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Sustainable Separation of Rare Earth Elements Using Green Solvent Systems

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Abstract: *The increasing demand for rare earth elements (REEs) in green technologies calls for more sustainable extraction and separation methods. Traditional solvent extraction relies heavily on toxic and flammable organic solvents, posing environmental and safety hazards. This study investigates the use of aqueous two-phase systems (ATPSs) composed of non-ionic surfactants and inorganic salts as green solvent systems for separating REEs (La, Pr, Nd) from transition metals (Fe and Al). The ATPSs based on Triton X-100 (TX-100) with ammonium thiocyanate ($(NH_4)SCN$) and ammonium sulfate ($(NH_4)_2SO_4$) demonstrated high efficiency and selectivity in separating metals under optimized phase ratios and pH conditions. Computational modeling using Density Functional Theory (DFT) provided molecular-level insights into the metal–ligand interactions, confirming the experimental observations. The findings reveal that these green ATPSs offer a promising, low-toxicity, and scalable alternative to conventional solvent systems in REE hydrometallurgy and recycling.*

Keywords*: Rare Earth Elements (REEs), Aqueous Two-Phase Systems (ATPS), Green Solvent Systems, Sustainable Extraction, Liquid–Liquid Extraction, Eco-friendly Solvent Extraction*

I. Introduction:

Rare earth elements (REEs), particularly light rare earths such as lanthanum (La), praseodymium (Pr), and neodymium (Nd), are vital components in various modern technologies. They are indispensable in permanent magnets for electric vehicles and wind turbines, phosphors for lighting and displays, catalysts, and advanced battery materials [1,2]. With the global shift toward renewable energy and electrification, the demand for REEs has grown exponentially, placing stress on traditional mining and extraction processes [3].

However, the separation of REEs from each other and from transition metal impurities such as iron (Fe) and aluminum (Al) remains a significant challenge. This is due to the high chemical similarity of the trivalent lanthanide ions, which differ only slightly in ionic radii and exhibit almost identical coordination chemistries [4]. Moreover, transition metals like Fe and Al can interfere in REE recovery due to their strong complexation tendencies and high affinity for common extractants, leading to co-extraction, solvent poisoning, or third-phase formation in traditional liquid–liquid extraction systems [5].

Conventional solvent extraction methods typically rely on organophosphorus extractants (e.g., D2EHPA, PC88A) dissolved in volatile and often toxic organic solvents such as kerosene or toluene [6]. These systems are not only environmentally harmful but also suffer from high operational costs, flammability risks, and complex multistage processes needed to achieve acceptable selectivity among REEs. Furthermore, they generate considerable chemical waste and pose hazards during solvent regeneration or disposal [7].

In light of these challenges, Aqueous Two-Phase Systems (ATPSs) have emerged as a promising, environmentally benign alternative for REE separation. An ATPS consists of two immiscible water-based phases, typically formed by mixing combinations of water-soluble polymers, surfactants, salts, or ionic liquids above critical concentrations under specific thermodynamic conditions [8]. The advantage of ATPSs lies in their biocompatibility, low toxicity, recyclability, and the elimination of organic solvents from the separation process [9].

Recent advances in the design of ATPSs have shown considerable promise for the selective partitioning of REEs from complex leachates derived from secondary sources such as electronic waste, spent batteries, or end-of-life magnets [10,11]. Systems based on non-ionic surfactants like Triton X-100, in combination with salts like ammonium thiocyanate (NH_4SCN), provide unique micellar environments capable of solubilizing metal–ligand complexes with high selectivity [12].

However, despite these advantages, the underlying mechanisms of metal extraction in ATPSs are not yet fully understood, particularly at the molecular level. The coordination behavior of REEs with various ligands, their solvation dynamics within surfactant micelles, and the role of phase ratio, pH, and ionic strength need to be thoroughly examined. Computational tools such as Density Functional Theory (DFT) have become valuable in elucidating the energetics and structures of metal–ligand interactions in ATPSs, supporting experimental findings with theoretical insights [13].

Therefore, this study focuses on developing and characterizing green ATPSs composed of Triton X-100 and various salts for the selective separation of REEs from Fe and Al. The approach combines experimental extraction trials with DFT modeling to understand the thermodynamics of complex formation and solvation. The goal is to advance ATPSs as a sustainable and scalable strategy for rare earth separation in the hydrometallurgical recycling of critical raw materials.

II. Materials and Methods

2.1 Chemicals and Synthetic Liquor

The aqueous two-phase systems (ATPSs) investigated in this study were prepared using high-purity reagents. The non-ionic surfactant Triton X-100 (p-tert-octylphenyl polyethylene glycol ether), with an average molar mass of 647 $g \cdot mol^{-1}$, was selected as the surfactant phase-forming component due to its proven ability to form micellar aggregates capable of solubilizing hydrophobic complexes [14,15].

The salts used to induce phase separation and act as extractants included ammonium thiocyanate (NH_4SCN), sodium thiocyanate (NaSCN), potassium thiocyanate (KSCN), and ammonium sulfate ($(NH_4)_2SO_4$), all of analytical grade (≥98% purity, Sigma-Aldrich or Synth, Brazil). Thiocyanate salts were chosen for their strong complexation ability with transition metals, particularly Fe^{3+}, and their unique chaotropic properties that disrupt water structure, enhancing hydrophobic complex formation [16, 17].

To simulate realistic leaching conditions from spent permanent magnets (urban mining feedstocks), a synthetic liquor was prepared by dissolving nitrate salts of rare earth elements (REEs)—lanthanum nitrate hexahydrate ($La(NO_3)_3 \cdot 6H_2O$), praseodymium nitrate hexahydrate ($Pr(NO_3)_3 \cdot 6H_2O$), and neodymium nitrate hexahydrate ($Nd(NO_3)_3 \cdot 6H_2O$)—each at a concentration of 1.0 $g \cdot kg^{-1}$. Ferric nitrate nonahydrate ($Fe(NO_3)_3 \cdot 9H_2O$) and aluminum nitrate nonahydrate ($Al(NO_3)_3 \cdot 9H_2O$) were added at 0.6 $g \cdot kg^{-1}$ to represent transition metal contaminants typically found in real leachates [18].

The pH of the solution was carefully controlled at either 1.00 or 2.00 using sulfuric acid (H_2SO_4, 98%) to prevent hydrolysis and precipitation of Fe^{3+} as ferric hydroxide ($Fe(OH)_3$), which can occur at higher pH levels [19]. A digital pH meter (Hanna Instruments, Model HI 2221) was used for precise pH adjustment.

2.2 ATPS Formation and Metal Extraction Procedure

The ATPSs were prepared by mixing predetermined proportions of the synthetic liquor, Triton X-100, and the chosen salt solution in 4 mL polypropylene tubes. Each system had a total mass of 4.0000 ± 0.0005 g, with mass ratios of the surfactant-rich phase to salt-rich phase (wUP/wLP) varied systematically—$1:1, 1:2, 1:3$, and $1:5$—to study the influence of phase volume on separation performance [20].

The mixtures were stirred manually followed by homogenization using a vortex mixer (Phoenix, Model AP56, 3,800 rpm) for 2 minutes. The samples were then centrifuged at 3,000 rpm for 10 minutes to achieve clear phase separation. The upper phase (surfactant-rich) and lower phase (salt-rich) were carefully separated and collected for analysis.

The concentrations of La, Pr, Nd, Fe, and Al in the lower phase were measured using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) (Agilent 725-ES), under the following conditions:

- Wavelengths: La (333.749 nm), Pr (418.948 nm), Nd (406.108 nm), Al (237.312 nm), Fe (259.940 nm)
- Power: 1 kW; Plasma flow: 15.0 $L \cdot min^{-1}$; Nebulizer pressure: 200 kPa

The concentrations in the upper phase were calculated by mass balance. The metal extraction percentage (%E) was calculated using Equation (1):

$$\%E = \left(\frac{n_{UP}}{n}\right) \qquad (1)$$

Where:

- n_{UP}- moles of metal in the upper phase
- n - total moles of metal in the system

In addition to %E, the distribution coefficient (D) and separation factor (S) were calculated:

$$D = \frac{\%E}{\frac{W_{UP}}{W_{LP}}(100-\%E)} \qquad (2)$$

$$S = \frac{D_X}{D_Y} \qquad (3)$$

Where $\frac{W_{UP}}{W_{LP}}$ are the masses of the upper and lower phases, and D_x and D_y are the distribution coefficients for species x and y, respectively. All experiments were conducted in triplicate, and average values were reported.

2.3 Computational Studies

To elucidate the extraction mechanisms at the molecular level, Density Functional Theory (DFT) calculations were carried out using the B97-3c composite method implemented in ORCA quantum chemistry software [21]. This method provides a balance between computational cost and accuracy, particularly for systems involving transition metals and lanthanides [22].

Three main reactions were modeled:

1. Stepwise complexation of metal ions with thiocyanate (SCN^-) at different substitution levels
2. Coordination of SCN^- ligands with a polyether (PEG-like) fragment mimicking TX-100's ethylene oxide chains
3. Solvation and partitioning behavior of metal–ligand complexes in different solvent environments

A hybrid solvation approach combining cluster and continuum models was used. The SMD solvation model simulated different solvent polarities (water, ethanol, diethyl ether, n-hexadecane) to approximate the heterogeneous micellar environment of TX-100 [23]. Transfer free energies ($\Delta G_{transfer}$) and partition coefficients (log P) were derived from these simulations to predict hydrophilic/hydrophobic tendencies of the complexes.

This theoretical data was correlated with experimental extraction values (ΔG_{app}), calculated from the distribution coefficients using:

$$\Delta G_{app} = -RT\ln(D) \tag{4}$$

Where RRR is the universal gas constant and TTT is the temperature (298.2 K). These analyses helped to establish the dominant factors—complexation, solvation, and micellar interaction—influencing selective metal extraction.

III. Results and Discussion

3.1 Preliminary Partitioning Performance

Initial screening of aqueous two-phase systems (ATPSs) composed of Triton X-100 (TX-100) and various salts highlighted the crucial role of the salt anion, cation, and solution pH in determining metal extraction behavior. Specifically, systems formed with NH_4^+ salts showed stark contrasts in extraction performance depending on the accompanying anion.

As shown in Figure 1A, TX-100-based ATPSs with ammonium thiocyanate (NH_4SCN) exhibited almost complete extraction of Fe (~100%) into the surfactant-rich (upper) phase, while the corresponding system with ammonium sulfate ($(NH_4)_2SO_4$) showed negligible metal extraction. This confirms that SCN^- is essential for effective complexation and partitioning of Fe and Al, while SO_4^{2-} fails to enable micelle-assisted separation [24-26].

Further comparison of SCN^- salts—NaSCN, KSCN, and NH_4SCN—revealed similarly high Fe extraction across all systems (≥98%), with Al showing moderate extraction (~40–60%), and rare earth elements (REEs: La, Pr, Nd) consistently below 10%, as illustrated in Figure 1B. This demonstrates that the cation (NH_4^+, Na^+, or K^+) has minimal effect on Fe extraction, but NH_4SCN showed slightly better REE suppression, indicating a minor advantage in selectivity[27].

Additionally, pH significantly affected extraction trends. At pH 1.00, co-extraction of REEs was slightly enhanced compared to pH 2.00, as shown in Figure 1C, likely due to increased proton competition for SCN^- binding sites. The best selectivity between Fe and REEs was achieved at pH 2.00, where REE extraction was minimized to <6%, and Fe remained fully extracted [28].

Together, these results confirm that:

- SCN^- is a critical complexing anion that forms hydrophobic $Fe(SCN)_n^{3-n}$ species,
- The surfactant micelles of TX-100 provide a hydrophobic environment favoring partitioning of these complexes,
- Fe^{3+} and Al^{3+} are selectively extracted, while REEs are largely excluded, especially under mildly acidic conditions.

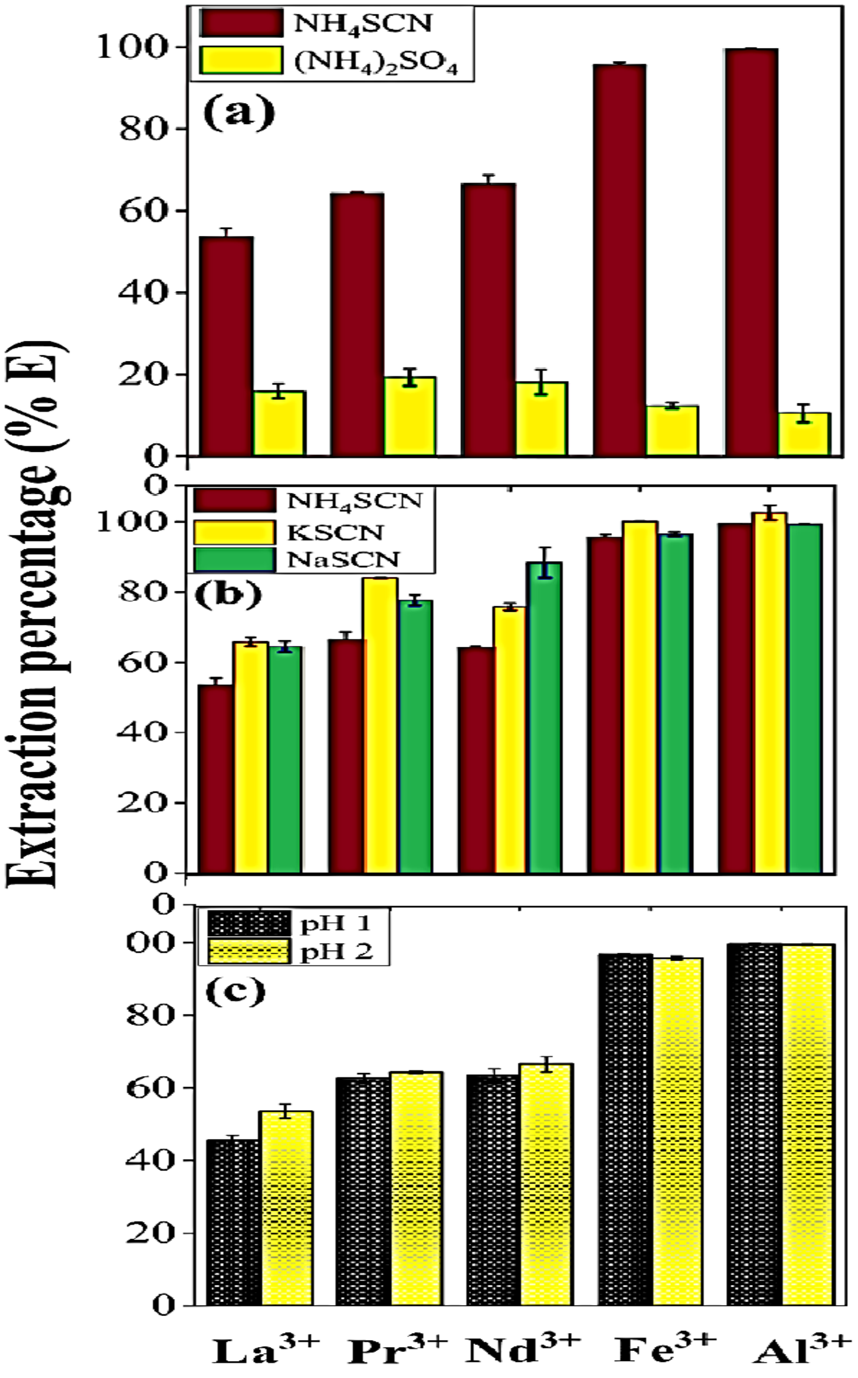

Figure 1: (A) Effect of salt anion (SCN^- vs. SO_4^{2-}); (B) Effect of SCN^- salt cation (NH_4^+, Na^+, K^+); (C) Effect of pH on extraction efficiencies of La, Pr, Nd, Al, and Fe using TX-100-based ATPSs.

These findings underscore the potential of SCN^--driven, micelle-based ATPSs for sustainable and selective separation of transition metals from REEs, providing an environmentally friendly alternative to conventional solvent extraction systems.

3.2 Optimizing REE/Transition Metal Separation

By modulating the phase ratio (wUP/wLP), the selectivity of ATPSs can be fine-tuned. When a 1:5 upper-to-lower phase ratio was used with TX-100 and NH_4SCN at pH 2.00, the system achieved highly selective separation:Fe: 98.6%, Al: 2.54%, La: 4.05%, Pr: 6.19%, Nd: 4.87% [29].

As shown in Figure 2, increasing the lower phase volume effectively suppressed co-extraction of REEs while maintaining complete Fe recovery. This adjustment in phase ratio enabled efficient pre-separation of Fe from

REEs, which is particularly beneficial for treating complex mixed-metal leachates derived from e-waste or recycled permanent magnets [30-33].
The calculated separation factors (S) confirmed the efficiency:S_Fe/La: 64.59, S_Fe/Pr: 44.20, S_Fe/Nd: 56.88. Such high selectivity values affirm the system's potential for sustainable hydrometallurgical applications.

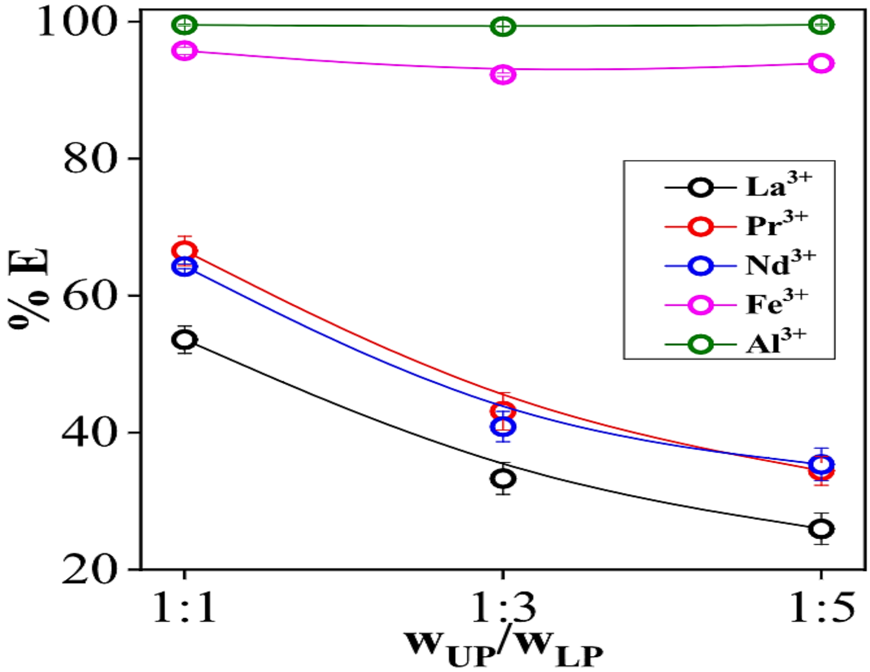

Figure 2: Extraction performance as a function of phase ratio (1:1 to 1:5) for Fe^{3+}, Al^{3+}, and REEs (La^{3+}, Pr^{3+}, Nd^{3+}) in the TX-100 + NH_4SCN system at pH 2.00.

3.3 Improved Selectivity Using Dual Salt Systems

To further enhance selectivity, a ternary aqueous two-phase system (ATPS) was developed by combining TX-100, $(NH_4)_2SO_4$, and water, with NH_4SCN added as an extractant. This dual-salt approach aimed to exploit synergistic effects between the surfactant micelles and salt-driven phase separation.

As shown in Figure 3, even small additions of NH_4SCN (as little as 0.5 mol·kg^{-1}) led to nearly quantitative extraction of Fe^{3+} into the TX-100-rich upper phase. In contrast, co-extraction of REEs and Al remained low (typically under 25%) across most concentrations, indicating selective complexation of Fe with SCN^- anions [34].

The effect of phase ratio (W_{UP}/W_{LP}) on extraction performance is illustrated in Figure 4. While Fe extraction remained consistently high across all phase ratios, increasing the volume of the upper (surfactant-rich) phase gradually enhanced REE and Al extraction, especially beyond a 2:1 ratio, suggesting phase ratio tuning as a tool to control selectivity.
Together, these findings reinforce the promise of dual-salt/surfactant ATPSs for green hydrometallurgy. The system offers efficient separation even at low extractant concentrations, reducing chemical load and providing a scalable pathway for selective pre-recovery of Fe from complex leachates generated from e-waste or permanent magnet recycling [35].

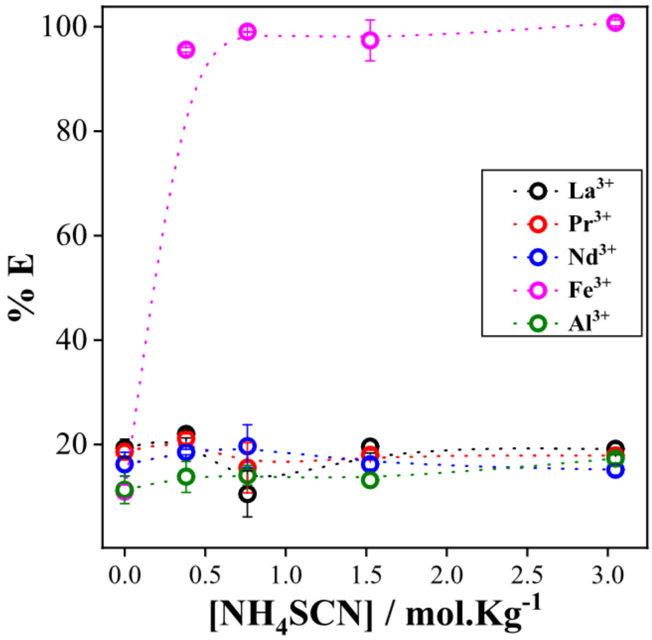

Figure 3: Metal extraction efficiencies (%) as a function of NH_4SCN concentration in the ternary ATPS (TX-100 + $(NH_4)_2SO_4$ + water) at pH 1.00.

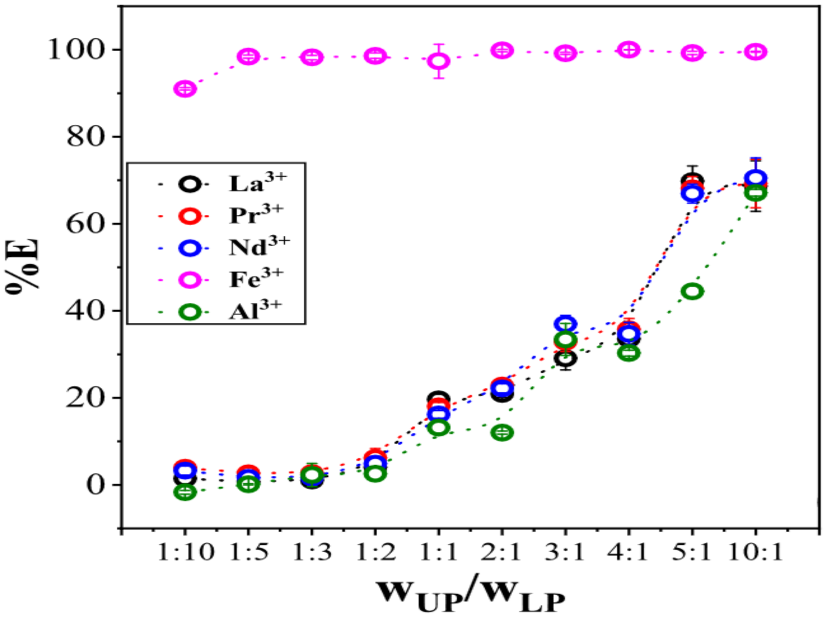

Figure 4: Metal extraction efficiencies (%) as a function of phase ratio (wUP/wLP) under fixed NH_4SCN concentration.

3.4 DFT Insights on Metal Partitioning Mechanisms

Density Functional Theory (DFT) simulations provided molecular-level insights into the selective partitioning behavior observed in the experimental ATPS extractions. These calculations focused on the interaction of various metal ions (Fe^{3+}, Al^{3+}, La^{3+}, Pr^{3+}, and Nd^{3+}) with thiocyanate (SCN^-) ligands in aqueous and organic-like environments.

The results indicated that SCN^- ligands preferentially form stable, tetrahedral $[Fe(SCN)_4]^-$ and $[Al(SCN)_4]^-$ complexes, which are more hydrophobic than the corresponding rare earth element (REE) complexes. This tetrahedral geometry is structurally well-suited to partition into the micellar (surfactant-rich) phase, such as that formed by TX-100[36].

As shown in Figure 5, the log P values (partition coefficients) of Fe–SCN^- complexes were significantly higher than those of Al or REE complexes, particularly as the number of SCN^- ligands increased from 1 to 4. These results were consistent across different solvents (diethyl ether, 1-octanol), mimicking the polarity range of the micellar environment.

In addition, the transfer free energy (ΔG_{transfer}) from water to organic solvents was most negative for Fe complexes, confirming that their partitioning into nonpolar or amphiphilic environments (such as TX-100 micelles) is thermodynamically favorable. This supports the experimental observation that Fe^{3+} is almost completely extracted into the upper phase of the ATPS, while REEs largely remain in the salt-rich lower phase [37].

This computational evidence reinforces the experimental selectivity trend:

Fe >> Al > La ≈ Pr ≈ Nd

— consistent with the increasing coordination strength and hydrophobicity of the metal–SCN^- complexes.

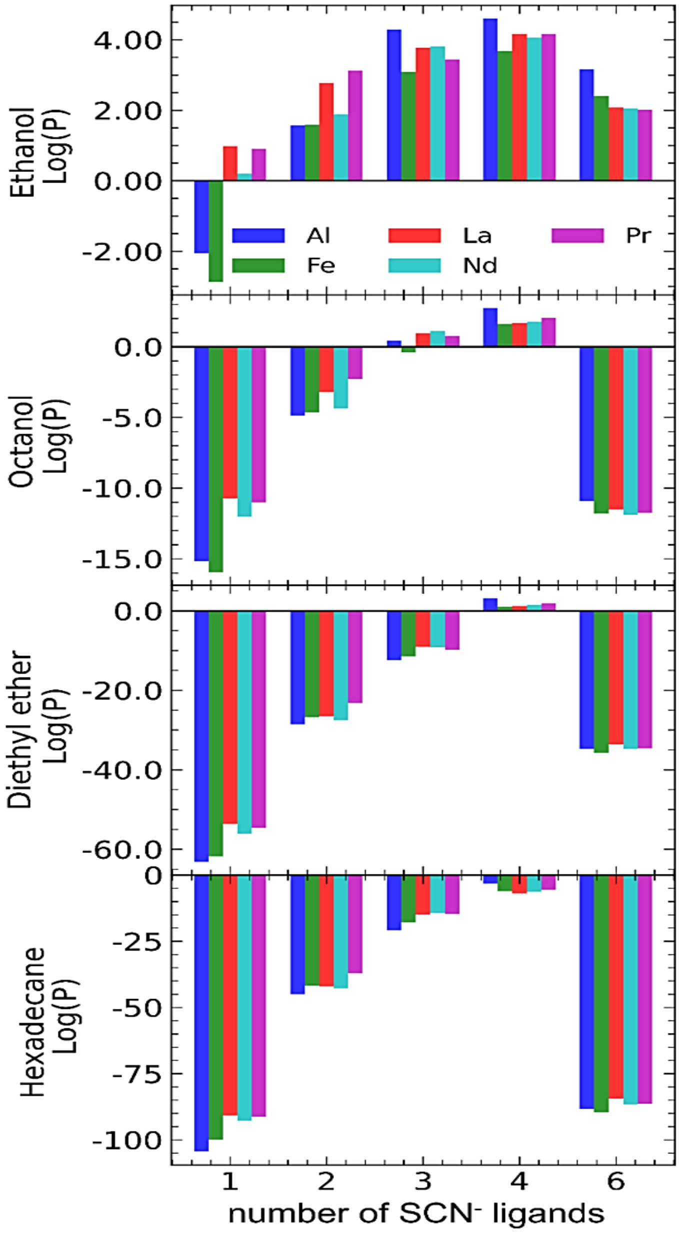

Figure 5: DFT-calculated partition coefficients (log P) for metal–SCN^- complexes (with 1 to 4 SCN^- ligands) in different solvents, showing that Fe complexes are the most hydrophobic and favor partitioning into nonpolar micellar environments.

3.5 Micellar Interaction and Physical Properties

In addition to chemical selectivity, the physical and interfacial properties of the aqueous two-phase systems (ATPSs) play a crucial role in determining their practical scalability and industrial applicability. The surfactant-rich phase formed by Triton X-100 (TX-100) micelles provides a highly amphiphilic environment, primarily due to its polyether (–CH_2CH_2O–) chains. These chains offer favorable interactions with hydrophobic Fe–SCN^- complexes, stabilizing them within the micellar core and facilitating selective extraction [38].

The incorporation of thiocyanate anions (SCN^-) was found to significantly influence the physical behavior of the system. Specifically, SCN^- reduced the viscosity of the TX-100-rich phase and simultaneously increased the interfacial tension between the surfactant-rich and salt-rich phases. These changes contributed to a faster phase disengagement time, which is beneficial for improving the efficiency of separation in batch or semi-continuous setups.

Despite these advantages, the system exhibited certain limitations that may affect its performance at scale. The density difference (Δρ) between the two phases was relatively low—approximately 0.03 g/cm³—which can hinder gravitational separation, especially in large-volume processes. Furthermore, the viscosity of the TX-100-rich upper phase remained relatively high, around 150 mPa·s, which may limit the throughput in continuous flow systems or complicate equipment design for industrial reactors [39].

Taken together, these observations highlight that while the micellar environment of TX-100 is chemically effective, some degree of process engineering—such as centrifugal separation, phase modifiers, or flow intensification strategies—may be required to fully realize the potential of ATPSs in sustainable rare earth element separation at an industrial scale.

IV. CONCLUSION:

This study demonstrates the potential of green aqueous two-phase systems (ATPSs) for the sustainable and selective separation of rare earth elements (REEs) from transition metals such as Fe and Al. Using non-ionic surfactant TX-100 combined with thiocyanate salts (SCN^-) and additional salts like $(NH_4)_2SO_4$, highly selective partitioning of Fe into the surfactant-rich phase was achieved, while REEs remained largely in the salt-rich aqueous phase. This enabled a simple yet effective pre-separation step in REE recovery workflows, which is particularly useful for complex leachates derived from recycled permanent magnets or e-waste.

The system demonstrated near-complete Fe extraction (up to 98.6%) with minimal co-extraction of REEs (below 6%), and separation factors (S_Fe/REE) exceeding 40, indicating excellent selectivity. Furthermore, density functional theory (DFT) calculations provided molecular-level insight into the mechanism of partitioning, confirming the formation of highly hydrophobic Fe–SCN^- complexes that spontaneously migrate into the TX-100 micellar phase.

Although the system exhibited some physical limitations—such as high viscosity and low density difference between phases—these can be mitigated through engineering interventions or alternative surfactant formulations. Overall, the dual-salt/surfactant ATPS strategy presented here offers a non-toxic, non-volatile, and environmentally benign alternative to conventional solvent extraction, paving the way for more sustainable hydrometallurgical processes for REE separation and recovery.

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