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Improvement in the performance of solvent extraction operation using solutions spray system

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Abstract: The solvent extraction (SX) method is a widely used hydrometallurgical technique for extracting metals from leach solutions. SX helps in the recovery and selective separation of rare earth elements (REEs), which is not possible in the case of physical separation techniques. However, traditional SX processes face challenges such as limited mass transfer between immiscible liquids and slow mixing and separation kinetics. To address these issues, a new solution spray system was developed to enhance mass transfer during extraction while also reducing mixing and separation times. This system generates uniform micro-droplets in the mixing column, creating a well-mixed emulsion of the two immiscible phases and improving mass transfer efficiency. Additionally, its redesigned settling section enhances phase separation, consistently producing a high-quality final product that is fully separated from the raffinate. The newly-designed solutions spray system and the conventional mixer-settler system were analyzed in extracting La, Nd, and Eu from the chloride solution using organic solvent DEHPA. It was found that the maximum extractions were 95.03%, 99.36%, and 99.98% for La, Nd, and Eu respectively from a feed solution having 2.0 pH with 0.303 M DEHPA, using solutions spray system. On the other hand. The maximum extractions were 98.60% for Eu, 89.25% for Nd, and 73.68% for La in the case of mixer-settler system. The solutions spray system showed better performance because of the production of mono-sized micro droplets of the feed streams. Furthermore, the separation was also efficient for the solutions spray system. McCabe-Thiele diagrams were also plotted to see the theoretical counter-current stages for extraction and stripping stages.

Keywords: hydrometallurgy, solvent extraction, solutions spray system, mixer-settler, REEs

1. Introduction

Rare earth elements (REEs) are crucial for advanced and greener technologies. Because of their exceptional characteristics, these elements are vital for almost every industry including defense, electric car batteries, wind turbines, chemical catalysis, magnets and many more. The demand of these elements will continue to rise with the growing global economic growth rates (Chen et al., 2022; Han et al., 2014; Li, 2019; Pathapati et al., 2023; Shetty et al., 2020; Talan & Huang, 2022). However, a continuous supply of these elements to the industries is a challenge because of their limited sources and lack of efficient extraction methods of REEs either from their ores or secondary sources. Moreover, the biggest challenge is the separation of individual REEs from each other because of their similar physical and chemical properties. The researchers are working on developing the most efficient, cost-effective, and environmentally friendly process to extract these elements to fulfill the industry's needs (Belova, 2017; Erust et al., 2022; Li, 2019; Shetty et al., 2020; Tunsu et al., 2015).

Several processing methods have been developed to extract REEs from commercially important ores of RE minerals. Solvent extraction (SX) is typically considered the best commercial approach for extracting REEs because it can handle massive volumes of diluted pregnant liquor, require relatively less energy, and show better extraction performance along with selective separation of REEs (Cantwell & Losier, 2002; Chen et al., 2022; Li, 2019; Pathapati et al., 2023; Suli et al., 2017; Traore et al., 2023).

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A number of equipment have been designed (Todd, 2014) and are being used in solvent extraction operations for the extraction of REEs, radioactive elements, and other industrial metals (Yelubay & Massakbayeva, 2017). The basic goals of these apparatuses are to improve the mass transfer between the immiscible feed streams, maximize the mass transfer rate, the separation of the product liquid phases, and control the loss of organic solvent (Hashemi-Moghaddam, 2023; Parvizi et al., 2016; Stevens & Pratt, 2000; Weatherley, 2020). The Mixer-settler and the spray columns are the two common techniques used for the solvent extraction operation because of their simplicity and cost-effectiveness (Hashemi-Moghaddam, 2023; Stevens & Pratt, 2000; Yelubay & Massakbayeva, 2017).

The advantages of the mixer-settler include its flexibility, efficiency, and simplicity. Whereas the drawbacks comprise poly-sized droplets production in the mixing section, and the longer time required for complete separation of the product streams. This leads to a bigger sized settling chamber, along with the loss of organic solvent (Duhamet & Commission, 2015; Gharehbagh & Mousavian, 2009; Hadjiev & Paulo, 2005; Kothari et al., 2012; Troshkin, 1996). The spray column is one of the oldest devices used for the solvent extraction operation (Frank Seibert & Fair, 1988; Hixson, 1950; Johnson et al., 1957; Pasteur & Koch, 2020). A key advantage of this system is the ability to produce uniform micro-droplets using a distributor (Stevens & Pratt, 2000). However, a major drawback is the back-mixing of the liquid phases (Almani et al., 2025; Pasteur & Koch, 2020; Sovilj et al., 2019; Todd, 2014). As the organic droplets rise, they carry some of the continuous phase with them, leading to axial mixing, which reduces the efficiency of the extraction process. To address this, the spray columns are modified to improve the coalescence-breakage cycle of the droplets and enhance mass transfer (Stevens & Pratt, 2000).

To address the limitations of traditional equipment, a new apparatus was developed to improve the efficiency of solvent extraction operations (Rahim & Arol, 2024). Initial studies on this system demonstrated promising extraction results and rapid separation of the product phases. The higher recoveries of the REEs with the solutions spray system compared to the mixer-settler technique are because of the generation of mono-sized micro-droplets in the case of the solutions spray system as shown in Fig. 1 (Shimogouchi et al., 2019).

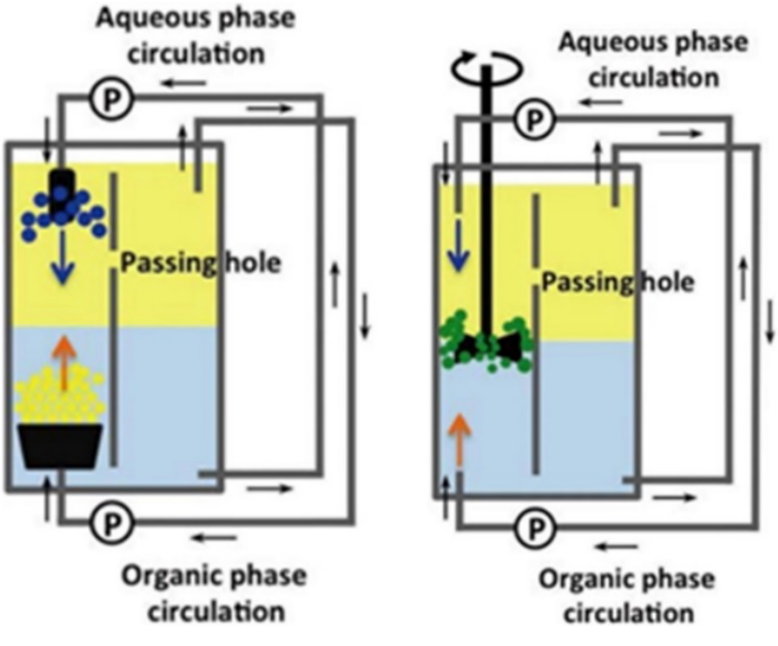

Fig. 1. Spraying system vs mechanical stirring system (Shimogouchi et al., 2019)

Upon spraying a liquid, the monodispersed droplets generated by the sparger undergo densification, randomly agglomerate together, and form emulsion. The aqueous product in the settling section is constantly transparent and free of organic droplets, making it possible to rapidly separate and clean product streams, as illustrated in Fig. 2. In contrast, the mechanical stirrer generates poly-dispersed droplets of both phases, making it difficult to distinguish between the different phases of the mixture (Shimogouchi et al., 2019). These unevenly sized droplets not only complicate mass transfer during the mixing process but also slow down phase separation. This is because of the slow settling of small droplets compared to the larger ones, requiring either a significantly larger cross-sectional area or an extended period of time for complete phase separation to occur.

In this research, the performance of this innovative setup will be evaluated alongside the conventional mixer-settler, with a focus on comparing their effectiveness in extracting REEs.

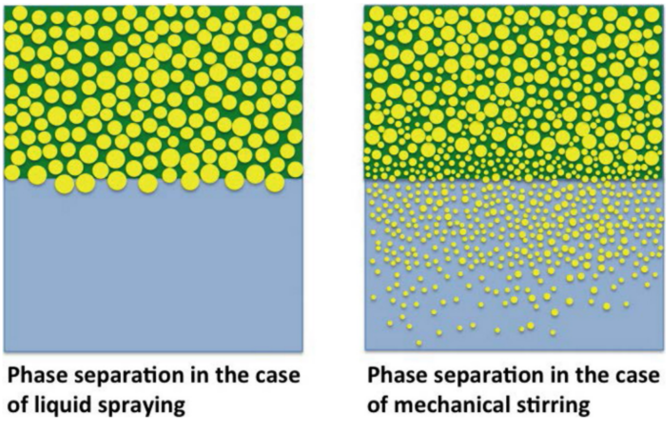

Fig. 2. Differences in the phase separation between droplets generated by spraying and mechanical mixing (Shimogouchi et al., 2019)

2. Materials and methods

2.1. Materials

To prepare the aqueous feed solution, lanthanum oxide (La_2O_3), neodymium oxide (Nd_2O_3), and europium oxide (Eu_2O_3) (all sourced from NATEN, Türkiye) were dissolved in 1.0 M hydrochloric acid (HCl, 37%). Each rare earth oxide (REO) was maintained at a concentration of 1.0 g/L. The organic phase consisted of di-(2-ethylhexyl) phosphoric acid (DEHPA), also from NATEN, Türkiye, used without any additional purification. To reduce its viscosity, DEHPA was mixed with kerosene (supplied by Etibakır A.Ş., Türkiye) at a volume ratio of 10:90 (10% DEHPA). The aqueous feed's pH was maintained between 0.50 and 2.0 using sodium hydroxide (NaOH).

2.2. Equipment and method

In the present study, the extraction of REEs was carried out using two methods: the newly developed "solutions spray system" and the conventional "mixer-settler." The extraction efficiency, measured in terms of percent extraction, was compared between these two techniques to evaluate their performance.

2.2.1. Solutions spray system

In this study, a solutions spray system (Fig. 3) was investigated with the aim of generating uniformly sized micro-droplets of the feed streams. These consistent droplet sizes not only enhance mass transfer during the mixing process but also improve phase separation and the overall quality of the final product (Shimogouchi et al., 2019).

This setup is divided into two distinct sections: the mixing section and the settling/separation section. In the mixing section, the two immiscible feed streams are sprayed counter-currently through spray nozzles, which generate uniformly sized micro-droplets. When these droplets mix, they form an emulsion, facilitating mass transfer. The emulsion then flows into the settling column under gravity, where the separation of the two phases is accelerated by a sudden increase in the diameter of the upper and lower sections. As the emulsion enters the settling column, partial segregation occurs between the products streams in such a way that aqueous droplets move downward, while organic droplets rise upward. During their upward movement, the organic droplets grow larger through collisions and coalescence. When these droplets reach the expanded cross-sectional area, their velocity drops significantly, allowing subsequent droplets to collide and merge, eventually forming a continuous organic phase free of aqueous raffinate. Similarly, the aqueous raffinate, now free of organic phase, is collected from the bottom outlet (Rahim & Arol, 2024).

In these experiments, 200 mL of both feed solutions were sprayed at a flow rate of 150 mL/min. The product solutions—DEHPA loaded with rare earth elements (REEs) and the aqueous raffinate—were collected continuously. The pH of the synthesized feed solution was adjusted using NaOH, varying it from 0.50 to 2.0, to see pH effect on the extraction process.

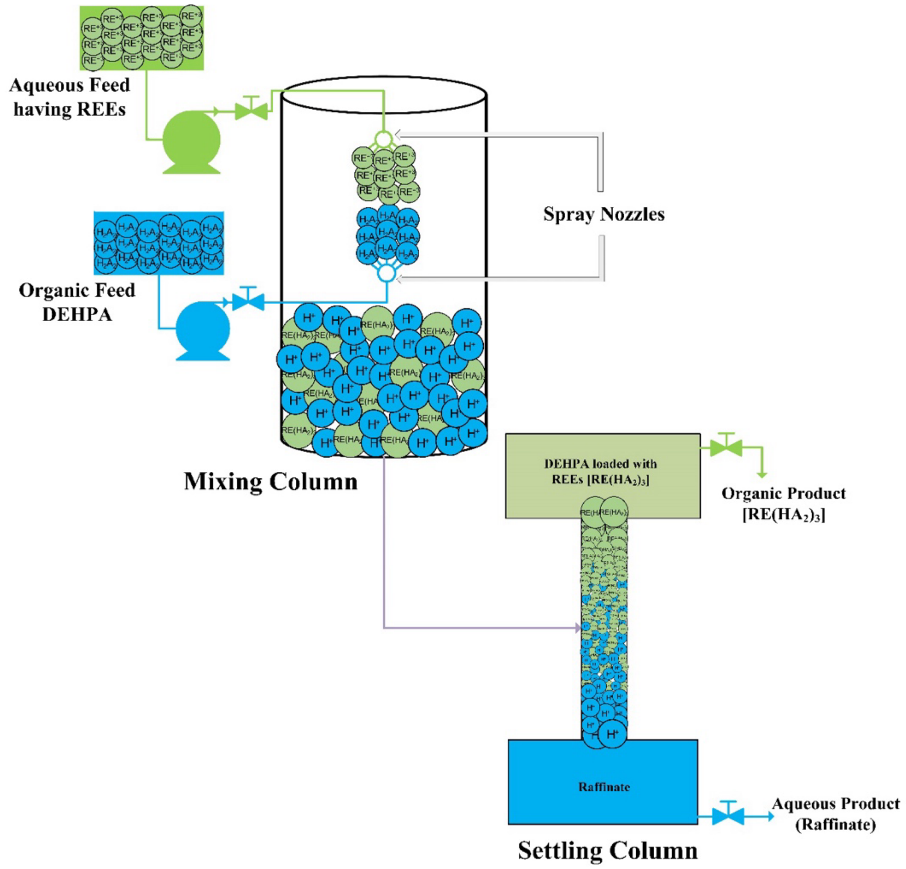

Fig. 3: Solutions spray setup (Rahim & Arol, 2024)

2.2.2. Mixer settler

A mechanical mixer was also used to blend the two immiscible phases, allowing for a comparison of REEs' extraction efficiency with the solutions spray system. In this approach, the aqueous and organic feeds were mixed in a beaker using a mechanical mixer set at 1400 rpm. The mixing process took 10 minutes, while complete phase separation required approximately 4 hours.

Similar to the solutions spray system, this method maintained constant conditions, including 100 mL of each feed stream and 1.0 g/L of each rare earth oxide (REO) in the aqueous feed. However, the pH was adjusted from 0.50 to 2.0 using NaOH.

2.3. Characterization and evaluation of the experimental data

The percentage extraction of REEs was measured to see the performance of these techniques and calculated by the following equation.

$$\%E = \frac{[RE]_{in} - [RE]_{eq}}{[RE]_{in}} \times 100 \tag{1}$$

Here, $[RE]_{in}$ represents the initial concentration of RE ions in the aqueous feed, and $[RE]_{eq}$ is the concentration in raffinate at equilibrium. These concentrations were measured with inductively coupled plasma optical emission spectroscopy (ICP-OES 5900). The pH of the aqueous solutions was determined with a pH meter (pH 211 microprocessor, Hanna Instruments). The FTIR spectrum of the organic phase before and after the REEs extraction was studied using Shimadzu IRTracerTM-100 Fourier Transform Infrared Spectrometer in the wavelength range of 500 – 4000 cm^{-1}. Finally, McCabe-Thiele diagrams were plotted to see the theoretical number of stages for the extraction of REEs.

3. Results and discussion

3.1. pH effect on REEs' extraction for the two mixing techniques

When DEHPA, one of the best-known acidic solvents, interacts with the aqueous solution containing rare earth ions (RE^{3+}), a cation exchange reaction occurs. Specifically, the hydrogen ions (H^+) from DEHPA are swapped with RE^{3+} in the aqueous solution. This exchange process can be represented by Equation (2).

$$RE_{aq}^{3+} + 3H_2A_2 \rightleftharpoons RE(HA_2)_3 + 3H_{aq}^{+} \tag{2}$$

Equation (2) indicates that the reaction tends to shift in the opposite direction as the system's acidity rises. This reverse shift hinders the deprotonation of DEHPA, which in turn reduces the extraction of REEs from the aqueous phase.

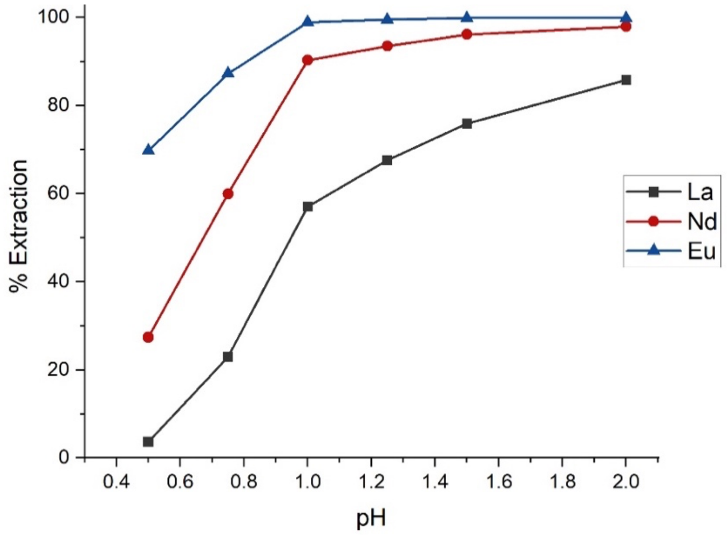

Fig. 4. pH effect on the extraction of REEs using solutions spray system (Rahim & Arol, 2024)

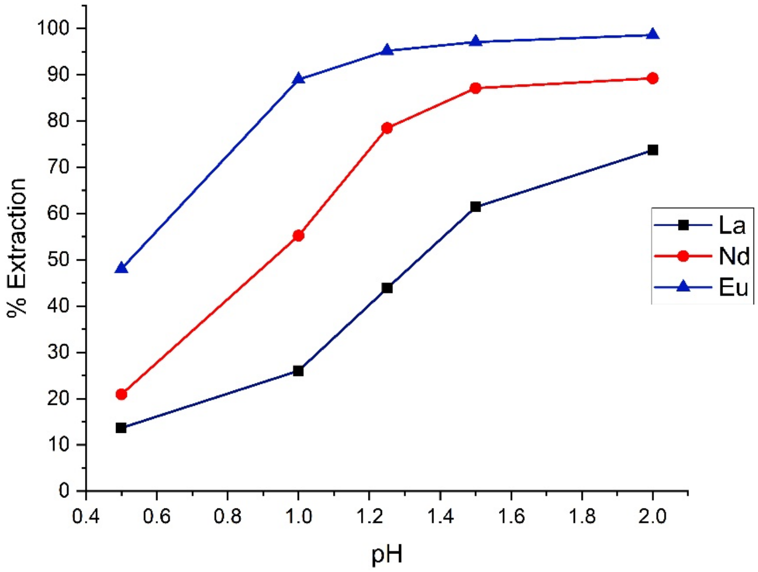

Fig. 5. pH effect on the extraction of REEs using mixer-settler

Fig. 4 and Fig. 5 show the extraction of REEs with the change in pH of the aqueous feed using DEHPA for the solutions spray system and the mixer-settler respectively. As predicted by Equation (2), the extraction efficiency (%E) increased with higher pH levels in both systems. Additionally, the extraction behavior varied depending on the ionic radii of the elements. Eu showed the highest extraction efficiency in both systems, followed by Nd and La. This trend is attributed to Eu's small ionic radius (0.950 Å) value, which gives it a stronger affinity to bond with DEHPA comparing Nd (0.995 Å) and La (1.061 Å) (Bautista, 1995; Han et al., 2014; Kislik, 2012).

In Fig. 4, which depicts the REEs' extraction using the solutions spray system, Eu extraction increased significantly as the pH rose from 0.50 to 1.0, reaching near-complete extraction at pH 1.0. Beyond this point, its extraction remained relatively constant regardless of further pH changes. For Nd, extraction efficiency rose sharply up to pH 1.25, after which the increase became minimal. In contrast, La extraction showed a steady rise with increasing pH, reaching its maximum recovery at pH 2.0. At this pH, the highest %E values achieved were 95.03% for La, 99.36% for Nd, and 99.98% for Eu. Fig. 5 presents the extraction results for the mechanical mixer system. Here, Eu extraction increased rapidly up to pH 1.0, after which the rate of extraction slowed, eventually reaching its maximum at pH 2.0. Similarly, La and Nd extraction increased steadily with pH, peaking at pH 2.0. The maximum extraction efficiencies for this system were 73.68% for La, 89.25% for Nd, and 98.60% for Eu.

To provide a clearer comparison of the recoveries between the two solvent extraction techniques, a bar chart is included as Fig. 6. This chart highlights the differences in extraction efficiency for La, Nd, and Eu across the two systems at varying pH levels.

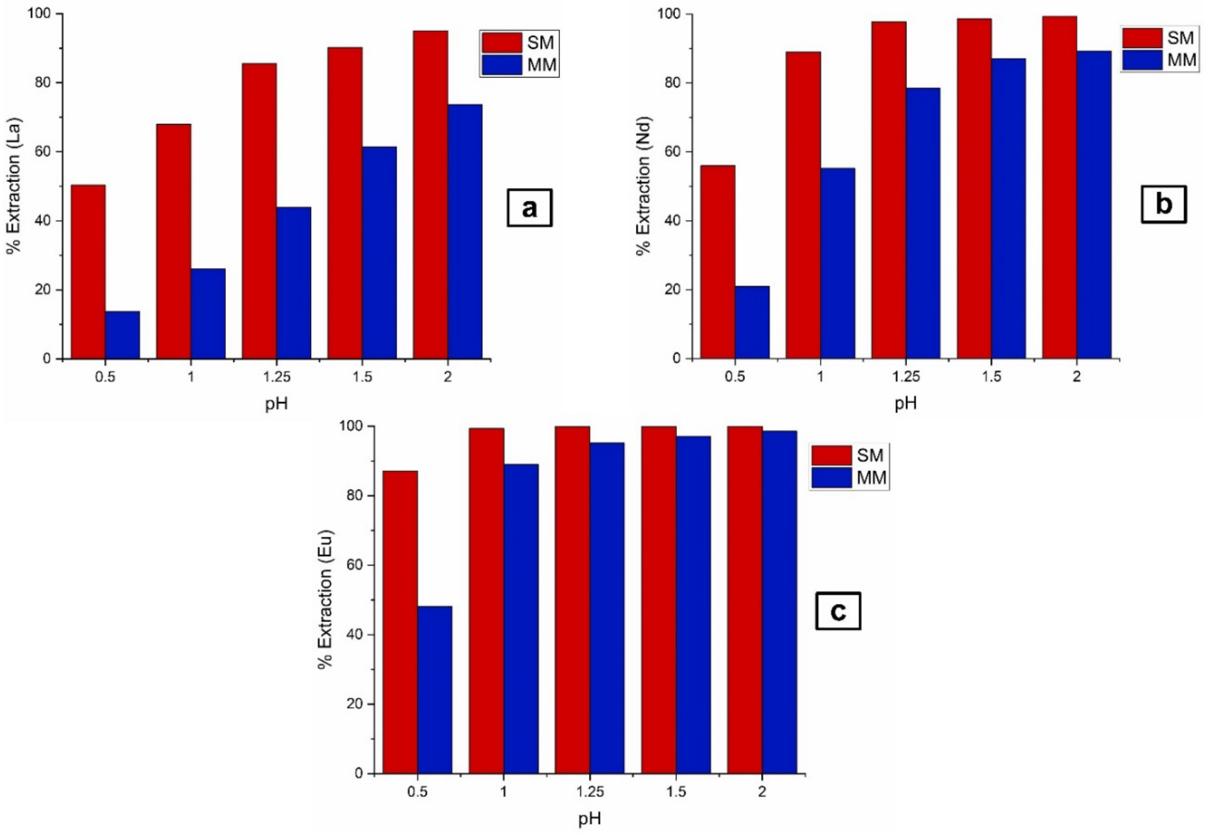

Fig. 6. Comparison of the spray mixing (SM) and mechanical mixing (MM) for %extraction of a) La, b) Nd, c) Eu

Fig. 6(a) illustrates the extraction efficiency of lanthanum (La) using both the solutions spray system and the mechanical mixing method. The data clearly shows that the solutions spray system outperformed mechanical mixing in terms of La recovery. This difference was particularly noticeable at lower pH levels, where the higher concentration of H^+ ions created stronger competition with RE^{3+} ions during the ion exchange process, making the spray system's advantage more pronounced. A similar trend is observed for neodymium (Nd) in Fig. 6(b), where the solutions spray system achieved higher extraction efficiencies compared to the mixer-settler technique. This further highlights the effectiveness of the spray system in extracting rare earth elements. In contrast, Fig. 6(c) demonstrates the extraction performance for europium (Eu). Here, the difference between the two systems is less significant due to Eu's exceptionally high affinity for DEHPA. This strong bonding tendency ensures that Eu is extracted efficiently regardless of the method used, minimizing the performance gap between the two techniques.

3.2. FTIR results

To study the complex formation of all three REEs with DEHPA, FTIR spectra of DEHPA were recorded before and after the loading of REEs for the solutions spray system and mechanical mixing.

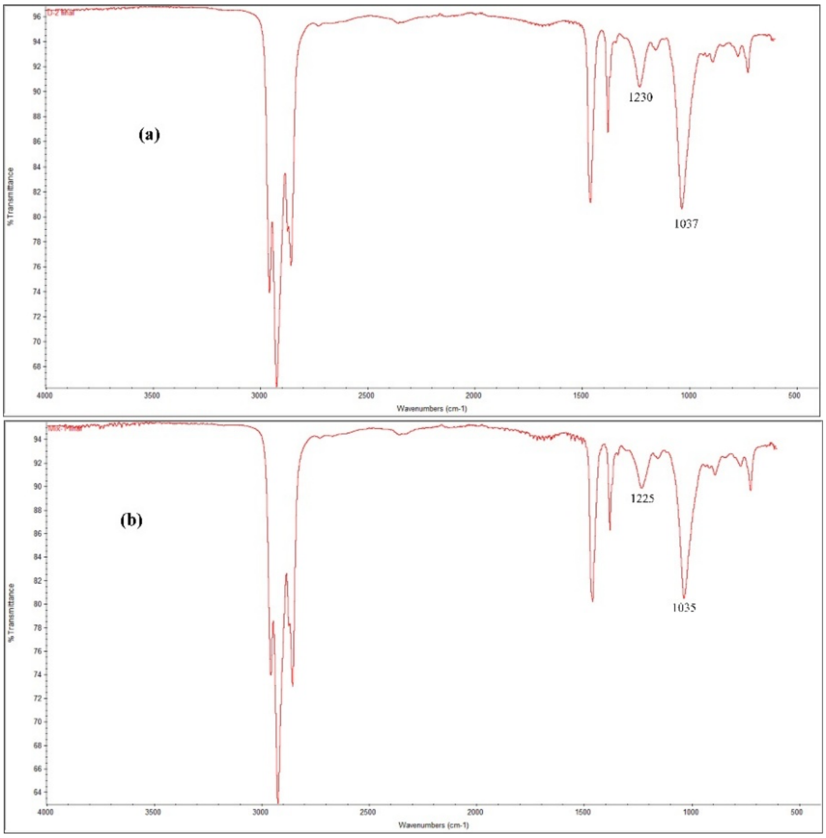

Fig. 7. FTIR spectra of DEHPA using the solutions spray system a) before loading of REEs, b) after loading of REEs

As shown in Fig. 7, the FTIR spectra for the solution spray system reveal that the P–OH stretching vibration, initially observed at 1037 cm^{-1} in pure DEHPA, shifts to 1035 cm^{-1} after the REEs are loaded. This shift indicates a cation exchange mechanism, where hydrogen ions from the P–OH group are replaced by RE metal ions. Additionally, the P=O peak shifts from 1230 cm^{-1} to 1225 cm^{-1}, confirming the formation of a P–O–RE bond alongside the cation exchange process. Similar trends of very small peak shifting have also been reported in the literature (Ariuntuya, 2018; Mishra & Devi, 2018, 2020; Zhang et al., 2014). Similar patterns were observed in the mechanical mixing method, as illustrated in Fig. 8. It is proposed that upon complexation with metal ions, dimeric DEHPA undergoes a ligand exchange mechanism wherein the hydrogen atom of the P-O-H group is substituted by RE ions. In addition, the oxygen atom of P=O group is also involved in coordination with RE ions.

3.3. McCabe-Thiele diagrams for solutions spray system

McCabe-Thiele diagrams were constructed to see the theoretical counter-current stages required for the complete extraction of REEs using the solutions spray system. These diagrams were plotted for aqueous-to-organic (A:O) phase ratio ranging from 1:2 to 5:1. The equilibrium isotherms were used to generate the diagrams, and the operating lines were based on an A:O ratio of 2:1. Fig. 9(a) illustrates the results of La extracion at a 2:1 A:O ratio, and the analysis revealed that a four-stage counter-current operation is necessary to achieve complete recovery of La from the aqueous feed into the DEHPA phase. In contrast, Fig. 9(b) shows that only a two-stage counter-current operation is sufficient to fully extract Nd from the aqueous phase. Finally, Fig. 9(c) demonstrates that Eu can be effectively extracted in a single-stage operation, leaving only 3.0 mg/L. This highlights the high efficiency of the solutions spray system for Eu extraction compared to La and Nd.

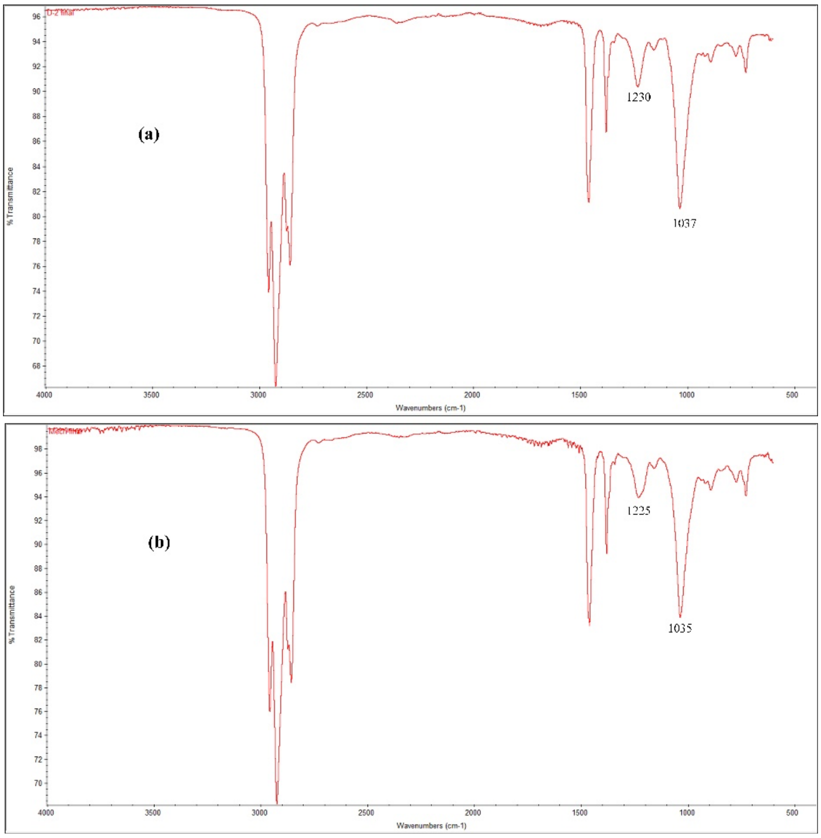

Fig. 8. FTIR spectra of DEHPA using the mechanical mixing system a) before loading of REEs, b) after loading of REEs

Similarly, stripping was also studied in terms of theoretical counter-current stages needed to recover REEs by plotting McCabe Thiele diagrams for the solutions spray system. For this study, HCl solution (2 M) was used as the stripping agent with A:O phase ratio varying from 2:1 to 1:4, and the results are shown in Fig. 10. According to the Fig. 10, all of the REEs under study were completely recovered while using same volumes of loaded DEPA and the stripping solution, whereas selective separation was achieved using A:O phase ratio of 1:2. As shown in Fig. 10(a), La was almost completely stripped in 4 and 8 counter-current stages during A:O phase ratio of 1:1 and 1:2 respectively. Furthermore, complete recovery of Nd was achieved in 6-stage counter-current operation as shown in Fig. 10(b) in the case of A:O phase ratio of 1:1, while recovery was incomplete in the case of A:O phase ratio of 1:2 leaving almost 250 mg/L unrecovered in the organic phase. Similarly, Eu was completely stripped in 6-stage counter-current operation with A:O phase ratio of 1:1, as illustrated in Fig. 10(c), and the saturation of the stripping solution achieved after 3-stages in the case of A:O phase ratio of 1:2, showing the inefficient stripping of Eu and leaving its maximum amount in the organic phase. It is therefore concluded from these results that the complete stripping of La, Nd, and Eu as a group will be attained with A:O phase ratio of 1:1, whereas their selective separation can be achieved while using higher volumes of organic phase compared to the volume of stripping solution (Rahim & Arol, 2024).

4. Conclusions

In this study, a newly developed solutions spray system was evaluated and compared with the traditional mixer-settler technique for extracting REEs from a synthetic solution. The solutions spray system demonstrated superior extraction efficiency for REEs compared to the mechanical mixer. This enhanced performance is attributed to the generation of uniform, mono-sized micro-droplets in the solutions spray system, which not only improved mass transfer but also facilitated faster and more effective phase separation, resulting in higher product quality.

The two solvent extraction techniques were studied to extract lanthanum, neodymium, and europium from an aqueous feed having 1.0 g/L of each REO. The pH of the feed solution was changed from 0.50 - 2.0 with the addition of NaOH. The organic solvent used was DEHPA having 0.303 M concentration. The feed solutions flow rate was 150 ml/min in the case of the solutions spray system, and the product solutions were received continuously from the separation column. Whereas, for the mixer-settler operation, the agitation speed was 1400 rpm, and it took 10 minutes to mix the two

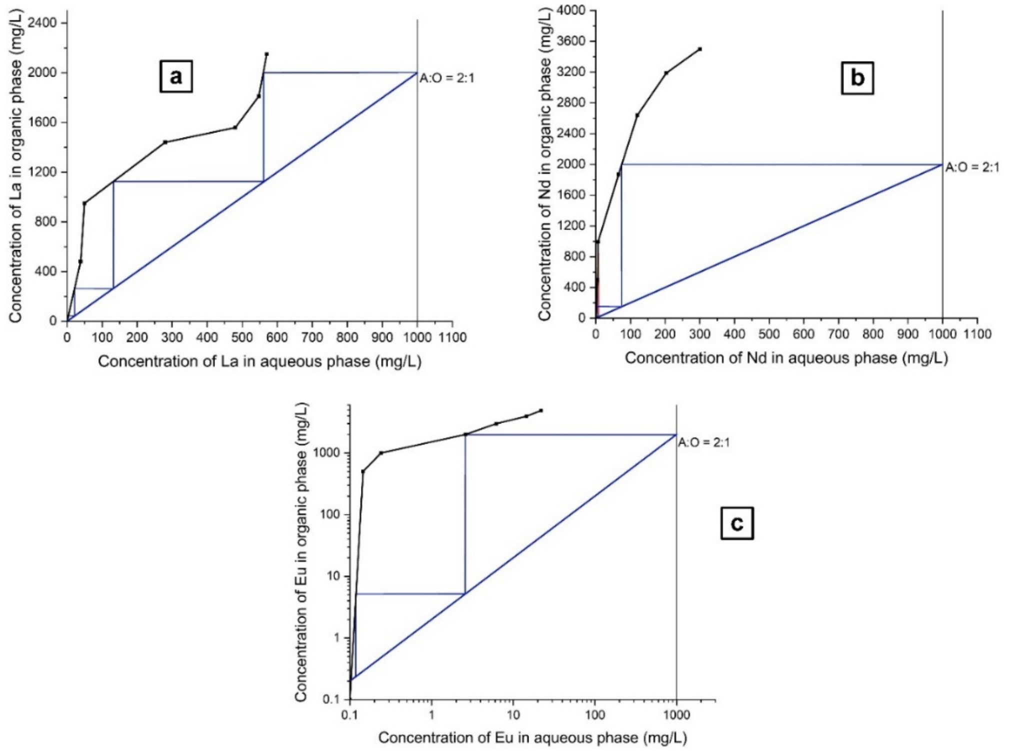

Fig. 9. McCabe Thiele diagram for the extraction operation in the case of the solutions spray system (Rahim & Arol, 2024)

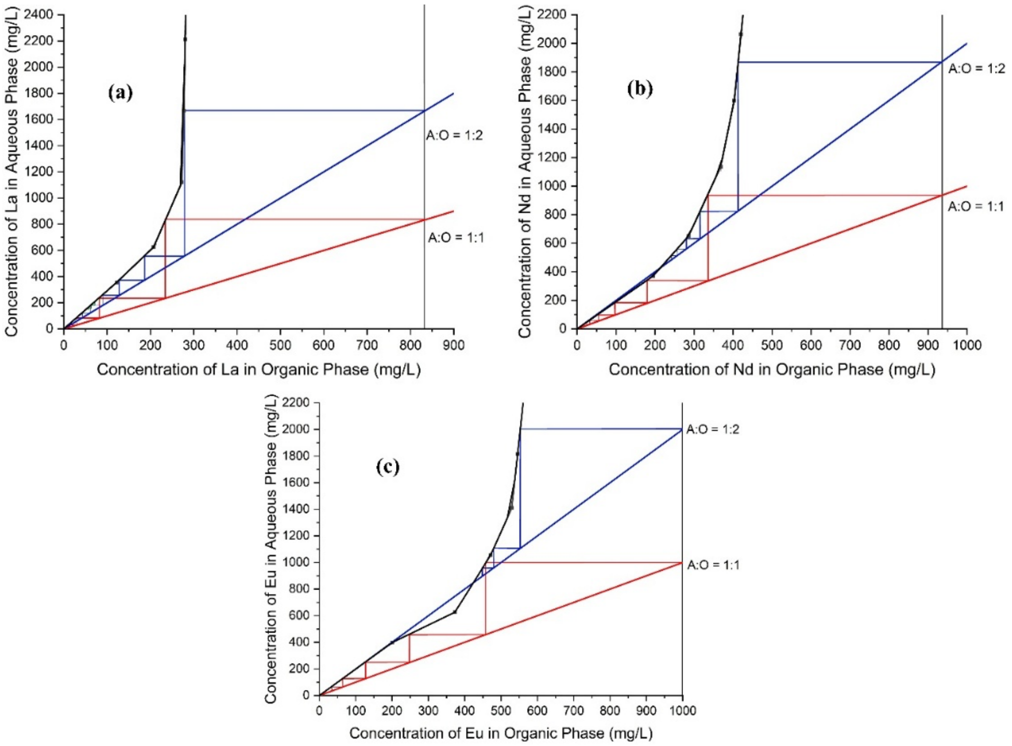

Fig. 10. McCabe Thiele diagram for the stripping operation in the case of the solutions spray system (Rahim & Arol, 2024)

immiscible feed solutions and 4.0 hours for their complete separation. In the case of the solutions spray system, the maximum extraction values were 99.98% for Eu, 99.36% for Nd, and 95.03% for La. Whereas these values were 98.60% for Eu, 89.25% for Nd, and 73.68% for La, in the case of mixer-settler. The better results with the solutions spray system are because of the production of mono-sized micro-droplets in the mixing section and the special design of the settling section, which enhanced the separation of the product streams and improved the quality of the products.

Finally, the number of theoretical counter-current stages were calculated using McCabe Thiele diagrams to see the complete extraction of REEs form the aqueous solution. These diagrams were also plotted for the stripping section to see the complete recovery and selective separation of REEs. All these diagrams provided insights into the extraction efficiency and the stripping process, further validating the advantages of the solutions spray system against the conventional mixer-settler technique.

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