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Extraction of Scandium and Removal of Iron from Bauxite Residue using Hydrochloric Acid Solution with/without Addition of EDTA

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Abstract

Scandium (Sc) is a strategic metal for its increasing demand for advanced materials applications. As a by-product of alumina production, bauxite residues possess a potential source of Sc. However, its high iron content hinders the Sc extraction efficiency. This study investigated the feasibility of Sc extraction from bauxite residue using hydrochloric acid (HCl) leaching process, with and without the addition of ethylenediaminetetraacetic acid (EDTA) as a chelating agent. Bauxite residue samples were characterized for their elemental composition using the X-ray fluorescence (XRF) spectroscopy. Subsequently, leaching experiments were conducted using 6M and 9M HCl solutions. The effect of EDTA on Sc extraction yield and iron dissolution was assessed. The XRF analysis revealed a significant iron content in the bauxite residue, confirming the need for effective iron removal. Hydrochloric acid was found to be effective in leaching iron (Fe) from bauxite residue, as confirmed by the high Fe content in the leachate, and a higher HCl concentration led to a higher Sc_2O_3 concentration in the residue. Although the addition of EDTA was effective in chelating iron, it also reduced Sc extraction efficiency. The leaching results suggested the use of 9M HCl without the addition of EDTA as the best leaching solution for Sc extraction, yielding a higher Sc recovery compared to extractions using 6M HCl and EDTA. These findings contribute to the understanding of Sc extraction from bauxite residue and provide valuable insights for developing efficient and sustainable recovery processes.

Keywords: *bauxite residue; EDTA; extraction; hydrochloric acid; leaching; scandium*

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INTRODUCTION

PT. Indonesia Chemical Alumina (ICA) is one of the mineral industries that produces alumina and its residue, which is called red mud. PT. ICA produces 300.000 tons of alumina by producing a large quantity of red mud amounting 240.000 – 450.000 tons every year, which is stored in its final stockpiling facility (Anugrah and Mamby, 2020). The mixture between the red mud stored in a stockpiling facility (landfill) class III and fly ash-bottom ash (FABA) from coal combustion residue at the PT. ICA is called bauxite residue (Eka et al., 2023).

In general, the accumulation of bauxite residue can cause significant harm to the environment due to its high heavy metals content and alkalinity (pH 10-12.5). One of the ways to overcome the accumulation of bauxite residue is to develop a comprehensive bauxite residue utilization technology for its use as a promising secondary resource. This residue contains important rare earth elements (REE), such as scandium, yttrium, and other rare earth elements. Thus, extracting these elements from bauxite residue can reduce their negative environmental impact (Wei et al., 2020; Li et al., 2022).

Scandium (Sc) is a critical REE with promising applications in advanced materials applications. Its unique properties, alloying capabilities, and electrochemical, electrical, and optical characteristics have made it a valuable resource. Due to its low natural availability, scandium is primarily recovered as a by-product from various industrial processes, including tungsten, titanium, and aluminum production. Among these sources, bauxite residue, a significant by-product of alumina production, stands out as a promising Sc source. This material typically contains REEs, with Sc concentrations ranging from 60 to 160 ppm, which represents over 90% of the total REEs value in the bauxite residue. Consequently, research efforts have focused on the development of efficient and sustainable methods for Sc extraction from bauxite residue to meet its growing demand as a strategic metal (Borra et al., 2015; Vind et al., 2018).

Several studies have investigated the direct leaching of Sc from red mud, a process that offers simplicity and a reduced acid consumption (Borra et al., 2015). Borra et al. (2015) reported Sc extraction yields of 70-80% using 6 N HCl as the leaching agent at room temperature for 24 hours. However, this method is associated with significant iron dissolution. To address this issue, Zhou et al. (2018) employed ethylenediaminetetraacetic acid (EDTA), as a chelating agent to selectively extract Sc, achieving a maximum extraction yield of 79.6% with HCl leaching.

Based on the abovementioned information, this study introduces a novel approach by combining red mud with fly ash-bottom ash. This modified bauxite residue will then be subjected to a two-stage process involving magnetic separation followed by hydrochloric acid leaching, with and without the addition of EDTA. This integrated approach is expected to reduce iron content and enhance Sc extraction yield. To optimize the process and determine the optimal conditions for Sc extraction from bauxite residue sourced from PT. ICA Kalimantan Barat, a comprehensive investigation into extraction and characterization techniques, is necessary. Two different hydrochloric acid concentrations were assayed their efficiency for Sc extraction, with and without the addition of EDTA. This approach provides valuable insights into the impact of varying acid concentrations on Sc recovery yields.

MATERIALS AND METHODS

Materials

The materials used in this research comprised bauxite residue from PT. Indonesia Chemical Alumina (ICA) West Kalimantan, distilled water, hydrochloric acid p.a 37% from Smart Lab Indonesia, and EDTA (ethylene diamine tetra acetic acid) from Sigma-Aldrich, Germany. The equipment used in this research was a 200-mesh sieve, an electric oven (Esco Isotherm Laboratory Ovens), a neodymium magnet (rectangular with dimensions 4×1×0.5 cm), a reflux set, and an X-ray Fluorescence, XRF PANalytical Epsilon 3.

Methods

Pretreatment of Bauxite Residue

Wet bauxite residue was initially sun-dried to reduce moisture content. The dried residue was then washed with distilled water to remove impurities and subsequently oven-dried at 105°C for 5 hours to achieve a constant weight. The dried residue was crushed and magnetically separated using a neodymium magnet to isolate non-magnetic components (Cahyono et al., 2019). The non-magnetic fraction was then sieved through a 200-mesh sieve, as shown in Figure 1, to obtain a homogeneous particle size distribution and enhance the leaching efficiency (Meng, et al., 2020; Li et al., 2022).

Figure 1. Non-magnetic bauxite Residue (200 mesh) after sieving

Extraction of Scandium by HCl Leaching

Leaching experiments were conducted in 300 mL flat-bottomed double-necked flasks equipped with reflux condensers and magnetic stirrers. In one set of experiments, EDTA was added to the HCl solutions at a ratio of 0.5:10 g/mL, while in the other set, no EDTA was added. (see Table 1).

Table 1. Variation of Sc Extraction Experiments

Sample code	Non-magnetic Bauxite Residue	6 M HCl	9 M HCl	EDTA
RM-00	✓			
RM-01	✓	✓		
RM-02	✓		✓	
RM-03	✓	✓		✓
RM-04	✓		✓	✓

Each HCl solution was heated to 80°C, and the bauxite residue was added. The non-magnetic bauxite residue (200 mesh) was poured to 6 M and 9 M HCl solutions at a solid-to-liquid ratio of 1:10 g/mL. The mixtures were stirred for three hours at a constant temperature. Subsequently, they were filtered to separate the solid residue from the liquid filtrate. The residues were washed with distilled water to neutrality and were further oven-dried at 105°C for 5 hours to a constant weight.

RESULTS AND DISCUSSION

Extraction of Scandium by HCl Leaching

This research used non-magnetic bauxite residue that was leached using HCl solvent for iron removal producing $FeCl_3$ (Borra et al., 2015). Leaching experiments were conducted using 6 M and 9 M HCl at a constant temperature of 80°C that were selected based on the work of Wei et al. (2020). The reaction equation that occurs in the leaching process is as follows (Wei et al., 2020):

$$Fe_2O_{3\,(s)} + 6HCl_{(aq)} \rightarrow 2FeCl_{3\,(aq)} + 3H_2O_{(l)} \quad (1)$$

Scandium in bauxite residue is typically present as oxides (e.g., Sc_2O_3) or incorporated into complex mineral matrices like ferrites and silicates. At 80°C, HCl dissociates completely, providing a high concentration of protons (H^+) that break down Sc-containing compounds into their soluble ionic forms and reacts with metal oxides and hydroxides (e.g., Fe_2O_3, Al_2O_3, TiO_2, and Sc-containing phases). The acidic environment provides H^+ ions that protonate and cleave bonds within these compounds, enabling their dissolution into their respective ionic forms. The leaching mechanism involving HCl can be represented by the following reactions:

$$Sc_2O_3 + 6HCl \rightarrow 2ScCl_3 + 3H_2O \quad (2)$$

$$Fe_2O_3 + 6HCl \rightarrow 2FeCl_3 + 3H_2O \quad (3)$$

The leaching process in this research used 6 M and 9 M, which were then filtered to obtain filtrate and residue (Figure 2). The filtrate is a reddish-yellow $FeCl_3$ solution. This result indicates the presence of high iron content dissolved in the solution (Habibi et al., 2023). The wet residue obtained with 6M HCl was brownish gray, and 9M HCl was gray. The meaning of both colors represents the level of Fe_2O_3 content, where the more concentrated the gray color in the residue, the less the Fe_2O_3 content is. The residue was then washed with distilled water and dried in an oven at 105°C for 5 hours to remove any remaining solvent in the residue (Figure 3).

Meanwhile, the non-magnetic bauxite residue was leached using an HCl solvent at the identical concentrations, with the addition of EDTA. EDTA, a hexadentate ligand, forms strong complexes with metal ions through its two tertiary amine groups and four carboxylate groups, thereby enhancing the leaching efficiency by stabilizing the metal ions in solution.

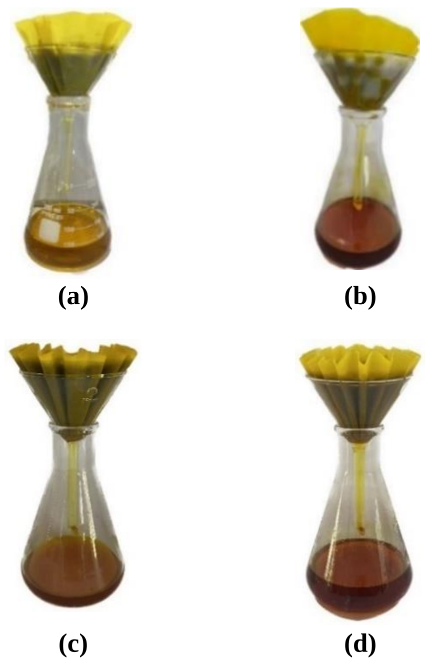

Figure 2. Filtration Results for Different Leaching Conditions: (a) 6 M HCl, (b) 9 M HCl, (c) 6 M+EDTA (d) 9 M+EDTA

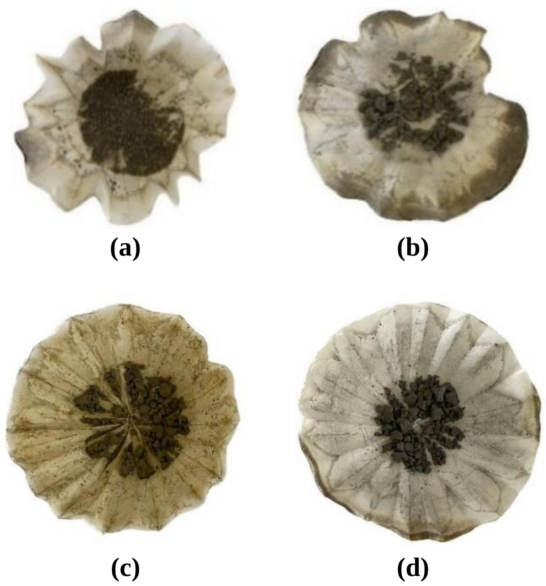

Figure 3. Dried Residues from Different Leaching Conditions: (a) 6 M HCl (b) 9 M HCl (c) 6 M+EDTA (d) 9 M+EDTA

The EDTA ligand binds to metal ions such as Fe^{3+} to form a stable complex, $[Fe(EDTA)]^-$ (Zhou et al., 2018). The structure of the $[Fe(EDTA)]^-$ complex is illustrated in Figure 4.

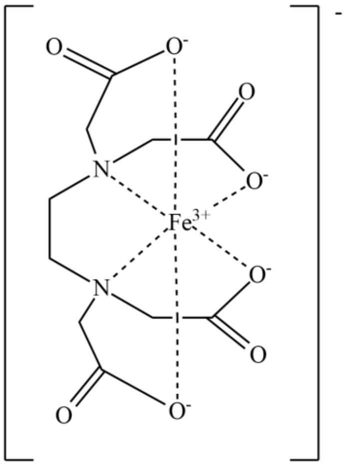

Figure 4. Complex Anion, [Fe(EDTA)-] (Jouyandeh et al., 2019)

EDTA was added to the leaching solution to chelate the iron ions and reduce their interference in the scandium extraction process (Zhou et al., 2018). After leaching with 6 M and 9 M HCl solutions, both with and without the addition of EDTA, the mixtures were filtered to separate the solid residues from the liquid filtrates. Regardless of the addition of EDTA, the filtrates remained reddish-yellow, indicating a high iron content (Habibi et al., 2023).) The wet residues obtained from the leaching processes with EDTA were brownish-gray and gray. These residues were washed with distilled water and oven-dried at 105°C for 5 hours (Figure 2).

Characterization of Bauxite Residue

X-ray fluorescence spectroscopy was employed to determine the elemental and oxide compositions of the non-magnetic bauxite residue, residues, and filtrates obtained from the leaching experiments.

XRF analysis was conducted on the following samples: 200-mesh non-magnetic bauxite residue (RM-00), residues and filtrates from 6 M HCl leaching (RM-01), 9 M HCl leaching (RM-02), 6 M HCl leaching with EDTA (RM-03), and 9 M HCl leaching with EDTA (RM-04). The XRF analysis results for the residues and filtrates are presented in Table 2 and Figure 5, respectively. The addition of EDTA to the leachate introduces a crucial step in the scandium recovery process, as EDTA, a strong chelating agent, exhibits a high affinity for Fe^{3+} ions. The addition of EDTA to the leachate results in the formation of a stable, water-soluble complex with Fe^{3+} ions, as represented by the following reaction:

$$Fe^{3+} + EDTA^{4-} \rightarrow [Fe(EDTA)]^- \quad (4)$$

Table 2. Oxides Composition in Filtrate RM-01, RM-02, RM-03, and RM-04

Oxides	RM-01 (%)	RM-02 (%)	RM-03 (%)	RM-04 (%)
Sc_2O_3	0	0	0	0
Fe_2O_3	29.01	18.72	20.26	20.24
SiO_2	1.38	2.72	2.21	2.26
TiO_2	0.81	0.56	0.6	0.66
Al_2O_3	4.36	8.01	17.63	6.13

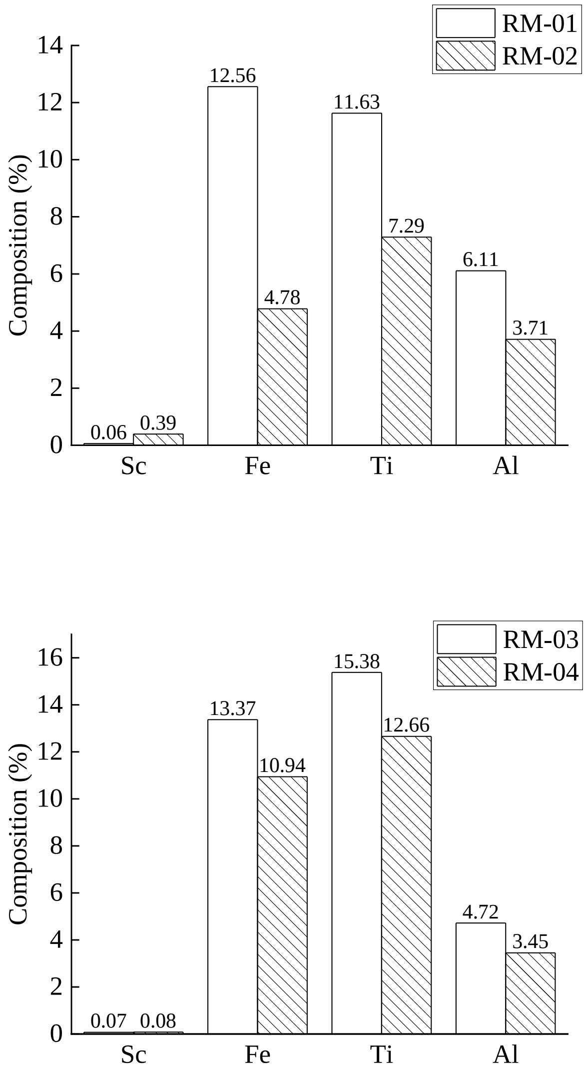

Figure 5. Oxides Composition in Residue RM-01, RM-02, RM-03, and RM-04

This complexation effectively sequesters Fe^{3+} ions from the solution, preventing their precipitation and potential interference with subsequent scandium recovery steps.

The RM-00 sample, a non-magnetic bauxite residue, contains a Fe_2O_3 composition of 10.83%. When compared to the bauxite residue sample before magnetic separation treatment, the sample contains a

Fe_2O_3 composition of 60.3% (Eka et al., 2023). This showed that magnetic separation treatment has succeeded in reducing the Fe_2O_3 composition from 60.3% to 10.83%.

Based on Table 2, the results of XRF analysis on all leaching filtrates, no Sc_2O_3 was detected. All filtrates obtained have a dominant Fe_2O_3 content, thus proving that Fe_2O_3 is soluble in an HCl solution. According to a previous study, Fe_2O_3 is more soluble in HCl solution than Sc_2O_3 (Borra et al., 2015).

As shown in Figure 5, XRF analysis revealed a significant increase in Sc_2O_3 content in all HCl-leached residues compared to the non-magnetic bauxite residue, which contains only 0.007% Sc_2O_3. The resulting Sc_2O_3 composition varied with HCl concentration: 0.06% for RM-01 and 0.39% for RM-02. This trend aligns with the currently established literature, which indicates that higher HCl concentrations lead to a substantial increase in scandium extraction (Wei et al., 2020). From their research, Wei, et al. (2020) obtained Sc_2O_3 composition of 0.00353% from in the extraction of Sc using HCl solution (6.5 M). The Sc_2O_3 composition in the RM-01 and RM-02 residue samples is much higher than that reported by Wei, et al. (2020). The use of 6M and 9M HCl in this research has succeeded in increasing the concentration of Sc_2O_3 obtained.

Residue samples RM-03 and RM-04, resulted from extraction using different HCl concentrations and EDTA as an iron chelating agent, exhibited Sc_2O_3 compositions of 0.07% and 0.08%, respectively. These values are lower than that of RM-02 (0.39%). Similarly, the Fe_2O_3 content was significantly lower in RM-02 (4.78%) compared to RM-03 (13.37%) and RM-04 (10.94%). However, EDTA's iron-chelating ability diminishes with increasing pH. Optimal chelation occurs at pH 1-2 (Zhou et al., 2018). As a result, the Fe_2O_3 content in RM-03 and RM-04 did not differ significantly, suggesting that HCl leaching without EDTA is more effective for increasing Sc_2O_3 content and decreasing Fe_2O_3 content. Based on these findings, 9M HCl was identified as the better leaching solution.

Although Sc_2O_3 extraction was successful, both TiO_2 and Al_2O_3 were remained in the residues. The TiO_2 content increased in all residues compared to the non-magnetic bauxite residue (3.48%). Therefore, further investigation into TiO_2 recovery through flotation can be suggested (Wei et al., 2020).

In contrast, the Al_2O_3 content in all residues decreased significantly compared to the non-magnetic bauxite residue (14.21%), as confirmed by Table 2, which shows that Al_2O_3 is partially soluble in HCl solution. Additionally, all samples, including the non-magnetic bauxite residue, exhibited high SiO_2 content (63.56%, 59.02%, 76.4%, 48.37%, and 59.32%, respectively). This is due to SiO_2's inherent insolubility and limited reactivity with acids other than HF. As a result, the SiO_2 concentration remained relatively unchanged throughout the leaching process (Mulyono, 2005). Consequently, the enrichment of SiO_2 in the flotation bauxite residue suggests its potential as a feedstock for white carbon black production.

CONCLUSION

The extraction of scandium from bauxite residue using HCl concentration with/without addition of EDTA has been carried out. From the research results, it can be concluded that HCl solution successfully leached iron (Fe) from the bauxite residue. A higher HCl concentration led to the attainment of a higher Sc_2O_3 concentration in the residue, suggesting an improved Sc extraction at higher acid concentrations. The addition of EDTA effectively reduced Fe content in the residue (RM-03 and RM-04) compared to leaching with HCl only (RM-01 and RM-02). The 9M hydrochloric acid solution was found as the best leaching solution for obtaining high Sc_2O_3 composition. While EDTA can be used as an iron chelating agent, its effectiveness diminishes with increasing pH during leaching.

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