

Laboratory Preparation of Alumina From Auchi kaolin

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ABSTRACT

In this study, the potential of extracting pure alumina from kaolin clay was examined. The kaolin clay was collected from Auchi, in Edo State, Nigeria. The ore was dug with a shovel and stored in bags for further processing. The kaolin sample was calcined at 850°C and held for 2 hours, the raw and the calcined kaolin were analysed using the XRD, XRF and FTIR to determine the percentage chemical composition. Loss on ignition, bulk density, water absorption, and dry and fired shrinkage was also determined on the sourced clay and the result recorded. 3kg of the calcined sample was placed in a jar mill and milled for 6 hours to obtain a very fine powder. 10g Sample of the milled calcined kaolin was combined with 100 ml of a 6 M HCl and placed in a conical flask and then placed on a digital magnetic stirrer at 90°C, 500 rpm for 4 hours to obtain a slurry. The resultant slurry was then diluted with 100ml of distilled water and this is filtered through a funnel to obtain AlCl₃. The filtrate was heated on a magnetic stirrer to a temperature of 90°C, drops of solution of 5M NaOH solution was added continuously to convert it into NaAlO₂. After filtration, 6 M HCl was added to the NaAlO₂ filtrate and stirred to bring the pH to 7, giving alumina. The extracted alumina was white and has a purity of 91.44%.

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I. Introduction

Kaolin, a naturally occurring clay mineral, is made up largely of silica (SiO₂) and alumina (Al₂O₃). It is widely distributed and used as a raw material in a variety of industrial applications because of its abundance and low cost (Kumari & Mohan, 2021). Despite its initial inertness, kaolin can be activated to extract important compounds like alumina, which is required for the production of aluminum and other industrial minerals (ElDeeb et al., 2021). The extraction of alumina from kaolin is a multistep process that begins with the material being activated through calcination. Calcination changes the crystalline structure of kaolin, resulting in metakaolin, an amorphous aluminosilicate with active chemical bonding (ElDeeb et al., 2021). This change is critical because it improves the reactivity of the material, making it more suitable for future extraction operations. Once kaolin has been transformed into metakaolin, the alumina can be separated via acid leaching. During leaching, acids such as hydrochloric acid (HCl) or sulfuric acid (H₂SO₄) are used to dissolve the alumina from the metakaolin structure, yielding an alumina-rich liquid and silica-rich solids (Tantawy and Alomari, 2019). This procedure is extremely reliant on a number of parameters, including acid concentration, temperature, and duration, all of which affect extraction efficiency and yield. The alumina-rich liquid obtained during the leaching process can then be further processed to recover pure alumina, which is required for aluminum production. In contrast, silica-rich solids can be used to synthesize zeolites and other useful minerals (Rahayu et al., 2018). Extensive research has been conducted to optimize the conditions for calcination and leaching, with the goal of increasing the efficiency of alumina extraction from kaolin. Innovations in this sector aim to increase the process's economic viability and environmental sustainability, making it an important area of research in industrial mineral processing (Kyriakogona et al., 2017). The alumina-rich liquid obtained from the leaching process can then be further processed to extract pure alumina, which is essential for producing aluminum. The silica-rich solids, on the other hand, can be utilized in the synthesis of zeolites and other valuable materials (Rahayu et al., 2018).

II. MATERIALS AND METHODS

MATERIALS

The various materials used for this project are

- Kaolin
 - Acids (HCl)
 - NaOH
-

- Beaker, round bottom flask, conical flask
- Heater
- Bursen burner
- Filter paper
- Stirrer
- Kiln
- FTIR, XRD, XRF machine

Experimental procedure

The kaolin considered in this study are sourced in Auchi Edo state Nigeria. The sites were visited and the kaolin were excavated with the use of shovel and stored in bags for further processing. The kaolin mineral nature varies from hard stone to soft mud depending on the accessory minerals. Kaolin sample was crushed, ground and screened to particles size below 90 μm . The kaolin was placed in a muffle kiln and calcined to a temperature of 850°C and held for 2hrs and was allowed to cool till the next day.

The screened raw sample of the kaolin and the calcined sample of the kaolin were presented for XRD, XRF and FTIR test and the result recorded. Samples of the clay were made into small cylindrical shapes and allowed to dry for a week and the weight determined. The test samples were fired to a temperature of 800°C and allowed to cool till the next day. The samples loss on ignition, bulk density, water absorption, and dry and fired shrinkage were determined and the results recorded.

3kg samples of the calcined kaolin fired at 850°C was placed in a jar milled and ground for 6 hours to obtain a very fine slurry. The slurry is the allowed to settle till the next day, decanted and dried to obtain a powder.

PHYSICAL TEST CARRIED OUT ON THE KAOLIN SAMPLE

Loss of ignition

Clay is a natural material made primarily of minerals, water, and organic components. When clay is exposed to high temperatures, such as during ignition or fire, it goes through a series of physical and chemical changes. When clay is heated, it loses weight, known as loss on ignition (LOI). The LOI of clay is mostly produced by the removal of water and organic molecules. To evaluate the loss on ignition of the sample clays, they were weighed and dried in an oven at 100°C for 2 hours. The dried samples were weighed and the weight recorded M1, which was then placed in a crucible and fired to a temperature of 800, 850, and 900°C and allowed to cool until the next day before it was then reweighed M2

$$LOI = \frac{\text{Dryweight} - \text{firedweight}}{\text{Dryweight}} \times 100$$

$$M1 - M2/M1 \times 100$$

Bulk Density

Bulk density, which measures a material's mass per unit volume, is commonly given in grams per cubic centimeter (g/cm^3) or kilograms per cubic meter (kg/m^3). To determine the bulk density of the clay samples, they were shaped into cylindrical kaolin bricks. The bricks were oven-dried at 90°C for 3 hours. The dry weight (WD) of the specimens was properly documented.

The dried cylindrical kaolin bricks were then burned in a furnace at 900°C. The mass (M), diameter (D), and height (H) of the burnt cylindrical kaolin bricks were measured with a scale and a ruler, and each value was recorded. The bulk density of the clay was then estimated using the following formula:

Bulk Density= Mass/Volume

Where M represents the mass of a sample and V is the volume of the cylindrical kaolin brick. The volume (V) is calculated using the formula $V=\pi r^2h$, where r is the radius and h is the height of the cylindrical kaolin brick.

Linear Dry shrinkage test

The linear dry shrinkage test is used to measure how much a clay specimen changes in size as it dries. Samples of clay were initially ground and allowed to air-dry for a few days. Using a weighing scale and a ruler, the mass and volume of water in each sample were calculated, and the measurements were noted. After that, the clay samples were formed into a bar and each one had a reference line marked on it before it was left to dry for five days outside.

The original parameters of the clay bar were measured and recorded using a ruler to determine its length (L). The clay bar was then placed in an oven for two hours, with the temperature adjusted to 90°C. The drying procedure was applied to the clay bar until it reached a constant length, which is a sign of full drying. Following full drying, the clay bar's final measurements were taken and recorded as L₂.

The linear dry shrinkage can be determined using the formula:

$$\text{Linear Dry Shrinkage} = \frac{L_1 - L_2}{L_1} \times 100$$

$$L_1 - L_2 / L_1 \times 100$$

Linear fired shrinkage test

The fired shrinkage test is used to measure how much a clay specimen's dimensions change following the calcination process. This test is very important in ceramics because it tells you how much a clay body has shrunk throughout the fire process. Four-square clay bars were used as samples to calculate linear dry shrinkage. The test was started by placing the clay bar samples in an oven that was preheated to 900°C and keeping them there for two hours. The clay bars then went through the fire process and were left alone until the next day so that they could cool. The ultimate dimensions of the clay bars were measured and recorded as L₃ after full burning and chilling.

Linear fired shrinkage test is calculated using the formula

$$L_2 - L_3 / L_2 \times 100$$

Water Absorption

The pace at which water is absorbed is determined by the percentage of water absorption. This characteristic is significant because it shows how much moisture the clay can take in without crumbling. Once a consistent weight was achieved, the test specimens created from the kaolin samples were dried in a secure and cool environment. Using a weighing balance, the samples were weighed sequentially and given the designation W₁. After that, each sample was placed in water one at a time and left to soak. After removing the samples for a full day, the remaining water was scraped off using a damp cloth, and the specimens were weighed once more W₂. Both the soaking and dried weights were measured and noted. The following formula was used to determine the percentage of water absorption:

Where: Dry weight = W₁ Soaked weight= Wet weight =W₂

$$\text{Water Absorption} = \frac{W_2 - W_1}{W_1} \times 100.$$

Leaching procedure

To create an extremely fine powder, 3 kg of the calcined sample was put in a jar mill and processed for 6 hours. To create a slurry, 10g of the milled, calcined kaolin sample was mixed with 100 ml of 6 M HCl, put in a conical flask, and stirred at 500 rpm for four hours at 90 °C using a digital magnetic stirrer. After diluting the resulting slurry with 100 milliliters of distilled water, it was filtered through a funnel to produce AlCl₃. To turn the filtrate into NaAlO₂, drops of a 5M NaOH solution were added after it was heated to 90°C on a magnetic stirrer. Following filtration, the NaAlO₂ filtrate was mixed with 6 M HCl and shaken to raise the pH to 7, which produced alumina and the result send for XRF test.



Figure1 :Calcined sample fired in the kiln



Figure 2: Samples in the beaker for leaching

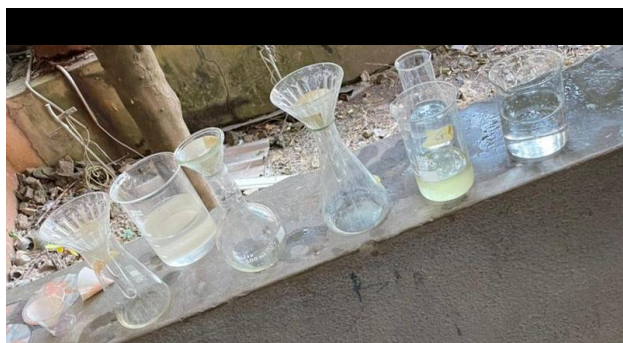


Figure 3 : Dealumination process of the samples



Figure 4: Result of obtained alumina from the filter paper

III. RESULTS AND DISCUSSION

PHYSICAL PROPERTY TEST RESULTS

Table 1 : Sample L.O. Idetermination

Samples	Mass Before firing (g)	Mass After Firing (g)	L.O. I
Auchi	500	434	13.2

Table 2: Determination of Bulk Density of the sourced clay after firing

Source	Length of a fired sample (h=cm)	Radius of fired sample (cm)	Mass of fired sample (g)	Volume of fired sample ($V = \pi r^2 h$)	Bulk density of fired sample (mass/volume)
Auchi	2.9cm	1.4	25.858	17.85	1.5

Table 3: Percentage water absorption determination of the sourced clay sample after firing.

Source	After firing weight (A)	Soaked weight (A)	% water Absorption (A)	After firing weight (B)	Soaked weight (B)	% water Absorption (B)
Auchi	25.058	32.144g	28.27	25.858	32.554g	25.89

Table 4: Determination of Bulk Density of the sourced clay after firing

	Length of fired sample (h=cm)	Radius of fired sample (cm)	Mass of fired sample (g)	Volume of fired sample ($V = \pi r^2 h$)	Bulk density of fired sample (mass/volume)
Auchi	2.8	1.4	25.058	17.243	1.50

Table 5 : Determination of linear dry shrinkage of the sourced clay after firing

	Moist length (cm)	Sample 1 (cm)	% Dry shrinkage	Sample 2 (cm)	% Dry shrinkage	Average percentage dry shrinkage rate (cm)
Auchi	10	9.1	9	9.1	9	9

Table 6 :Determination of fired shrinkage of the sourced clay after firing

	Dry length Sample 1 (cm)	Fired length sample 1 (cm)	% fired shrinkage	Dry length Sample 2 (cm)	Fired shrinkage sample 2	% fired shrinkage	Average percentage dry shrinkage rate (cm)
Auchi	9.1	7.8	14.28	9.1	7.9	13.18	13.73

Table 7. Composition Analysis (oxide composition) in weight percent of Auchi kaolin by XRF

	SiO ₂	TiO ₂	MgO	Fe ₂ O ₃	MnO	K ₂ O	Al ₂ O ₃	
Raw Sample	63.698	2.808	-	4.119	0.013	-	25.678	
Calcined sample	60.768	2.465	-	0.248	0.031	2.690	27.319	

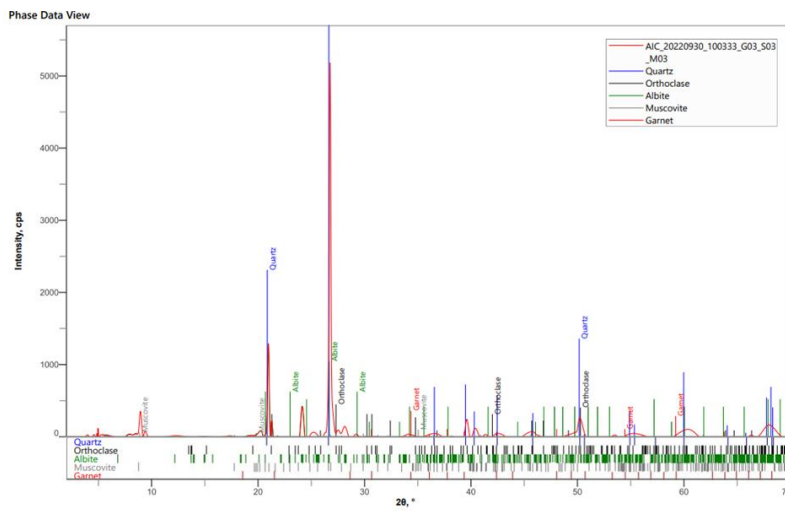


FIGURE 5: XRD result of Auchiclained clay

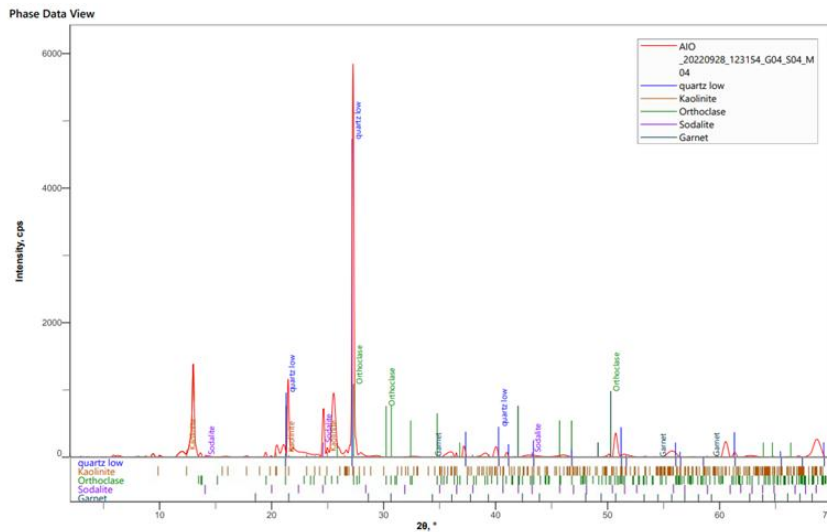


FIGURE 6: XRD result of Auchi raw clay

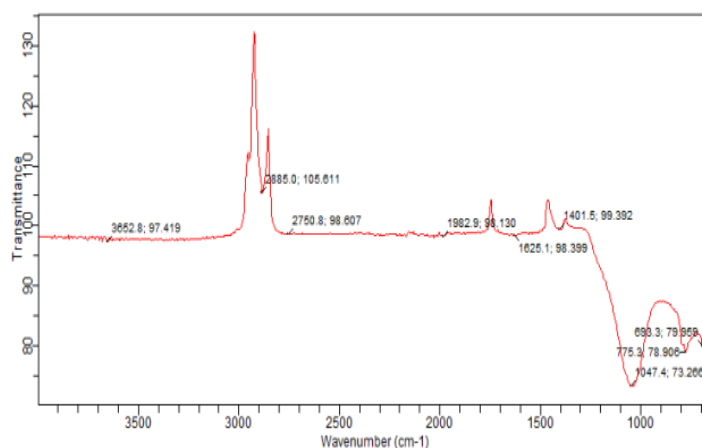


FIGURE 7: FTIR result of Auchicalcined clay

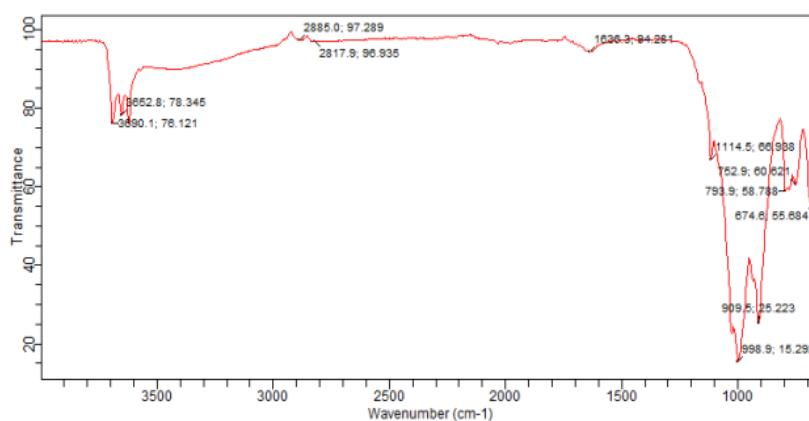


FIGURE 8: FTIR result of Auchi raw clay

Table 8. Composition Analysis XRF (oxide composition) in weight percent of Alumina Obtained from Auchi Kaolin sample.

Material	SiO ₂	Cl ₂	MgO	K ₂ O	Fe ₂ O ₃	Al ₂ O ₃	
Composition	0.98	3.45	-	0.190	0.14	91.44	

IV. Discussion:

The XRD results in Table 7 demonstrate that the percentage alumina concentration of Auchi kaolin raw and calcined samples is 25.678 and 27.31, respectively. The percentage of alumina obtained from the kaolin as provided by (Alafara 2020) is a suitable kaolin for alumina extraction. The increase in alumina percentage in the calcined kaolin is caused by the burning off of other organic compounds found in the kaolin at the site. Calcination increases the proportion of alumina in calcined kaolin samples.

Figures 7&8 show the results of the FTIR. When the results were compared to Mohammed's (2019) FTIR results, it was discovered that in the FTIR spectrum of kaolin, the band at 1105 cm⁻¹ is due to the asymmetric stretching vibrations of Si-O-Si, and the band at 1025 cm⁻¹ is due to the alternating stretching vibrations of Si-O-Si and Al-O-Al. The 908 cm⁻¹ band is created by bending Al-O-H vibrations. The symmetric Si-O-Si stretching vibration corresponds to the band at 683 cm⁻¹.

The calcined white powder was characterized using X-ray fluorescence. The chemical analysis using XRF showed that alumina is the predominant ingredient. The X-ray fluorescence in Table 8 shows γ -alumina as the main ingredient, with traces of SiO₂, K₂O, and Fe₂O₃. The XRF analysis highlights the presence of Cl-phase in the γ -alumina, indicating inadequate product washing prior to characterization. According to Table 8, the Al₂O₃ wt.% extraction in the final product is 91.44%, which is greater than the alumina extraction (63%) reported by (Ibrahim et al., 2018). The increase in surface area due to serious milling of the calcined kaolin before leaching might cause the increase alumina content. The impurities in γ -alumina are related to the presence of Cl₂ at 3.45 wt.% along with traces of SiO₂ (0.98%), K₂O (0.190) and Fe₂O₃ (0.14%).

V. Conclusion

This study highlights the leaching process as a superior method for extracting alumina from kaolin. The methods discussed demonstrated alumina extraction rates exceeding 85–95%. This efficiency is considered favorable and effective, offering an alternative to the Bayer process. The simplicity of the leaching process, requiring fewer personnel, lower energy input, and yielding high surface area and purity, is strongly endorsed. It is noteworthy that the widespread availability of kaolin, coupled with the high alumina content in most kaolin, underscores the importance of initiating commercial alumina production from kaolin through the leaching method, particularly for industrial applications such as refractories.

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