Sains Malaysiana 54(10)(2025): 2509-2523
http://doi.org/10.17576/jsm-2025-5410-14

Solid-Liquid Extraction of Rare Earth Elements from Indonesian Wacopek Bauxite Tailings by the Sulfuric Acid Leaching and Precipitation Methods

(Pengekstrakan Pepejal-Cecair Unsur Nadir Bumi daripada Amang Bauksit Wacopek Indonesia dengan Kaedah Larut Lesap Asid Sulfurik dan Pemendakan)

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Received: 6 October 2024/Accepted: 11 August 2025

ABSTRACT

The high demand of rare earth elements (REEs) and geopolitical issues affecting their supply. Thus, a number of research have been conducted to find some secondary resources for producing of REEs. The work is aimed to extract of total REEs (TREEs) from Indonesian Wacopek bauxite tailing by solid-liquid extraction using sulfuric acid with concentrations being 1.5 to 5 M and the precipitation methods. Leaching and enrichment of total REEs from the bauxite tailings employed sulfuric acid as the solvent, where precipitation occurred in two stages using sodium sulfate and sodium phosphate as well as leaching kinetic have been investigated. The final extracted REEs are in the forms of their hydroxides. The experimental was conducted in the batch system by varying leaching time (5 to 60 min) and temperature (25 to 60 °C). Metal separation from the leachate was carried out by a solid-liquid extraction in H_2SO_4 at a liquid/solid (L/S) ratio of 20 mL/g, according to the optimal operating temperature at 60 °C, leaching time of 30 min, concentration of H_2SO_4 of 3 M, leachate volume of 500 mL, and L/S ratio of 20 mL/g. In the total REEs precipitation process with subsequent NH_4OH-sodium sulfate and NH_4OH-sodium phosphate precipitant system yielded 0.23 g and 9.8 g of REEs-sulfate and REEs-phosphate, respectively. The final REE-hydroxides gave the maximum total REEs yield as high as 90.75%. Leaching kinetics study was described by the shrinking core model for unchanged spherical particle sizes, and acid leaching occurred in two steps that governed by chemical reaction and diffusion process. The activation energy in the first chemical process was characterized to be 10.1 kJ/mol and the second diffusion process was 15.5 kJ/mol. In this work, the solid-liquid extraction process used sulfuric acid to bind to the TREEs contained in the Wacopek dried bauxite tailing potential for further separation of TREEs.

Keywords: Batch system; bauxite tailing; solid-liquid extraction; sulfuric acid leaching; total rare earth elements

ABSTRAK

Permintaan yang tinggi untuk unsur nadir bumi (REE) dan isu geopolitik telah menjejaskan bekalannya, justeru, banyak penyelidikan telah dijalankan untuk mencari beberapa sumber sekunder untuk menghasilkan REE. Penyelidikan ini bertujuan untuk mengekstrak jumlah REE (TREE) daripada amang bauksit Wacopek Indonesia melalui pengekstrakan pepejal-cecair menggunakan asid sulfurik dengan kepekatan 1.5 hingga 5 M dan kaedah pemendakan. Larut resap dan pengayaan jumlah REE daripada amang bauksit telah menggunakan asid sulfurik sebagai pelarut dengan pemendakan berlaku dalam dua peringkat menggunakan natrium sulfat dan natrium fosfat serta penyelidikan kinetik larut lesap telah dikaji. Ekstrak akhir REE adalah dalam bentuk hidroksida. Uji kaji telah dijalankan dalam sistem kelompok dengan masa larut lesap (5 hingga 60 minit) dan suhu (25 hingga 60 °C) yang berbeza-beza. Pengasingan logam daripada larut lesap dilakukan dengan pengekstrakan pepejal-cecair dalam H_2SO_4 pada nisbah cecair/pepejal (L/S) 20 mL/g, mengikut suhu operasi optimum pada 60 °C, masa larut lesap 30 minit, kepekatan H_2SO_4 3 M, isi padu larut resap 500 mL/S dan nisbah L/S 20 mL/g. Dalam proses pemendakan jumlah REE dengan sistem pemendakan NH_4OH-natrium sulfat dan NH_4OH-natrium fosfat seterusnya menghasilkan 0.23 g dan 9.8 g sulfat REE dan fosfat REE. Hidroksida REE akhir memberikan hasil maksimum jumlah REE sehingga 90.75%. Kajian kinetik larut lesap telah diterangkan oleh model teras mengecut untuk saiz zarah sfera yang tidak berubah dan asid larut lesap berlaku dalam dua langkah yang dikawal oleh tindak balas kimia dan proses penyebaran. Tenaga pengaktifan dalam proses kimia pertama dicirikan sebagai 10.1 kJ/mol dan proses penyebaran kedua ialah 15.5 kJ/mol. Dalam kertas ini, proses pengekstrakan pepejal-cecair menggunakan asid sulfurik untuk mengikat kepada TREE yang terkandung dalam amang bauksit kering Wacopek dan berpotensi untuk pemisahan selanjutnya bagi TREE.

Kata kunci: Amang bauksit; jumlah unsur nadir bumi; larut lesap asid sulfurik; pengekstrakan pepejal-cecair; sistem kelompok

INTRODUCTION

According to European and American agencies, rare earth elements (REEs) are among the most critical elements (Couturier et al. 2024). REEs are essential for modern technologies, including defense, energy (such as wind power, electric vehicles, low-energy lighting, and hybrid vehicles) (Schreiber et al. 2021), as well as the medical and economic sectors. These elements play a vital role in the transition from the fossil fuel era to a decarbonized future. The lanthanide family, which includes scandium (Sc), yttrium (Y), and the elements from lanthanum (La) to lutetium (Lu) with atomic numbers 21, 39, and 57-71, respectively, possess unique optical, electronic, and electromagnetic properties. These characteristics make them highly valuable for a wide range of applications in electronics, manufacturing, medicine, renewable energy, and other advanced technologies, such as optical devices, catalysts in petroleum refining, synthetic products, medical imaging, lamps, lasers, magnets, projectors, and X-ray intensifying screens (Guyonnet et al. 2015). In addition to REEs, other critical minerals such as lithium (Li), cobalt (Co), nickel (Ni), silicon (Si), graphite, and zinc (Zn) are projected to have strong market growth through 2050, driven by the demand for low-carbon energy technologies and net-zero emission targets (Kusrini et al. 2024). REEs and these critical minerals, particularly in the form of their metal oxides, are key industrial materials used in sectors such as catalysis, magnetic materials, and biomedicine. The annual demand for these metal oxides was generally predicted to grow by more than 5% starting in 2020 (Zhou et al. 2017).

Notably, China is the leading producer of REEs and holds significant control over nearly the entire global REE value chain, making the global supply highly vulnerable. Due to these economic and geopolitical dynamics, the primary extraction and production of REEs have often overlooked environmental and social concerns, particularly those related to high energy consumption and substantial waste generation (Couturier et al. 2024). The REE sources are mostly monazite, bastnasite, and xenotime ores (Demol et al. 2019). The development of recycling and recovery of REEs from minor or secondary resources and/or industrial wastes has become increasingly important (Kusrini et al. 2024; Rizos, Righetti & Kassab 2024; Vuppaladadiyam et al. 2024; Xiao et al. 2025). Exploration of different potential REE sources, such as mining wastes, bauxite tailings was found to contain traces of REEs (mainly La and Y) (Kusrini et al. 2024). Various methods have therefore been applied to separate and enrich the content of REEs using precipitation, ion-exchange, and liquid-liquid and solid-liquid extraction. Liquid-liquid extraction using an N-pivot tripodal ligand (Mahanty et al. 2018) and adsorption using activated carbon from banana peels (Kusrini et al. 2018a), dried powder from durian peels (Kusrini et al. 2019a), and pectin as adsorbents (Kusrini et al. 2020a, 2018b) offer green methods recovering REEs. Similarly, the recovery of REEs from contaminated waters by living seaweed (*Gracilaria gracilis*) has also been reported (Jacinto et al. 2018).

Since the high demand of REEs and geopolitical issues may affect their supply, many efforts have been devoted to find some secondary resources for producing REEs. The promising approaches include recycling REEs from manufacturing by-products, discarded consumer goods, and extraction of REEs from industrial waste (Binnemans et al. 2013; Cunningham, Etherington-Rivas

& Azimi 2024). Recycling of industrial wastes has also been investigated to reduce the environmental impact of conventional mining and to increase the economic value of REEs (Rizos, Righetti & Kassab 2024; Vuppaladadiyam et al. 2024; Xiao et al. 2025). REEs are commonly found in a mixture of some elements with other minerals in ores. The separation of individual REEs from the minerals is difficult because of their similar chemical properties and small variation in their atomic number (Jacinto et al. 2018). The sources of REEs are scarce in many countries, and separation of REEs from secondary resources such as bauxite tailings has not been studied extensively.

In Bintan Island, Indonesia, the existence of bauxite as residual and abandoned tailings is abundant, where about 1.292 million tons of bauxite tailings are present as deposit on soil surfaces (Kusrini et al. 2020b). This has become environmental issues, as the presence of the bauxite tailings in the aquatic water environments are hazardous to aquatic life and ecosystems (Evans 2016). Although bauxite tailings are rich in REEs (Borra et al. 2016), currently, the bauxite tailings are applied in limited scales, especially for minor use in cements and ceramics.

Solid-liquid extraction using sulfuric acid was selected for separation of total REEs from many resources such as monazite, bastnasite, xenotime, and other less common REES including loparite (Demol, Ho & Senanayake 2018), apatite (Battsengel et al. 2018), tailing bauxite (Kusrini et al. 2020b, 2020e) and Belitung silica sand (Kusrini et al. 2020d), sand (silicate) particles (Poscher et al. 2014). However, the kinetic studies related the REE extraction from bauxite tailings have not been yet reported and investigated in details. Borra et al. (2016) reported that increasing the concentration of acid, the leaching yield of REEs increases, as the greater acid concentrations enhances the dissolution of Fe into the leachate solution.

Sulfuric acid and other sulfate-based solutions are primarily selected due to their cost-effectiveness and widespread availability as a mineral acid reagent (Cunningham, Etherington-Rivas & Azimi 2024). Poscher et al. (2014) also investigated the use of different types of strong acids, including HNO_3, HCl, and H_2SO_4, to extract REEs from sand (silicate) particles with the highest level of extraction for Ce (99%) and La (98%). The chemical composition of bauxite tailings is complex, containing mainly metal oxides of Al, Fe, and Ti and minor amounts of REEs (Kusrini et al. 2020e). The metal composition often depends on the origin and source of the mineral ores, including the operational conditions for the production of $Al(OH)_3$ from bauxite minerals (Rivera et al. 2019).

This study presents the solid-liquid extraction of total REEs, including La, Ce, Nd, Sm, and Y from Indonesian Wacopek bauxite tailings with the subsequent sulfuric acid leaching and precipitation methods. In this study, a batch system reactor is used for the extraction of La, Ce, Nd, Sm, and Y, while the precipitation reactions using sodium sulfate and sodium phosphate were employed to obtain the solid concentrated REE-hydroxides. The role of sulfuric acid concentration, leaching temperature, and leaching time were also studied to optimize the extraction of total REEs from the bauxite tailings.

EXPERIMENTAL DETAILS

MATERIAL

The bauxite tailings used in this work were obtained from Wacopek, Bintan Island, Indonesia. Powdered Na_2SO_4 (97%) and Na_2HPO_4 (99.5%) were purchased from Sigma Aldrich (USA). Other chemical reagents, such as sulfuric acid (H_2SO_4, 97%), sodium hydroxide (NaOH) pellets, and ammonium hydroxide (NH_4OH) of analytical grade were purchased from Merck (Germany).

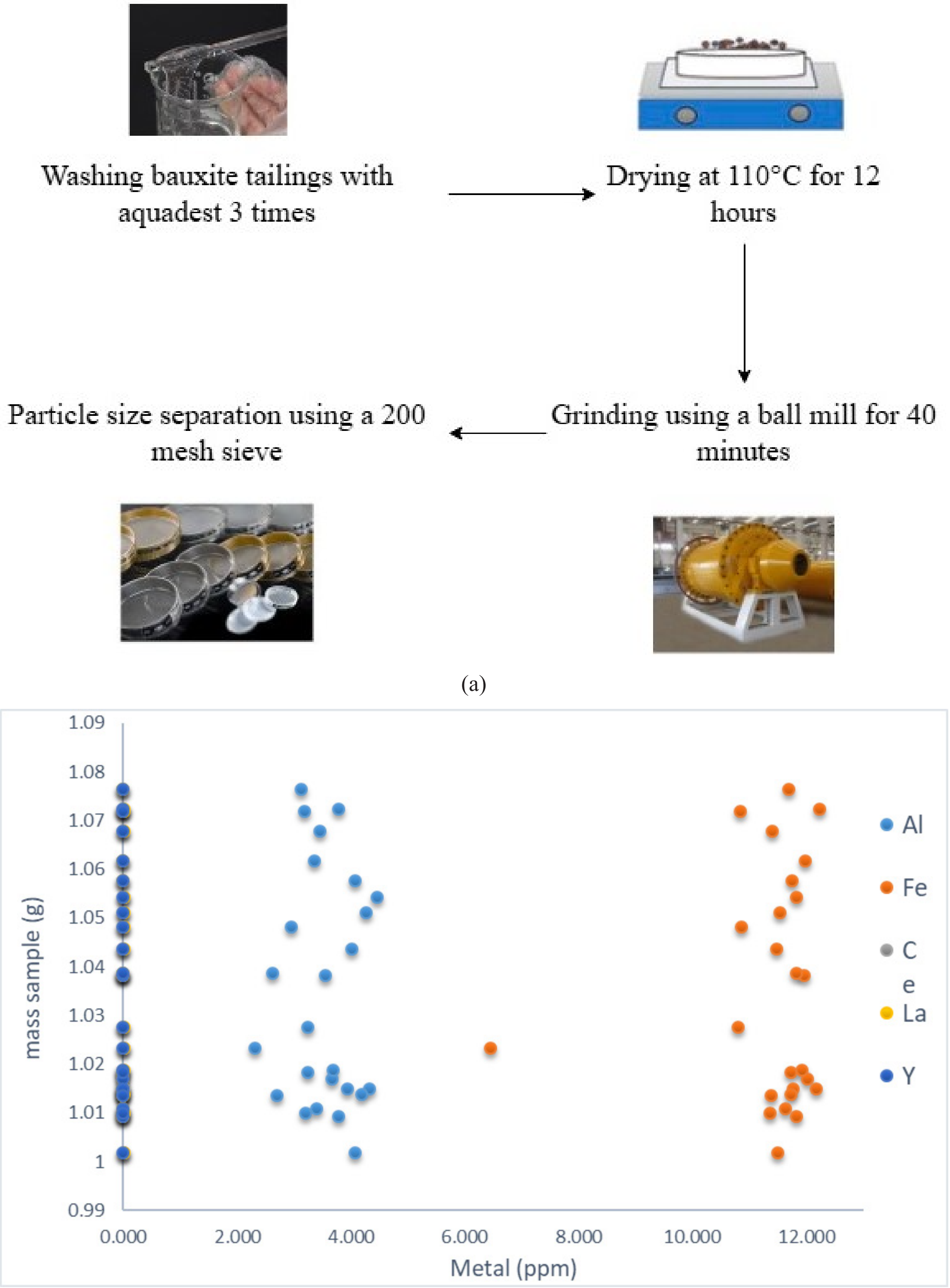
PRETREATMENT OF BAUXITE TAILINGS

Pretreatment of bauxite tailings involved triple washing with distilled water, followed by drying at 110 °C for 12 h. The dried tailings were ground using a ball-mill for 40 min and then filtered to obtain particles with 200 mesh size. The pretreatment process is presented in Figure 1(a). The metal concentrations of Al, Fe, Ce, La, and Y were detected in the bauxite tailing using ICP-OES (Figure 1(b)).

SOLID-LIQUID EXTRACTION USING SULFURIC ACID

In the leaching experiments, the effects of sulfuric acid concentration, leaching temperature, and leaching time on the leaching efficiency of REEs were studied. In all experiments, the mixture was stirred using a magnetic stirrer. Leaching experiments were carried out at different intervals of time between 0 and 60 min to study the effect of leaching time on the extraction of REEs from the bauxite tailings. The liquid-solid (L/S) ratio was fixed at 20:1 mL/g. The pH and volume of the liquid after the leaching process were measured.

The H_2SO_4 solution was diluted to various concentrations (1.5, 2. 3, 4, and 5 M) with the final volume of H_2SO_4 being 500 mL. Into each beaker containing 500 mL of sulfuric acid with the various concentrations was added 25 g dried bauxite tailings. The reaction system occurred in the batch reactor which was continuously stirred at 250 rpm with a magnetic stirrer. The mixture was kept at a desired temperature, either 25, 40, or 60 °C. The liquid was sampled from the solid-liquid system using Whatman paper No. 40 at various leaching times (5, 10, 15, 20, 30, and 60 min) for the kinetic study. The solid-liquid extraction is illustrated in Figure 2(a). The leachate from all samples were characterized by Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) to estimate metal concentrations (in ppm or mg/L), REE extraction yield, and the sample weight content (%). The ICP-OES results enabled estimation of the weight (%) using Equation (1) as follows;



(a)

(b)

FIGURE 1. (a) pretreatment of Wacopek tailing bauxite, and (b) The metal concentrations of Al, Fe, Ce, La, and Y were detected in the bauxite tailing using ICP-OES

$$(AxV/(\frac{M}{1000\frac{g}{kg}}))\ x\ D)\ /10000 = Wt\%\ TREEs \qquad (1)$$

where A is the concentration of REEs in samples obtained from ICP-OES measurements (in mg/L); V is volume of solution produced (in L); M is sample weight of solids from the leaching process (in g); and D is dilution coefficient. The REE extraction yield from each condition of the acid leaching was calculated using Equation (2);

$$Yield\ REEs\ (\%) = \frac{Wt\%_{Leach}}{Wt\%_{Solid}}\ x\ 100\% \qquad (2)$$

According to Equation (2), the yield of REEs can be simply estimated by;

$$Yield\ REEs\ (\%) = \frac{0.0047}{0.0051}\ x\ 100\% \qquad (3)$$

$$Yield\ REEs\ (\%) = 90.75\% \qquad (4)$$

KINETIC STUDY

Kinetic study of the solid-liquid extraction was divided into two parts, namely chemical reaction and diffusion reaction stages, as described by following Equations (5) - (7):
Chemical reaction;

$$k_1 t = 1 - (1 - X_B)^{1/3} \qquad (5)$$

Diffussion reaction stage;

$$1 - 3(1 - X_B)^{\frac{2}{3}} + 2(1 - X_B) \qquad (6)$$

$$X_B = \frac{Concentration\ of\ REEs\ extracted\ in\ leachate}{Initial\ concentration\ of\ REEs} \qquad (7)$$

where k_1 is reaction rate constant at the chemical reaction stage and X_B is the fraction of converted rare earth elements in Wacopek bauxite tailing.

PRECIPITATION REACTION

Further experiments employed the best operating condition for leaching of TREEs in batch system at temperature of 60 °C and leaching time of 30 min. The volume of leachate of 300 mL was sampled and then the pH of leachate was adjusted to pH 3 using NH_4OH under constant stirring at 500 rpm at room temperature. Then, Na_2SO_4 was added into the leachate REE-sulfate resulting from solid-liquid extraction at a mole ratio of 5:1 with heating at 80 °C for 1 h under stirring at 500 rpm. The first precipitate was filtered using a filter paper (Whatman No. 40), followed by drying at 100 °C for 12 h. The first precipitated was named as P1. The process of precipitation reaction is illustrated in Figure 2(b).

The filtrate from the first precipitation reaction (F1) was added into NH_4OH solution to reach the pH 5 under stirring at 500 rpm. Then, this F1 filtrate was added into Na_3PO_4 solution with stoichiometric mole ratio of 5:1 under heating at 80 °C with stirring at 250 rpm for 1 h. The precipitated formed from the second precipitation process was dried at 100 °C for 12 h, which was labelled P2. Finally, the P1 and P2 were diluted using NaOH (12 M) with stoichiometric ratio 15: 1 under stirring at 250 rpm for 15 min at 70 °C, and were kept for a fixed residence time (1 h). After precipitation and solid-liquid separation, the precipitated P1 and P2 was washed with hot water until it reached neutral pH and dried at 100 °C for 12 h. The final of total REE-hydroxides ($REE(OH)_3$) was analyzed using ICP-OES.

CHARACTERIZATIONS

The total REEs concentration for all samples was determined using inductively coupled plasma optical emission spectrometry (ICP-OES, Agilent technology 7000). Samples were dissolved in aqua regia (HCl: HNO_3 = 3:1) solution. The activation energy of the solid-liquid extraction reaction was determined using the shrinking core model analysis method (Kim et al. 2014).

RESULTS AND DISCUSSION

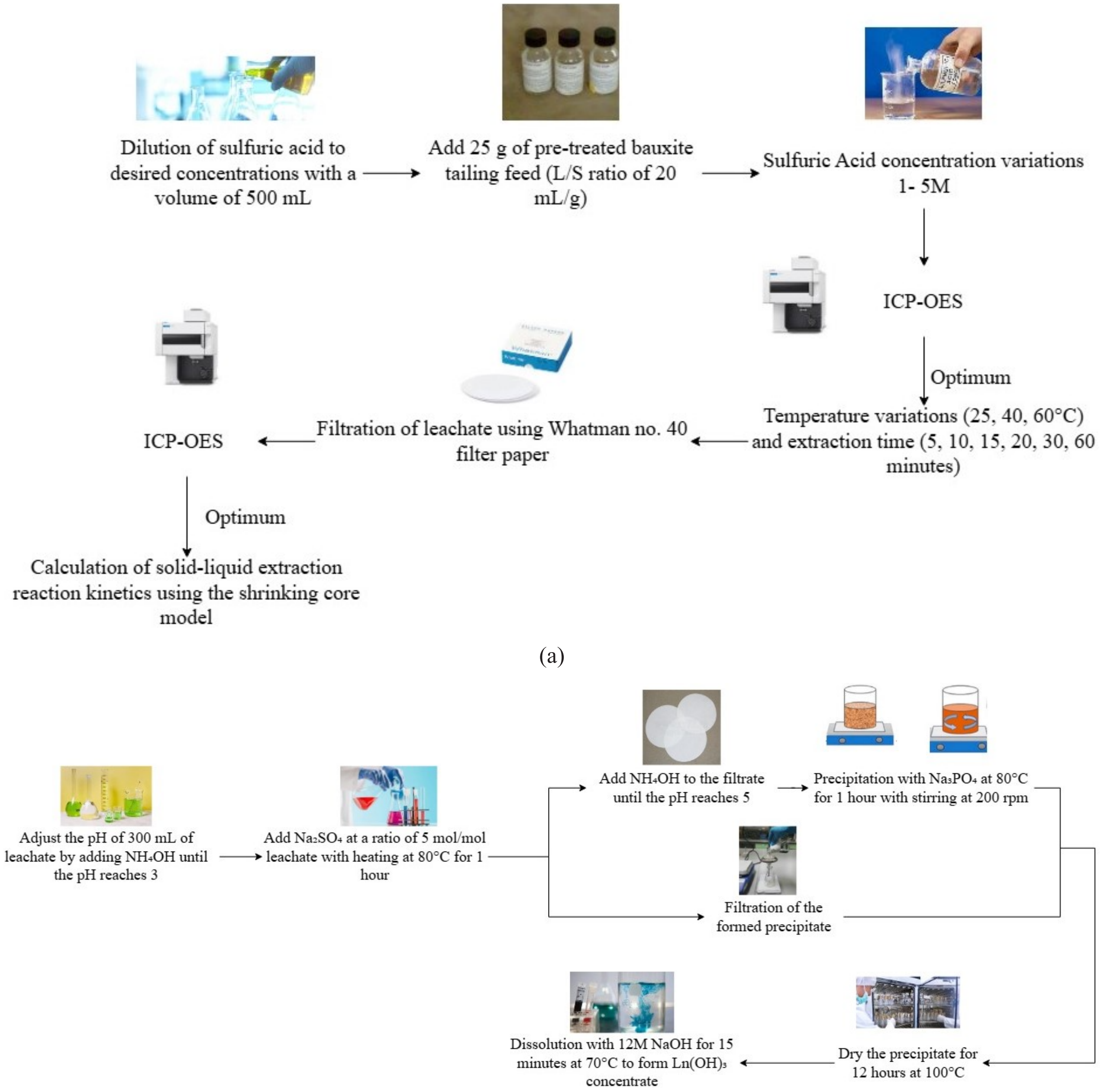
PRETREATMENT OF BAUXITE TAILINGS AND ITS COMPOSITION

Chemical analysis of the major elements (Al, Si, Fe and Ti) and other minor metals in bauxite tailings was determined using X-ray fluorescence spectroscopy, as reported by Kusrini et al. (2020c). However, the metal impurities such as Al and Fe was also detected in bauxite tailing according to the ICP-OES analysis (Figure 1(b)).

At this stage, only three rare earth elements (La, Ce, and Y) were detected using ICP-OES. These may be the low concentration of total REEs and limited availability of the standard for determination of REEs. The particle size of tailing bauxite was maintained up to 200 mesh (74 μm) in order to increase the surface area of the solid contact with the sulfuric acid extractant. Thus, the particle reduction can facilitate the diffusion process of sulfuric acid solution reacted with REEs due to the small intraparticle barriers. In addition, smaller particle sizes can increase the distribution of heat reduction when the extraction process takes place due to its exothermic reaction (Johner et al. 2018).

SOLID-LIQUID EXTRACTION

To observe the effect of sulfuric acid concentration in the acid leaching process, an operating condition with a leaching temperature of 80 °C and leaching time of 60 min was used. This condition has been previously reported by Kim et al. (2014). The reaction between REEs and sulfuric acid was assumed as in Equation (8):



(a)

(b)

FIGURE 2. (a) solid-liquid extraction process of REEs and (b) precipitation reaction of REEs

$$REE_2O_{3(s)} + 3H_2SO_{4(aq)} \rightarrow REEs_2(SO_4)_{3(aq)} + 3H_2O_{(l)} \quad (8)$$

Figure 3 shows the effect of sulfuric acid concentration towards the leachate that contains REEs. The REEs extracted using sulfuric acid with variable concentration from 1.5 to 5 M generally increase with incremental acid concentration (0.43, 1.10, 1.56, 0.56, and 0.42 ppm) that peaks at 3 M H_2SO_4 (1.56 ppm), corresponding to the batch system. This relates to the highest content of lighter rare earth elements (LREEs) including Sm (1.12 ppm), followed by Nd (0.35 ppm), Ce (0.04 ppm), La (0.04 ppm) and heavier rare earth elements (HREEs) especially Y (0.02 ppm). These values are significantly lower than those found for the concentration total REEs in leachate under leaching temperature of 60 °C with a leaching time of 30 min (Ce 20.69 ppm, Sm 3.05 ppm, Y 3.04 ppm, La 0.39 ppm, and Nd 1.92 ppm).

In this work, the best sulfuric acid concentration was 3 M, where the trends herein concur with that reported by Kim et al. (2014). Increasing the acid concentration is also related to the pH of TREEs dissolution, where increasing the pH (decreased acid concentration) will cause the deposition of metal impurities including iron (Fe). The extraction behaviour process of REEs is influenced by temperature and time, where higher reaction temperature will enhance collisions and the metal dissolution process in acidic media. The reaction time affects because it takes the optimum time in the process of diffusion of sulfuric acid into pore sites of particles to dissolve the total REEs. The longer interaction time of sulfuric acid with total REEs results in more optimal dissolution process. Sulfuric acid (H_2SO_4) is one type of acid that has a high effectiveness because it is easy to bind metal ions and is not destructive to dried bauxite tailings, where it does not pollute leachate. This finding is similar to observations reported by Kim et al. (2014) and Poscher et al. (2014). By contrast, H_2SO_4 is a reagent for acid leaching of bauxite residue (red mud) to produce Y, La, and Nd with lower yield (less than 20 wt.%), as compared with HCl (yield above 95 wt.%) (Rivera et al. 2019). This is due to the formation of a double sulfate ($NaREE(SO_4)_2.nH_2O$ which has a lower adsorption capacity onto the surfaces of Si/Al-oxides (Rivera et al. 2019). They also reported that extraction of Sc from slags can reach 90% using H_2SO_4 at 150 °C, whereas HCl can produce a extraction yield of Sc up to 80% at 120 °C (Rivera et al. 2019).

The extraction level (%) is different for each total REEs based on their association with different minerals and their ionic radii (Borra et al. 2015). It is noted that bauxite is not completely soluble in the acid solutions, but the extraction is high for Y, followed by Dy, Nd, Sc, La, and Ce. This result suggests that the extraction depends on the type REEs and the acid concentration employed (Borra et al. 2015). In this study, the highest recovery value for enrichment and extraction of total REEs from the Wacopek bauxite tailings gave a yield of 90.75% at leaching temperature of 60 °C and leaching time of 30 min, producing a weight of solid total REEs of 5.1×10^{-3} g.

Figure 4(a) showed the best leaching time to obtain the highest REEs content at 60 °C. Greater temperatures enhance the solubility and the solvent diffusivity, which increase the level of extraction. At high temperatures, the solvent viscosity is reduced, making it easier for solvent to diffuse into a solid matrix and the solute diffuses out of the mineral matrix (McCabe, Smith & Harriott 2005). The total REEs leaching efficiency decrease at 40 °C (Figure 4(a)) that occurs due to external factors during the acid leaching. At temperatures higher than 60 °C, the contact between particles of bauxite tailings increases, reducing the energy release that can raise the solvent temperature (Taggart, Hower & Hsu-Kim 2018). The R^2 was obtained through linear regression using a 3rd order polynomial.

Furthermore, a greater leaching time enhances the diffusion process of sulfuric acid for the total REEs extracted. Increasing the leaching temperature and leaching time results in greater kinetic energy and collisions of the sulfuric acid solvent with REEs, accelerating dissolution of metals to achieve optimal conditions. The content of total REEs in leachate with optimal operating conditions at 60 °C for leaching time of 30 min is Ce (20.69 ppm), Sm

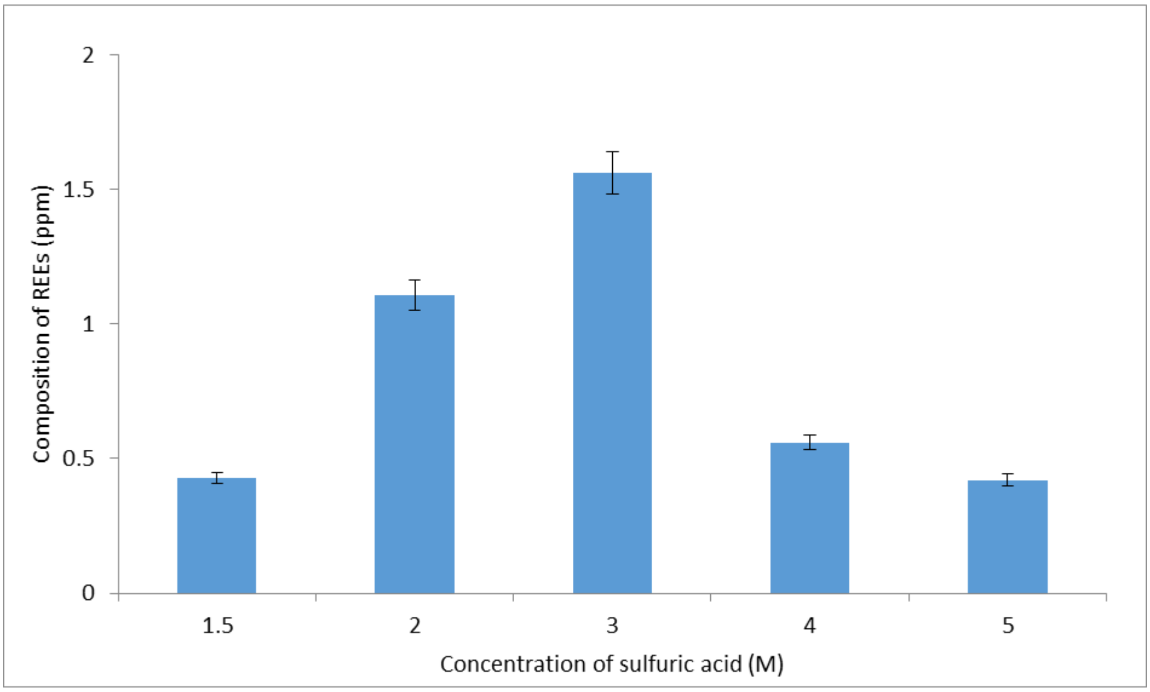
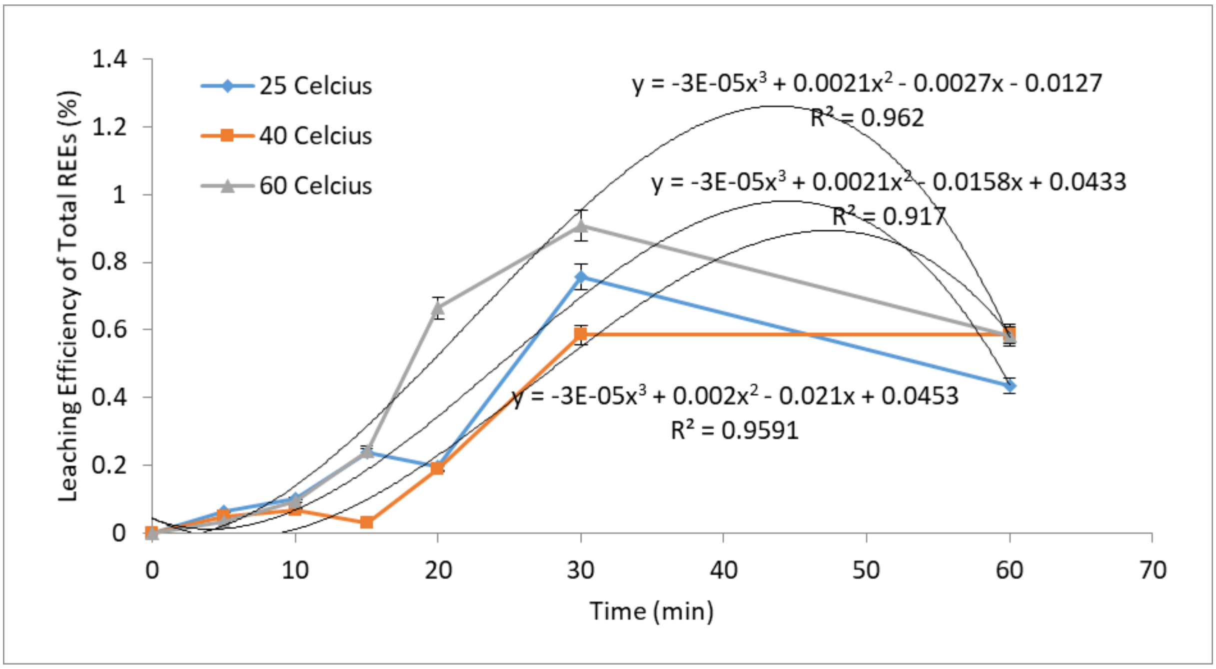
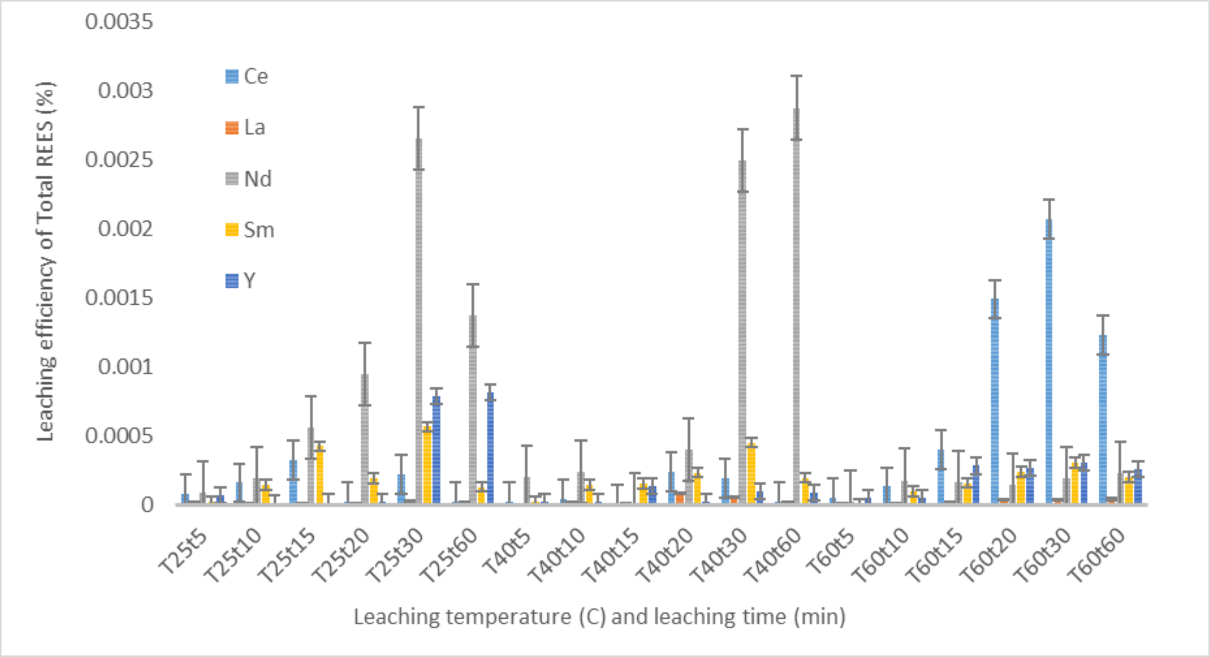


FIGURE 3. Effect of sulfuric acid concentration on TREEs extracted with variable leaching conditions: 80 °C, leaching time (60 min) and L/S ratio of 20 mL/g



(a)



(b)

FIGURE 4. (a) The influence of leaching time and temperature on the leaching efficiency of TREEs using H_2SO_4 (3M), and (b) the effect of temperature and time towards the leaching efficiency

(3.05 ppm), Y (3.04 ppm), La (0.39 ppm), and Nd (1.92 ppm). The leaching time of 30 min is the most optimum because it shows that the highest leaching efficiency for various temperatures with the following total REEs leaching efficiency: 4.25×10^{-5} at 25 °C, 3.28×10^{-5} at 40 °C, and 2.91×10^{-5} at 60 °C. However, the total leaching efficiency of REEs decreases to the following values: 2.35×10^{-5} at 25 °C, 3.2×10^{-5} at 40 °C, and 1.96×10^{-5} at 60 °C for 60 min leaching time. Since the condition of total REEs saturation was achieved in the solution, higher concentrations of sulfuric acid dissolve other mineral impurities, such as Fe, Si, Al, and Ca which have low ionization energies and more binding with the impurity metals (Borra et al. 2015).

Different composition of total REEs at different leaching temperatures and times with the operating conditions; H_2SO_4 concentration is 3 M, volume of H_2SO_4 is 500 mL, L/S ratio is 20 mL/g, and dried bauxite tailings is mesh-size 200, as presented in Figure 4(b). The influence of leaching temperatures (25 to 60 °C) toward the colour of leachate liquids was observed as the color of leachate liquid is darker at higher temperatures.

EFFECT OF LEACHING TIME AND LEACHING TEMPERATURE ON pH AND VOLUME OF LEACHATE

Increasing the leachate pH results in longer leaching time and greater leaching temperature that will cause stronger metal binding with sulfuric acid. However, low leaching pHs of sulfuric acid solution produce unstable REEs, irrespective of the leaching rate. Increasing pH from 0.1 to 1.0 has a significant impact to increase the extraction efficiency of total REEs. In other words, extraction of REEs is easier at higher pH (stable pH for extraction REEs is close to 1.0) (Qianlin et al. 2018). In addition, the production of water molecules from the reaction of sulfuric acid with metal oxide also causes an increase in pH because the acidity of sulfuric acid will continue to decrease due to the increasing water content (Equation 9). Increasing temperature for the endothermic reaction will lead to increase the product formation along with water molecules.

$$REE_2O_{3(s)} + 3H_2SO_{4(aq)} \rightarrow REE_2(SO_4)_{3(aq)} + 3H_2O_{(l)} \quad (9)$$

However, increasing the pH near 5 can increase the solubility of Ca and Mg metal impurities, which affect the extraction of REEs (Johner et al. 2018), showing the need for pH optimization for the extraction of REEs. Greater leaching occurs from 25 to 60 °C and longer leaching time from 5 to 60 min, where the volume of leachates is changed. At 25 °C and a leaching time from 5 to 60 min, the leachate volume is reduced between 448 and 428 mL. At 40 °C, a leaching time from 5 to 60 min and a leachate volume of between 428 and 418 mL occurs. At 60 °C and a leaching time from 5 to 60 min, the volume of leachate is 440 and 404 mL. At 40 °C with a leaching time of 20 min, the volume of leachate is 392 and 368 mL, respectively. High volumes indicate that only a small amount of sulfuric acid reacts with total REE metals. Total rare earth metals extracted until an operating temperature of 40 °C with a leaching time of 15 min has not been optimized. Increasing temperature in the leaching process causes the sulfuric acid to evaporate, so the volume of the extracted product will decrease. This cause longer filtering process of the liquid leachate on filter paper.

KINETIC STUDY OF ACID LEACHING USING SULFURIC ACID

The acid leaching process with sulfuric acid can partially or fully extract REEs from tailing bauxite and bauxite residue. Thus, the intra-particle diffusion transports the metals from the solid phase via fractures, cracks and pores of the particles to the solution, which regulate the leaching process (Kusrini et al. 2019b; Rivera et al. 2019). To determine the activation energy of the solid-liquid extraction, the shrinking core model analysis method (Kim et al. 2014) was used and assumed to form a spherical particle to obtain the reaction kinetics between solvent and REEs.

The kinetics of the reaction between tailing bauxite and solvent can be modeled in two stages, chemical reaction and diffusion processes. The concentration of sulfuric acid can be ignored during a chemical reaction if the sulfuric acid added to the reactor exceeds the stoichiometric ratio of the reaction. A reaction was governed by the diffusion rate that can be ascertained by observing the initial and final diameters after the reaction is almost similar, thus modeling with the shrinking core models can be determined for constant particle size (Kim et al. 2014).

In this method, chemical reaction and diffusion processes are studied. Chemical reaction can be modeled in the following equation. At the chemical reaction stage, the leaching process is expressed by Equation (10):

$$k_1 t = 1 - (1 - X_B)^{\frac{1}{3}} \quad (10)$$

With the least square method, a slope value (k) was obtained for a plot of $1-(1-X_B)^{1/3}$ versus time (t), and the k values at the different leaching temperatures were further obtained, as shown in Figure 5(a). The activation energies (E_a) were then determined based on Equation (11).

$$\ln k_1 = \ln A - \frac{Ea}{RT} \quad (11)$$

where k_1 is the leaching rate constant for surface reaction; A is a pre-exponential factor; R is the universal gas constant; T is reaction temperature; and E_a is the activation energy.

A plot of $\ln k_1$ to 1/T produces a straight line with a slope of $-E_a/R$ and the intercept on the y-axis is ln A. The leaching activation energy (E_{a1}) of REEs was calculated to be 10.1 kJ/mol. During the chemical reaction process, REE oxides on the surface sites were extracted. The remaining layer does not react after the initial chemical reaction, which becomes saturated and is resistant to H_2SO_4 solution. Therefore, the process of diffusion into the pores of bauxite tailings can be used as a rate determination step for the second stage of a solid-liquid extraction reaction. The reaction rate can be written as the rate of H_2SO_4 diffusion through the tailing bauxite (Equation 12);

$$k_2t = 1\text{-}3(1\text{-}X_B)^{2/3} + 2(1\text{-}X_B) \tag{12}$$

The k_2t value at various leaching temperatures based on the plot of $k_2t = 1\text{-}3(1\text{-}X_B)^{2/3} + 2(1\text{-}X_B)$ versus time (t) is shown in Figure 5(b). The k value is proportional to the temperature rise up to 60 °C, so the reaction rate of REE-sulfates formation from the REE-oxides will accelerate at 60 °C. The activation energy (E_{a2}) value was obtained from the linear plot of ln k_2 vs. 1/T.

In this work, the activation energy of a diffusion-controlled process (E_{a2}) was 15.5 kJ/mol, which is near to the value for a diffusion-controlled process with an E_a value of 13 kJ/mol (Kusrini et al. 2019b). If the reaction mechanism reaction is governed by chemical-controlled process, the E_a value is higher, where the activation energy was more than 43 kJ/mol (Kusrini et al. 2019b).

Table 1 shows the kinetic data and models, correlation coefficients (R^2) and rate constant (k, min^{-1}) for the leaching of REEs at different temperatures. The R^2 values was obtained through linear regression using a 3rd order polynomial. From the kinetics data of the reaction at the chemical reaction stage and diffusion, it can be seen that the reaction was controlled by the diffusion stage. The reaction rate was controlled by the slowest stage. At the diffusion stage, it has a value of the reaction rate constant (k_2) which is smaller than the value of the reaction rate constant (k_1) in the chemical reaction stage. The R^2 is closed to 1 for the chemical reaction stage and the diffusion stage at temperature of 333.15K or 60 °C.

PRECIPITATION REACTION

To produce REEs in solid form, two precipitation reactions using sodium sulfate and sodium phosphate salts as the precipitating reagents was carried out. According to the industrial plants, the precipitation method was used, as this method has some advantages, such as simple process, low cost, and feasibility (Wu et al. 2023). A pretreatment of the leachate via precipitation method is generally used to separate REEs from other metal ions. After precipitation, the filter liquors are then obtained for REE extraction.

The precipitation process was carried out with the sodium sulfate as the excessed reagent as shown in Equation (13). The first precipitation reaction with sodium sulfate begins with adjusting the pH of the leachate to pH 3 with the addition of NH_4OH. The addition of NH_4OH was carried out drop by drop to avoid the buffer being missed because the alkaline used was classified as a weak base. If it is missed in the buffer limit, the leachate solution will be very basic. The optimum leachate (300 mL) was obtained at 60 °C for leaching time of 30 min after addition of 15 drops of NH_4OH to reach pH 3. In this study, NH_4OH was selected in this reaction because it is very soluble base in sulfate solution, and the resulting NH_4^+ ions will not contaminate the precipitated product. According to the Equation (13), the sodium sulfate (Na_2SO_4) will act as the excessed reactant and the $REEs_2(SO_4)_3$ is a limiting reagent. Thus, to obtain the high yield of $Na_2SO_4.REEs_2(SO_4)_3.2H_2O$, the amount of sodium sulfate is five times than the amount of REEs present in leachate solution and produced in 5:1 mole ratio.

$$\begin{gathered} REEs_2(SO_4)_3 + Na_2SO_4 + 2H_2O \rightarrow Na_2SO_4. \\ REEs_2(SO_4)_3.2H_2O \end{gathered} \tag{13}$$

We assumed that the mass of REE-sulfate in the leachate solution is considered to be the same as with mass of total REEs in the initial sample. If the addition of sodium sulfate which is smaller than 5:1 mole ratio will result in less optimal precipitation or incomplete precipitation of REEs will occur due to the limited reagent. Addition of excess sodium sulfate will cause the formation of impurities. During the precipitation process with sodium sulfate, a stirring of 200 rpm was carried out to assist the formation of sludge. The faster the stirring will cause the formation of precipitated grains, where the smaller the size will yield a greater surface contact area.

The precipitate formed was dried at 100 °C for 12 h to remove residual water. Incorporated into NaOH (12 M) with a ratio of moles to REEs being 15:1 was required in order to convert the sodium metal salt into REE-hydroxide. In turn, the process can facilitate separation of REEs into single element with high purity and to dissolute impurities contained in precipitated REE-sulfate (Equation 14).

$$\begin{gathered} NaREEs(SO_4)_2.xH_2O_{(s)} + 3NaOH_{(aq)} \rightarrow \\ REEs(OH)_{3(s)} + 2Na_2SO_{4(aq)} + xH_2O_{(l)} \end{gathered} \tag{14}$$

The REE-hydroxide precipitates was filtered using a Whatman filter paper No. 40, followed by oven drying for 12 h at 100 °C to remove the moisture content from the reaction. In the first of precipitation reaction, a solid precipitated of REEs (P1) was formed with weight of 0.23 g. The small amount of P1 caused by the very small amount of REEs in leachates is out of balance with the number of moles of sodium sulfate precipitating agent. Wacopek bauxite tailings as raw material, precipitated P1 from the first precipitation reaction using sodium sulfate, P2 from the second precipitation reaction using sodium phosphate, and the REE-hydroxide product are shown in Figure 6.

The filtrate F1 still contains REEs, which requires a second precipitation reaction. The F1 was adjusted to pH 5 by adding NH_4OH (ca. 2 mL). The purpose of adjusting

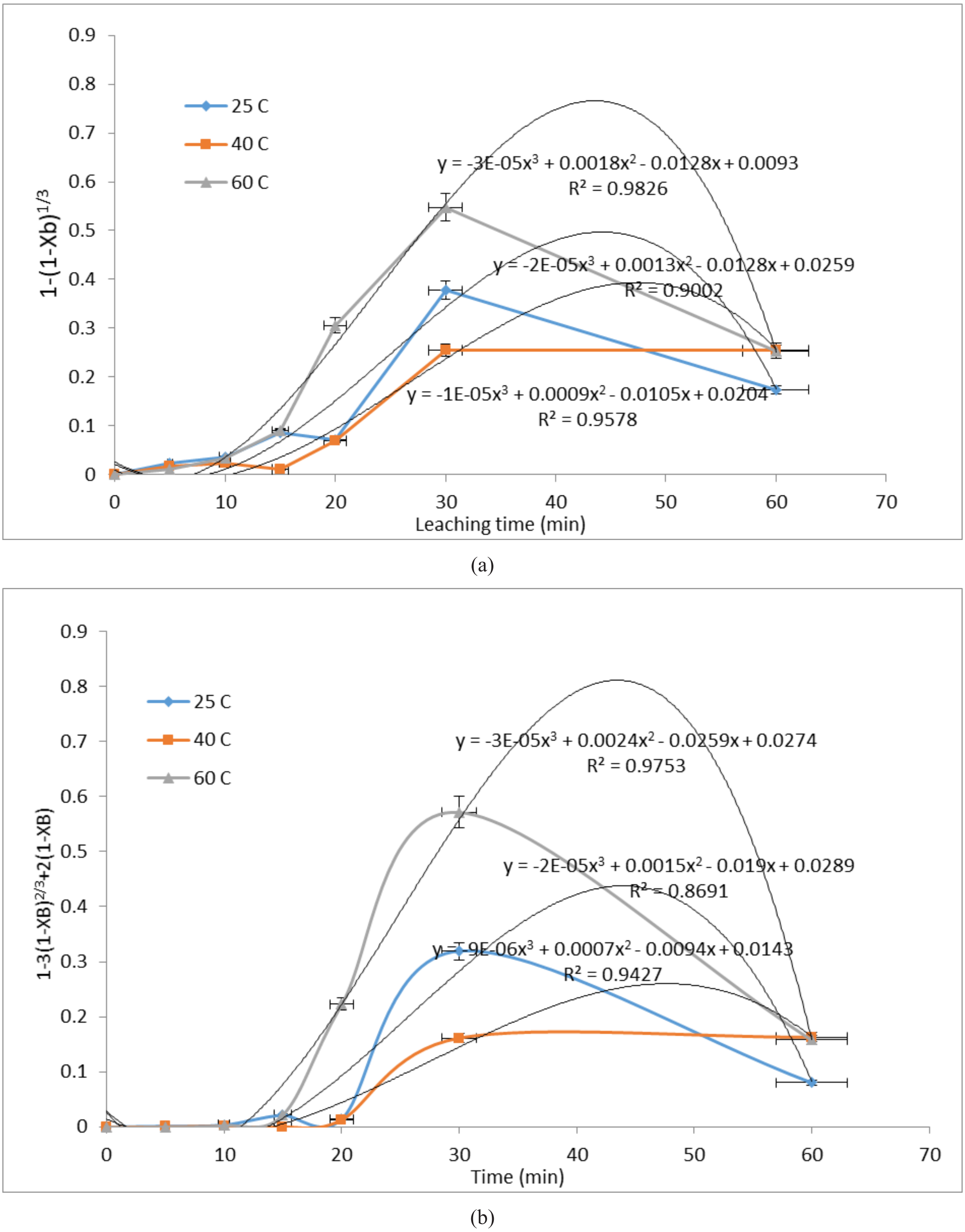


FIGURE 5(a). Plotting of 1- $(1-X_B)^{1/3}$ against extraction time to show the chemical reaction stage and (b) Plotting of $1-3(1-X_B)^{2/3} + 2(1-X_B)$ against leaching time to show the diffusion stage at variable temperatures

TABLE 1. Kinetics models, correlation coefficients (R^2) and rate constant (k, min^{-1}) for leaching of total REEs at variable temperatures

	Chemical reaction stage			
Temperature (T)	1/T	k	Ln k	R^2
298.15	3.35×10^{-3}	3.9×10^{-3}	-5.55	0.9002
313.15	3.19×10^{-3}	5×10^{-3}	-5.29	0.9578
333.15	3.0×10^{-3}	6×10^{-3}	-5.12	0.9826
	Difussion stage			
	1/T	k	Ln k	R^2
298.15	3.35×10^{-3}	2.54×10^{-3}	5.97	0.8691
313.15	3.19×10^{-3}	3.29×10^{-3}	5.72	0.9427
333.15	3.0×10^{-3}	4.9×10^{-3}	5.32	0.9753

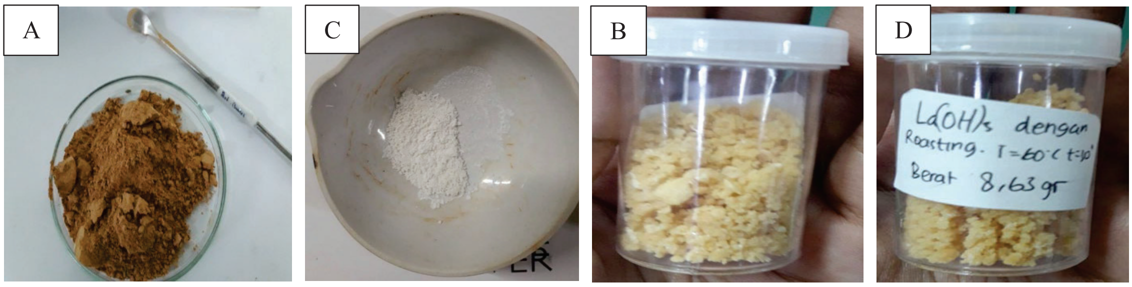


FIGURE 6. Wacopek bauxite tailings 200 mesh size (A), Precipitated P1 from the first precipitation reaction using sodium sulfate (B) and P2 from the second precipitation reaction using sodium phosphate (C) and TREE-hydroxide (D)

to pH 5 is to favour the precipitation of REE-phosphate product. The amount of Na-phosphate added is the result of a stoichiometric comparison between Na-phosphate and REEs found in leachate, which is a 15:1 mole ratio. The addition of Na-phosphate which is smaller than 15 mole/mole ratio will result in incomplete precipitation of the REEs present or inefficient precipitation. The addition of excess Na-phosphate favours the formation of other phosphate metal impurities, where the second precipitation reaction is shown in Equation (15):

$$\mathrm{REE(SO_4)_n^{3-2n}}{}_{(aq)} + \mathrm{PO_4^{3-}}{}_{(s)} \rightarrow \mathrm{REEPO_{4(s)}} + \mathrm{nSO_4^{2-}}{}_{(aq)} \quad (15)$$

The precipitate obtained from the second precipitation reaction (P2) was put into a 12.5 M NaOH solution with REEs in a 15:1 mole ratio. The purpose of adding REE-phosphate into NaOH solution is to change the structure of NaREE-phosphate salt into REE-hydroxide to facilitates the process of separating REEs with single elements that have higher purity and to dissolve impurities present in REE-phosphate that is precipitated. The P2 was dried at 100 °C for 12 h to remove the moisture content formed during the reaction, where the yield of P2 was 9.8 g.

CONCLUSION

REEs were classified as the most critical materials nowadays and in the future in terms of supply risk and their economic importance, thus, extraction and recovery of total rare earth elements (REEs) from Indonesian Wacopek bauxite tailings were urgent to be evaluated. This study was conducted at the laboratory scale to produce a greater concentration with enrichment of REEs, which are considered as critical minerals, aiming to extract total REEs from Wacopek bauxite tailings using solid-liquid extraction by sulfuric acid system and precipitation reaction using sodium sulfate and sodium phosphate, respectively. The experiments were carried out by studying the effects of leaching time and temperature on the leaching kinetics. The best conditions to extract of REE-hydroxide is 3 M sulfuric acid as solvent at 60 °C for the leaching time of 30 min. In the precipitation process using sodium sulfate and sodium phospate, with precipitates P1 (0.23 g) and P2 (9.8 g) are formed, respectively. The total REEs content in leachate at the optimum conditions are 20.69 ppm (Ce), 3.05 ppm (Sm), 3.04 ppm (Y), 0.39 ppm (La), and 1.92 ppm (Nd). The largest yield of total REEs (La, Ce, Sm, Nd, and Y) was 90.75%. The leaching kinetic model of total REEs follows the shrinking core model and controlled by diffusion with energy activation of 10.1 kJ/mol and 15.5 kJ/mol, respectively. Based on the kinetic study, the dissolution of total REEs is inhibited and its content in the leaching liquid was reduced by the particle size, leaching temperature, leaching time, pH and physical treatment under stirring.

ACKNOWLEDGMENTS

This work was supported by Kementerian Pendidikan Tinggi, Sains dan Teknologi under grant award PT-LP Nomor: PKS-608/UN2.RST/HKP.05.00/2025. Our contributions is as follows; EK: Conceptualization, supervision, methodology, writing - review & editing; MA & FA: Performed the experiments, preparing the data, drafting an article & preparing the figures; YM: Supervision & methodology; NS, S, DD & ESN: Interpreted the data and review; LDW & AU: Writing-review, validation, and interpreted the data; ABP & PI: Characterization and Interpreted the data; AR: Providing the sample Wacopek bauxite tailing.

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