. The strategic design of this scrubbing section, including the number of stages, is paramount for optimizing overall process efficiency and product purity.

While the importance of multi-stage scrubbing in REE separation is acknowledged, detailed investigations into optimizing the high-stage scrubbing cascades for specific REE oxalate systems remain crucial. In this study, D2EHPA, diluted in sulfonated kerosene, was employed as the extractant of choice. We aimed to optimize dissolution parameters for REE oxalate (REOx), specifically focusing on the HCl concentration and solid-to-liquid ratio. Subsequently, following the optimization of these dissolution parameters, a comprehensive 10-stage scrubbing process was meticulously designed and conducted, with 1.0 M HCl introduced at each stage, to purify the extracted REEs and evaluate the efficacy of such an extensive scrubbing cascade.

2. Experimental

2.1 Experimental materials

The rare earth oxalate (REOx) cake utilized in this study was taken from undisclosed location in Malaysia. All experimental procedures were carried out at the Mineral Research Centre in Ipoh, Perak.

2.2 Experimental process and analysis

Approximately 1 kg of rare earth oxalate (REOx) was prepared for the solvent extraction process (SXP). The sample was manually ground using a ceramic mortar and pestle to achieve a finer particle size. All solid sample was sent to the Gold, Rare Earth & Material Technopreneurship Centre (GREAT), Faculty of Bioengineering and Technology, Universiti Malaysia Kelantan (UMK) for REE contents analysis. An Inductively Coupled Plasma Mass Spectrometer (ICP-MS) (NexION 5000, Perkin Elmer, USA) was used to determine the REE contents. This study highlighted the influences of extraction REE from dissolution parameters such as concentration and organic-to-aqueous (O:A) phase ratio. Prior to the solvent extraction and scrubbing processes, the REOx underwent the dissolution process using hydrochloric acid (HCl). The initial weight of the filter paper was recorded. REOx was placed in a beaker and mixed with HCl using a magnetic stirrer (HS0707V2, Favorit, Italy) at speed setting no.1 for 10 minutes. The volume (mL) and mass (g) ratio of liquid (HCl) to solid (REOx) was investigated from 2:1, 4:1, 6:1, 8:1 and 10.1 and the HCl

concentration from 0.2 M, 0.4 M, 0.6 M, 0.8 M, 1.0 M, 1.2 M, 1.4 M, 1.6 M and 1.8 M was investigated. After mixing with different concentrations of HCl, the solution was left to sediment for a few minutes, then filtered using filter paper (diameter: 125mm and pore size: 8µm). The residue was dried, and the final weight was recorded. The solution is known as pregnant leach solution (PLS).

The 10 stages of scrubbing in solvent extraction were investigated to separate light and heavy REEs based on the optimum parameters obtained for the dissolution. All experiments were performed at a room temperature (22˚C ± 3˚C). The prepared PLS was mixed with a specific volume of diluted organic extractant in a separating funnel flask, and the mixture was shaken at a speed of 350 rpm for 15 min to load REEs with the organic phase. Then the liquids were separated into an organic and an aqueous phase by a separatory funnel. After two-phase separation, the concentration of REEs in the aqueous phase was analysed using ICP-MS. The REEs concentration in the organic phase was obtained by mass balance and calculated by the following Equation (1):

$$E\% = \frac{C_{ini} - C_{final}}{C_{ini}} x\ 100\%$$

where E% represents the efficiency of REEs extracted into the organic phase, C_{ini} is the REE concentration in the PLS, C_{final} remained REEs concentration in the aqueous phase (raffinate) after extracting REEs into the organic phase. The reaction between REEs and cation-exchanging extractants can be expressed as Equation (2).

$$REE^{3+} + 3(HL)_2 = REE(HL_2)_3 + 3H^+$$

3. Results and Discussion

3.1 Elemental Composition by X-ray Fluorescence (XRF)

XRF analysis indicated that rare earth oxalate (REOx) is predominantly composed of rare earth oxides (REOs), with a minor proportion of other common oxides in Table 1. The most abundant components are Nd_2O_3 (26.00 mass%), La_2O_3 (22.30 mass%), and Y_2O_3 (20.90 mass%). These three alone accounts for over 69% of the total measured composition. The high concentrations of Nd_2O_3 and La_2O_3 observed in the sample are consistent with typical compositions of rare earth mineral concentrates derived from bastnasite ores [4]. Other notable rare earth oxides include Pr_6O_{11}, Sm_2O_3, Gd_2O_3, Dy_2O_3, CeO_2, Er_2O_3, and Yb_2O_3, each present at concentrations above 1 mass%. Common rock-forming or minor industrial components like CaO, SiO_2, Al_2O_3, Fe_2O_3, and SO_3 are present in much smaller quantities, generally below 1 mass%. Trace Elements is a small amounts of Tb_4O_7, Eu_2O_3, Tm_2O_3, Ho_2O_3, Lu_2O_3, NiO, K_2O, PbO, and Cl. A high TREO value (96.59%) indicates that the rare earth oxalate is highly concentrated in rare earth elements.

Table 1. The composition of rare earth oxalate

Component	**Nd_2O_3**	**La_2O_3**	**Y_2O_3**	**Pr_6O_{11}**	**Sm_2O_3**	**Gd_2O_3**	**Dy_2O_3**	**CeO_2**	**Er_2O_3**	**Yb_2O_3**
Result (mass%)	**26.00**	**22.30**	**20.90**	**6.10**	**5.13**	**4.45**	**3.54**	**3.10**	**1.87**	**1.38**
Component	**CaO**	**SiO_2**	**Al_2O_3**	**Tb_4O_7**	**Eu_2O_3**	**SO_3**	**Tm_2O_3**	**Fe_2O_3**	**Ho_2O_3**	**Lu_2O_3**
Result (mass%)	**0.95**	**0.93**	**0.79**	**0.57**	**0.55**	**0.53**	**0.21**	**0.20**	**0.17**	**0.12**
Component	**NiO**	**K_2O**	**PbO**	**C1**						
Result (mass%)	**0.09**	**0.08**	**0.04**	**0.01**						

3.2 Crystalline Phase Identification by XRD

X-ray diffraction (XRD) was employed to identify the crystalline phases of the rare earth oxalate (REOx) sample. This technique provides unique diffraction patterns for crystalline materials, enabling the identification of their atomic and molecular structures [5]. The obtained XRD patterns strongly corroborated the X-ray fluorescence (XRF) findings of a rare earth-rich sample as shown in Fig. 1, precisely identifying the crystalline forms of the detected Light Rare Earth Elements (LREEs), such as Lanthanum (La) and Neodymium (Nd), and Heavy Rare Earth Elements (HREEs), such as Yttrium (Y). The presence of multiple distinct rare earth oxalate phases (e.g., separate La, Nd, Y phases) suggests a complex co-precipitation behavior, where different REEs may form slightly varied oxalate structures or hydrate states. Furthermore, the XRD analysis revealed the presence of impurity oxalate phases, including ammonium cadmium zirconium oxalate hydrate. This finding underscores common challenges in achieving high-purity rare earth concentrates, often attributed to the similar chemical behavior of certain impurity ions to rare earths during precipitation [6]. The identification of these impurity-containing oxalate phases (specifically involving Cd, Zr, Pb, Na, Co, and Rb) provides a more detailed understanding of the nature of the trace contaminants initially detected by XRF. This detailed insight is crucial for refining the subsequent purification process. Additionally, the identification of a sulfate-containing lanthanum phase (Lanthanum Sulfate Oxalate Hydrate) directly aligns with the SO_3 observed in the XRF analysis, indicating that sulfate was not merely a residual component from the solution but actively participated in the precipitation.

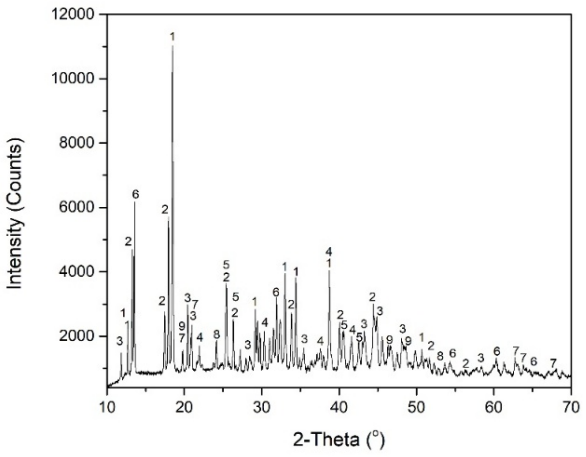

Fig. 1. X-ray diffraction for the rare earth oxalate with indication (1) Copper Lanthanum Oxalate Hydrate, (2) Lanthanum Oxalate Hydrate, (3) Lanthanum Sulfate Oxalate Hydrate, (4) Rubidium Yttrium Oxalate Hydrate, (5) Neodymium Aqua Oxalate Hydroxide Hydrate, (6) Ammonium Cadmium zirconium Oxalate Hydrate, (7) Lead Titanium Oxide Oxalate Hydrate, (8) Sodium Cobalt Oxalate and (9) Linbergite

3.3 Quantification of Rare Earth Elements in Solution by ICP-MS

Table 2 presents the content of the REE in the oxalate. The total rare earth elements (TREE) in the raw REOx sample measured 9656.50 mg/kg. This high concentration confirms the suitability of the sample as a promising source material for REE extraction. Light rare earth elements (LREEs) constitute the largest fraction at 5537.45 mg/kg, representing approximately 57.35% of the TREE. Within the LREE group, Lanthanum (La) and Neodymium (Nd) are the most abundant, with concentrations of 2300.99 mg/kg and 2262.55 mg/kg, respectively. Praseodymium (Pr) also contribute significantly at 597.63 mg/kg. Heavy rare earth elements (HREEs) is the second most abundant group at 3171.77 mg/kg, accounting for approximately 32.85% of the TREE. Yttrium (Y), often grouped with HREEs due to its similar chemical properties, is particularly prominent at 2251.78 mg/kg, indicating its significant presence in the sample. Dysprosium (Dy) and Erbium (Er) are also present in notable amounts at 369.02 mg/kg and 204.45 mg/kg, respectively. Lastly, medium rare earth elements (MREEs) represent the smallest fraction at 947.28 mg/kg, or approximately 9.81% of the TREE. Gadolinium (Gd) and Samarium (Sm) are the primary contributors within this group. The dominance of LREEs (particularly La and Nd) alongside a substantial HREE content (driven largely by Y) is characteristic of many REE deposits, including those found in ion-adsorption clays or certain weathered crusts [18]. The high concentrations of Nd and Pr, specifically, are of significant industrial interest due to their critical role in manufacturing high-performance permanent magnets, which are vital for electric vehicles and renewable energy technologies. The presence of significant Y also adds value, as it is crucial for various high-tech applications [16].

Table 2. The elemental result of rare earth oxalate.

GROUP	*ELEMENT*	*Raw Sample (mg/kg)*
LREE	La	2300.99
	Ce	376.28
	Pr	597.63
	Nd	2262.55
MREE	Gd	440.43
	Sm	435.06
	Eu	71.79
HREE	Y	2251.78
	Sc	0.25
	Tb	63.49
	Dy	369.02
	Ho	70.98
	Er	204.45
	Tm	27.58
	Yb	160.53

	Lu	23.69
LREE		5537.45
MREE		947.28
HREE		3171.77
TREE		9656.50

3.4 Effect of Hydrochloric Acid Concentration on Rare Earth Element Dissolution

Acidity is a pivotal parameter in solvent extraction, playing a determinant role in controlling selectivity [10]. A lower range of HCl concentrations of 0.2 M - 1.8 M has been study in this research to dissolve rare earth oxalate. This is corroborated by Y. Zhou et al., who investigated the initial concentration of HCl for REE leaching at 3 M. The extraction efficiency with 0.5 M HCl swiftly declined to zero after attaining its peak value at 2 minutes. This may arise from the insufficient H^+ in the solution being consumed to form a precipitate. Conversely, an initial HCl concentration of 3 mol/L resulted in the reaction reaching a plateau too quickly, hindering a comprehensive study of the kinetics and reaction mechanisms [9].

In contrast to some general dissolution trends where increasing HCl concentration, up to a certain level improves the dissolution efficiency and rate [11], this study observed a different behavior concerning the extraction efficiency of rare earth elements. Specifically, the concentration of rare earth elements decreased with increasing HCl concentration in the aqueous phase, indicating a diminished transfer into the organic phase. This suggests that lower HCl concentrations are more advantageous for the subsequent extraction of rare earth ions. Consequently, 0.2 M HCl was strategically employed in this study to facilitate better separation during the extraction process. This choice is supported by other research; Carlos et.al. demonstrated that a more diluted acid solution, such as 0.25 M HCl, could enhance metal extraction, specifically improving the extraction level for Gd/Eu using DEHPA [8]. Similarly, Sadia et. al. found that the extraction of Eu^{3+} and Y^{3+} is favorable achieved through solvation in lower acid solution [7]. It is important to acknowledge that the overall effectiveness of these concentrations is highly dependent on various factors, including the specific REE oxalate's crystallinity, particle size, the desired dissolution rate, temperature, and the presence of impurities in the solution.

Fig. 2 illustrate that the highest extraction efficiency in this study was achieved with 0.2 M HCl, its selection as the optimal concentration. These results not only corroborate that lower acid concentrations facilitate better separation but also underscore the practical benefits of reduced chemical usage, thereby enhancing the overall sustainability of the process. Therefore, 0.2 M HCl was precisely chosen for all subsequent experiments.

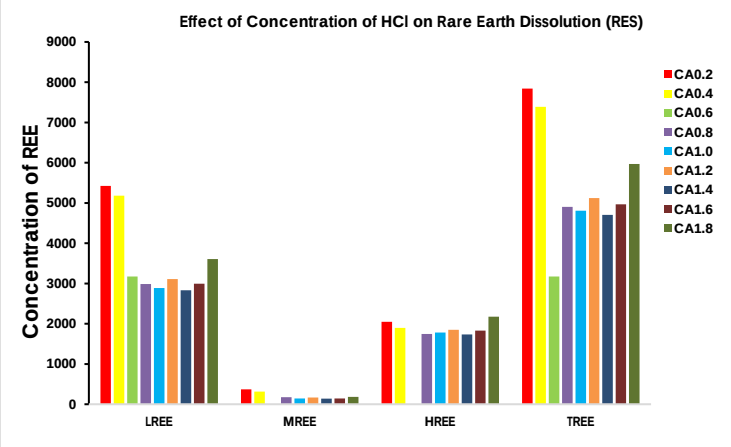

Fig. 2. Bar chart of the LREE, MREE and HREE of the effect on the concentration of HCl to rare earth dissolution.

3.5 Effect of Liquid-to-Solid Ratio on REE Dissolution

The liquid-to-solid (L/S) ratio is an important factor in improving the mass transfer rate and extraction efficiency of TREEs [12, 13]. It is generally understood that a higher L/S ratio generally improves the dissolution efficient by providing more reactant (acid) per unit of solid, reducing the buildup of dissolved REE that can inhibit further dissolution, and improving solution mixing. The following condition were used, 1M HCl and 15 minutes as the mixing timing. A range of liquid-to-solid ratios has been examined including 2:1, 4:1, 6:1, 8:1 and 10:1. As shown in Fig. 3, the increase in the volume of HCl diminished the extraction efficiency of elements, suggesting that the acid becomes the limiting agent in the dissolution reaction. The extraction efficiency of REEs decreased with an increasing liquid-solid ratio. At a low acid volume (2:1 ratio), the extraction efficiency increased significantly, achieving more than 78% leaching efficiency for REEs such as Y (78.9%), Nd (78.64%), Yb (78.29%), Pr (78.01%), and Er (78.0%) when using an HCl concentration of 1 mol/L. However, further increases in the liquid-to-solid ratio (from 2:1 to 10:1) reduced extraction efficiencies. In all cases, it is evident that the extraction efficiency of Y, Nd, Yb, Pr and Er is greater at lower liquid-to-solid ratios.

According to Y. Zhou et al., the dissolution mechanism proceeds via the diffusion of hydrochloric acid through the liquid phase to react with the REE-rich solid. Increased stirring time significantly influenced this process by promoting the growth of interconnected holes within the REE-rich phase particles. This development of internal porosity facilitated the fragmentation of the particles and their subsequent dissolution into smaller pieces [9]. The low extraction efficiency of REEs when the L/S ratio is above 4:1, and the lack of significant increase after 10 min, can be attributed to several factors. This is believed that an increase in the liquid-to-solid ratio increased the viscosity, which indicated higher mass transfer between the liquid. This indicates that the diffusion of acid to the reaction surface may already exceed its saturation concentration. It also tends increasing the impurity in the leachate and potentially leading to the formation of insoluble precipitates that entrap or co-precipitate with rare earths. Not only this, processing dilute rare earth element (REE) leachate solutions (REEs < 1%) from unconventional sources presents significant environmental and economic challenges for liquid-liquid extraction. These

challenges stem from the requirement for elevated aqueous-to-organic phase ratios, which in turn demand larger separation units (implying higher capital expenditure), prolong equilibration times, intensify energy consumption, and increase extractant dispersion losses into the aqueous systems [14].

Fig. 3 displayed that the extraction efficiency improved as the L/S ratio decreased. Consequently, a L/S ratio of 2:1 was considered optimal. Initial higher ratios were indeed suboptimal for overall performance and specific condition with equilibrium limitations which made 2:1 more favorable. This is common for ion-adsorption clays or specific mineralogy where less extracted is sometimes better to avoid co-dissolving impurities or over-saturating the solution [16]. At lower L/S ratios and acid concentrations, the leaching process was found to be diffusion-controlled, characterized by a low activation energy. However, beyond a certain L/S threshold, the activation energy increased significantly, suggesting a transition to a chemically reaction-controlled regime where further increases in L/S did not enhance the reaction rate [15].

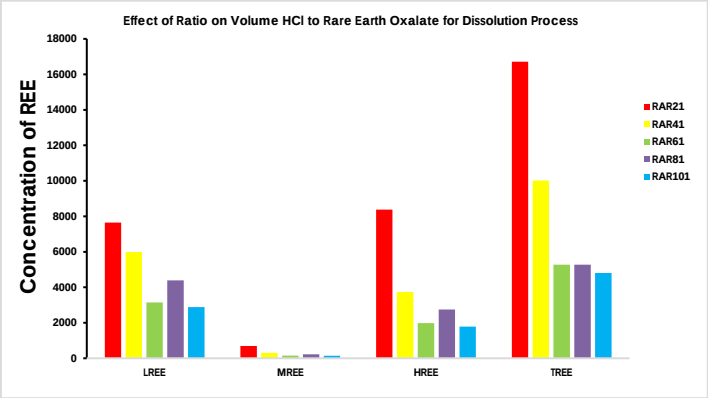

Fig. 3. Bar chart of the LREE, MREE and HREE of the effect on the volume HCL to rare earth oxalate ratio for the dissolution process.

3.6 Effectiveness of 10-Stage Scrubbing on REE Purification

Following the optimization of the dissolution process, the extraction of REEs from the Pregnant Leach Solution (PLS) with D2EHPA was carried out. An organic-to-aqueous (O/A) phase ratio of 1:1 was maintained, and a concentration of 1M HCl was introduced at each of the 10 scrubbing stages. This study specifically designed a solvent extraction (SX) circuit featuring 10 scrubbing stages to achieve high purity of REEs.

The primary purpose of employing numerous scrubbing stages is to enhance purity. More stages provide increased opportunities for contact and equilibrium, which allows for more complete removal of impurities while minimizing the loss of valuable REEs to the scrub raffinate. This approach aligns with studies such as Valente et al. [17], who implemented models and optimization methods to separate a feed mixture containing REEs. Their work demonstrated that high purities (99.52% for Yttrium and 85.41% for Lanthanum) are achievable using multi-stage combinations, including 8-12-3 and 10-3-5 stage configurations for loading, scrubbing, and stripping, respectively. Thus, a 10-stage scrubbing process is

considered a viable and effective strategy in simulated, optimized flowsheets utilizing D2EHPA or similar extractants for high-purity REE separation.

Experimentally, the initial impurity concentration (comprising Fe, K, Mg, Mn, and Na) in the PLS after dissolution was recorded at 131.98 mg/L. After the 10th scrubbing stage, 94.50% of these impurities were successfully removed. Notably, the recovery of Light Rare Earth Elements (LREEs) progressively increased throughout the scrubbing stages, achieving an overall recovery above 97% by the final stage. The findings further confirm the effectiveness of the scrubbing process, as La and Ce recoveries notably increased to 100% from 99.84% and 99.83%, respectively, at the 5th stage. Furthermore, Pr and Nd reached near-complete extraction at the 6th stage, achieving 99.97% and 99.99% recovery, respectively.

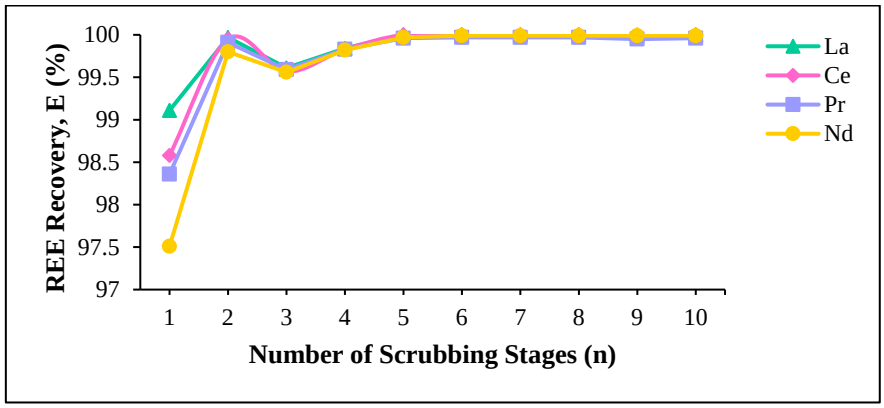

Fig. 4. Effect of scrubbing stages on La, Ce, Pr and Nd recoveries.

4. Conclusion

This study successfully characterized a rare earth oxalate (REOx) sample and rigorously optimized key parameters for its dissolution and subsequent purification. XRF and ICP-MS analyses confirmed the REOx sample's significant total rare earth element (TREE) content of 9656.50 mg/kg, with LREEs (5537.45 mg/kg, primarily La and Nd) being the most abundant, followed by HREEs (3171.77 mg/kg mainly Y) and MREEs (947.28 mg/kg). XRD analysis further elucidated the complex crystalline nature of the REOx, identifying distinct LREE (La, Nd) and HREE (Y) oxalate phases, alongside various impurity oxalate phases (Cd, Zr, Pb, Na, Co, Rb) and a sulfate-containing lanthanum phase, corroborating the XRF findings and highlighting the challenges in achieving high purity. The initial dissolution of REOx using HCl was systematically optimized, revealing that solid to liquid ratio of 10:1 yielded an impressive 92.02% TREE extraction, while an optimal HCl concentration of 0.2 M achieved 86.98% TREE separation. These findings underscore the importance of carefully balancing acid volume and concentration to achieve high dissolution efficiency while also considering economic and environmental factors. Critically, this study then demonstrated the exceptional effectiveness of a 10-stage scrubbing process using 1.0 M HCl for the purification of the extracted REEs. This extensive scrubbing proved highly efficient in removing co-extracted impurities, with 94.50% of initial impurities (Fe, K, Mg, Mn, Na) successfully removed after the 10th stage. Concurrently, the process achieved excellent recovery of valuable LREEs into the organic phase, with overall LREE recovery exceeding 97%. Specifically, La and Ce recoveries reached 100% by the 5th stage, and Pr and Nd attained near-complete recovery (99.97% and 99.99%, respectively) by the 6th stage. These results confirm that multi-stage scrubbing approach, particularly with a well-designed 10-stage cascade, is a highly effective strategy for producing high-purity REE products from complex oxalate feedstocks. The study not only provides optimized dissolution parameters but also validates the critical role of extensive scrubbing in achieving the stringent purity requirements for modern REE applications, such as

those in high-performance magnets and low-carbon technologies. This research contributes valuable data to the development of more efficient and selective hydrometallurgical processes for REE recovery from increasingly complex sources.

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