

The recovery of rare earth elements and titanium using direct atmospheric leaching and digestion methods from low-grade monazite sources

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ABSTRACT

Rare earth elements (REEs) have an increasingly wide range of applications in various industries. This study investigates the dissolution of REEs using two methods: direct atmospheric leaching and digestion with sulfuric acid, aiming to compare the efficiency of both approaches. Prior to leaching experiments, magnetic and gravity pre-concentration processes were applied to the sample to enhance the grade of rare earth elements. The total concentrations of REEs, titanium, and iron in the monazite low-grade sample were 0.88%, 3.88%, and 8.25%, respectively. In the direct leaching method, the effects of various parameters, such as temperature, time, acid concentration, and the liquid-to-solid ratio on the recovery of REEs and associated metals were examined. Based on the experiments, the optimal conditions for these parameters were found to be an acid-to-solid ratio of 1.5, a liquid-to-solid ratio of 1.5, a temperature of 125°C, and a duration of 3 hours. Under these optimal conditions, the recovery rates for REEs, titanium, and iron were 80.1%, 89%, and 81.2%, respectively. For the digestion method using sulfuric acid, influential factors, such as digestion temperature, sulfuric acid-to-solid ratio, water leaching time, water leaching-to-solid ratio, water leaching temperature, and water leaching duration were investigated. The optimal conditions for digestion and subsequent leaching were determined to be a sulfuric acid-to-solid ratio of 1.2, a digestion temperature of 220°C, a digestion time of 1 hour, a water-to-solid ratio of 1.5 during leaching, a leaching time of 1 hour, and a leaching temperature of 30°C. Under these optimal conditions, the recovery rates for REEs, titanium, and iron were 91.4%, 88.2%, and 72.4%, respectively.

Keywords: Rare earth elements (REEs), Leaching, Digesting, Sulfuric acid, Titanium.

1. Introduction

Rare earth elements (REEs) are a group of 17 metallic elements, including the lanthanides, scandium, and yttrium that play a crucial role in modern technologies (Echeverry-Vargas & Ocampo-Carmona, 2022). Their unique electronic, magnetic, and optical properties make them essential in high-performance magnets, electric vehicle batteries, wind turbines, catalysts, and advanced electronic devices (Mwewa et al., 2022). The increasing global demand for REEs, particularly in clean energy and high-tech industries, has intensified research efforts to develop efficient and sustainable extraction and recovery methods. However, REE extraction presents significant challenges due to their complex mineral associations and the environmental concerns associated with traditional processing techniques (Kusrini et al., 2018; Julapong et al., 2023).

Monazite is one of the primary sources of REEs and is commonly processed using sulfuric acid digestion or direct atmospheric leaching. However, other REE-bearing minerals, such as bastnäsite, xenotime, loparite, allanite, parisite, apatite, and gadolinite, have also been studied for their potential in REE extraction (Kumari et al., 2015). Among these, monazite is widely utilized due to its relatively high REE content and established processing methods. However, the presence of radioactive

elements, such as thorium and uranium in monazite complicates its processing and requires careful waste management strategies (Alves et al., 2021).

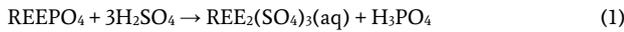
Several studies have focused on improving the efficiency and environmental sustainability of REE extraction from monazite. Sulfuric acid digestion, one of the most widely used methods, involves treating monazite with concentrated sulfuric acid at high temperatures (200–300°C) to convert REEs into soluble sulfates. However, this method presents challenges, such as incomplete REE conversion and the generation of hazardous waste. Alternative leaching techniques, including direct leaching with sulfuric acid, have been explored to enhance dissolution rates and reduce processing temperatures, thereby improving both efficiency and safety. Additionally, hydrometallurgical processes, such as sulfuric acid baking and alkaline digestion, have been compared to determine the most effective method for different grades of monazite. Studies indicate that lower-grade monazite concentrates are often processed using sulfuric acid digestion, whereas alkaline digestion is more suitable for higher-grade materials (Berry et al., 2018; Demol et al., 2018).

In sulfuric acid digestion, after monazite reacts with sulfuric acid, the

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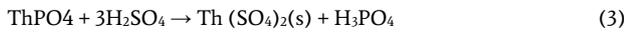
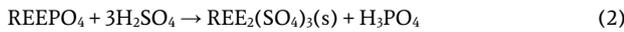
resulting rare earth sulfates must be separated from impurities through a water leaching step. Water leaching plays a critical role in REE recovery by dissolving the REE sulfates into solution, while leaving insoluble residues, such as thorium and iron, behind (Sadri et al., 2017). The efficiency of this step depends on factors, such as the water-to-solid ratio, leaching temperature, and leaching duration, which influence REE recovery and impurity separation. Optimizing these conditions enhances overall REE extraction efficiency and reduces contamination from non-REE elements (Jha et al., 2016).

Another method for REE recovery is direct atmospheric leaching, in which monazite is treated with sulfuric acid under atmospheric pressure conditions to dissolve REEs selectively while minimizing impurity dissolution (Al Sheidi et al., 2024). The direct leaching reaction with sulfuric acid can be represented as follows (Equation 1):



This reaction indicates that monazite's phosphate structure is broken down under acidic conditions, releasing REEs into solution, while phosphoric acid (H_3PO_4) is generated as a byproduct. The efficiency of this process depends on various factors, including acid concentration, temperature, leaching time, and the liquid-to-solid ratio (Hou et al., 2025; Peelman et al., 2016).

The chemical reactions involved in sulfuric acid digestion and subsequent water leaching include the breakdown of the phosphate structure in monazite to form rare earth sulfates and phosphoric acid (Equations 1, 2, and 3):



To enhance REE recovery, key parameters, such as the acid-to-solid ratio, reaction temperature, reaction time, and water leaching conditions must be optimized. Higher temperatures generally improve digestion efficiency, but excessive heating can lead to unwanted byproducts. Similarly, the choice of water leaching parameters affects the final recovery rates of REEs and the purity of the extracted solution (Zhang et al., 2016; Berry et al., 2018).

This study investigates and compares direct atmospheric leaching and sulfuric acid digestion for REE recovery from low-grade monazite sources. By systematically optimizing key processing parameters, the research aims to enhance REE extraction efficiency, reduce environmental impact, and contribute to the development of more sustainable and economically viable REE recovery technologies.

2. Material and methods

2.1. Mineralogical studies and sample analysis

To conduct mineralogical studies, polished and thin sections of a low-grade monazite ore sample from Central Iran (Saghand) were prepared and examined using a ZEISS Axioplan 2 polarized light microscopy with transmitted and reflected light (Figure 1). Prior to these studies, magnetic and gravity pre-concentration processes were applied to the sample to enhance the grade of rare earth elements. The studies revealed that plagioclase, accompanied by tremolite and phlogopite, is the most significant mineral in the sample. Other observed minerals include chlorite, titanite, quartz, pyrite, and dolomite. Ilmenite and magnetite are the primary metallic minerals, comprising approximately 2% to 4% of the sample. The ilmenite mineral was predominantly found interlocked with titanite. Trace elements, such as cerium, lanthanum, and yttrium were present in very small amounts and appeared as bright zones within the ilmenite and titanite.

Further analyses were conducted using a scanning electron microscopy (SEM) and SEM/EDX analysis, revealing that rare earth elements (REEs) occur as monazite minerals in extremely fine particles (smaller than 5 microns), interlocked with titanite and ilmenite.

The sample, labeled as LP12, was analyzed using an ICP-OES device

to determine the grade of rare earth elements and other metals. The total REE and other metal contents in sample LP12 are shown in Table 1.

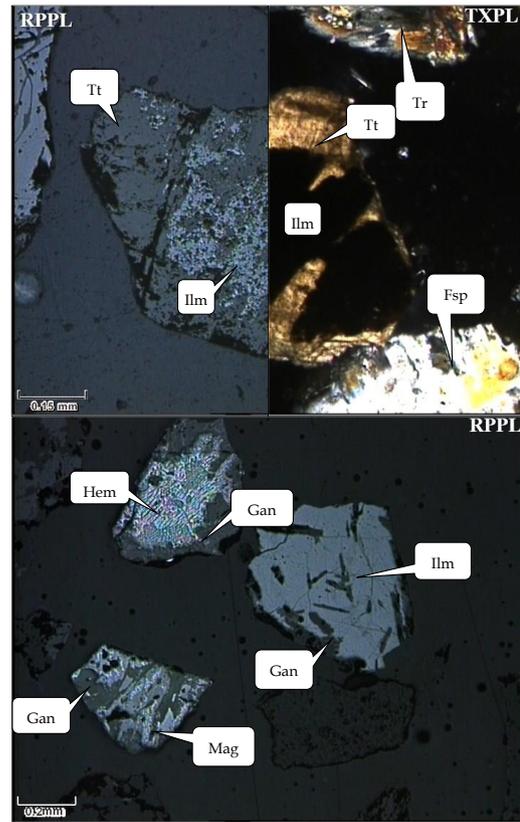


Figure 1. Microscopic images depicting the interlocking of magnetite, hematite, ilmenite, and titanite with gangue minerals.

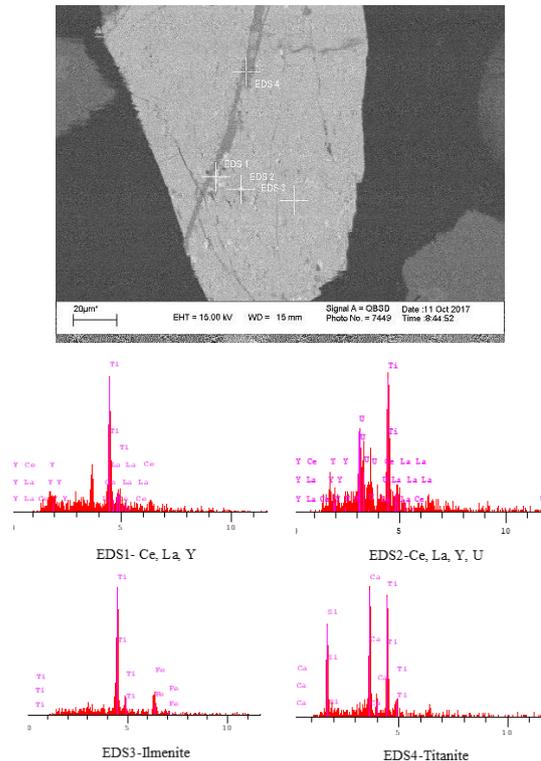


Figure 2. SEM image and SEM/EDX results of Ilmenite, Titanite and Monazite ores.

The sample, labeled as LP12, was analyzed using an ICP-OES device to determine the grade of rare earth elements and other metals. The total REE and other metal contents in sample LP12 are shown in Table 1.

Table 1. The total grade of rare earth elements and the metals titanium and iron in sample LP12.

Sample	ΣREE(ppm)	Ti(%)	Fe(%)
LP12	8837	3.875	8.25

After grinding, the sample had a d_{80} particle size of 23 microns, which was used for leaching and digestion experiments. The particle size distribution analysis of sample LP12 is depicted in Figure 3.

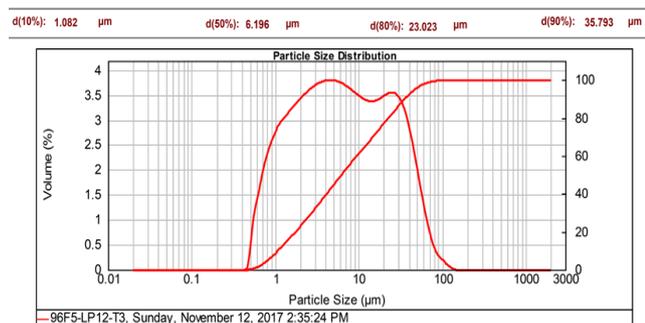


Figure 3. The particle size distribution of sample LP12 using a Laser Diffraction Particle Size Analyzer.

2.2. Direct leaching method

The direct leaching of REEs from the sample was conducted using sulfuric acid under stirring conditions. After leaching, the pregnant leach solution (PLS) was separated from the solid using vacuum filtration, followed by washing the solid residue with water. The leach residue and the volume-adjusted PLS were sent for chemical analysis to calculate the recovery rates. The leaching experiments were carried out in 500 mL Erlenmeyer flasks with condensers to prevent water evaporation.

The effects of key factors, such as leaching time, temperature, solid-to-liquid ratio, and sulfuric acid concentration, were investigated using the Taguchi experimental design method. The leaching test conditions and their results are summarized in Table 2.

2.3. The digestion method using sulfuric acid and leaching.

Unlike leaching, which is a single-step process, the digestion of rare earth elements involves two distinct stages. In the first stage, the sample is thoroughly mixed with concentrated sulfuric acid at room temperature. Then, the temperature is increased to promote the digestion process. After the digestion is completed, the second stage involves water leaching of the digested material to transfer the rare earth elements into the aqueous phase. To evaluate the efficiency of digestion using sulfuric acid, various digestion experiments were conducted on the sample using sulfuric acid. The key factors influencing the digestion process were examined, including digestion temperature, leaching time, sulfuric acid-to-solid ratio, water leaching-to-solid ratio, water leaching temperature, and water leaching time.

The experimental conditions for digestion and the values for each factor are presented in Table 3. The experiments were designed using the Taguchi experimental design method.

3. Results and discussion

3.1. Direct leaching method

In Figure 4, the average effect of each factor is shown while keeping other factors at their average levels. Based on the average effects, it is determined that temperature has the greatest impact on the recovery of rare earth elements. Additionally, after temperature, acid concentration has a greater influence on the recovery of rare earth elements compared to other factors.

Based on the results obtained from laboratory activities, it was determined that temperature is the most influential factor in the recovery of rare earth elements. Therefore, for subsequent experiments, an increase in temperature to higher levels was examined.

The results also indicate that increasing the temperature from 90°C to 125°C under identical conditions enhances the recovery of rare earth elements from 72% to an average recovery of 81%. Based on the conducted leaching experiments, it was determined that temperature has the most significant impact on the recovery of rare earth elements. The highest recovery was observed at the boiling point of the solution (110–125°C). Additionally, factors, such as acid concentration, liquid-to-solid ratio, and leaching time also influence the recovery process.

According to the experiments, the optimal conditions for these factors were found to be an acid-to-solid ratio of 1.5, a liquid-to-solid ratio of 1.5, and a leaching time of 3 hours at 125°C. Under these optimal conditions, the recovery rates for rare earth elements, titanium, and iron were 80.1%, 89%, and 81.2%, respectively.

Table 2. The direct leaching test conditions and their results.

Num	Acid to Solid ratio (gr/gr)	Digestion Time (h)	Digestion Temperature	Water to Solid ratio in Leaching (ml/gr)	Leaching Temperature	Leaching Time (h)
1	1	5	220	6	50	3
2	1.2	1	260	6	50	1
3	1	3	180	8	50	1
4	1	3	180	6	30	3
5	0.8	1	220	8	50	5
6	1	1	180	6	70	5
7	1.2	1	220	4	70	3
8	0.8	3	220	6	70	1
9	1	3	220	4	30	5
10	1.2	5	180	4	50	5
11	0.8	5	180	8	70	3
12	1	1	260	8	30	3
13	0.8	5	260	6	30	5
14	0.8	3	260	4	50	3
15	1.2	3	260	8	70	5
16	1.2	5	220	8	30	1
17	0.8	1	180	4	30	1
18	1	5	260	4	70	1

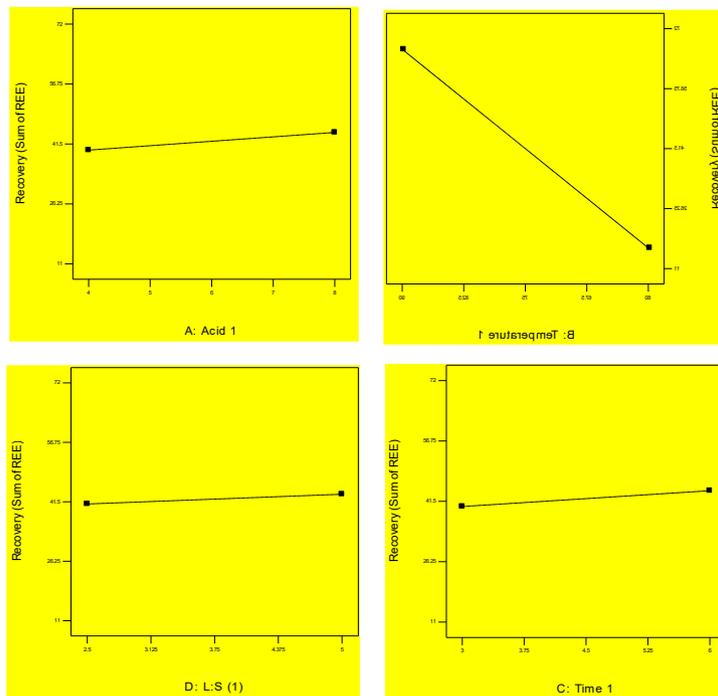


Figure 4. The average effect of leaching factors on the recovery of rare earth elements.

3.2. Direct leaching method

The results of the digestion experiments conducted (Table 3) are presented in Table 4. This table includes the recovery rates of rare earth elements, titanium, and iron.

Table 4. The results of the digestion experiments designed using the Taguchi experimental design method.

Num	ΣREE Recovery (%)	Ti Recovery (%)	Fe Recovery (%)
1	84.6	83.8	73.6
2	87.0	88.0	75.2
3	83.7	85.7	83.7
4	88.2	85.3	85.2
5	66.3	62.0	61.3
6	67.0	63.4	69.6
7	53.8	51.1	58.1
8	64.7	60.9	68.3
9	75.7	72.2	75.6
10	84.6	89.8	84.9
11	77.7	38.4	69.5
12	69.8	62.4	65.9
13	70.5	61.4	69.9
14	59.0	53.8	62.2
15	85.6	79.3	80.9
16	64.8	46.1	55.5
17	43.1	28.1	43.4
18	86.8	84.4	77.0

The results obtained from the digestion and leaching experiments, analyzed using Dx7 software, indicate that the absence of a condenser and the drying of the pulp have the greatest impact on the recovery of rare earth elements, leading to an increase in their recovery. After the effect of the condenser, the sulfuric acid-to-solid ratio has the next highest impact, where increasing the acid ratio enhances the recovery of

rare earth elements. The effect of each factor on the recovery of rare earth elements is shown in Figure 7. Other factors have a lesser impact on the recovery rate, with their effects summarized as follows:

- Increasing the digestion time slightly increases the recovery of rare earth elements.
- Increasing the digestion temperature also slightly increases the recovery of rare earth elements.
- Increasing the leaching time does not affect the recovery of rare earth elements within the selected time range.
- Leaching temperature has an inverse effect, where increasing the temperature slightly reduces the recovery.
- Increasing the water-to-solid ratio during leaching slightly increases the recovery of rare earth elements.

Based on the analysis of the digestion experiment results, the conditions for maximum recovery of rare earth elements were determined. The optimal experimental conditions include a digestion temperature, a leaching time of 1 hour, a sulfuric acid-to-solid ratio of 1.2, a water leaching-to-solid ratio of 4, a water leaching temperature of 30°C, and a leaching time of 1 hour.

For further investigation, the effects of digestion temperature and the sulfuric acid-to-solid ratio were examined. In these experiments, temperatures of 220°C and 260°C and acid-to-solid ratios of 1.2 and 1.5 were evaluated. Additionally, in another experiment, the effect of increasing the digestion time from 1 hour to 2 hours was assessed.

The results of these experiments, comparing digestion and leaching conditions with the optimal conditions, showed that increasing the temperature from 200°C to 260°C and increasing the sulfuric acid-to-solid ratio from 1.2 to 1.5 did not enhance the recovery of rare earth elements. Furthermore, the results indicated that extending the digestion time from 1 to 2 hours had no effect on the recovery of rare earth elements.

The effect of reducing the water-to-solid ratio in leaching and the sulfuric acid-to-solid ratio was also investigated in other experiments. In these tests, water-to-solid ratios of 4 and 1.5 in leaching and acid-to-solid ratios of 1.2 and 1 were examined.

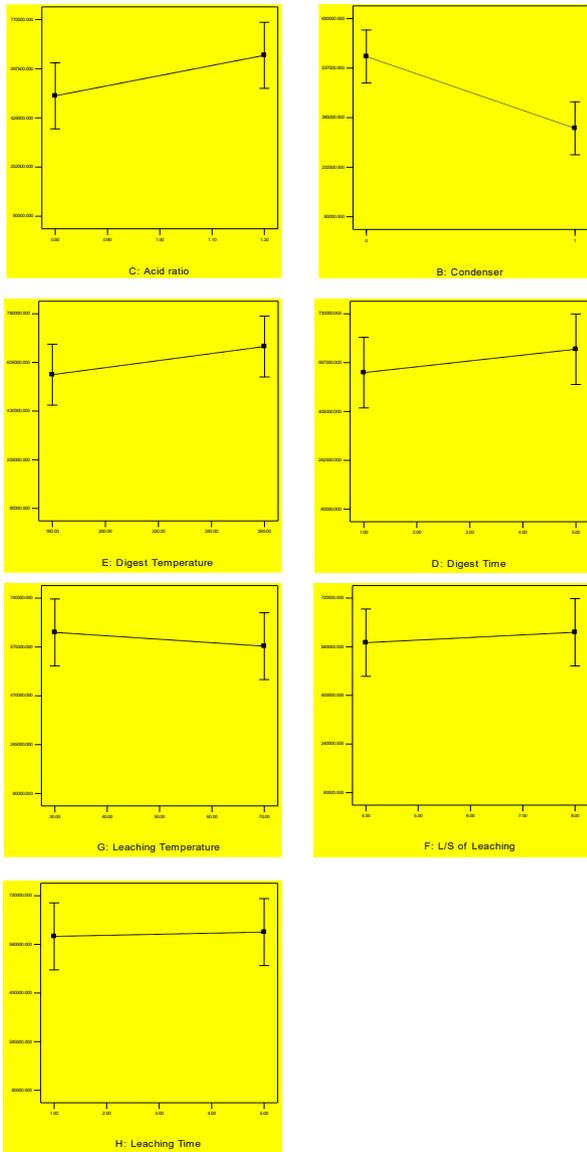


Figure 5. the effect of digestion and leaching factors on the recovery of rare earth elements.

The results indicate that reducing the acid ratio from 1.2 to 1 leads to a decrease in the recovery of rare earth elements and titanium. However, it was also found that reducing the water-to-solid ratio in leaching does not reduce the recovery of rare earth elements. Therefore, to increase the concentration of rare earth elements in the PLS (Pregnant Leach Solution), the water-to-solid ratio can be reduced.

In another experiment, the effect of increasing the digestion temperature was investigated, where the digestion temperature was set at 400°C. The other conditions for digestion and leaching remained the same as the optimal conditions.

The results show that increasing the temperature to 400°C does not result in a significant increase in the recovery of rare earth elements, and the optimal digestion temperature can be considered 220°C. However, increasing the digestion temperature reduces the recovery of iron.

The results of the digestion experiments show that not using a condenser and allowing the pulp to dry has the greatest impact on increasing the recovery of rare earth elements. Following the effect of the condenser, the sulfuric acid-to-solid ratio has the next highest influence, where increasing the acid ratio improves the recovery of rare earth elements..

4. Conclusions

Based on the experiments conducted at this stage, the optimal conditions for digestion and subsequent leaching were identified. These conditions include a sulfuric acid-to-solid ratio of 1.2, a digestion temperature of 220°C, a digestion time of 1 hour, a minimum water-to-solid ratio of 1.5 during leaching, a leaching time of 1 hour, and a leaching temperature of 30°C. Under this optimal digestion and leaching conditions, the recovery rates of rare earth elements, titanium, and iron are 91.4%, 88.2%, and 72.4%, respectively.

For direct leaching, the optimal conditions include a sulfuric acid-to-solid ratio of 1.5, a liquid-to-solid ratio of 1.5, and a leaching time of 3 hours at 130°C. Under these conditions, the recovery rates of rare earth elements, titanium, and iron are 80.1%, 89%, and 81.2%, respectively.

The recovery achieved using the digestion and leaching method is higher than that obtained through sulfuric acid leaching at high temperatures. Moreover, the digestion method consumes less acid compared to high-temperature sulfuric acid leaching. Therefore, the digestion and leaching method has been identified as a more suitable approach for the dissolution of rare earth elements and titanium.

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