

COMPARATIVE STUDY OF ALUMINA AND IRON OXIDE EXTRACTED FROM AKU CLAY BY HYDROCHLORIC ACID LEACHING

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Abstract: The extraction of alumina and iron oxide from Aku clay by hydrochloric acid leaching was carried out. The raw Aku clay was calcined and both samples were characterized to determine and compare their physicochemical properties, and then analyzed using atomic absorption spectrophotometer to determine the metallic ions present in the clay. Both raw and calcined clay samples were leached by mixing each sample with adequate amount of hydrochloric acid of required concentration. The leached liquor was then collected and filtered using Whatman filter paper; after which 5mls of the filtrate was then withdrawn and analyzed using AAS to determine the amount of alumina and iron oxide present. SEM image taken before and after acid treatments revealed some cracks in the residual clay; indicating that leaching was effective and successful. The results obtained showed that Aku clay contained an appreciable amount of alumina and iron oxide.

Keywords: Aku Clay, Leaching, Alumina, Iron Oxide, Hydrochloric Acid.

1. INTRODUCTION

The discovery of aluminium and iron have given industrial production and manufacturing a great boost as this was a major improvement over the use of stone and wood for construction (Udeigwe *et al.*, 2015). Normally, Alumina and iron oxide are produced from bauxite and iron ore respectively. However, due to the fact that the amount of these raw materials in Nigeria are not sufficiently abundant to meet up with producing the required amount of iron and aluminium, and because of high volume of waste generated from the production of alumina from bauxite, there is need to search for alternative but sufficient local raw materials from which iron oxide and alumina can be extracted.

One of the numerous raw materials distributed on a large scale in the country is clay (Ogbuagu *et al.*, 2007). It is abundant and can be found virtually in all states of Nigeria. Clay is one of the raw materials that have much of alumina and iron oxide. Ajemba and Onukwuli (2012) noted that most clays contain about 25-40% alumina and up to about 30% iron oxide. Clays are essentially alumina/silicates which have resulted from weathering of rocks. Clays have high affinity for silica and because of these, it is difficult to separate alumina from silica and this presents the big problem in treatment of clays (Udeigwe *et al.*, 2015).

There are various chemical procedures adopted over the years for the treatment and separation of alumina from silica, and iron oxide from iron ore. Most researchers have used different sintering and acid-extraction processes for the leaching of alumina and iron oxide from clays. Ajemba and Onukwuli (2012), reported that the use of some inorganic and organic acids for alumina dissolution (leaching) is feasible, efficient and sustainable. Researches conducted over the years have shown that hydrochloric acid is more advantageous over other acids especially in leaching alumina.

Nigeria has enormous clay deposits with high alumina and iron oxide content. Aku clay from Aku community in Igbo-Etiti local government area of Enugu state, has the potential of providing large amount of alumina and iron oxide, if harnessed properly. The research is therefore aimed at evaluating the viability of Aku clay for alumina and iron oxide production by hydrochloric acid leaching.

Leaching is the process of extracting soluble constituents from a solid by means of a solvent (Coulson and Richardson, 2002). In the chemical industry, it has a variety of commercial applications, including separation of metals from their ores using acid, and sugar from sugar beets using hot water.

2. MATERIALS AND METHOD

2.1 Materials and Equipments:

The local clay used in this work was obtained from Aku community in Enugu state. The materials used in the experimental work include distilled water, sodium hydroxide and hydrochloric acid. The equipment used in this work are pestle and mortar, muffle furnace, oven, sieves, glass beakers, conical flasks, stop watch, scanning electron microscope (SEM), atomic absorption spectrophotometer (AAS), pycnometer, filter paper, round bottom flask fitted to a magnetic stirrer and pipette.

2.2 Methods:

2.2.1 Preparation of clay:

The raw clay was mined manually and also manually separated from the debris. The clay was sun dried for 2 days and later oven dried at 105°C for 1hour. It was then ground and sieved using 100µm sieve. The clay was calcined at temperatures between 600°C and 1000°C for a period of 1hr.

2.2.2 Leaching process:

The calcined clay and the raw clay both at 100µm particle size and 50°C leaching temperature underwent leaching process by mixing each sample with 3M acid concentration and 8cm³ acid/1gm clay liquid to solid weight ratio. The mixture was then stirred at a constant speed of 500rpm at 60minutes contact time. After each leaching process, the leached liquor was collected and filtered using Whatman filter paper. 5mls of the filtrate was then withdrawn and analyzed using AAS to determine the amount of alumina and iron oxide present. The remaining filtrate was used in the extraction of alumina and iron oxide.

2.2.3 Extraction of iron oxide:

Iron oxide was extracted according to the method of Rasmita and Mamata (2011). The filtrate was introduced into a 250ml conical flask. The pH meter was calibrated using buffer solutions of the following, pH 4, pH 6.8 and pH 9.8 as supplied by the manufacturer. The meter was initially used to determine the pH of the solution. It was found to be acidic. 1N sodium hydroxide solution was added to the leachate in drops until the pH was brought to 10. As the pH of the medium increased, reddish brown precipitates of iron (III) hydroxide were crystallized. The solution was filtered and the crystals collected in filter paper. The crystals (iron (III) hydroxide) were washed with 50ml of distilled water and dried in laboratory hot air oven. The whole dried residue was transferred to a porcelain crucible and converted to ash in a muffle furnace at 600°C for 4 hours. The iron (III) hydroxide was then converted to iron (III) oxide.

$$\% \text{ Iron oxide} = \frac{\text{wt of iron oxide crystallized out}}{\text{theoretical weight of iron oxide present in the raw/calcined clay}} \times 100 \quad (1)$$

2.2.4 Extraction of alumina:

Alumina was extracted according to the method of Ajemba and Onukwuli (2012). The filtrate from which iron (III) hydroxide was crystallized out was evaporated to dryness and the residue ignited at 1100°C to form alumina (Al₂O₃) and it was weighed. Aluminum ion was confirmed according to the method of Larson (2008). The dried residue was dissolved in 0.5M nitric acid. 0.5M barium chloride solution was added in drops, which gave a white precipitate. 1M sodium hydroxide solution was added in drops and warmed with powdered aluminum. The fume given off was tested with damp red litmus paper, which turned the litmus paper blue.

$$\% \text{ Alumina} = \frac{\text{wt of residue}}{\text{theoretical weight of alumina present in the raw/calcined clay}} \times 100 \quad (2)$$

2.3 Characterization of raw and calcined clay samples:

The physical properties of the raw and calcined clay samples were carried out using ASTM D7263-09(2017) methods. Atomic absorption spectrometry (AAS) was used to measure the concentration of elements present in the sample. Atomic absorption is so sensitive that it can measure down to parts per billion of a gram ($\mu\text{g dm}^{-3}$) in a sample. Scanning electron microscopy (SEM) was used to determine the size and morphology of the clay sample. The SEM micrograph was obtained using JOEL scanning electron model JSM 6400. Scanning electron microscopy recorded at 15KV with 8000x magnification.

2.4 Characterization of the leached extracts:

The leached extracts were analyzed using atomic absorption spectrophotometer (AAS). The sample in liquid form was analyzed at the wavelengths of 309.3nm for aluminium and 248.3nm for iron. Atomic absorption spectrophotometer works with cathode lamps. Each metal has a cathode lamp that provides the energy to absorb the atomized sample.

3. RESULTS AND DISCUSSIONS

3.1 Physico-chemical characterization and properties of the raw and calcined clays:

Physico-chemical properties of the raw and calcined clay samples are presented in Table 3.1.

Table 3.1: Physico-chemical characterization of raw and calcined clay samples

Sample	pH	Surface Area	Bulk Density (g cm^{-3})	Carbon (%)	Organic Matter (%)	Loss on Ignition (%)	Particle Density (g cm^{-3})	Colour	Total Porosity (%)
Raw Clay	5.66	768.30	1.79	2.03	6.01	9.50	2.33	Light Brown 8/1	23.60
Calcined Clay	7.50	847.15	1.91	1.22	3.63	11.50	3.55	Brownish gray 4/1	46.25

Table 3.1 shows that most of the properties increased after calcination except carbon and organic matter. According to Gray et al. (2014), the porosity increases after calcination due to increase in bulk density; thus enabling the leaching solvent to easily permeate the clay and enhance leaching.

3.2 Atomic absorption spectrophotometer (AAS) characterization of the clay samples:

The AAS characterization was performed to identify and quantify the chemical composition of the minerals present in the clay sample.

Table 3.2: The AAS characterization of raw and calcined Aku clay samples

Component	Composition (mg/g) of raw Aku clay	Composition (mg/g) of calcined Aku clay
Al_2O_3	30.75	31.5
SiO_2	10.95	11.00
Fe_2O_3	24.8	25.1
CaO	0.64	0.65
MgO	0.46	0.49
Na_2O	1.85	1.89
TiO_2	1.65	1.69
Total	71.1	72.32

Table 3.2 shows that the major substances in raw and calcined Aku clays are alumina, silica and iron (iii) oxide; while there are traces of other metals. In comparison, the result shows that the essential metals in the raw clay slightly increased after calcinations, and this could be as a result of reduction in the carbon and organic matter content during this process.

3.3 SEM results for raw, activated and leached clay samples:

Morphology or structural compositions of the solid sample could be seen through the use of SEM as shown in Figures 3.1, 3.2 and 3.3. Figures 3.1, 3.2 and 3.3 revealed that clay is a bulk of microstructure, which in turn is composed of homogeneously distributed network of small filamentous crystals; indicating presence of minerals.

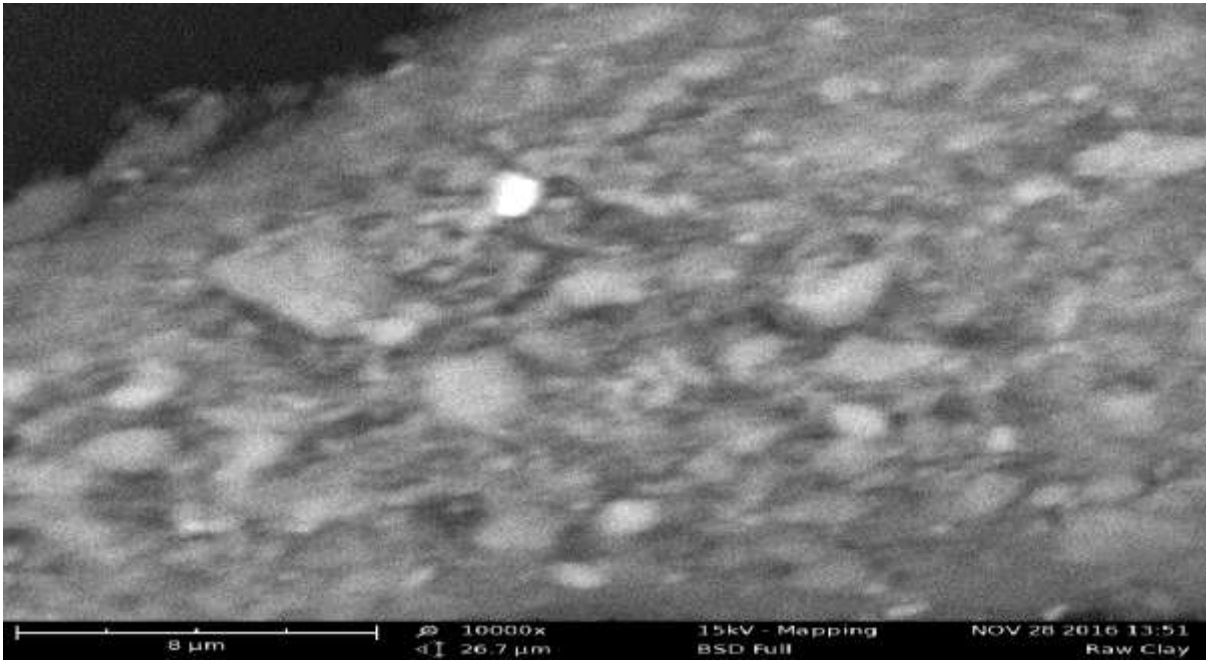


Fig 3.1: Result of SEM for the raw clay sample

From plate 3.1, it could be seen that the pores are more opened after the calcinations process

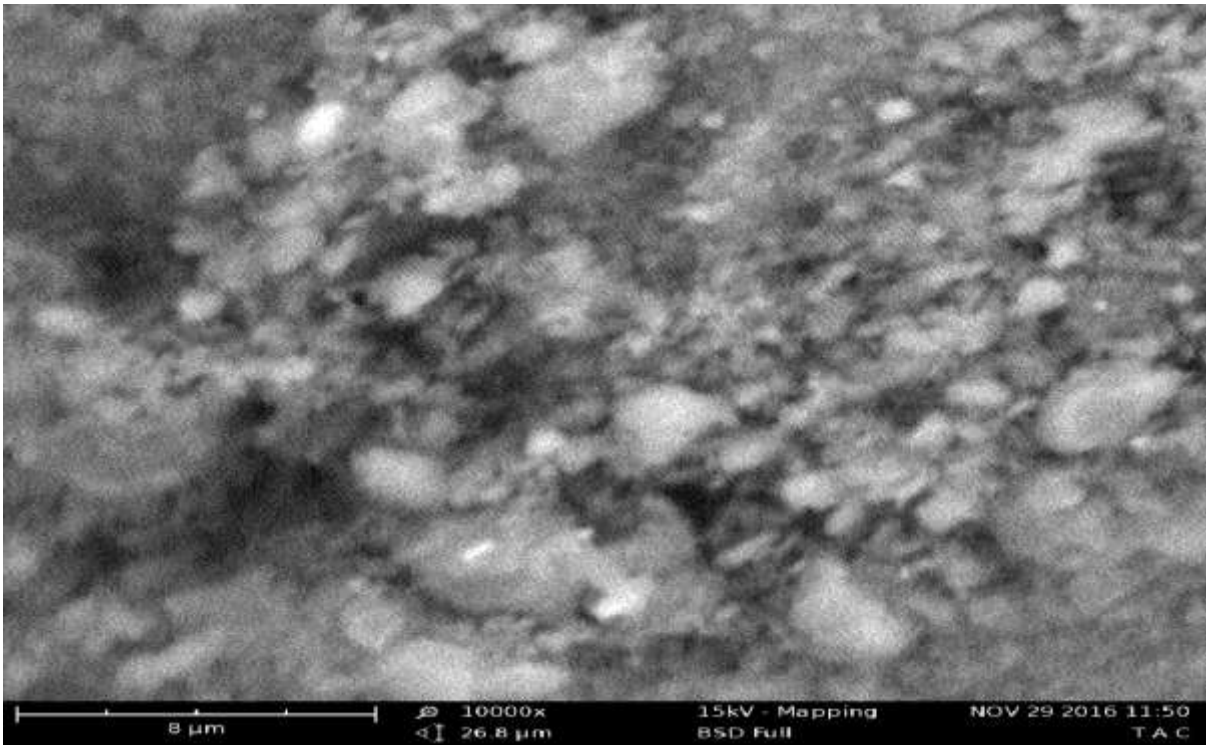


Fig 3.2: Result of SEM for the activated clay sample

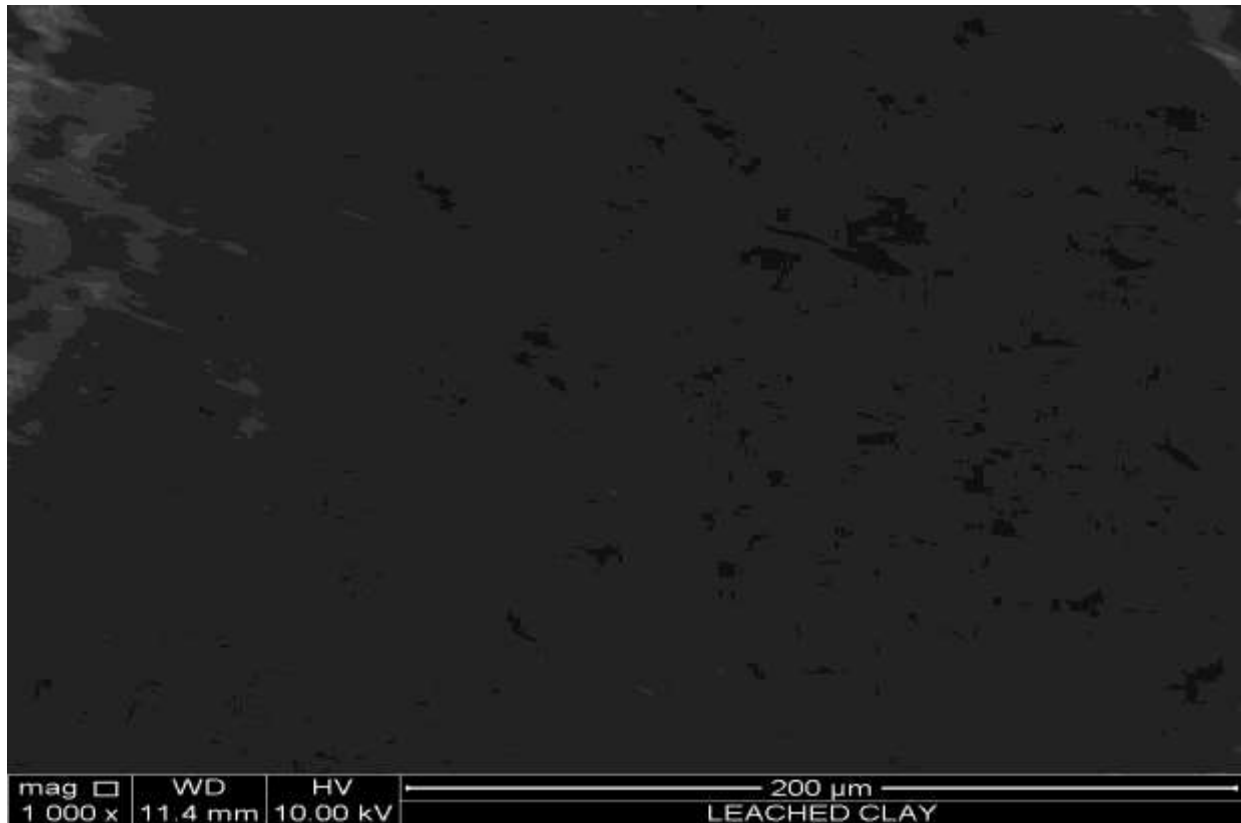


Fig 3.3: Result of SEM for the leached clay sample.

From plate 3.3, it could be observed that the pores became shrunk after leaching showing that some of the metallic oxides were leached out during this process.

4. CONCLUSIONS

The result showed Aku clay has high abundance of alumina and iron oxide; implying that it is a good alternative source of alumina and iron oxide for industrial application. The characterization results revealed that both raw and calcined Aku clay samples are rich in alumina and iron oxide deposits, though the process of calcination increased the purity and the amount of the major elements in the raw clay sample. The increase in many of the components of the clay after calcination process revealed that it was necessary to increase the properties of the clay while reducing its carbon or organic matter content. Calcination was necessary in increasing the porosity; thus enabling the leaching solvent to easily permeate the clay and enhance leaching. The results also revealed that Aku clay contains more alumina than iron oxide, and that hydrochloric acid is very suitable for its leaching.

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