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Research Article

Optimization and Kinetics of Terbium Leaching from Lapindo Mud using Sulfuric Acid as the Leaching Agent

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Abstract

This study investigated the impact of solid/liquid ratio, solvent concentration, temperature, and leaching time on the recovery of rare earth elements (REEs), particularly terbium, from Lapindo mud using sulfuric acid as a leaching agent. The objective was to optimize the leaching conditions and identify the most appropriate kinetic model for describing the extraction process. Leaching experiments were conducted under various solid/liquid ratios, sulfuric acid concentrations, temperatures, and time. The findings revealed that the maximum terbium recovery of 94.51% was achieved at a solid/liquid ratio of 0.5, and 18 M sulfuric acid was used as the leaching agent for the extraction process at 200 °C for 30 minutes. Kinetic analysis proved that the Zhuravlev-Leshokin-Templeman (ZLT) model best described the leaching process. The calculated reaction's apparent activation energy (E_a) was 27.96 kJ/mol, indicating that a combination of chemical reactions and diffusion mechanisms controls the leaching process. These insights are crucial for optimizing the extraction of terbium and other REEs from Lapindo mud, offering significant potential for industrial applications in recovering valuable materials from waste sources.

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Keywords: Kinetics; Lapindo mud; Leaching; Rare earth elements; Terbium

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1. Introduction

Rare earth elements (REEs), a collective term for 17 chemically similar elements, which include lanthanum (La), cerium (Ce), praseodymium (Pr), neodymium (Nd), promethium (Pm), samarium (Sm), europium (Eu), gadolinium (Gd), terbium (Tb), dysprosium (Dy), holmium (Ho), erbium (Er), thulium (Tm), ytterbium (Yb), lutetium (Lu), scandium (Sc), and yttrium (Y) [1,2]. These elements naturally co-occur in mineral deposits and share similar physical and chemical

properties, such as high electrical conductivity, slight variations in solubility, and predominantly trivalent oxidation states [3]. Often described as "industrial vitamins," REEs exhibit unique magnetic, optical, and electronic properties, making them indispensable in high-technology industries. Their applications span sectors such as nuclear technology, electronics, aerospace, and renewable energy production [4,5]. Global demand for REEs has surged over the past 15 years, doubling to approximately 125,000 tonnes annually, with projections estimating a rise to 315,000 tonnes by 2030 [6]. This significant growth is primarily driven by the increasing

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adoption of green technologies and advanced electronic devices.

REEs are classified into light rare earth elements (LREEs), ranging from lanthanum (La) to europium (Eu), and heavy rare earth elements (HREEs), ranging from gadolinium (Gd) to lutetium (Lu) and yttrium (Y). HREEs hold greater economic importance because of their specific roles in critical applications, such as laser crystals, optical amplifiers, advanced alloys, ceramics, and medical technologies [7]. Among REEs, terbium(III) oxide stands out for its ability to enhance material properties, especially in glass formulations. The distinct atomic and electronic configurations of terbium contribute to glass's unique structural and optical characteristics. At the same time, its redox flexibility, which shifts between the +3 and +4 states, is advantageous for catalytic soot oxidation. The Tb3+ ion's green luminescence, with emission bands at 489 nm, 543 nm, 584 nm, and 622 nm, has made terbium a focus in diagnostics, therapeutics, magnetism, and fluorescence research, mainly through isotopes 149Tb, 152Tb, 155Tb, and 161Tb [8-11]. Terbium is a versatile rare earth element with extensive applications across various industries. It plays a vital role in electronics and display technologies, particularly in green phosphors for displays, LCD backlight units, and plasma panels [12,13]. Terbium is also crucial in advanced technologies, such as solid-state devices, fuel cells, magneto-restrictive alloys, laser systems, and medical theranostics [14,15].

Chemical leaching for Tb extraction, despite its high costs associated with significant reagents and water consumption, remains well-suited for industrial applications [16]. Its effectiveness, scalability, and straightforward implementation make it a practical choice for large-scale operations. Unlike mechanical activation, which demands considerable energy input and specialized equipment, chemical leaching utilizes widely available reagents and conventional processing techniques, reducing the complexity of industrial setups [17]. Similarly, while sequential

digestion offers high recovery rates and environmental benefits, it often involves more intricate processes that may not be as easily integrated into existing industrial workflows [18]. As such, the reliability, adaptability, and proven track record of chemical leaching render it a preferred method for industrial-scale operations despite its higher operating costs.

Studying terbium's leaching behavior and kinetic analysis is essential, particularly in the context of various ore sources. Previous research has explored the leaching behavior of rare earth elements (REEs) such as dysprosium (Dy) and ytterbium (Yb) from zircon tailings [19,20], red mud [21], spent catalysts and REEs from fly ash and bottom ash [22,23]. A notable source of heavy rare earth elements (HREEs), including terbium, is Lapindo mud—a continuously erupting mud volcano located in the Porong subdistrict of Sidoarjo, East Java, Indonesia, which has been active since May 2006 [24,25]. Elemental composition of Lapindo mud presented in Table 1.

The potential for terbium extraction from this mud presents a significant opportunity for resource recovery. Therefore, investigating the leaching behavior of terbium from Lapindo mud, focusing on kinetic analysis, is crucial for indepthunderstanding the underlying leaching mechanisms and optimizing extraction processes.

The reactions between monazite (REE-phosphate) and sulfuric acid, as well as between terbium phosphate and sulfuric acid, are critical in understanding the leaching processes of rare earth elements. The reaction with monazite is represented by Equation (1), whereas the specific interaction between terbium phosphate and sulfuric acid is detailed in Equation (2) [26,27].

$$2REEPO_4 + 3H_2SO_4 \rightarrow REE_2(SO4)_3 + 2H_3PO_4$$
 (1)
 $2TbPO_4 + 3H_2SO_4 \rightarrow Tb_2(SO4)_3 + 2H_3PO_4$ (2)

In this study, terbium was extracted from Sidoarjo's Lapindo mud. The drying process was a critical step in preserving the elemental content. Following preparation, the mud was leached with

Table 1.	Elemental	composition	of I	Lapindo	mud
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Element	Mg	Al	Si	S	Cl	K	Ca	Ti	V	Mn
Conc.	0.217%	4.734%	15.980%	0.423%	1.487%	1.653%	2.7%	0.768%	250.4 ppm	0.153%
Element	Fe	Cu	Zn	Ga	As	Br	Rb	Sr	Y	Zr
Conc.	10.434%	55 ppm	237.4 ppm	53.2 ppm	41 ppm	194.8 ppm	176.9 ppm	0.126%	52.9 ppm	503 ppm
Element	Nb	Sn	Te	Ba	Се	Pm	Eu	Gd	Tb	Но
Conc.	15.9 ppm	254.5 ppm	94.3 ppm	381.9 ppm	158.9 ppm	233.3 ppm	458.6 ppm	430.5 ppm	0.189%	141.1 ppm
Element	Er	Pb	Th	U						
Conc.	391.8 ppm	26.5 ppm	35.5 ppm	17.6 ppm						

sulfuric acid. This study aimed to assess the impact of the solid/liquid ratio, leaching agent concentration, temperature, and time on REE recovery and to identify the most suitable kinetic model for describing terbium extraction from Lapindo mud using sulfuric acid solution.

2. Materials and Methods

2.1 Materials and Analysis

The materials utilized in this study included Lapindo mud sourced from Porong (Sidoarjo), 95-97% sulfuric acid (H₂SO₄) supplied by Merck & Co., Inc., aquadest from the PSTA-BATAN kernel fabrication laboratory, and filter paper. The analysis involved various instruments, including laboratory glassware, a hot plate with magnetic stirrer (IKA C-MAG HS 4), a ball mill (planetary ball mill type, Changsha Tianchuang Powder Technology Co. Ltd.), a furnace (vertical lift door type, produced by PT. Suhaterm), a sieve, an analytical balance, a thermometer, and an X-ray fluorescence (Epsilon 4 Panalytical) were used for material characterization.

2.2 Terbium Leaching

The influence of various parameters on terbium leaching from Lapindo mud was systematically investigated. The effect of the solid/liquid ratio was assessed by varying the Lapindo mud: sulfuric acid ratio from 1/1 to 1/4 g/mL (0.25-1), with leaching performed in a stirred reactor at 200 °C using 18M sulfuric acid for 60 minutes. The impact of the solvent concentration was evaluated by adjusting the sulfuric acid concentration within the range of 10.8-18 M while maintaining a constant solid/liquid ratio of 1/2 g/mL under the same temperature and duration. The effect of temperature on leaching was examined by varying the process temperature between 100-200 °C, using 18 M sulfuric acid at a 1/2 g/mL

solid/liquid ratio for 60 minutes. Finally, the influence of leaching time was explored over intervals ranging from 0 to 60 minutes, with the reaction conducted at 200 °C, a solid/liquid ratio of 1/2 g/mL, and 18 M sulfuric acid.

2.3 Kinetic Analysis

The leaching kinetics were assessed by altering the leaching temperature at 100, 150, and 200 °C. At each temperature, samples were collected at specific intervals during the leaching process, specifically at 0, 5, 10, 15, 20, 30, and 60 minutes. The time intervals were adjusted to account for the reaction kinetics during the leaching process. Initially, a 5-minute interval was used to closely observe the significant increase in Tb leaching, as the reaction occurs rapidly in the early stages. For the subsequent period (20-30 minutes), a 10-minute interval was employed to maintain a balance between capturing sufficient data points and experimental efficiency. Finally, a 30-minute interval was adopted between 30-60 minutes as the reaction rate slowed, and fewer data points were necessary to characterize the leaching process during this phase. Sulfuric acid was utilized as the solvent in the leaching process of Lapindo mud. The recovery and recovery fraction of terbium through leaching was calculated using Equations (3) and (4), respectively. Additionally, the leaching residue was analyzed to determine the remaining terbium content in the solid residue.

$$Recovery(\%) = \frac{Xo - Xi}{Xo} \times 100$$

$$X = \frac{Recovery}{100}$$
(3)

$$X = \frac{Recovery}{100} \tag{4}$$

where X_0 is leaching feed mass (g) multiplied by the concentration of terbium in the feed (%), and X_i is leaching residue mass (g) multiplied by the concentration of terbium in the residue.

Table 2. The equation for finding the t value for various models

Model	Time, t
Chemical reaction control	$1-(1-X)^{\frac{1}{3}}$
Ash diffusion control	$t = \frac{1 - 3(1 - X)^{\frac{2}{3}}}{k_s}$
Zhuravlev, Leshokin and Templeman	$t = \frac{k_a}{\left(1 + \frac{1}{2}\right)^2}$
	$t = \frac{\left(\frac{1}{(1-X)^{\frac{1}{3}}} - 1\right)}{k_{sa}}$
Mix model	$t = \frac{\frac{1}{3}(\ln(1-X)) + (1-X)^{-\frac{1}{3}} - 1}{k_m}$

The kinetic models utilized in this study are based on the phenomena described by the shrinking core model (SCM). Kinetic analysis was performed by fitting each kinetic model in Table 2 to the experimental data. The model that best fits the data was subsequently selected to determine the leaching control mechanism, and the corresponding k value was used to calculate the activation energy (E_a) via the Arrhenius equation, as shown in Equations (4-5). In Table 2, trepresents time (in minutes), X is the recovery fraction, k_s denotes the apparent rate constant for chemical reaction control (in min-1), ka is the apparent rate constant for ash diffusion control (in min⁻¹), $k_{\rm sa}$ refers to the apparent rate constant for Zhuravlev-Leshokin-Templeman control min⁻¹), and $k_{\rm m}$ stands for the apparent rate constant for the mixed model (in min-1).

$$k = k_0 \exp \frac{-E_a}{RT} \tag{4}$$

$$\ln k = \ln k_0 - \frac{E_a}{2} \frac{1}{2} \tag{5}$$

k = $k_0 \exp{\frac{-E_a}{RT}}$ (4) $\ln k = \ln k_0 - \frac{E_a}{R} \frac{1}{T}$ (5) where k_0 is a frequency factor (min-1), E_a is apparent activation energy (kJ/mol), R is gas constant (8.314 J.mol⁻¹.K⁻¹), and *T* is temperature (K).

3. Results and Discussion

3.1 Characterization of the Lapindo Mud Composition

Lapindo mud, an industrial byproduct, has been identified as a significant source of rare elements (REEs), with concentrations of terbium (Tb), gadolinium (Gd), erbium (Er), cerium (Ce), holmium (Ho), and yttrium (Y) (Table 3). Among these elements, terbium has the highest concentration, accounting for 0.189% by weight and approximately 61.66% of the total REE content within the mud. The cumulative REE concentration in the Lapindo mud is quantified at 0.31% (wt.%). The REEs content in Lapindo mud surpasses that found in coal fly ash, where the concentration of REEs is approximately 404 ppm [28].

3.2 Optimization of Terbium Leaching

This study optimized the leaching process to determine the optimum conditions for LTJ recovery, focusing on variables such as the solid/liquid ratio, leaching agent concentration, temperature, and leaching time. The results for variations in the solid/liquid ratio are presented in Figure 1. The effect of the solid/liquid (S/L) ratio

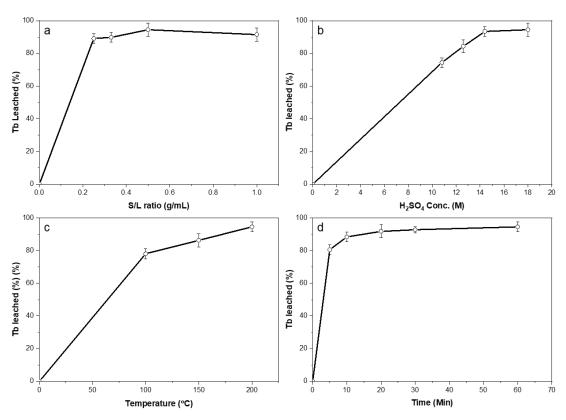


Figure 1. Effects of the (a) S/L ratio, (b) H₂SO₄ concentration, (c) temperature, and (d) leaching time on Tb leaching

Table 3. REEs composition of Lapindo mud

Element	Tb	Gd	Er	Се	Но	Y
Concentration (wt.%)	0.189	0.0430	0.0391	0.0158	0.0141	0.0052

on terbium (Tb) leaching recovery is significant, as shown in Figure 1(a). The highest recovery, 94.51%, was observed at an S/L ratio of 0.5. An increase in acid concentration and volume generally enhances recovery; however, the recovery decreases as the S/L ratio increases. This decrease is attributed to increased slurry viscosity, which hinders the mobility of sulfate ions and the transfer of Tb ions into the solution. An increase in the concentration of solids within a slurry corresponds to heightened viscosity and yield stress, primarily due to the intensified particle-particle interactions associated with higher solid content [29]. This phenomenon aligns with the principles of mass transfer, where solvent diffusion toward solid particles is impeded by higher viscosity and density, ultimately slowing the leaching process [30].

The impact of the H₂SO₄ on Tb leaching as illustrated in Figure demonstrated that increasing the H_2SO_4 concentration resulted in more excellent Tb recovery. This improvement is due to the accelerated reaction rates at elevated solvent concentrations, where a greater density of molecules leads to more frequent molecular thereby enhancing the reaction kinetics. The study revealed that the highest Tb recovery, 94.51%, was achieved at a H₂SO₄ concentration of 18 M. From a technical and economic perspective, an H₂SO₄ concentration of 14 was selected as the optimal leaching agent concentration, as further increases did not result in a significant improvement in Tb recovery.

The effect of temperature on Tb leaching recovery, as depicted in Figure 1(c), shows that higher temperatures result in increased Tb recovery. This trend is attributed to the increased reaction rates at elevated temperatures, where the increased kinetic energy of the reactant

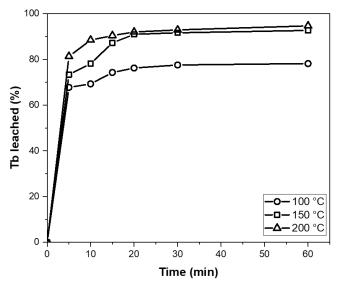


Figure 2. Percent recovery plot with time at 100, 150, and 200 °C for Terbium

particles leads to greater mobility of sulfate ions. Consequently, the transport of Tb ions into the solution is accelerated. The study revealed that the highest Tb recovery, 94.51%, was achieved at a temperature of $200~^{\circ}\mathrm{C}$.

As shown in Figure 1(d), the effect of leaching time on Tb recovery reveals that Tb recoveries increased significantly within the first 20 min and then plateaued with extended leaching durations. A recovery of 91.79% was achieved at 20 minutes, with the highest recovery of 94.51% occurring at 60 minutes. This trend is attributed to the prolonged contact between the feed and the solvent, which enhances the leaching process, although the recovery rate slows as the process approaches equilibrium. A leaching duration of 30 minutes was determined to be optimal, as extending the duration further did not yield a significant improvement in recovery.

3.3 Leaching Kinetics of Terbium from Lapindo Mud using Sulfuric Acid

Figure 2 shows the leaching recovery of Tb at various temperatures, revealing that at 100 °C, recovery increased significantly within the first 20 min but was lower than leaching at 150 °C and 200 °C. Leaching at 150 °C achieved substantial Tb recoveries within 30 minutes, whereas at 200 °C, significant recoveries were observed within just 15 minutes. These results indicate that higher temperatures accelerate the leaching process and enhance the interaction between H₂SO₄ and REEs, as previously discussed. The recovery data were subsequently used for kinetic analysis via four models: chemical reaction control, ash layer diffusion control, the ZLT model, and a mix model. The kinetics study was conducted across a temperature range of 100-200 °C and a leaching time range of 5-30 min, with longer leaching times showing minimal additional recovery. Based on these observations, it can be concluded that the optimal leaching temperature is 200 °C for a duration of 30 minutes. Prolonging the leaching process beyond this point does not improve Tb recovery, as equilibrium has likely been reached.

Figure 3 presents the fits of various kinetics models to the leaching time data, with the accuracy of each model assessed via the coefficient of determination (R²) values listed in Table 2. Compared with ZLT (Figure 1(a)) and mix models (Figure 1(d)), the chemical reaction control (Figure 1(a)) and ash layer diffusion control (Figure 1(b)) models exhibit poor linear fits. Notably, the ZLT model yields the highest R² values, with values of 0.9202, 0.9009, and 0.9219 for temperatures of 100 °C, 150 °C, and 200 °C, respectively. This calculation suggests that using sulfuric acid, the ZLT model most accurately describes the kinetics of the Tb leaching process from Lapindo mud.

As shown in Figure 3, this research found that the kinetic model close enough to the kinetics of the leaching process is the ZLT model. This statement is supported by evaluating the model using the value of R² for the relationship between each model with time. Table 4 shows the R² value of each kinetic model used in the study. The ZLT model, predicated on the assumption of pseudofirst-order kinetics, posits that the leaching rate of the target element is directly proportional to the reactant concentration and the surface area of the solid material. It assumes a uniform particle size distribution, facilitating a consistent leaching rate

across all the particles. The model further presumes that the surface area of the solid remains constant throughout the leaching process, implying negligible changes in particle size or shape due to dissolution. Additionally, it assumes uniformity in the leaching reaction across the solid material, disregarding local variations in reactivity or surface properties. Finally, the model assumes that mass transfer limitations, such as diffusion through a boundary layer, do not significantly influence the overall leaching rate, a condition typically valid in well-mixed systems [31,32].

Table 4. R^2 and k value of various kinetic models

TZ: 4: 1.1	\mathbb{R}^2			k (min ⁻¹)		
Kinetic model	100 °C	150 °C	200 °C	100 °C	150 °C	200 °C
$1 - (1 - X)^{1/3}$	0.8932	0.8562	0.8073	0.0034	0.0089	0.0058
$1 - 3(1 - X)^{\frac{2}{3}} + 2(1 - X)$	0.8977	0.8570	0.8054	0.0046	0.0131	0.0086
$\left(\left(\frac{1}{(1-X)^{\frac{1}{3}}}\right)-1\right)^2$	0.9202	0.9009	0.9219	0.0089	0.0602	0.0560
$\frac{1}{3}(\ln(1-X)) + (1-X)^{-\frac{1}{3}} - 1$	0.9148	0.8930	0.8973	0.0029	0.0154	0.0132

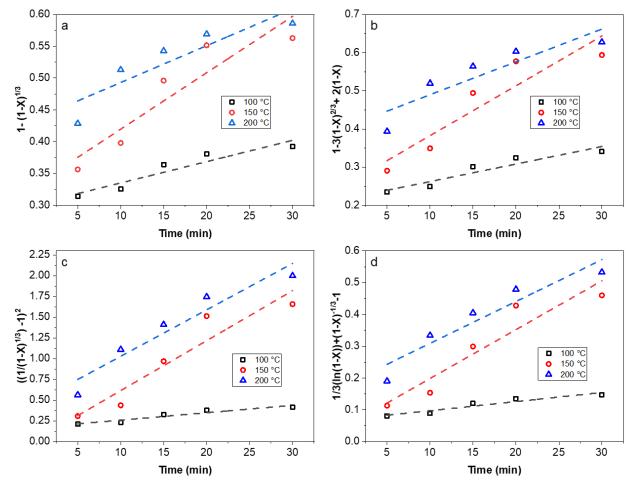


Figure 3. Leaching time vs kinetic model: (a) chemical reaction control, (b) ash layer diffusion control, (c) ZLT kinetic model, and (d) mix model

The apparent activation energy (Ea) for terbium (Tb) leaching from Lapindo mud using H_2SO_4 solution was determined from an Arrhenius plot (ln k vs. 1/T) to be 27.96 kJ/mol (Figure 4), indicating a moderate activation energy for the leaching process. The non-linear correlation in Figure 4 between $\ln k$ and 1/T can be attributed to the deviation in Tb recovery behavior during leaching at 100 °C compared to the consistent behavior observed at 150 °C and 200 °C. This discrepancy affects the linearity of the Arrhenius plot.

The pre-exponential factor (k_0) was calculated as 94.86 min⁻¹, leading to the kinetic equation

$$\left(\left(\frac{1}{(1-X)^{\frac{1}{3}}} \right) - 1 \right)^2 = 94.68 \ min^{-1} \exp\left(-\frac{27.96 \frac{kJ}{mol}}{RT} \right) . t$$

This finding is critical for scaling up the Tb leaching process from Lapindo mud, providing essential parameters for industrial application. Previous research has established that an Ea value of less than 20 kJ/mol indicates that the leaching process is controlled primarily by chemical reactions. In contrast, an E_a value exceeding 42 kJ/mol suggests control by ash layer diffusion [32,33]. The present study, which adheres to the ZLT kinetic model, interprets the leaching process as a mixed mechanism involving reaction and diffusion, as evidenced by the moderate E_a value of 27.96 kJ/mol.

4. Conclusions

The leaching behavior of terbium (Tb) from Lapindo mud using H_2SO_4 was investigated under various leaching conditions. The analysis revealed that Lapindo mud contains 0.31% rare earth elements (REEs), with Tb accounting for 0.1890%. A remarkable leaching recovery of 94.51% was achieved at an S/L ratio of 0.5, an H_2SO_4 concentration of 14 M, and at a temperature of 200

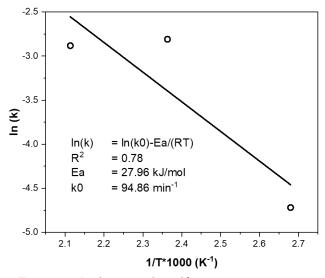


Figure 4. Arrhenius plot of kinetics equation

°C for 30 minutes. The leaching process conformed to the ZLT kinetic model, indicating a mixed reaction and diffusion control mechanism, with an apparent activation energy of 27.96 kJ/mol. These findings are crucial for the scale-up of the Tb leaching process from Lapindo mud, providing essential parameters for its industrial application.

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CRedit Author Statement

Author Contributions: H. Supriadi formulates the concept and writes the original manuscript, S. Suyanti and W. Astuti conduct the formal analysis and validation, T. Handini, V. S. H. Sujoto and G. Prameswara review and visualize the the manuscript. All authors have read and agreed to the published version of the manuscript.

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