Kinetics Study of Yttrium Leaching from Zircon Tailings Using Sulfuric Acid

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Abstract: *From the analysis of zircon tailings using X-Ray Fluorescence (XRF), Yttrium is a rare earth element (REE) with the highest concentration compared to other REEs. The purpose of this study is to determine the best kinetic model for describing how sulfuric acid extracts Yttrium from zircon tailings. Leaching temperatures of 200, 250, and 300 °C were used to determine the kinetics. Samples were obtained at 0, 20, 40, 60, 80, 100, and 120 min for each temperature. This study discovered that the chemical reaction model's kinetics are the most closely related to those of the leaching process. The evaluation of the model utilizing the coefficient of determination (R^2) on the relationship between each model and time lends support to this conclusion. The activation energy (E_a) of the leaching process is determined by the Arrhenius plot between ln k and 1/T. In the Yttrium leaching procedure, the E_a value is 14.42 kJ/mol. The chemical reaction model was in charge of the leaching process, according to the E_a value. The premise of the chemical reaction model is that chemical reactions regulate the rate of the reaction.*

Keywords: *zircon tailings; leaching; rare earth elements; kinetics; Yttrium*

■ INTRODUCTION

Rare earth elements (REEs) are strategic materials that are hard to come by and have a wide range of applications. The usage of REE, either singly or in combination, is on the rise at the moment. This is due to the fact that REE has strong mechanical properties, a high melting point, and a sizable cross-section for neutron absorption. Because of this, REE is widely used in many technological domains [1]. Fifteen elements with atomic numbers ranging from 57 to 71 are referred to be REEs or lanthanides. Due to their comparable chemical and physical characteristics, two more elements, scandium and yttrium, are also regarded as belonging to the same group [2]. REEs are derived from monazite and xenotime, which are found in zircon tailings. Consequently, processing the tailings is necessary to increase their economic value [3].

As "essential" minerals for contemporary industrial uses, REE is currently in the limelight for raw materials policy. Numerous combinations of REEs are utilized in a variety of sectors, including phosphorus (Eu, Y, Nd, Tb, Er, Gd), metal alloys (La, Ce, Pr, Nd, Y), catalysts (La, Ce), and magnets, which all have a high demand (Nd, Pr, Dy, Sm). Prior to production, REE-containing minerals must undergo downstream processing and chemical separation. High-purity REE oxides can be purchased through this technique and sold to the manufacturing sector [4].

Known as industrial vitamins, REEsare a unique non-renewable resource. Some of its physicochemical characteristics have significant strategic implications and are widely applicable in the sectors of permanent magnet materials, petroleum, chemical, metallurgy, textile, and ceramics. The demand for REE is rising yearly in a number of nations due to the rapid development of numerous advanced and novel technologies [5]. The Department of Energy (DOE) has classified some of them as essential components for the clean energy economy. From 2015 to 2025, Ce and La were discovered to be near-critical, while five REEs, i.e., Dy, Tb, Eu, Nd, and Y, were revealed to be critical. China currently controls 95% of the global REE production market, making up the majority of the REE market globally. In 2017, there was no mining of REE in the United States, and from 2013 to 2016, China supplied 78% of imports. The Chinese government's export policies have a significant impact on the price volatility of REEs traded internationally [6]. Rare earth markets have recently been migrating away from older, primarily Light REE-using applications, like catalysts, and toward newer, Heavy REE-using applications, including permanent magnets used in wind turbines and hybrid/electric vehicles [7].

Yttrium is one of the most important REEs, used in a wide range of items like fluorescent lighting, cubic zirconia jewelry, fighter jet engines, industrial, medical, and graphic technology, as well as electronic parts for missile defense systems. The cathode ray tubes (CRTs) from televisions and computers are one of the most important secondary sources of yttrium. Even though LCD and LED panels have completely replaced outmoded equipment, there is still a tremendous amount of human waste that needs to be dealt with [8].

Yttrium is used extensively in a variety of fields, including metallurgy, electronics, ceramics, laser technology, and fluorescent materials. Yttrium (Y) and lanthanides always coexist in rare earth minerals due to their similar chemical characteristics, especially in the ion-adsorption type rare earth deposits [9]. One of the most significant REEs, Y, is also the second most common REE in the crust of the earth after cerium. Because it has the highest thermodynamic affinity for oxygen, Y has
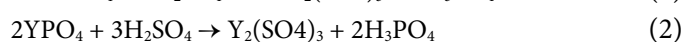
been employed in phosphors, ceramics, and metallurgy. Many nations and regions, including the United States, China, Japan, and the European Union, have designated Y as a critical metal [10].

Chemistry's field of "reaction kinetics" is concerned with the rate of reactions and the variables that influence that speed. The change in the concentration of the reactants or reaction products over a given period of time is used to express the reaction rate. An essential component of a chemical process that helps explain how the process works are the study of reaction kinetics. The method of managing the reaction rate, kinetic parameters (reaction rate constants or diffusion coefficients), activation energies, and equations or kinetic models can all be known from the findings of the study of reaction kinetics. The process scale-up steps can be carried out on an industrial scale based on the findings of kinetic studies. For constructing both chemical process but also for leaching processes, kinetic examinations of the reactions are crucial. The majority of leaching procedures include interactions between liquid and solid phases [11].

The pyrometallurgical and hydrometallurgical approaches are the two basic techniques for recycling REE. The hydrometallurgical techniques now employed to recover REE include dense medium centrifugation, flotation recovery, precipitation, selective Y dissolution by strong acidic and basic solutions, and solvent extraction. Because so many different metals are present, the pyro-metallurgical approach frequently takes a lot of energy, and it is difficult to isolate and recover specific metals. The pyrometallurgical recovery of Y includes feed preparation, blending and pelletizing, reduction, incineration, melting, and casting. In addition to oxidized REEs, volatile organic compounds (VOCs), furan gasses, and dioxins, which all complicate cleanup and have adverse effects on the environment, this multi-step process needs a lot of energy and necessitates a high temperature. In comparison to pyrometallurgical methods, hydrometallurgy operations are significantly more profitable because of their low operating costs, potential for leachate recovery, and decreased air pollution levels [12].

Leaching is one of the most effective methods in extractive metallurgy for removing REE from their ores by dissolving them in an appropriate leaching agent and then further processing them using both conventional and cutting-edge methods [13]. Leaching is frequently utilized in a variety of sectors, particularly the mining sector. The pretreatment of the solid particles to be extracted depends on the characteristics of the solvent used, the distribution of the solvent in the solid, and the nature of the solid particles [14]. The process of separating REE by the acid method can be carried out with sulfuric acid, nitric acid, and perchloric acid. However, sulfuric acid is commonly used because the price is relatively low, and the process is simpler than using nitric acid or perchloric acid [15].

Recovery of REE like Y from actual wastes has already been accomplished using hydrometallurgical techniques like acid leaching. For leaching agents, including sulfuric acid, nitric acid (HNO_3), and hydrochloric acid (HCl), different extraction efficiencies have been recorded (H_2SO_4). Hydrogen peroxide, cyanide, iodine, thiourea, halides, thiosulphate, and other substances are examples of additional leaching agents [16]. It was determined that the process of relocating Y into the solution was more effective in H_2SO_4 based on the data reported by authors in different media (HNO_3, HCl), so this leaching medium was chosen as the right one for this investigation [17]. Chemical reactions that occur during leaching using sulfuric acid [18]:

$$2REEPO_4 + 3H_2SO_4 \rightarrow REE_2(SO4)_3 + 2H_3PO_4 \qquad (1)$$
$$2YPO_4 + 3H_2SO_4 \rightarrow Y_2(SO_4)_3 + 2H_3PO_4 \qquad (2)$$

The sulfuric acid-based separation of monazite has a long history. The separation of REE and thorium from coastal sand monazite was first developed in the early to mid-1900s. In this study, the monazite in the reactor was stirred at a temperature of 200–245 °C, using a weight ratio of acid and concentrate varying from 1.5:1 to 3:1. From this study, it was concluded that a minimum temperature of about 200 °C is required for almost complete decomposition of monazite. While the minimum acid and concentrate ratio required is 1.6:1 [19].

Similar studies have been carried out in the research on the processing of Bangka monazite with the acid method by digestion using sulfuric acid. The dimensions of the monazite ore, the amount of sulfuric acid used, the temperature, the length of time, and the amount of water used for washing are among the factors considered. The results showed that the optimum digestion conditions were monazite ore size ~ 58 μm, the weight ratio of ore:sulfuric acid is 1:2.5, digestion temperature 190 °C, digestion time 3 h and washing consumption 8 times the weight of monazite feed with digested recovery U = 99.90%, Th = 99.44%, REE = 99.54% and PO_4 = 99.88% [18].

Research related to sulfuric acid digestion is research on the effect of digestion temperature. In this study, temperature variations were carried out between 200–800 °C. From this research, it can be concluded that digestion at 300 °C produced amorphous phosphate deposits, while the dissolved REEsincreased to 99% [15]. Research on the separation of REEs from zircon tailings has also been carried out. The initial stage of the research was the alkaline fusion process; the feed ratio of NaOH: zircon tailings is 1:1. The result of the alkaline fusion process is used as leaching feed using HCl. From these results, the optimum conditions for the alkaline fusion zircon tailings leaching process were obtained at a temperature of 60 °C, HCl concentration of 1 M, and a ratio of yield of the alkaline fusion: HCl is 1:10 for 7.5 min. Y recovery reached 87%, and the activation energy value (E_a) was 20.21 kJ/mol [20].

The solvent used in this study was sulfuric acid. Meanwhile, the reactor used in this study was an autoclave without an agitator, and this study was conducted without pretreatment. From the results of the study of reaction kinetics, the process of controlling the reaction rate, kinetic parameters, activation energies, and equations or kinetic models can be known. From the results of kinetic studies, the process scale-up stages can be carried out on an industrial scale.

■ EXPERIMENTAL SECTION

Materials

The materials used in this study include zircon tailings from PT. Monokem Surya, H_2SO_4 (95–97%) produced by Merck & Co., Inc., distilled water produced

by PSTA-BATAN kernel fabrication laboratory and filter paper.

Instrumentation

The instruments used in this study were a ball mill (planetary ball mill type, Changsha Tianchuang Powder Technology Co. Ltd.), furnace (vertical lift door type, produced by PT. Suhaterm), autoclave, laboratory glassware (Thermo scientific Heraeus), sieve, analytical balance, and X-Ray Fluorescence (NEX QC RIGAKU, S/N QC 1202).

Procedure

Determination of leaching kinetics

The determination of leaching kinetics was carried out by varying the leaching temperature with the variation of 200, 250, and 300 °C. At each temperature, samples were taken at a leaching process of 0, 20, 40, 60, 80, 100, and 120 min. The solvent used in the zircon tailings leaching process was sulfuric acid.

Analysis of leaching result

The leaching residue was analyzed using X-Ray Fluorescence to determine the levels of each element. Determining the percentage of recovery is carried out using the following equation:

$$\text{Re cov ery} = \frac{X_0 - X_i}{X_0} \times 100\% \tag{3}$$

with, X_0 = mass of leaching feed x concentration of element, % and X_i = mass of leaching residue x concentration of element, %.

Data interpretation

Determination of the fitting of the kinetic model to the experimental results (trend line) is done by comparing each kinetic model with the experimental data. The kinetic data will be plotted in the form of a graph of the relationship between the kinetic model and time to get the most appropriate model. The kinetic model obtained is then used to calculate the value of the constant k, as well as plots of ln k and 1/T, to get the E_a value.

Each kinetic model was compared with experimental data. After obtaining the constant value k, the value of time (t) is calculated again by dividing the value obtained from various models by the value of k. The

equation for finding t values for various models is presented in Table 1.

Furthermore, the calculated t value is compared with the t value at various recovery fractions (x) in the experimental results.

■ RESULTS AND DISCUSSION

Characterization of Zircon Tailings Composition

The raw material used in this research is zircon tailings from PT. Monokem Surya. The initial stage of this research is the drying process of zircon tailings which will later be used as research feed. Drying was carried out at 110 °C for 3 h. After the drying process, a filtering process is carried out to remove impurities. In the early stages of the study, analysis was also carried out using X-Ray Fluorescence (XRF). The concentration of each REE per total REEs concentration is presented in Fig. 1.

As shown in Fig. 1, it can be seen that the levels of each REE per total REEs level are presented; it can be seen that the highest concentration of metal is Y at 35%, followed by Ce metal at 27%, La metal at 12%, and Nd at 10%.

Based on the results of the characterization of zircon tailings using X-Ray Diffraction (XRD) can be seen that the xenotime content is 42.6%, where xenotime
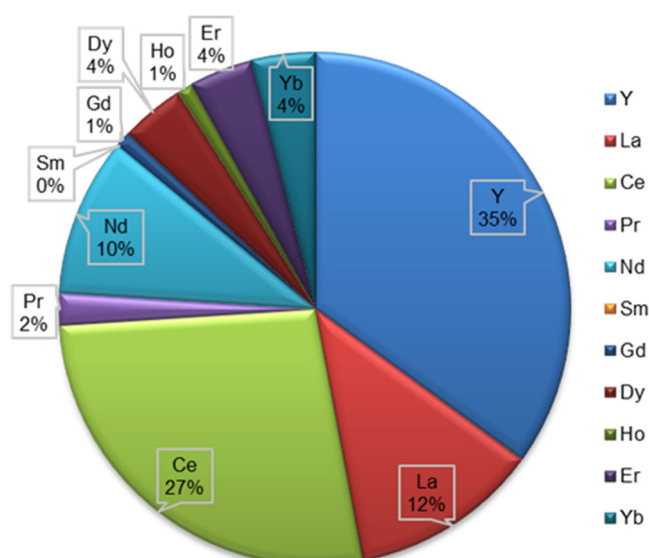

Fig 1. The concentration of each REE per total REEs concentration

Table 1. The equation for finding the t value for various models [21]

Model	Time, t
Chemical reaction control	$t = \dfrac{1 - (1-x)^{\frac{1}{3}}}{k_s}$
Ash diffusion control	$t = \dfrac{1 - 3(1-x)^{\frac{2}{3}} + 2(1-x)}{k_a}$
Zhuravlev, Lesokhin, and Templeman	$t = \dfrac{\left(\frac{1}{(1-x)^{\frac{1}{3}}} - 1\right)^2}{k_{sa}}$
Jander cylindrical	$t = \dfrac{\left(1 - (1-x)^{\frac{1}{2}}\right)^2}{k_{aj}}$
Jander 3D	$t = \dfrac{\left(1 - (1-x)^{\frac{1}{3}}\right)^2}{k_{ab}}$
Dickinson	$t = \dfrac{\left(\frac{1}{(1-x)^{\frac{1}{3}}}\right) - 1}{k_{ad}}$

Where t is time (min), x is the recovery fraction, k_s is the apparent rate constant for the chemical reaction control (min^{-1}), k_a is the apparent rate constant for the ash diffusion control (min^{-1}), k_{sa} is the apparent rate constant for the Zhuravlev-Lesokhin-Templeman control (min^{-1}), k_{aj} is the apparent rate constant for the Jander cylindrical control (min^{-1}), k_{ab} is the apparent rate constant for the Jander 3D control (min^{-1}), and k_{ad} is the apparent rate constant for the Dickinson control (min^{-1})

is a mineral that contains Y, and it was another mineral with the highest composition in the zircon tailings followed by monazite-Ce (34.2%), zircon (17%), anatase (4.3%), rutile (1.2%), and cerianite (0.7%) [20].

Leaching Kinetics of Yttrium from Zircon Tailings Using Sulfuric Acid

Determining the leaching kinetics was carried out by varying the leaching time to 0, 20, 40, 60, 80, 100, and 120 min. At each leaching time, the processes were carried out at 200, 250, and 300 °C. Leaching was carried out with a grain size of zircon tailings of 119 μm and a liquid/solid ratio of 1 mL/g for each temperature variation (grain size of zircon tailings and liquid/solid ratio obtained from the optimization results in previous studies). The results of the leaching of REEs at each temperature can be seen in Fig. 2.

As shown in Fig. 2, it can be seen that for temperatures of 300 °C, the recovery trend obtained increased drastically from 0 to 60 min, then after reaching 60 min the recovery slowly increased. For temperatures of 250 and 200 °C, the recovery trend obtained increased

drastically from 80 to 100 min. From the recovery trend of the three temperatures, it can be seen that temperature greatly influences the time needed to achieve a significant increase in recovery. Increasing temperature, the value of the reaction rate constant will increase, and the reaction rate will be faster because the reaction rate constant is very dependent on temperature. The data obtained from the study were then used for kinetics evaluation.

In this study, an evaluation of the leaching kinetics of REEs was carried out using the developed model to predict the leaching process of rare earth elements from zircon tailings. Several models were used to evaluate the leaching process are Chemical reaction model, the Ash diffusion model, the Zhuravlev-Lesokhin-Templeman model (Z-L-T), the Jander Cylindrical model, the Dickinson model, and the Jander 3D models. The leaching kinetics models are often used to describe the leaching process in the extraction of elements from ores.

Each kinetic model was compared with experimental data. After obtaining the constant value k,
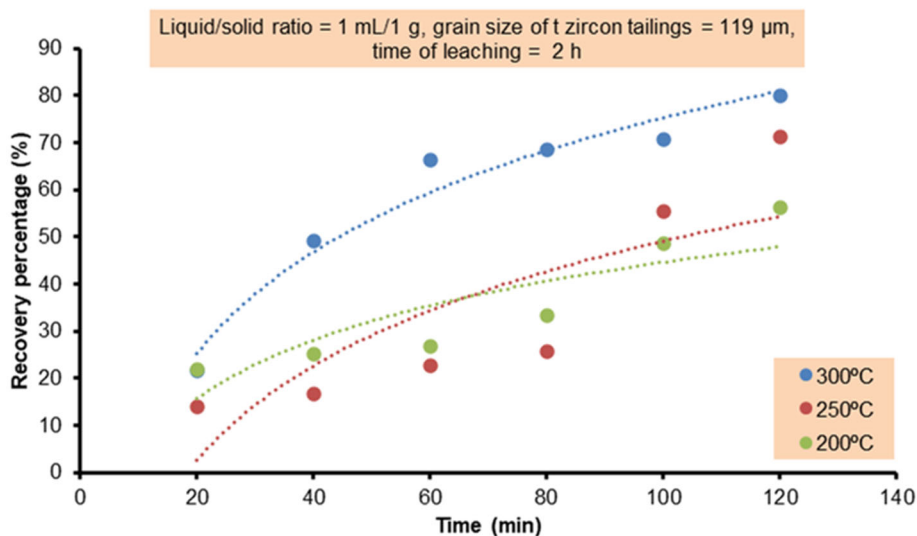
Fig 2. Percent recovery plot with time at 300, 250, and 200 °C for Yttrium

the value of time (t) is calculated again by dividing the value obtained from various models by the value of k. Furthermore, the calculated t value is compared with the t value at various x in the experimental results. A comparison of the accuracy of the use of the model in this study can be seen in Fig. 3.

This study discovered that the chemical reaction model is the kinetic model that comes the closest to the kinetics of the leaching process, as illustrated in Fig. 3. The examination of the model utilizing the R^2 value on the relationship between each model over time lends support to this. The R^2 value for each kinetic model
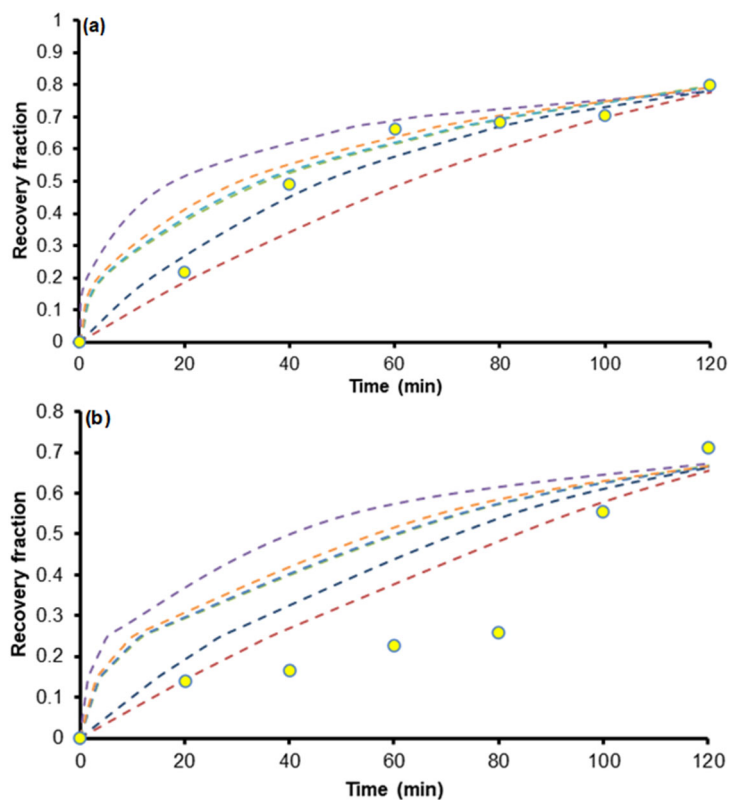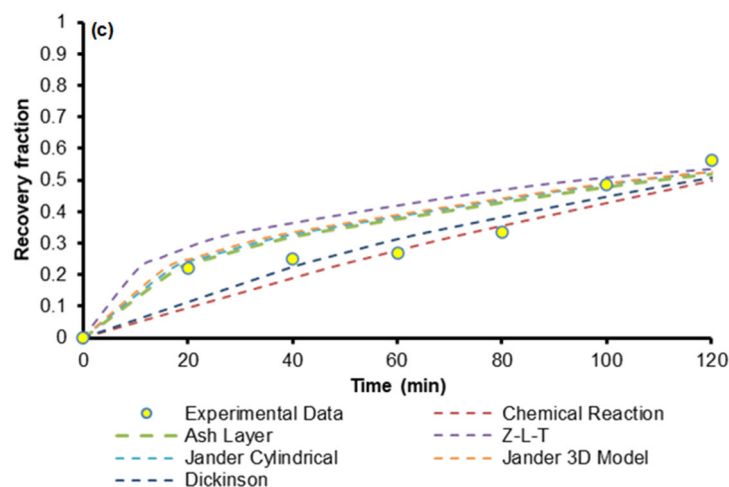
Fig 3. Recovery fraction plot with time for fitting the kinetic model at (a) 300, (b) 250, and (c) 200 °C for Yttrium

Table 2. R^2 value of various research models

Kinetic Model	Temperature (°C)	R^2
Chemical Reaction	300	0.9412
	250	0.8234
	200	0.7964
Ash Layer Diffusion	300	0.9352
	250	0.6814
	200	0.6939
Zhuravlev, Lesokhin, and Templeman	300	0.8638
	250	0.5952
	200	0.6155
Jander 3D	300	0.9298
	250	0.6599
	200	0.6745
Jander Cylindrical	300	0.9345
	250	0.6756
	200	0.6887
Dickinson	300	0.9157
	250	0.7655
	200	0.7731

employed in the study is shown in Table 2.

As shown in Table 2, it can be seen that the appropriate kinetic model is the chemical reaction model because it has the highest coefficient of determination (R^2) when compared to other kinetic models. The chemical reaction model assumes that chemical reactions control the rate of the reaction.

Based on the results of the leaching process approach with the models used, the most suitable model can be used to calculate E_a value. The plot between models

$(1 - (1 - x)^{1/3}$ and time in Fig. 4 was used to find the value of the apparent rate constant for the chemical reaction control (k_s), as the slope from the curve of model and time. From the calculation results obtained, the value of k_s at each temperature of 0.0038 (300 °C), 0.0022 (250 °C), and 0.0019 (200 °C). The equations are as follows:

Chemical reaction control:

$$k_s.t = 1 - (1 - x)^{1/3} \tag{4}$$

where t is time (min), x is the recovery fraction, and k_s is the apparent rate constant for the chemical reaction control (min^{-1}).

The E_a was determined by using Arrhenius plots:

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

$$k_s = \ln A - \frac{E_a}{RT} \qquad (6)$$

$$E_a = -R * \left(\text{slope from the curve of } \ln k_s \text{ vs } \frac{1}{T} \right) \qquad (7)$$

where E_a is the activation energy (J/mol), R = 8.314462 (J/(mol.K)), k_s is the apparent rate constant for the chemical reaction control (min^{-1}), A is the frequency factor, and T is the leaching temperature (K).

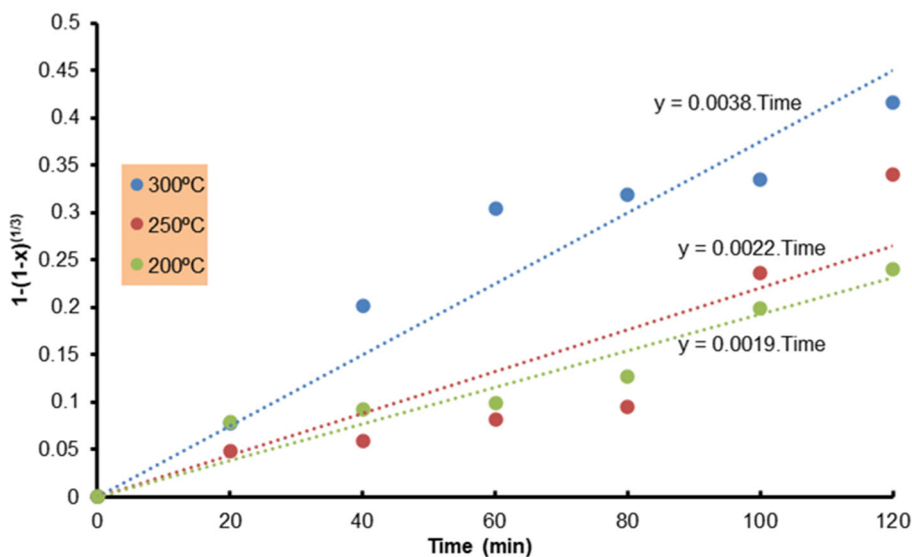The Arrhenius plot in Fig. 5 was used to find the E_a of the leaching process. The E_a value for the leaching of

Fig 4. Recovery fraction of suitable model plot with time at 300, 250, and 200 °C for Yttrium
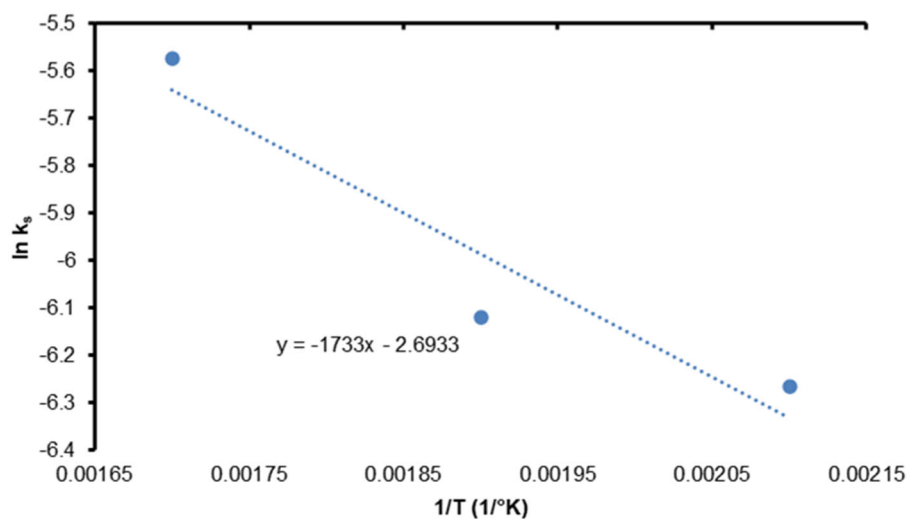

Fig 5. Arrhenius plot

Table 3. The value of E_a from previous research

Researcher	E_a, kJ/mol	Sample
[2]	26.95	Concentrate after roasting treatment
[20]	20.21	Alkaline fusion treatment results

REEsfrom zircon tailings using sulfuric acid was 14.42 kJ/mol. The value of E_a from previous research is presented in Table 3.

The chemical reaction will govern the leaching process if the E_a value is less than 20 kJ/mol, whereas diffusion through the ash layer will control it if the E_a value is greater than 40 kJ/mol [22]. From the results of the study, if it is seen from the obtained E_a value, the model that is suitable for the leaching process of Yttrium from zircon tailings using sulfuric acid is the chemical reaction model.

■ CONCLUSION

According to the results of fitting the kinetic model, the chemical reaction model that governs is comparable to the sulfuric acid-assisted Yttrium leaching process from zircon tailings. The evaluation of the model based on the value of the coefficient of determination (R^2) on the correlation between each model and time also supports this. Sulfuric acid's E_a value was 14.42 kJ/mol for the leaching of Yttrium from zircon tailings. The E_a value demonstrated that the leaching process was within the control of the chemical reaction model.

■ ACKNOWLEDGMENTS

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