

## YTTRIUM RECOVERY: A SELECTIVE LEACHING STRATEGY FROM FUSED XENOTIME

*Roshasnorlyza Hazan\**, Mimi Anisa Romli, Zakiah Zamri, Nur Hanisah Asri, Khaironie Mohamed Takip, Nur Aqilah Sapiee, Norhazirah Azhar, Wilfred Paulus, and Jacqueline Kones

Materials Technology Group (MTeG), Industrial Technology Division, Agensi Nuklear Malaysia, Bangi, 43000 Kajang, Selangor, MALAYSIA.

\*Correspondence author: roshasnorlyza@nm.gov.my

### ABSTRACT

*This research investigates a selective leaching strategy aimed at efficient recovery of yttrium (Y) from fused xenotime in response to the increasing demand for this essential rare earth element (REE) in various high-tech applications. The development of selective leaching strategies is vital for the sustainable and economically viable production of Y. The proposed research adopts a hydrometallurgical approach, utilizing selective leaching to recover Y from fused xenotime while minimizing the dissolution of other matrix components, thereby simplifying separation processes. The leaching process requires careful control of several factors, including the selection of leaching agents (hydrochloric acid (HCl), nitric acid (HNO<sub>3</sub>), sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), and acetic acid), acid concentration, duration, temperature, and solid-to-liquid ratio, to optimize the recovery and selectivity of Y. The fused xenotime and the leachates were analyzed using X-ray fluorescent (XRF) techniques to determine the elements present in the samples. Experiments were conducted at room temperature for one hour with varying molarities of leaching agents; the maximum recovery for Y was found to be 79%, 61%, 58%, and 36% for leaching with 4 M HNO<sub>3</sub>, 2 M HCl, 3 M acetic acid, and 1 M H<sub>2</sub>SO<sub>4</sub>, respectively. However, uranium (U) began to appear in the leachate at low concentrations (0.2 M) of H<sub>2</sub>SO<sub>4</sub> and acetic acid and at 1.5 M of HNO<sub>3</sub> and HCl. Additionally, thorium (Th) began to be detected in the leachate after using 0.5 M H<sub>2</sub>SO<sub>4</sub>, 2 M acetic acid, 2 M HNO<sub>3</sub>, and 6 M HCl. Therefore, it is recommended to use 1.5 M HNO<sub>3</sub> for leaching the fused xenotime, as this concentration can effectively prevent the dissolution of U and Th while still achieving a high recovery of Y (66%). Following the selective leaching stage, solvent extraction techniques can be utilized to further purify the Y-rich leachate and eliminate any residual contaminating metal ions that may have co-dissolved during the leaching process.*

**Keywords:** Alkaline fusion; rare earth element (REE); recovery; selectivity; XRF

### INTRODUCTION

In recognition of its special qualities as a rare earth element (REE), yttrium is in high demand on the international market. Furthermore, the aforementioned study aligned with Malaysia's Sustainable Development Goals (SDG) in several areas, including clean water and sanitation, affordable and clean energy, industry, innovation and infrastructure, and climate action (DOSM, 2019). Through the National Advanced Materials Technology Road Map 2021–2030, Malaysia aims to attract RM100 billion in investment from various investors by 2030, resulting in the creation of 4000 jobs and RM747.2 billion in revenue (MOSTI, 2022). This initiative also contributes to a specific goal in a flagship program for environmental and natural resource management as part of the Dasar Teknologi Nuklear Negara 2030 vision (Nuklear Malaysia, 2021). Furthermore, Wawasan Nuklear Malaysia for Nuclear Science and Technology is implementing strategic core number 1 to enhance industrial

competitiveness (MOSTI, 2023). Y provides excellent functionality in numerous green and high-tech components (Haxel et al., 2002). Some of the applications of yttrium are solid oxide fuel cells (SOFC) (Vasconcellos et al., 2006), superconductors (Basuki et al., 2020), chemically stable substrates, crucible materials for melting reactive metals, cutting tools, infrared windows, thermal barrier coatings, solid-state laser host materials, and catalyst support (Gribov et al., 2023). However, limited processing knowledge (Jordens et al., 2013), sustainability of locally produced yttrium in purity, and cost-effectiveness from the upstream sector up to its commercialization are the primary obstacles to yttrium's commercialization. Since Malaysia has abundant natural sources of amang and is rich in yttrium (27–41%), it could be utilized as the raw material for yttrium production (Amer et al., 2016 and Mohamed and Saleh, 2017) depending on its origin.

Yttrium separation and purification can be achieved through various methods, such as solvent extraction and precipitation. In a rich yttrium pregnant leach solution, 50–60%  $Y_2O_3$  may be obtained in a single leaching process (Xaba et al., 2018). However, Y in nature coexist with the other REE gang, Th and U. Separating individuals became a major challenge for the industry. Therefore, selective leaching could facilitate the separation process by controlling the dissolution of Y and other elements. In this research paper, the efficient recovery of Y while minimizing the dissolution of other matrix components was studied.

## MATERIALS AND METHODS

### Materials

The xenotime ores were collected from Kinta Valley, Perak, Malaysia. Table 1 displays the major elements present in the xenotime ore and fused xenotime. Other chemicals, such as 37% HCl, 68%  $HNO_3$ , 98%  $H_2SO_4$  and 99% acetic acid manufactured by Merck, Fisher Chemical, R&M Chemical, and HmbG Chemical, respectively, were analytical grade.

Table 1. Elemental composition of xenotime.

Element	Elemental composition (wt. %)	
	Ore	Fused
<u>Rare Earth elements (REE)</u>		
Y	21.98 ± 0.18	52.72 ± 0.32
Gd	1.374 ± 0.08	3.06 ± 0.24
Tm	ND	2.07 ± 0.58
Ho	0.66 ± 0.06	2.40 ± 0.01
Yb	5.57 ± 0.04	9.32 ± 0.34
Nd	1.96 ± 0.08	3.00 ± 0.57
Ce	1.79 ± 0.10	2.30 ± 0.55
<u>Radioactive elements</u>		
Th	0.45 ± 0.01	0.92 ± 0.19
U	0.38 ± 0.01	0.91 ± 0.08
Other elements	65.83 ± 0.10	23.29 ± .019

\*ND = not detected

### Selective Acid Leaching

Under predetermined ideal conditions, xenotime samples were fused from an earlier work with NaOH (Hazan et al., 2025). Subsequently, different acid molarities for acid-leaching parameters were analyzed to ascertain their optimal values for selective leaching of heavy rare earth elements (HREE). Each of the experiments was carried out in a closed system at room temperature (25 °C) for an hour. The leached product was thoroughly rinsed and filtered using deionized water. XRF was employed to analyze both the leachate and its solid residue. Only HREE, which solubilizes the leachate in the absence of thorium and uranium, will advance to the subsequent phase.

### Analytical techniques

The elemental analysis was performed by XRF spectroscopy using EDX-7000 by Shimadzu for both liquid and solid samples. All samples were prepared in triplicate to ensure repeatability. The data's uncertainty was documented according to the recommendations of the EDX-7000 program. The recovery of elements (R%) in leachate was calculated based on equation 1.

$$\% \text{ leaching recovery of REE} = \frac{\text{amount of element in leachate (g)}}{\text{initial amount of element in fused xenotime (g)}} \times 100 \quad (1)$$

## RESULTS AND DISCUSSION

### Effect of HCl Concentration on REE Recovery

The REE contents were measured at different HCl molarity acids. Figure 1 illustrates the recovery of rare earth elements (REE) after leaching fused xenotime using various concentrations of HCl. As may be seen in Figure 1(a, b, d), Y recovered 60% upon leaching between 1.5 M and 4 M HCl; Gadolinium (Gd) recovered 70% upon leaching with 1.5 M HCl; and Holmium (Ho) recovered ~58% upon leaching between 2 M and 4 M. Due to Y and Gd's increased solubility in slightly stronger acidic conditions, Y and Gd most likely showed higher leaching effectiveness in 1.5 M HCl than other REEs. However, the solubility of most REE decreased upon increasing the HCl molarity due to the development of stronger complexation and precipitation between REE and chloride into REE-chloride complexes. These conditions could limit the further dissolution of REE in the system. Other REEs may experience a maximum recovery of 28% thulium (Tm) upon leaching with 4 M HCl. Oversaturation and the common-ion effect were the causes of Tm's decreased dissolving ability. The common-ion effect is the result of an equilibrium when any of the reaction elements in the reaction are shared in the reacting system (Ayogu et al., 2020). Meanwhile, the recovery of Neodymium (Nd) keeps increasing upon leaching with 12 M HCl (Figure 1(c)). It was due to the greater solubility of Nd under strong acidic conditions.

### Effect of HNO<sub>3</sub> Concentration on REE Recovery

Figure 2 shows the leaching recovery of fused xenotime using different HNO<sub>3</sub> concentrations. Figure 2 indicates that REE recovery was in equilibrium after a certain HNO<sub>3</sub> molarity. Notably, the highest Gd recovery of 97% was observed upon leaching with 1.75 M HNO<sub>3</sub>. Gd likely had higher leaching in 1.75 M than other REE due to its greater solubility. The maximum Y recovery of 79% was observed when leaching with 4 M HNO<sub>3</sub>. On the other hand, cerium (Ce) starts to recover with 6 M HNO<sub>3</sub> leaching. It means that Ce needs a higher acid concentration to increase proton activity to ensure higher dissolution efficiency. This experiment indicates that Ce and Yb were leached out using HNO<sub>3</sub>. As compared to HCl leaching, both elements were detected. The results indicated that the HNO<sub>3</sub> was able to break down the fused xenotime structure more effectively and overcome the common-ion effect for Ce and Yb.

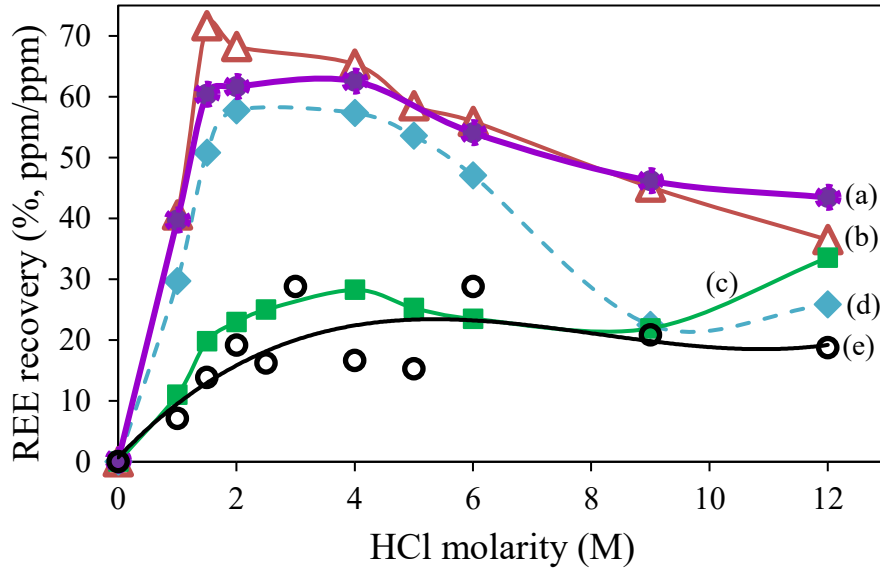


Figure 1 Effect of HCl concentration on REE recovery ((a) Y, (b) Gd, (c) Nd, (d) Ho, and (e) Tm after HCl leaching of fused xenotime

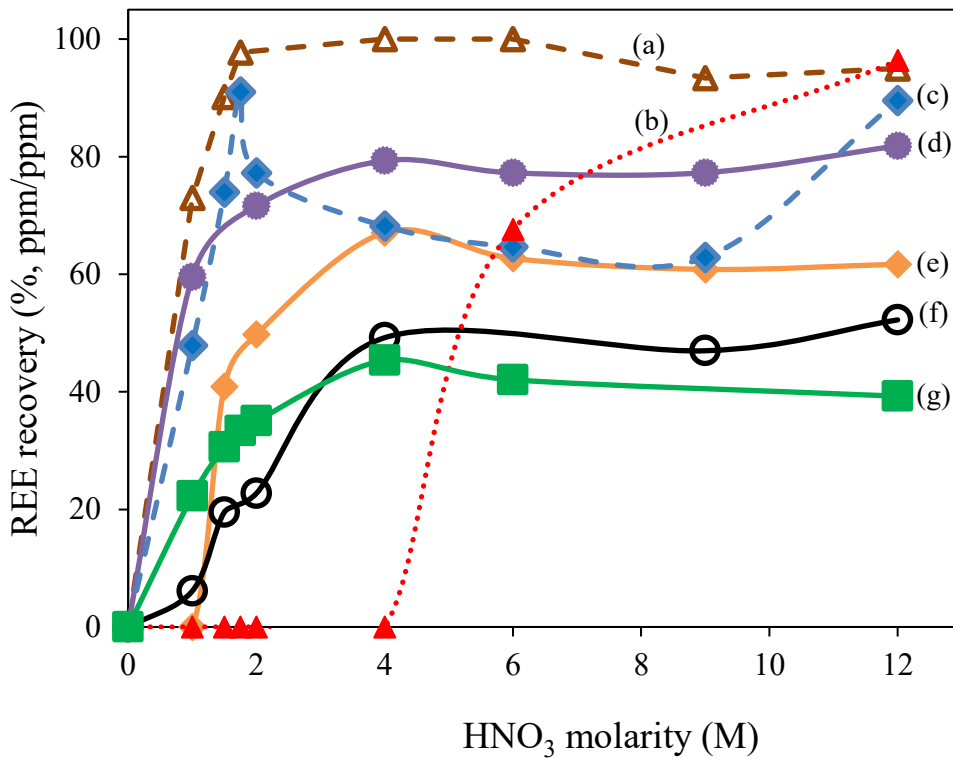


Figure 2 Effect of HNO<sub>3</sub> concentration on REE recovery ((a) Gd, (b) Ce, (c) Ho, (d) Y, (e) Yb, (f) Tm, and (g) Nd) after HCl leaching of fused xenotime.

### Effect of H<sub>2</sub>SO<sub>4</sub> Concentration on REE Recovery

The effect of different concentrations of H<sub>2</sub>SO<sub>4</sub> on the leaching of fused xenotime was investigated from 0 M to 12 M H<sub>2</sub>SO<sub>4</sub> and was shown in Figure 3. The recovery of most rare earth elements (REE) was lower than 40% when compared to that of another leaching agent. In this experiment, Y just recovered 50% between 0.5 M and 4 M H<sub>2</sub>SO<sub>4</sub> leaching. REE likely had lower leaching recovery in H<sub>2</sub>SO<sub>4</sub> due to its specific chemical behavior or complexation in fused xenotime. There was precipitate existing after the leaching process, and it can be seen in the H<sub>2</sub>SO<sub>4</sub> leachate. This phenomenon was not preferred, as it will affect the solvent extraction stage in the future. It was the result of leached REE in H<sub>2</sub>SO<sub>4</sub> tending to precipitate back in the leachate. In contrast, the recovery of Ce was 100% when leaching with 4 M to 6 M H<sub>2</sub>SO<sub>4</sub>. Therefore, H<sub>2</sub>SO<sub>4</sub> is suitable to leach Ce from fused xenotime between 4 M and 6 M, as other REE recovery was low (10 to 50% recovery only). This finding suggests that H<sub>2</sub>SO<sub>4</sub> was not suitable to leach REE from fused xenotime except for Ce.

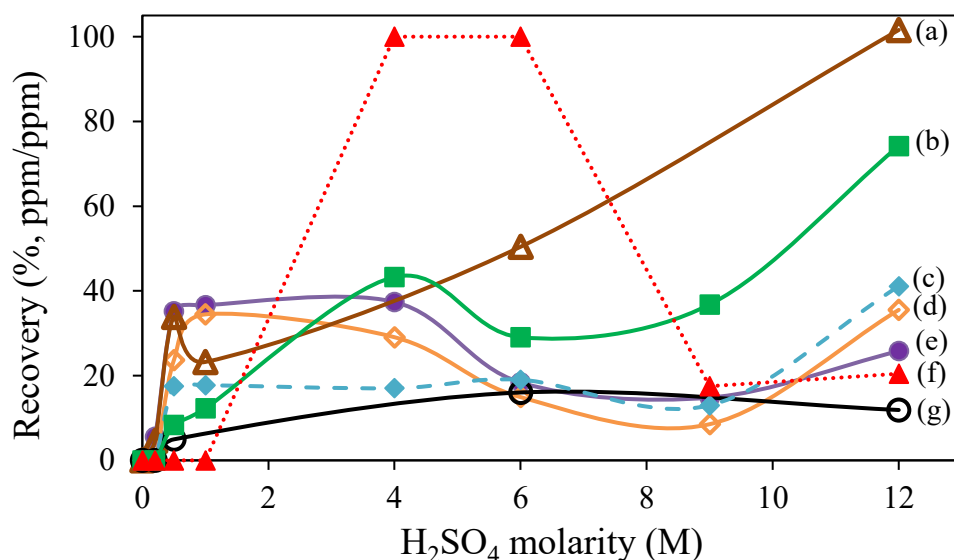


Figure 3 Effect of H<sub>2</sub>SO<sub>4</sub> concentration on REE recovery ((a) Gd, (b) Nd, (c) Ho, (d) Yb, (e) Y, (f) Ce, and (g) Tm) after HCl leaching of fused xenotime.

### Effect of Acetic Acid Concentration on REE Recovery

The leaching recovery results under various acetic acid molarities were shown in Figure 4. At 1 M acetic acid, Y just recovered 50%. Most REE recovery was low (<40%). In 2024, Zhang et al. reported that adding some acetic acid to the leaching process of aluminium (Al) could effectively reduce the REE losses. Such results could suggest that a small amount of acetic acid could assist the leaching process by controlling and selectively leaching the unwanted elements rather than being the major leaching agent. The leaching experiment was not continued for higher concentrations of acetic acid due to thorium (Th) and uranium (U) recovery starting at 2 M and 0.2 M, respectively, according to Figure 5. For REE, its leaching recovery was lower in acetic acid, as it is less reactive in acetic acid compared to Th and U. This phenomenon will be discussed further in the next section.

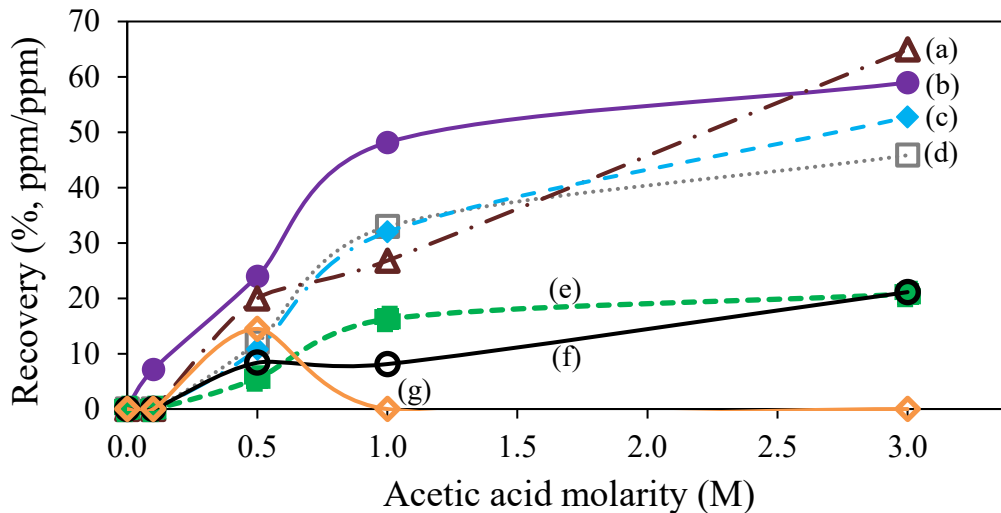


Figure 4 Effect of acetic acid concentration on REE recovery ((a) Gd, (b) Y, (c) Ho, (d) Dy, (e) Nd, (f) Tm, and (g) Yb) after HCl leaching of fused xenotime.

#### Effect of different acid concentrations on Th and U dissolution

The results of applying different acid concentrations on Th and U dissolution from fused xenotime are shown in Figure 5. The idea is to leach out REE into the leachate while preventing Th and U from dissolving into the leachate and maintaining both elements in the solid phase. The dissolution of U began to appear in the leachate at low concentrations (0.2 M) of H<sub>2</sub>SO<sub>4</sub> and acetic acid and at 1.5 M of HNO<sub>3</sub> and HCl. Whereas, Th began to be detected in the leachate after using 0.5 M H<sub>2</sub>SO<sub>4</sub>, 2 M HNO<sub>3</sub>, and 6 M HCl. This comparison reveals that Th and U have higher acid affinity in H<sub>2</sub>SO<sub>4</sub>, as both elements dissolve in H<sub>2</sub>SO<sub>4</sub> at low concentrations. Th was not dissolved in acetic acid, as it needs higher acidity to break down its binding affinity.

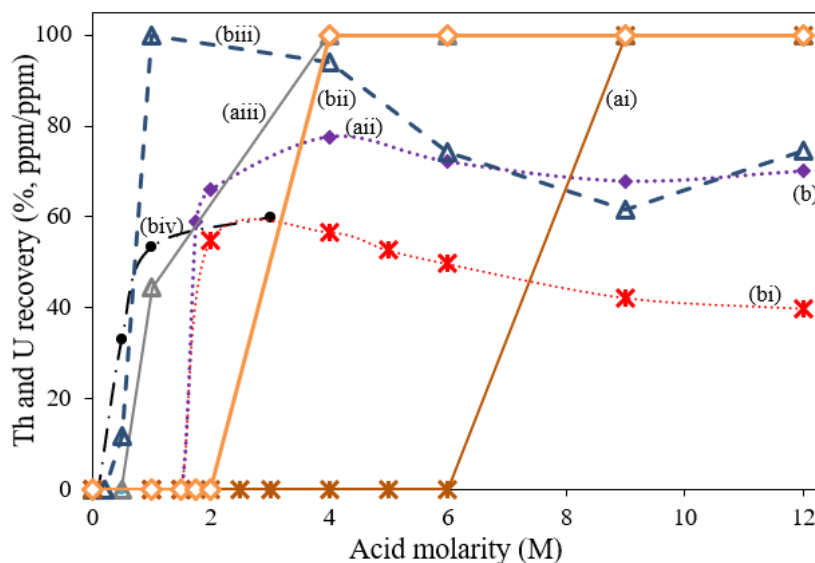


Figure 5 Effect of different acid concentrations on (a) Th and (b) U dissolution from fused xenotime using (i) HCl, (ii) HNO<sub>3</sub>, (iii) H<sub>2</sub>SO<sub>4</sub>, and (iv) acetic acid.

This study reveals that maximum recovery for Y was found to be 79%, 61%, 58%, and 36% for leaching with 4 M HNO<sub>3</sub>, 2 M HCl, 3 M acetic acid, and 1 M H<sub>2</sub>SO<sub>4</sub>, respectively. From this study, it is recommended to use 1.5 M HNO<sub>3</sub> for leaching the fused xenotime, as this concentration can effectively prevent the dissolution of U and Th while still achieving a high recovery of Y (66%). With this research, the fused xenotime, when properly fused and leached, can effectively recover REE in the leachate while preventing dissolution of Th and U to ensure the products are radioactively free.

For selective leaching, all elements offer some compelling insights into the underlying mechanisms of rare earth element leaching from a xenotime matrix. The general trend observed for elements was an initial increase in recovery with increasing acid molarity, followed by a peak, and then a decrease or levelling off at higher acid concentrations. This classic profile in acid leaching suggests a balance of several physicochemical processes. The initial increase in recovery (0 M to ~2 M acid) occurs at lower acid concentrations, as the primary mechanism is the protonation and dissolution of the rare earth compounds, likely present as oxides, hydroxides, or complex compounds within the fused xenotime matrix. The fused xenotime would have been liberated more because of acid attack due to structural changes from the fusion process. Acid solution provides H<sup>+</sup> ions that react with the mineral complexes, breaking down the crystal lattice within the fused xenotime and releasing the REE ions into the aqueous phase. The dissolution reaction can be generally represented as acid concentration increases, the availability of H<sup>+</sup> ions rises, driving the dissolution equilibrium forward and enhancing the kinetics of leaching, thus leading to higher recovery (Zhao et al., 2024).

The peak recovery is represented in the second stage by the optimal balance between sufficient acid concentrations for dissolution and other factors that might hinder recovery at higher concentrations (Maltrana and Morales, 2023). At this point, the rate of dissolution is maximal, and the rare earth ions are stable in solution, likely as hydrated ions or simple chloride complexes. In the last stage of acid leaching, a decrease or levelling off in recovery (above ~4 M acid) occurs due to the decline in REE recovery at higher acid concentrations. This is a critical observation that can be attributed to several factors, such as the common ion effect or salting out, the formation of less soluble chloride complexes, and the kinetics of precipitation. In common ion effector salting out, at very high ionic strengths (high acid concentration), the activity coefficients of the dissolved REE ions can decrease significantly. These decreases can lead to a "salting out" effect, where the solubility of the rare earth solutions (or other less soluble REE species) in the highly concentrated acid solution decreases, potentially causing precipitation. Whereas the formation of less soluble chloride complexes happens at very high concentrations, REE ions might form highly stable, but less soluble, complex species with chloride ions (for example, anionic chloro-complexes). The formation of these species could shift the equilibrium away from the free, highly soluble REE<sup>3+</sup> ions, leading to reduced recovery if these complexes are less stable or prone to precipitation under these conditions. In addition, in kinetics of precipitation (Romano et al., 2025), the very high concentration of hydrogen and chloride ions might also favour the formation of new, less soluble phases or a shift in the reaction equilibrium that precipitates some of the dissolved REEs.

However, Nd shows a moderate recovery, while Ho and Tm exhibit significantly lower recoveries. This difference is mechanistically important. Rare earth elements have slightly different chemical properties due to their varying ionic radii and electronic configurations (lanthanide contraction). These differences affect their solubility, complexation, and chemical binding in the matrix. Some REE solutions might be inherently less soluble at high acid concentrations than others. Also, multiple elements' existence would form complexes of varying stability with chloride ions, influencing their speciation and solubility in concentrated acid. The initial distribution and chemical binding of these multiple elements within the fused xenotime might also vary and affect the leaching process. This phenomenon would impact elements released in the aqueous phase during leaching. Some complexes might be more structurally integrated or exist as different mineral phases (Muhammad et al., 2025).

This finding provides valuable insights for optimizing the single-stage acid selective leaching process. Understanding these mechanistic behaviours is key to developing more efficient and environmentally viable hydrometallurgical processes, aligning with the goal to enhance yttrium supply locally.

## CONCLUSION

In conclusion, the maximum recovery for Y was determined to be 79%, 61%, 58%, and 36% for leaching with 4 M HNO<sub>3</sub>, 2 M HCl, 3 M acetic acid, and 1 M H<sub>2</sub>SO<sub>4</sub>, respectively. Utilizing 1.5 M HNO<sub>3</sub> for leaching fused xenotime is advisable, since this concentration efficiently inhibits the dissolution of U and Th while attaining a substantial recovery of Y (66%).

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