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Extraction of gadolinium and neodymium from tayan red mud

Crystonius Simbolon[1], Panut Mulyono[1*], Sutijan[1], Himawan Tri Bayu Murti Petrus[1], Widi Astuti[2], Esti Handini[3]

[1] Department of Chemical Engineering (Sustainable Mineral Processing Research Group), Universitas Gadjah Mada, Yogyakarta, Indonesia
[2] Chemical Research Center, Indonesian Research and Innovation Agency (BRIN), South Tangerang, Banten, Indonesia
[3] Department of Geological Engineering, Universitas Gadjah Mada, Yogyakarta, Indonesia
[*] Corresponding author email: pmulyono@ugm.ac.id

Abstract

Red mud is a waste generated from the alumina production process. Red mud was previously categorized as hazardous waste due to its high alkalinity. In general, the mineral composition of red mud consists of Fe_2O_3 (30-60%), Al_2O_3 (10-20%), SiO_2 (3-50%), Na_2O (2-10%), CaO (2-8%) and TiO_2 in amounts no greater than 25%. This composition varies widely around the world depending on the origin of the bauxite and red mud characterization results also have a rare earth element content of at least 0.03%. Rare earth element is classified as a critical metal, i.e. an essential material used and subject to supply risks as demand increases over time. Indonesia already produces at least 4.4 million tons of red mud per year, so more and more REY will be wasted if we do not utilize the existing red mud. Laboratory-scale experiments were conducted to recover gadolinium and neodymium from Tayan red mud in sulfuric acid in atmospheric pressure acid leching condition. Maximum recovery of gadolinium (91.3%) was recorded with 3 M H_2SO_4 at 90°C, solid/liquid ratio of 1/15 g/L and agitation rate of 700 rpm in 2 hours. While 81.5% neodymium recovery was achieved at 90°C and solid/liquid ratio of 1/15 g/L in 2 M H_2SO_4.

Keywords

Red mud, Leaching, Gadolinium, Neodymium

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Introduction

Globally, the aluminum production process occurs in 2 stages, namely in the Bayer process which is a process of refining bauxite ore to obtain aluminum oxide (alumina) and the Hall-Heroult process is a process of smelting aluminum oxide to produce pure aluminum. In addition to producing aluminum oxide (alumina), the Bayer process also produces bauxite residue called red mud [1]. Based on data from the Indonesian Central Bureau of Statistics, processing bauxite through the Bayer process produces approximately 0.8-2.5 tons of red mud waste for every ton of alumina obtained. Indonesia currently has a bauxite processing industry in West Kalimantan and will continue to be built. In 2021, the industry will produce 4.3 million tons of Chemical Grade Alumina (CGA) per year and 1.2 million tons of Smelter Grade Alumina (SGA) per year, resulting in approximately 4.4 million tons of red mud waste per year. Red mud contains alkaline compounds with high pH, is very fine and sometimes contains harmful minor elements that cause serious problems for the environment. Therefore, research for the handling and utilization of red mud is very intensive [2].

Ore processing can be done by hydrometallurgical methods, such as the Caron process, high pressure acid leaching (HPAL), and atmospheric pressure acid leaching (APAL). The use of this hydrometallurgical method has the advantage of producing a much purer primary product compared to using metallurgical methods. Of the three examples of processes obtained by hydrometallurgy, the APAL process is considered more economical if applied on an industrial scale than the other two. This is due to the use of atmospheric pressure in the process, resulting in low energy requirements and operational costs. To be applied on an industrial scale, a study of the kinetics of this APAL process must be carried out because by studying the kinetics of the process, the design parameters of the extractor are obtained and this information is important for the design stage. The APAL method was used in consideration of the optimum conditions suitable for red mud samples and acid reagents that bind Rare earth metals [3].

The 17 chemical elements that make up the Rare Earth Elements (REE) group include 15 members of the lanthanide series, scandium, and itrium. These elements are actually abundant in the Earth's crust, despite being referred to as "rare." However, they are rarely found in economically viable mining concentrations. Due to their distinct magnetic, optical, and electrical characteristics, rare earth elements (REEs) are crucial in many contemporary technological applications. These elements are divided into two categories: heavy REEs (HREE), which include europium and lutetium, and light REEs (LREE), which include lanthanum and samarium [4][5].

Neodymium (Nd), which has an atomic number of 60 and extremely potent magnetic characteristics, is a significant element in this group. Neodymium-iron-boron (NdFeB) permanent magnets, which are utilized in hard drives, wind turbine generators, electric motors, and electronic gadgets like cell phones, are made using a lot of neodymium. These magnets are crucial for downsizing technology because of their extremely high magnetic strength in spite of their small size. Neodymium is also utilized in the production of glass colorants, lasers, and lenses. Another element in the REE group, gadolinium (Gd), has a pronounced paramagnetic property with an atomic number of 64. Due of gadolinium's capacity to enhance the picture quality of bodily tissues, this element is frequently utilized in medical technology, particularly as a contrast agent in magnetic resonance imaging (MRI). Gadolinium is also utilized in the production of metal alloys to enhance their resistance to heat and corrosion, fluorescent displays, and nuclear fuel as a neutron absorber. Both gadolinium and neodymium are crucial for advancing green energy, medical applications, and technological innovation.

In previous studies, scandium recovery can reach 40 wt% at red mud in Belgium with a single stage dry digestion method using HCl solvents and can increase REE concentrations when using multi-stage methods from 8 mg/L to 20 mg/L [6]. At Indian red mud, lanthanum recovery can be obtained as much as 99% using 3M H_2SO_4 at 35°C and cerium recovery is obtained at 99.9% at 75°C [7]. The same research was also conducted on Indonesian red mud, the recovery of scandium obtained was 90.97% and could recover iron and aluminum by 32.34% and 75.23% respectively [8]. Several previous studies have chosen inorganic acids as leaching acids, but there are also those who carry out a multistage leaching process using organic acids and produce high recovery values. In the multistage leaching process on red mud from Australia, the purification results of the leaching process with inorganic acids are then re-extracted using organic acids such as D2EHPA and Cyanex272. Scandium recovery values reached more than 99% with almost no iron extracted, but this process needs to be considered because it is costly [9].

Based on the research that has been done, it has not provided information on the optimum conditions in the process of separating REE components in red mud in Indonesia. Red mud was chosen because the mineral industry in Indonesia is increasingly producing aluminum, so the red mud produced will also be a lot. In addition, based on previous research proves that with a very small composition of REE in red mud can still be separated with a high conversion rate. In previous research, the method used in the leaching process of red mud from Indonesia was high pressure acid leaching (HPAL). Whereas in hydrometallurgy there is also an atmospheric pressure acid leaching (APAL) process which is considered more economical, so the method used in this study is APAL to find the optimum conditions in obtaining the results of the leaching process with high conversion.

In general, there are several factors that can affect the outcome of the leaching process such as temperature, pressure, catalytic effect, nature of reactants, concentration of reactants, chemical composition of the metal compounds, nature of gangue, etc. Depending on the scale of view each parameter acts differently, so among all the parameters the most important are the concentration of leaching agent, temperature and particle size. Based on previous literature, H_2SO_4 was selected as the most selective leaching agent to recover REEs in red mud [10][11][12]. In addition, the higher concentration of leaching agent and temperature will also greatly affect the leaching results, but there is a limit to the highest optimum point for a certain concentration of leaching agent and temperature [13][14][15].

Materials and Methods

Raw Material Characterization

Characterization of Red mud samples was carried out in 2 analyses. First is the characterization of solids X-ray fluorescence (XRF) analysis. Analysis is done to determine the composition of the main elements in red mud. The second analysis is the analysis of liquid digesting results using Inductively Coupled Plasma (ICP-OES) analysis. The analysis was conducted to accurately determine the REE content.

Red mud samples were first dried using an oven for approximately 3 hours (adjusting the initial condition of the sample) at 105°C to ensure that the red mud samples to be used were completely dry. After that, the dry red mud will be reduced in size using a mortar and pestle, then the size of the red mud is uniformed to a size of +200 mesh using a sieve shaker.

Leaching experiments

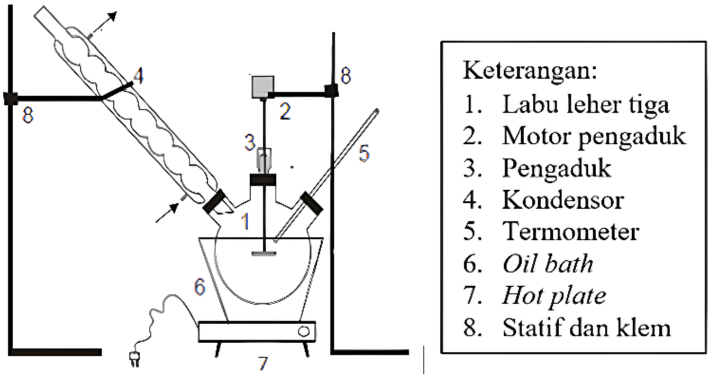

Figure 1. Design scheme of leaching experiment

The red mud sample that has been prepared will be put into a three-neck flask that has been prepared in accordance with the series of leaching equipment. The leaching process is carried out according to the variable variations that have been determined with the parameters of solid/liquid ratio, temperature and reagent concentration. The leaching process was carried out for 120 minutes for all variable variations shown in Table 1 using a syringe filter. After the leaching process, a filtering process was carried out to separate the filtrate and residue using a vacuum filter. The residue obtained will be dried in an oven, while the filtrate will be measured in volume.

The product is analyzed to determine the REY content using ICP-MS analysis and the residue is analyzed by XRF analysis. Samples of leaching results will be taken and diluted using demineralized water. The diluted sample is homogenized. The diluted sample is filtered using a syringe filter to avoid the presence of impurity solids and is ready for analysis. The measurement uncertainty of temperature on the thermometer with a ±1°C limit, along with the mineral composition analysis of red mud using XRF and ICP-MS, will yield different results because red mud from different locations will have different mineralogical structures.

Table 1. Variable of the leaching experiments

Exp. number	Variables	
	[H2SO4] (M)	Temperature (°C)
1	1.5	30
2	1.5	65
3	1.5	90
4	2	30
5	2	65
6	2	90
7	3	30
8	3	65
9	3	90

Results and Discussion

The chemical composition of major elements in solid samples, including iron, silica, calcium, aluminum, and titanium from red mud, was analyzed using X-ray fluorescence (XRF), while the concentrations of REEs in solid samples were determined using Inductively Coupled Plasma Mass Spectroscopy (ICP-MS), as presented in Table 2.

Table 2. Chemical composition of the red mud

Compound	Major elements (%)					Minor elements (mg/L)	
	Al_2O_3	SiO_2	Fe_2O_3	TiO_2	CaO	Nd	Gd
Conc. Unit	12,718	17,99	60,59	3,738	2,936	0.047	0.54

In this study, the leaching efficiency of element i (η_i,%) in the leaching solution was determined using Equation (1), as shown below:

$$Leaching\ eff. = \frac{C_i \times V}{C_0 \times m_0} \times 100 \tag{1}$$

where c_i (mg/L) represents the concentration of element i in the leaching solution, V (L) denotes the volume of the leaching solution, while C_{0i} (g/g) and $m_0(g)$ correspond to the content of element i in the original red mud and the mass of the original red mud, respectively.

Table 3. Leaching efficiency calculation results

Concentration M	Temperature (°C)	Efficiency (%)	
		Nd	Gd
	30	15.96	19.10
1.5	65	31.12	53.03
	90	55.85	72.34
	30	19.95	26.10
2	65	35.90	66.60
	90	81.46	82.07
	30	23.94	29.59
3	65	48.67	68.77
	90	75.00	91.30

Effect of acid concentration

Experimental results indicate that neodymium achieves optimal leaching efficiency at a 2 M acid concentration, as shown in Figure 2, while gadolinium exhibits maximum leaching efficiency at 3 M acid concentration, as illustrated in Figure 3. Specifically, the highest neodymium leaching efficiency (81.5%) was obtained using 2 M H_2SO_4, and the highest gadolinium leaching efficiency (91.3%) was achieved with 3 M H_2SO_4 under the following conditions: S/L ratio = 1/15, temperature = 90°C, duration = 2 hours, and agitation rate = 700 rpm. The extraction percentages of neodymium and gadolinium were calculated based on their concentrations using mass balance analysis. At a concentration of 3 M H_2SO_4, there is a decrease in neodymium leaching efficiency which is due to the decrease in solubility in concentrated H_2SO_4 solution can be explained by the concept of solvation: as the electrolyte concentration increases, fewer water molecules are available to aid in the dissolution process because they become tightly bound to the cations and anions in the solution. This phenomenon is known as the salting-out effect.

Effect of temperature

Figure 2 and Figure 3 illustrate that the leaching efficiency of neodymium and gadolinium from red mud reaches its peak at 90°C. For instance, the highest recovery of neodymium (81.5%) and gadolinium (91.3%) is achieved at 90°C under the following conditions: S/L ratio = 1/15, agitation rate = 700 rpm, and a duration of 2 hours. The leaching efficiency of elements exhibited a rising trend with increasing reaction temperature, highlighting temperature as a key factor influencing the extraction process. The findings demonstrated that higher temperatures enhanced the leaching reaction activity. With an increase in temperature, particle diffusion into the leaching agent became faster, and the interfacial reaction rate between the mineral particles and the leaching reagent increased, promoting particle dissolution. The results of the leaching process with varying temperatures, according to theory and previous research on temperature parameters, show that as the temperature increases, the recovery value of REE (Rare Earth Elements) also increases, but only up to the optimum temperature point. The same applies to acid concentration, where increasing the concentration raises the recovery, but only up to an optimum limit.

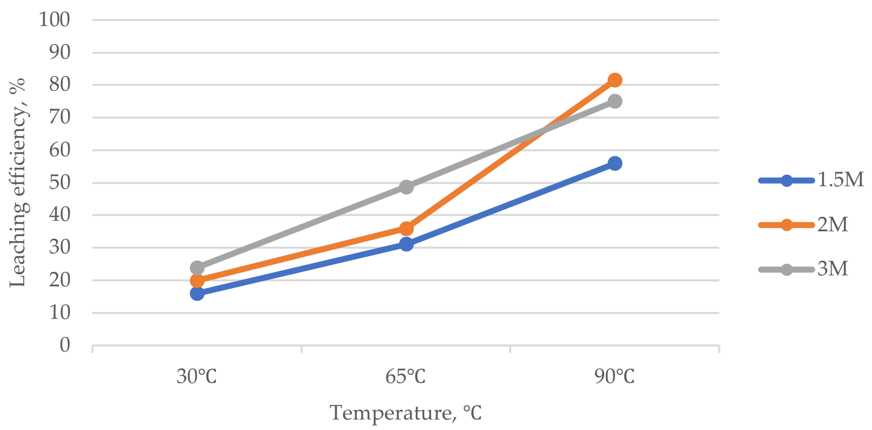

Figure 2. Leaching efficiency of neodymium

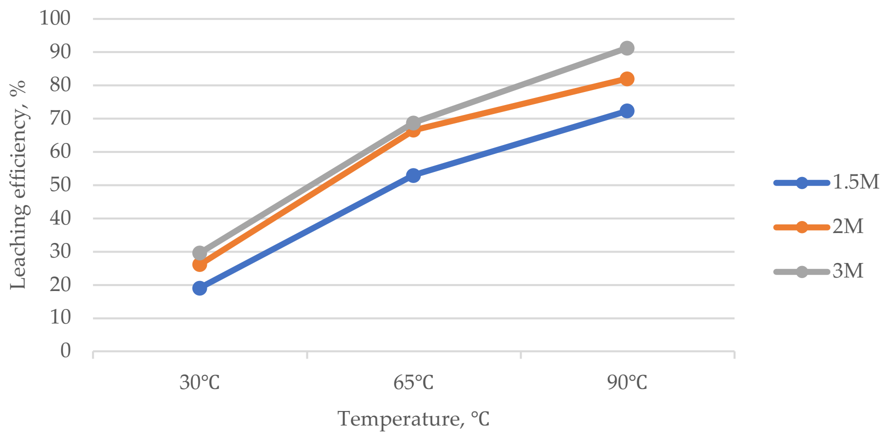

Figure 3. Leaching efficiency of gadolinium

Conclusion

Based on the results of the research that has been carried out, rare earth metals contained in red mud have the potential to be recovered. Where it can be seen that the resulting leaching efficiency can reach more than 80%. The optimum conditions obtained from the results of the study were at a temperature variation of 90°C using 2M sulfuric acid, where the leaching efficiency of Neodymium and Gadolinium was 81.5% and 82.07%, respectively. This study highlights the potential for extracting technologically significant rare earth elements (REEs) from the readily available bauxite residue produced during industrial alumina manufacturing.

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