



## Recovery of Critical Elements (Dysprosium and Ytterbium) from Alkaline Process of Indonesian Zircon Tailings: Selective Leaching and Kinetics Study

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**Abstract.** Rare earth metals are important today, especially for heavy rare earth elements (HREEs). Dy and Yb, included in the HREE group, are widely used for permanent magnets and sensors. On the other hand, accumulated solid waste of zircon tailings is an environmental issue in mineral processing. Valuable minerals such as rare earth elements (REEs) also remain in the tailings. This study aims to determine the optimum conditions of the experimental parameters and obtain an appropriate kinetic model. The leaching process was carried out in a flat bottom three-neck flask equipped with a thermometer and reflux condenser. Observation of the effect of process conditions on recovery and kinetics analysis obtained satisfactory results. In this study, Dy and Yb leaching from solid REE-hydroxide using HCl solution afforded a total of 61.71% and 74.55% recovery of Dy and Yb, respectively, at operating conditions of 1 M HCl, 60°C, 10 g/100 mL (solid-to-liquid ratio), at 60 min (leaching duration), and 150 rpm (stirring speed). It was found that the leaching process was controlled by chemical reactions with apparent activation energies of 31.68 kJ/mol and 11.95 kJ/mol for Dy and Yb leaching, respectively.

**Keywords:** Dysprosium; Kinetics analysis; Leaching; Ytterbium; Zircon tailings

### 1. Introduction

The need for clean processes is increasing as industrial development progressively turns green. Among the elements used to support the green industry are rare earth elements (REEs). The need for REEs is increasing while primary sources such as bastnaesite, xenotime, and monazite mining are decreasing. REEs are relatively abundant in the earth's crust but not in large concentrations compared to other minerals. REE-oxide production in 2019 and 2020 was 220,000 tones and 240,000 tones, respectively, while the

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doi: [10.14716/ijtech.v14i4.4960](https://doi.org/10.14716/ijtech.v14i4.4960)

worldwide REEs reserve reached 120,000,000 tones (U.S. Geological Survey, 2021). Developments in optimally processing REEs, and efforts to discover alternative secondary sources, must be encouraged.

Dysprosium (Dy) and ytterbium (Yb) are two elements found in the lanthanide group. Dy and Yb are classified as heavy rare earth elements (HREEs) based on their atomic numbers. These metals are included in the list of critical metals and are among the most widely used in the energy sector (Guyonnet *et al.*, 2015). Dy metal is used in permanent magnets, phosphors, and metal halide lamps, while Yb is widely used for optical lenses, pressure sensors, and lasers (Lucas *et al.*, 2014). The need for Dy and Yb metals in the future is predicted to increase (Supriadi *et al.*, 2023).

REEs demand is predicted to increase by 2035 to reach around 374,000 metric tons, with the most used as permanent magnet products at approximately 80,000 metric tons (Yang *et al.*, 2017). In contrast to Dy, Yb has begun to be widely used for memory devices and as a catalyst in the industry to replace other catalysts categorized as too toxic and polluting.

Currently, the primary sources of REEs are the minerals bastnaesite, monazite, and xenotime. Bastnaesite is the primary source, with REEs content of approximately 57% (Boulanger, Bazin, and Turgeon, 2019). Monazite and xenotime are often found in minerals bearing cassiterite and zircon (Trisnawati *et al.*, 2022). The presence of this mineral is usually concentrated in the by-products of cassiterite/zircon processing (Gediga *et al.*, 2019). REEs processing from zircon by-products solve solid waste accumulation and add value to the zircon beneficiation process.

Several studies have observed the REEs leaching kinetics from different material sources. The REEs leaching mechanism is usually influenced by the ore properties, mass transfer behavior, and reaction kinetics. In general, three models can describe the leaching process: homogeneous, shrinking core, and grain models (Petrus *et al.*, 2022; Wanta *et al.*, 2022; Liu *et al.*, 2012). Shrinking-core models can be used to describe the leaching process for systems containing non-porous solid particles and reactant fluids.

Research into REEs leaching has been conducted extensively. Extraction was carried out using various secondary REE sources. Stopic and Friedrich (2016) researched the kinetics of yttrium leaching from ceramic waste dust. Under optimal operating conditions, a yttrium dissolution of 98.6% was achieved. Yttrium dissolution can be described using a kinetic model based on a chemical reaction as the limiting step. The observed activation energy was 31.2 kJ/mol. Obuz *et al.* (2018) studied the leaching kinetics of REEs in complex ores. The reaction constants for the solutions of Ce, La, Nd, and Pr were determined. Research on REE recovery from secondary sources from Indonesia has also been carried out. Rosita *et al.* (2019) observed the yttrium recovery from coal fly ash (CFA) using sulfuric acid, while Prihutami *et al.* (2020) also recovered yttrium from CFA using citric acid with the highest recovery of 83.35%.

This study aims to investigate the leaching of Dy and Yb from secondary sources (zircon tailings) and determine optimal conditions for HREE leaching using HCl from REE-hydroxide. The study also aims to analyze the kinetics of the leaching process and obtain recovery data, such as a kinetic model and its activation energy value.

## 2. Materials and Methods

### 2.1. Roasting

The raw material is zircon tailings from Monokem Surya, Ltd., mined and processed in Bangka Island and Central Kalimantan. The zircon tailings sample contained monazite and xenotime minerals. The Dy and Yb contents in the zircon tailings sample were 1.45% and 1.29%, respectively (Prameswara *et al.*, 2020; Trisnawati *et al.*, 2020). REE-hydroxide was prepared by reacting zircon tailings with NaOH flakes at a temperature of 450°C for 3 hours

and a 1:1 wt ratio of zircon tailings and NaOH flakes. The roasting process was carried out in a muffle furnace. Our previous work has thoroughly described the REE-hydroxide preparation stage, sample characterization, mineral composition analysis, and elemental analysis (Prameswara *et al.*, 2020).

### 2.2. Leaching experiment

The leaching process was carried out in a flat bottom three-neck flask equipped with a thermometer, reflux condenser, and hot plate with magnetic stirrer Thermo Scientific Cimarec. The experimental schematic illustration is presented in Figure 1. Leaching was performed under several operating conditions: temperature, solid-to-liquid ratio (S/L), and acid concentration. A total of 5 g of REE-hydroxide with a particle size of 44  $\mu\text{m}$  was leached for each data collection. Analytical grade HCl (37%, Merck & Co., Inc.) was used. The leaching process was carried out in 0.125–2 M HCl concentration, temperatures ranging from 30–110°C, and a solid-to-liquid (S/L) ratio of 5–25 g/100 mL, a stirring speed of 150 rpm. Based on several studies, higher stirring speed does not significantly affect REE recovery (Kurnia *et al.*, 2016). Elemental analyses of Dy and Yb were performed on the leaching residue. Leaching recovery was calculated using Equation 1.

$$\text{Leaching Recovery} = \frac{x_0 - x_i}{x_0} \times 100 \% \quad (1)$$

where  $x_0$  is the REE concentration in the feed multiplied by the mass of the feed (%), and  $x_i$  is the REE concentration in the residue multiplied by the mass of the residue (%). Elemental analysis was performed using an X-ray fluorescence spectrometer (Rigaku NEX QC, S/N QC1202) on the solid residue. Residue characterization at the optimum leaching process was carried out using Rigaku Miniflex 600 Benchtop X-ray diffractometer (XRD). The leaching process was repeated twice to ensure an acceptable measurement error (less than 10%).

### 2.3. Kinetic analysis

Kinetic analysis was performed using a shrinking-core model. The kinetic models to be tested are the chemical reaction limiting step, diffusion through the ash layer limiting step, and the diffusion model developed by Zhuravlev, Leshokin, and Templeman (ZLT). These three models are expressed in Equations (2)–(4), respectively.

$$1 - (1 - x)^{\frac{1}{3}} = kc \cdot t \quad (2)$$

$$1 - 3(1 - x)^{\frac{2}{3}} + 2(1 - x) = kd \cdot t \quad (3)$$

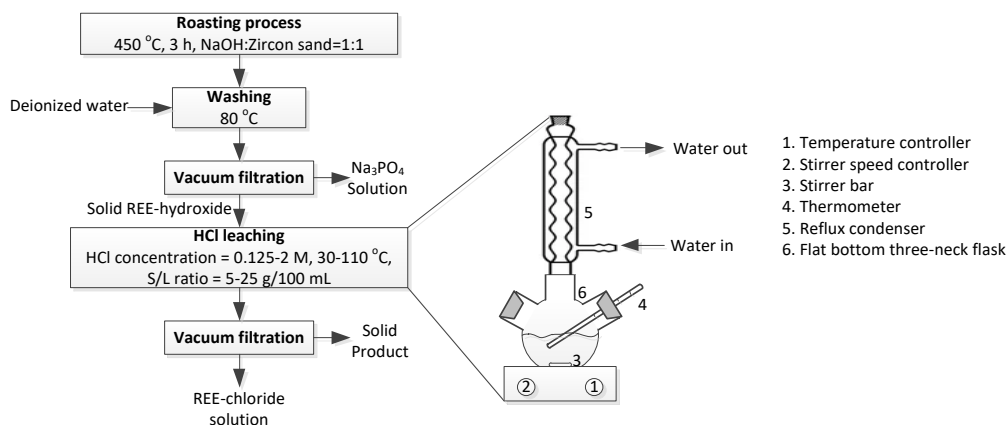
$$\left[ \frac{1}{(1-x)^{\frac{1}{3}} - 1} \right]^2 = kz \cdot t \quad (4)$$

where  $x$  is the percentage leaching recovery divided by 100.  $kc$ ,  $kd$ , and  $kz$  are the rate constant for the chemical reactions control kinetics model, diffusion through the ash layer kinetic model, and ZLT kinetic model, respectively. While  $t$  (min) is the leaching duration. The kinetic model selected to determine the kinetic parameters (frequency factor and activation energy) is the model that exhibits the highest R-squared value. The kinetic parameters were evaluated using the Arrhenius equation (Equations 5 and 6).

$$k = A \exp\left(-\frac{E_a}{RT}\right) \quad (5)$$

$$\ln k = \ln A - \frac{E_a}{R} \frac{1}{T} \quad (6)$$

where  $k$  is the rate constant ( $kc/kd/kz$ ),  $A$  is the frequency factor,  $E_a$  is the activation energy ( $\text{kJ/mol}$ ),  $R = 8.314462 \text{ (J/(mol}\cdot\text{K))}$ , and  $T$  is the leaching temperature ( $\text{K}$ ). The activation energy was obtained from the slope of the curve  $1000/T$  vs.  $\ln k$ , and the frequency factor was obtained from the intercept.

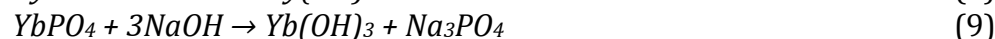
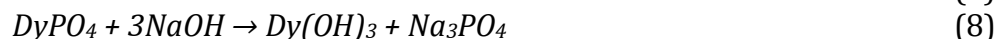
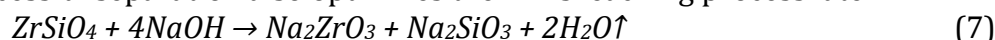


**Figure 1** Experiment scheme

### 3. Results and Discussion

#### 3.1. Roasting

At this stage, REE-phosphate and zirconium silicate contained in monazite and xenotime naturally react with the NaOH flakes. As mentioned in [da-Silva, Dutra, and Afonso \(2012\)](#), the typical reaction is as follows (Equations 7-9). Based on our previous research, there was a decrease in the zircon concentration in the leaching residue. This indicates that zircon also reacted in the alkaline fusion process. The phosphate concentration is also reduced, indicating that most of the phosphate that binds to REE reacts with NaOH to produce  $\text{Na}_3\text{PO}_4$  ([Prameswara \*et al.\*, 2020](#); [Trisnawati \*et al.\*, 2020](#)). Separating the major and unwanted elements from the final product appears effective during the alkaline fusion process. This successful separation also optimizes the REEs leaching process later.



#### 3.2. Effect of operating conditions on Dy and Yb leaching recovery

REEs leaching of solid REE-hydroxide was carried out to carefully observe the recovery behavior under several parameters such as temperature, HCl concentration, and solid-to-liquid ratio. The typical reaction for dissolving REE-hydroxide in an HCl solution is shown in Equations (10) and (11). Observations were also carried out on different types of acid solvents under the same operating conditions. In leaching using a sulfuric acid solution, 59.11% and 70.73% of Dy and Yb were recovered, respectively. The recovery behavior in leaching using  $\text{HNO}_3$  also indicates a lower REEs recovery than that using HCl. Sulfuric acid has a higher viscosity than hydrochloric acid, which causes the wetting rate of solid particles to decrease. As a result, the leaching reaction rate becomes slower. Moreover, sulfuric acid requires a two-step dissociation reaction to release  $\text{H}^+$  into the solution, whereas hydrochloric acid dissociates more rapidly into the solution ([Yang and Honaker, 2020](#)). Therefore, the HCl solution was chosen as the solvent for the REEs leaching process of REE-hydroxide solids, a valuable product of the alkaline fusion process on zircon tailings.



Observations were made on the effect of the leaching operating conditions on Dy and Yb recovery. As shown in Figure 2a, the recovery for Dy and Yb increased drastically in the HCl concentration range of 0.125–1 M. The recoveries for Dy and Yb reached 47.38% and 72.74%, respectively. The recoveries did not show any increase in HCl concentrations above 1 M. Dy and Yb recovery reach plateau shows that the limiting effects have been reached. Increasing the HCl concentration would only increase the dissolution of unwanted elements, such as Si, P, and Al (Walawalkar, Nichol, and Azimi, 2016).

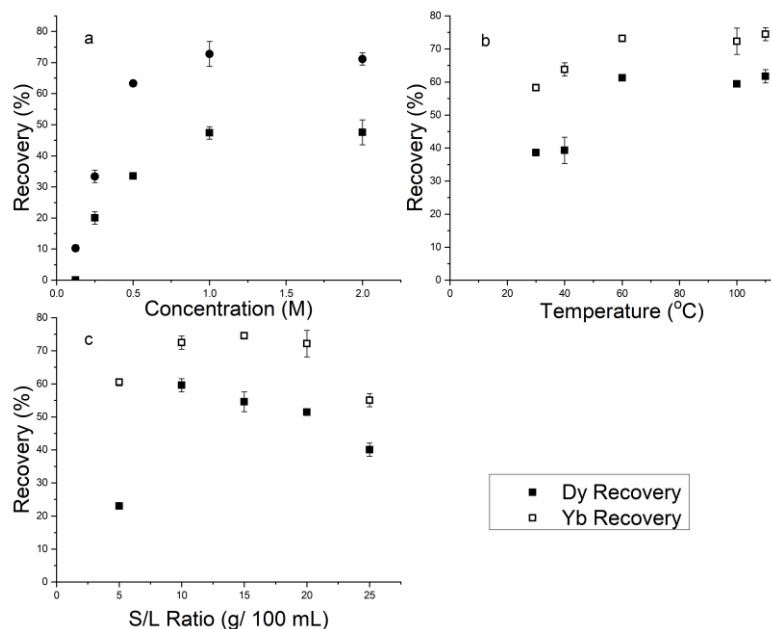
**Table 1** Thermodynamic parameter for leaching Dy(OH)<sub>3</sub> and Yb(OH)<sub>3</sub>

Dy(OH) <sub>3</sub> + 3HCl = DyCl <sub>3</sub> + H <sub>2</sub> O			Yb(OH) <sub>3</sub> + 3HCl = YbCl <sub>3</sub> + H <sub>2</sub> O		
T (°C)	ΔH (kcal)	ΔG (kcal)	T (°C)	ΔH (kcal)	ΔG (kcal)
30	-7.36	-5.81	30	-23.14	-21.78
40	-7.50	-5.76	40	-23.75	-21.72
50	-7.62	-5.70	50	-24.32	-21.65
60	-7.73	-5.64	60	-24.85	-21.56
70	-7.83	-5.58	70	-25.36	-21.45
80	-7.93	-5.51	80	-25.85	-21.33
90	-8.03	-5.44	90	-26.34	-21.20
100	-8.14	-5.37	100	-26.83	-21.05
110	-8.27	-5.29	110	-27.33	-20.89

The effect of temperature on the recovery of Dy and Yb is shown in Figure 2b. Increasing the temperature increased the recovery of Dy and Yb. The higher the temperature, the higher the kinetic energy of the reactants, causing the REE-hydroxide solid to collide and react faster with the HCl solution. The increase in recovery occurred in the temperature range of 30–60°C. Furthermore, there was no increase in the recovery of Dy and Yb. The increase in the leaching temperature did not increase the Dy and Yb recoveries. Temperatures above 90°C decrease REE leaching recovery by causing the solvent to evaporate, reducing the amount of solvent needed for the reaction. Additionally, high temperatures generate new issues in the reaction due to the solvent's boiling point. The exothermic nature of the REE-hydroxide leaching reaction with HCl is indicated by a negative value of enthalpy (ΔH) obtained from the database and a negative value of Gibbs free energy (ΔG), indicating the reaction can occur spontaneously. The low activation energy value (section 3.3) shows that the rate of heat generated during the reaction is fast.

Figure 2c indicates that an increase in recovery occurred at a solid-to-liquid (S/L) ratio of 10 g/100 mL. Subsequently, there was a decrease in the recovery of Dy. However, Yb recovery remained at 74.55% until the S/L ratio was 20 g/100 mL. Meanwhile, at an S/L ratio of 10 g/100 mL, Dy recovery reached 59.56% and then slowly decreased as the S/L ratio increased. Yb recovery is higher than Dy recovery at the same operating condition. This is because the solubility of Yb is higher than the solubility of Dy. In general, the solubility increases with increasing atomic number (Kim *et al.*, 2018). The decrease in recovery occurs because the limit for REEs solubility has been reached, but other major elements continue to dissolve. The increase in the S/L ratio will lead to an increase in the density of the solution, which will then decrease the mobility of the Cl<sup>-</sup> ion. Therefore, it will be slowing down the rate of REE<sup>3+</sup> transfer into the solution body. Thus, the product contains higher concentrations of major elements such as P, Zr, Si, and Al at a higher S/L ratio (Dupont and Binnemans, 2015). Therefore, an S/L ratio of 10g/100 mL was chosen as the optimum S/L ratio.

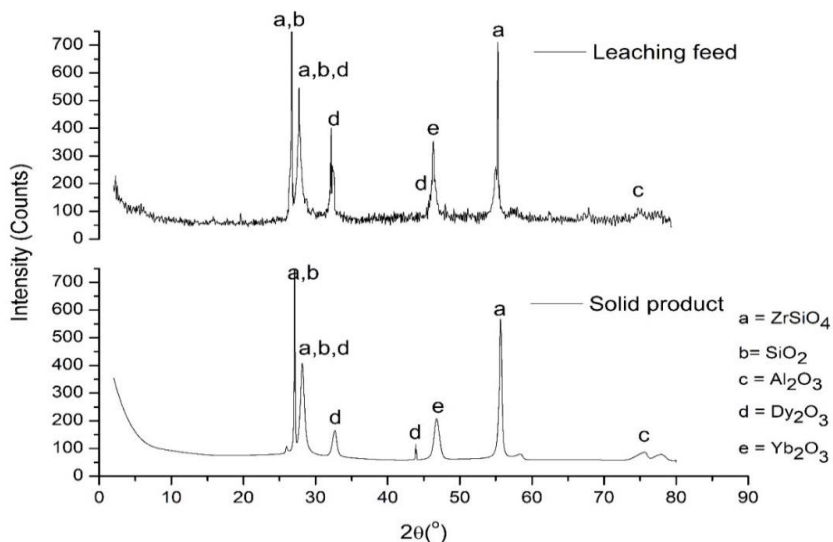




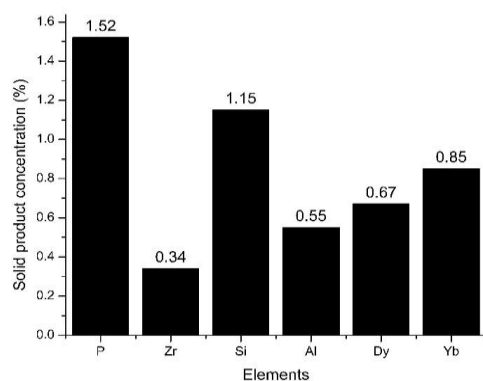
**Figure 2** Effect of leaching operating conditions on Dy and Yb recoveries: (a) effect of HCl concentration (80°C, S/L=10 g/100 mL, 150 rpm, and 60 min); (b) effect of temperature (2 M HCl, S/L=10 g/100 mL, 150 rpm, and 60 min); and (c) effect of S/L ratio (2 M HCl, 80°C, 150 rpm, and 60 min)

The components in the leaching residues were analyzed using XRD to determine the components contained. The results of the XRD analysis are presented in Figure 3. ZrSiO<sub>4</sub> and SiO<sub>2</sub> represent the initial content of the tailings. The REE concentration is not significantly different from that of the undesirable elements. These data accurately indicate that most of the REEs are in the dissolved form in REE-Cl<sub>3</sub>.

Elemental analysis was performed on solid product samples (residual leaching) under optimum operating conditions. Figure 4 shows the concentrations for some major impurity elements and HREEs (Dy and Yb) in the solid product. Major elements also appear to be much dissolved in the leaching process under optimum leaching conditions because the solubility of P, Zr, Si, and Al in HCl is higher than Dy and Yb.



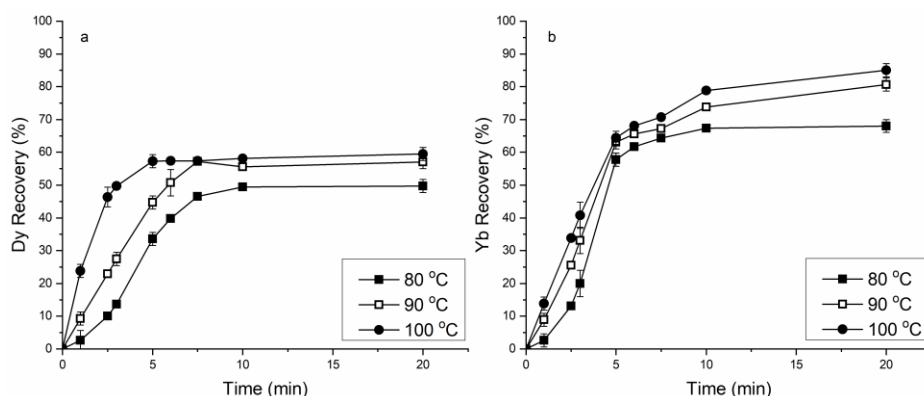
**Figure 3** XRD analysis of leaching feed and residue (solid product) at optimum operating conditions (1 M HCl, 60°C, 10 g/100 mL, and 60 min)



**Figure 4** Elemental analysis on the solid product at optimum operating conditions (1 M HCl, 60°C, 10 g/100 mL, and 60 min)

### 3.3. Kinetics analysis of Dy and Yb leaching process

Kinetic analysis was performed for the Dy and Yb leaching processes at a temperature of 80–100°C. A shrinking-core model was used to describe the leaching process. The limiting step or control in this leaching kinetic model describes the most dominant process that occurs. Chemical reaction control, ash layer diffusion control, and ZLT kinetics models evaluated the REEs leaching recovery. The kinetics analysis was performed for a leaching duration of 0–7.5 minutes because there was no significant recovery increase above 7.5 minutes. Figures 5a and 5b show that the recovery of Dy and Yb constantly increased up to 7.5 min and then reached a plateau with a longer leaching duration.



**Figure 5** Dy (a) and Yb (b) Recovery (2 M HCl, S/L = 10 g/100 mL, and 150 rpm)

The selection of the most suitable kinetic model was based on the highest R-square value. In leaching Dy from solid REE-hydroxide using an HCl solution, the highest R-square value was found in the chemical reaction limiting step kinetics model (Table 2). As much as 0.97485, 0.99851, and 0.90976 at 80°C, 90°C, and 100°C, respectively. In the kinetics analysis using the chemical reaction limiting step model, the value of the rate constant ( $k_c$ ) for the Dy leaching process was 0.024 min<sup>-1</sup>, 0.034 min<sup>-1</sup>, and 0.043 min<sup>-1</sup> for 80, 90, and 100 °C, respectively.

For Yb leaching, the kinetics model that showed the highest R-square value was the chemical reaction limiting step kinetics model. The R-squared values for 80°C, 90°C, and 100°C were 0.94964, 0.97875, and 0.98763, respectively. The rate constant ( $k_c$ ) values were 0.040 min<sup>-1</sup>, 0.046 min<sup>-1</sup>, and 0.050 min<sup>-1</sup> for 80, 90, and 100 °C.

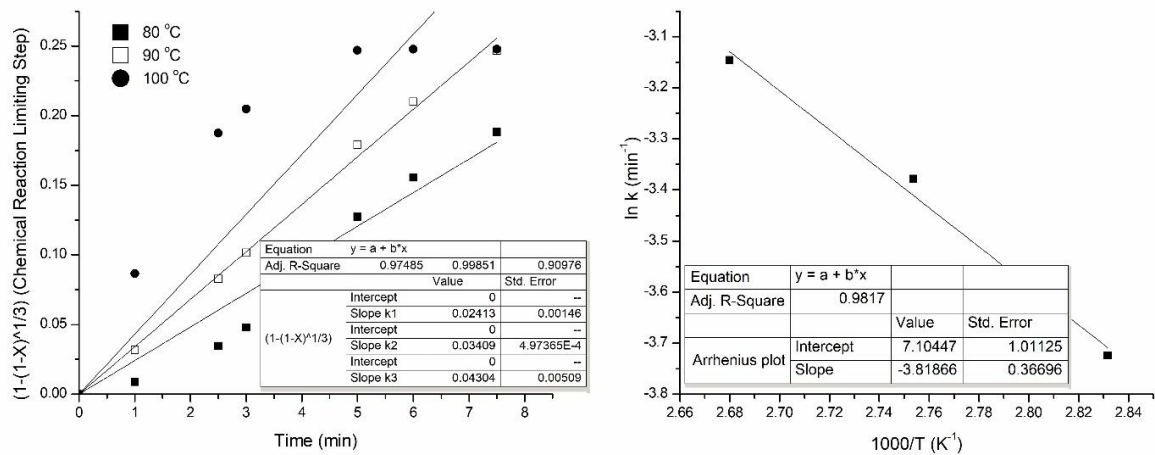
Stopic and Friedrich (2016) have also reported that the chemical reaction limiting step kinetics model is more suitable than the diffusion-based kinetics model for the HREE (yttrium) leaching process using an HCl solution. Kim *et al.* (2014) found that REEs leaching recovery followed a chemical reaction limiting step kinetics model in the first 20 minutes,

likely due to the temperature's significant effect. Linearisation was satisfactory at all temperatures studied (60, 80, and 100°C), with a straight line starting from the origin at each temperature (intercept = 0).

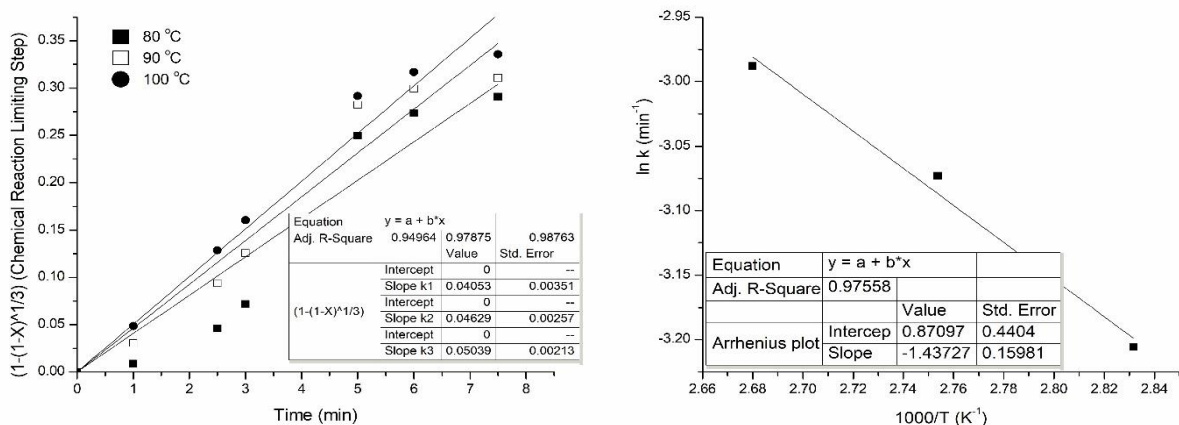
The leaching kinetic parameters  $D_y$  and  $Y_b$ , such as the activation energy ( $E_a$ ), were determined graphically using the Arrhenius plot in Figures 6 and 7. The slope value of the Arrhenius graph is the value of  $-E_a/R$ , where  $R$  is the gas constant ( $R = 8.314472 \text{ J}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}$ ). The activation energy values of leaching  $D_y$  and  $Y_b$  were 31.68 kJ/mol and 11.95 kJ/mol, respectively.

**Table 2** Selection of the leaching kinetic model listed

		Chemical reaction limiting step			Ash layer diffusion limiting step			ZLT		
		80°C	90°C	100°C	80°C	90°C	100°C	80°C	90°C	100°C
Dy leaching	$R^2$	0.9749	0.9985	0.9098	0.8844	0.9511	0.9501	0.8513	0.9053	0.9646
	k	0.0241	0.0341	0.043	0.01	0.0179	0.0256	0.0054	0.0114	0.0175
Yb leaching	$R^2$	0.9496	0.9788	0.9876	0.8873	0.9292	0.9633	0.8748	0.9028	0.9291
	k	0.0405	0.0463	0.0504	0.026	0.0313	0.0356	0.0202	0.0261	0.0316



**Figure 6** Kinetics analysis on  $D_y$  leaching, chemical reaction limiting kinetic model vs time (left) and Arrhenius plot using  $k$  data on chemical reaction limiting kinetic model (right)



**Figure 7** Kinetics analysis on  $Y_b$  leaching, chemical reaction limiting kinetic model vs time (left) and Arrhenius plot using  $k$  data on chemical reaction limiting kinetic model (right)

The activation energy value of  $D_y$  is greater than that of  $Y_b$ , indicating that  $D_y$  is more difficult to dissolve (Meshram, Pandey, and Mankhand, 2016). Activation energy is the



minimum energy required for a reaction to occur. When molecules collide, kinetic energy can stretch and break the bonds that cause reactions to occur.

The higher the activation energy, the stronger the bond between molecules, the more complex the reaction, and the smaller the recovery obtained.

#### 4. Conclusions

Several operating conditions such as HCl concentration, leaching temperature, and the S/L ratio of the leaching process were observed. Leaching kinetics were also carried out to obtain the kinetic parameters for the Dy and Yb leaching processes from REE-hydroxide. The optimal conditions for leaching occurred at 1 M, 60°C, and 10 g/100 mL HCl with a fixed process condition of 150 rpm and a leaching duration of 60 min. The Dy and Yb recoveries were 61.71% and 74.55%, respectively, under these operating conditions. The leaching process was found to follow a chemical reaction as a limiting step. The apparent activation energy ( $E_a$ ) obtained from the kinetics model was 31.68 kJ/mol and 11.95 kJ/mol for Dy and Yb, respectively.

#### Acknowledgments

The authors are grateful to Universitas Gadjah Mada for supporting this research. This study was funded by RIIM (Riset dan Inovasi untuk Indonesia Maju), contract number B-807/II.7.5/FR/6/2022 and B-6952/III.10/KS.00.00/6/2022.

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