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EFFECT OF CONCENTRATION SULFURIC ACID ON ALUMINA EXTRACTION FROM NAPA SOIL

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Abstract: West Sumatra is one of Indonesia's provinces with abundant and diverse natural potential. Based on data from the Department of Energy and Mineral Resources, West Sumatra province has a rich potential for metal and non-metallic minerals. Some types of minerals include gold lead (Au), zinc (Zn), manganese (Mn), coal, ironstone, and others. In iron ore, metal minerals contained iron minerals and gangue minerals such as silica and alumina. Napa soil is one of the materials found in West Sumatra containing aluminosilicate with a SiO_2/ Al_2O_3 ratio of 63.20 %/ 16.55 %. It also contains TiO_2, CaO, and K_2O. Extraction of alumina generally using HCl or H_2SO_4 solvent. In previous research, HCl has been used as a solvent in extracting alumina from napa soil. Based on the literature search, no one has used H_2SO_4 as a solvent in the extraction of alumina from napa soil; therefore, it is necessary to conduct research as a material for further study. Therefore, this study will be conducted on variations in the concentration of acid solvents to determine the optimum amount of alumina products in the extraction of alumina in napa soil. In this study, the optimum results obtained alumina extraction with variations in the sulfuric acid concentration at a concentration of 6.0 M, with a % yield of 26.73%. The XRF analysis of alumina extraction showed that the Al2O3 content increased by 72.89%. Characterization of Alumina by FTIR indicated the presence of Al-O, Al-OH.

Keywords: *Napasoil, Alumina, Extraction, Sulfuric Acid.*

INTRODUCTION

West Sumatra is one of Indonesia's provinces with abundant and diverse natural potential. Based on data from the Department of Energy and Mineral Resources, West Sumatra Province has a rich potential for metal and non-metallic minerals. Some types of minerals include gold (Au), zinc (Zn), manganese (Mn), coal, ironstone, and others. In iron ore, metal minerals contained iron minerals and gangue minerals such as silica and alumina[1].

In addition to Al_2O_3, there are other forms of metastable Al_2O_3 structures, such as γ, ρ, χ, θ, η, and κ- Al_2O_3. This type of transformation of Al_2O_3 can result from the heat treatment of aluminum hydroxide or aluminum salts[2].

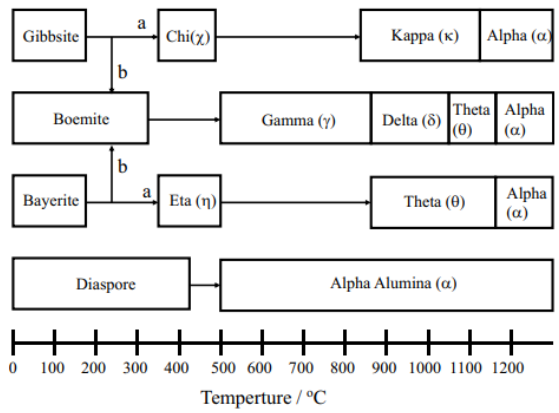

Figure 1. Transition structure of alumina and aluminum hydroxide

Napa soil is a group of aluminosilicate minerals with a very high content of alumina (Al_2O_3) and silica (SiO_2), but it has not been used commercially. Napa soil is also produced by nature from feldspar weathering and is influenced by exogenous energy so that it moves away and separates from the parent rock. Napa soil comes from nature in the form of inorganic solids with a large surface area; from the chemical composition analysis, it contains high amounts of alumina and silica [3].

Napa soil is one of the materials found in West Sumatra containing aluminosilicate with a SiO_2/ Al_2O_3 ratio of 63.20 %/ 16.55 %. It also contains TiO_2, CaO, and K_2O. The availability of napa soil is quite a lot in West Sumatra, such as 50 City Regency, Solok Regency, Pesisir Selatan Regency, Tanah Datar Regency, but until now, its utilization has not been maximized; people only use it as a diarrhea medicine and stomach pain medicine. Therefore, research using napa soil needs to be developed so that this material has a high commercial value as an economic support for local communities[4].

One way that can be done is by extracting alumina from napa soil. Alumina extraction can be done by several methods, such as sol-gel, hydrothermal, and other methods. Based on research conducted[5], it is reported that the extraction of alumina is carried out using an alkaline mixture (NaOH + $Ca(OH)_2$) through the hydrothermal method. This study showed that the extraction ratio of aluminum will increase with increasing reaction temperatures, calcium-silicon ratios, and solid-liquid ratio. The alumina extraction ratio can reach 91.3% by optimizing the washing conditions. A study conducted by ElDebb in 2019 [6 explained the process of

extracting alumina from kaolin ore through a washing process using sodium bicarbonate solution as a washing agent. However, before the extraction process is carried out, a sintering process is carried out to change the phase of kaolin into metakaolin.

Table 1. Extraction Alumina With Different Leachead

Authors	Scheme	Results
(Tripathy et al, 2019)	Leaching using NaOH, followed by leaching using H_2SO_4 + NaF	Product: alumina, with calcium silicate by-products
(ElDeeb et al, 2019)	Leaching Of Sodium Carbonate ($NaCO_3$)	The optimal conditions for sintering kaolin and limestone loads to produce the highest recovery of alumina are 1360 sintering temperatures.
(Cao et al., 2022)	A hydrothermal process does the extraction of alumina from kaolin with NaOH and lime. Washing temperature: 180 for 2 hours	The maximum alumina leaching ratio is 33.64% at 20g /L NaOH.
(Shoppert et al., 2023)	Leaching sulfuric acid with the addition of $(NH_4)_2SO_4$ helps speed up the reaction.	< 20% at temperature (90–110), liquid to solid ratio (5–10), and washing time (15–120 minutes).
(Al-Zahrani, 2009)	The samples were calcined and then extracted using HCl 6M.	Alumina extraction 32%
(Cui et al., 2015)	Calcined samples were extracted using HCl with a S/L ratio of 1/20 at a speed of 500 rpm, and the residue was added to HCl with a ratio of 1/20.	Leaching 90 minutes 45°C wash yields 94.5%

[6]–[11].

The solid-liquid extraction process on napa soil material is done by the acid method, generally using HCl or H_2SO_4 solvent. In previous research, HCl has been used as a solvent in extracting alumina from napa soil [12]. In this study, the largest extraction of alumina was at a concentration of HCl 1.5 M with a yield of 25%. Based on the literature search, no one has used H_2SO_4 as a solvent in the extraction of alumina from napa soil; therefore, it is necessary to conduct research as a material for further study. Therefore, this study will be conducted on variations in the concentration of acid solvents to determine the optimum amount of alumina products in the extraction of alumina in napa soil and alumina characterization with various instruments.

RESEARCH METHODS

Tools

It also has the following features: me-100 JAW CRUSHER 5"X 8" brand crusher, 200 mesh sieve, oven, magnetic stirrer, analytical scale, Ph meter, Buchner funnel, pore oven, kurs, *arloo* glass, 1000 ml Beaker, 250 and 400 ml Erlenmeyer, 10 ml and 25 ml measuring cups, 100 and 250 ml measuring flasks, volume pipette, straw Hatch, stirring rod, and spatula. Then, The instrument is designed based on X-ray fluorescence (XRF) and Fourier Transform Infrared (FTIR) limits.

Ingredients

This study uses the napa soil of South Pesisir Regency, chloride acid, sulfuric acid, sodium hydroxide, aquades, and *whattman* 42 filter paper.

Method

1. Leaching Acid

A total of 80 g of calcined napa soil samples were extracted with 240 mL of H_2SO_4 sulfuric acid solution with a concentration of 2M, 4M, 6M, and 8M within 3 hours at a temperature of 95°C at a speed of 500 rpm the solution will form a slurry, cool for a few minutes then filter using a Buchner funnel the residue is washed with the aquades [13].

2. Addition of NaOH

The sodium aluminate solution is heated to boil the leaching acid Al2(SO4)3 solution with sodium hydroxide (NaOH) 5 M at a speed of 500 rpm, then filtered using a Buchner funnel.

3. Purification Alumina

After filtration, 6 M HCl was added to the sodium aluminate ($NaAlO_2$) solution with stirring to pH 7, and a residue of aluminum hydroxide/ $Al(OH)_3$ was formed, characterized by a white deposition. The mixture is allowed to stand for 18 hours. Then, continue filtering over the Buchner funnel to obtain $Al(OH)_3$.

4. Hydrothermal

Aluminum hydroxide $Al(OH)_3$ has been filtered at a temperature of 110°C oven for 2 hours. To obtain alumina, calcined at a temperature of 1100ºC for 2 hours. Then, the sample obtained is a metal oxide (Al_2O_3).

Research Design

The research design is presented in a flow diagram like figure 2 below.

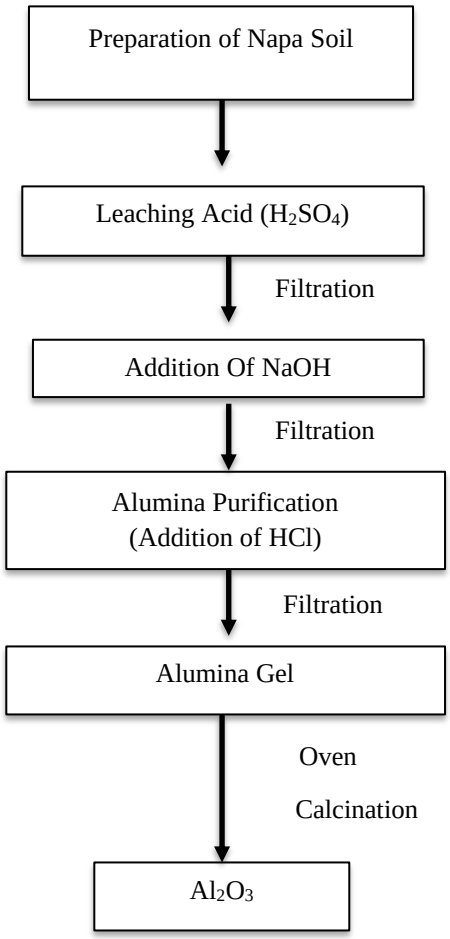

Figure 2. Research Design

RESULTS AND DISCUSSION

The extraction of alumina from Pesisir Selatan napa soil minerals using leaching acid extraction with sulfuric acid is carried out in two stages. The first stage is the preparation of Pesisir Selatan napa soil, and the second is extraction. In the first stage, napa soil is prepared by grinding and continued calcination. In the first stage, napa soil is prepared by grinding and continuing calcination. Grinding aims to change physical napa soil from chunks into fine powder with a fineness of 70 µm[10].

Effect Of H_2SO_4 Concentration On Yield

The extraction process using sulfuric acid varies, as shown in Table 2.

Table 2. Effect Concentration H_2SO_4

Yield weight (g)	Concentration H_2SO_4(M)			
	2.0	4.0	6.0	8.0
	10.92	15.77	21.39	18.65

The relationship between concentration and alumina yield can be quantitatively known from the calculation of research data. Variations in the concentration of sulfuric acid determined in the study showed that the higher the sulfuric acid concentration, the greater the weight of the resulting alumina yield. The higher the sulfuric acid concentration, the more H+ ions are contained, so the Al atoms are also more soluble. The presented graph of the effect of H_2SO_4 concentration on the yield is shown in Figure 3.

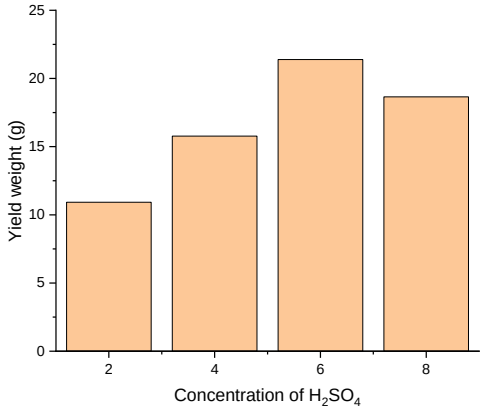

Figure 3. Effect Of H_2SO_4 Concentration On Yield

It is seen that alumina extraction increases at pH 6 and decreases after pH 6. At a concentration of 2.0 M of sulfuric acid, we obtained alumina weighing 10.92 grams with a percentage of 13.65%. At a concentration of 4.0 M, we obtained alumina sulfuric acid weighing as much as 15.77 grams with a percentage yield of 19.71%. At the concentration of sulfuric acid affected by 6.0 M, there was a significant increase in the extraction results obtained, reaching as much as 21.39 grams with a percentage of 26.73%. But at pH 8.0, sulfate masala decreased, and alumina produced only as much as 18.65, with a percentage of 23.31%.

Characterization by X-Ray Fluorescence

The comparison of oxide mineral composition before and after alumina extraction is shown in Table 3.

Table 3. Comparison Of Mineral Composition Between Before And After

Sample	Contain (%)				
	Al_2O_3	SiO_2	Fe_2O_3	CaO	MgO
S1	28.78	59.38	3.96	0.37	1.08
S2	31.16	62.70	4.04	0.24	1.14
S3	72.98	0.81	0.02	0.97	-

Description:
S1: Napa Soil Before Calcination
S2 : Napa Soil After Calcination
S3 : Alumina

In the stage of extraction, alumina is by four steps:
1. Leaching acid

At this stage, alumina will react with acids such as hydrochloric acid or sulfuric acid. $Al_2O_3 + 3H_2SO_4 \rightarrow Al_2(SO_4)_3 + 3H_2O$ [14] Products of leaching acid, such as slurry, which contains very fine passive silica and $Al_2(SO_4)_3$, are then separated by filtration and washed with distilled water.

2. Alkali Fusion

The alkaline fusion method is a method of alumina extraction in which NaOH is used as a solvent to precipitate elements such as Fe, Ca, Ti, and others at pH 13.

$Mn^+ + nOH^- \rightarrow M(OH)n\downarrow$ [15]

$Al^{3+} + 4OH^- \rightarrow AlO_2^-$ [16]

3. Precipitation

Alumina that has been extracted after forming sodium aluminate purification by adding HCl to form pure alumina [17].

4. Hydrothermal

Hydrothermal is a technology for crystallizing materials (chemical compounds) directly from aqueous solutions by control (temperature, pressure, and composition) [18].

It can be seen from Table 3. that after calcination, levels of Al_2O_3, SiO_2, and Fe_2O_3 increased, while levels. CaO decreases after calcination. On the extraction process alumina, Al_2O_3 levels increased, while SiO_2 levels disappeared.

The analysis with the XRF test method in Table 3 showed that the sample of S3 (alumina extract) contains considerable levels of Al_2O_3 (72.98%), while SiO2 has lost levels. With the loss of SiO_2 levels, it was concluded that this study has succeeded in taking and separating alumina from - other content, especially the mineral SiO_2. Alumina levels have increased due to impurities such as insoluble silica occurring simultaneously in the leaching acid process so that these silica impurities are left in the residue.

Referring to Table 3 states that Mineral impurities other than Al contained in the extraction.

Alumina decreased due to when done. Acid washing 4 times per 100 ml, minerals such as Si, Fe, Ca, Mg, and other impurities remain in the residue. In addition to the acid washing factor, acid solvents sulfuric are also free from impurities: others, especially Si, whose content is quite large.

Characterization by Fourier Transform Infra-Red

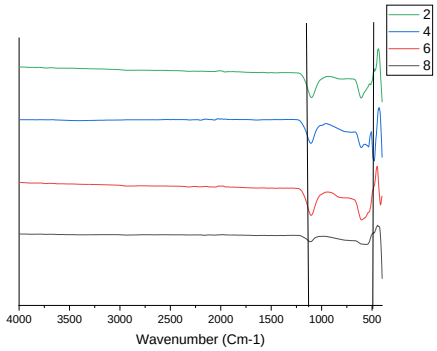

Figure 4. Spectra FTIR Alumina

FTIR characterization carried out the identification of functional groups from the extraction of alumina (Al_2O_3). Several absorption peaks appear in the infrared spectrum in each independent variable set. The wavenumber 4000-1250 cm^{-1} shows the vibrational absorption bands of –OH groups that bind octahedral Al atoms; vibration of Al-O bond elongation appears in the wavenumber region 850-650 $cm^{-1,}$ which has a symmetrical range [19]. Based on some literature, it states that the peak of vibration absorption and bending of Al-O is found in the wavenumber area of 738 cm-1 and 620 cm^{-1}[20]. In other literature, the rise of IR absorption in the 775 cm-1 and 590 cm-1 wavenumber is the vibration and bending of Al-O.

CONCLUSION

In this study, the optimum results obtained alumina extraction with variations in the sulfuric acid concentration at a concentration of 6.0 M, with a % yield of 26.73%. The sample of S3 (alumina extract) showed considerable levels of Al_2O_3 (72.98%), while SiO2 lost levels. Characterization of Alumina by FTIR indicated the presence of Al-O, Al-OH. Alumina that has been extracted will be used as a modifier to manufacture electrodes in the voltammetry method.

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