

# Physicochemical And Mineralogical Characterization Of Use Abat, Ibiono Ibom Clay For Industrial Application

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**Abstract**—This paper examined physicochemical and mineralogical characterization of Use Abat, Ibiono Ibom clay for industrial application. 5kg each of the clay was obtained from both Ikot Usan and Ikot Edet deposits. The clays were processed into powder form and used for the analyses. They were characterized using X-ray diffraction (XRD), X-ray fluorescence (XRF), and physical analysis. The XRD and XRF analysis of sample A gave Quartz, Halloysite, and Strengite while sample B gave Quartz, Nacrite-1Md, and Berthierine-1M. ASTM specifications were used for the physical analysis and gave average results for sample A, Atterberg test using ASTM-D4318 (liquid limit test-51.14%, plastic limit-31.69%, shrinkage test-15.96%), natural moisture content using ASTM-D2216 (20.14%), compaction test moisture content (24.59%) using ASTM-D4718/D4718 specifications, particle size (39.1g) using ASTM-D3740. The physical analysis of sample B gave Atterberg test (liquid limit test-61.63%, plastic limit-38.28%, shrinkage test-16.07%), natural moisture content (22.15%), compaction test moisture content (23.06%), particle size (39.1g).

**Keywords**—Clay Minerals, Clay Characterization, Kaolin, X-ray diffraction (XRD), X-ray fluorescence (XRF)

## 1.0 INTRODUCTION

This research location, Use Abat village, is in Ibiono Ibom Local government of Akwa Ibom State, Nigeria. The community is largely inhabited by farmers and petty traders and the clay deposit is largely unexplored because the terrain lies in valleys, making it difficult to access.

Meanwhile, the most visible investment in clay processing in Akwa Ibom State are the now defunct Quality Ceramics Ltd in Ikot Ebom, Itam, and the Nigeria Newsprint Manufacturing Company (NNMC) at Oku Iboko, both in Itu Local Government Area. A sustainable effort in reviving these two major industries will require ensuring the availability of raw materials to sustain production and this will include exploring the clay deposits in communities such as Use Abat, to complement other available clay deposits in the State.

The Nigerian economy has been largely dependent on crude oil, but the current economic realities have called for diversification and investment in the country's abundant solid minerals sector. Nigeria has a considerable wealth of natural resources with an estimated reserve of over 50 proven minerals [1]. Geological survey of the country's mineral deposits has proven the country to be rich in Tinstone, Columbite, Limestones, Bitumen, Lead-zinc ores, Coal, Clays, Iron Ore, Gold, and Marble all discovered in various parts of the country [2]. Clay is a naturally occurring solid material that is in abundance throughout the country's sedimentary basins, constituting 50% of the non-metallic mineral deposits [3]. Akwa Ibom State has rich natural mineral deposits with hydrocarbon being the most prominent [4].

The industrial viability of clay minerals is largely dependent on their physical and chemical properties. The formation of clay is attributed to the decomposition of igneous rock which results in the mixture of gases and vapor in the earth crust [5]. Clay rich in kaolinite (aluminum, silicate hydroxide) has enormous industrial potential and application. Kaolinite is chemically composed of aluminum, silicon, and water, with the formula  $Al_2Si_2O_5(OH)_4$ . Clay varieties are determined based on their plasticity when moist and also when subjected to high temperatures. Clay is the principal raw material in the production of ceramics. The production of ceramics with clay involves several unit operations which include molding, pounding, rolling, flattening, coiling, cutting, and extruding. The process starts with processing the clay to remove impurities such as scrap metals, sand, stones, plant materials, and others that can affect the quality of the ceramic [6].

## Ceramics

Clay is used in the production of earthenware, porcelain, and bricks [7]. Ceramics has excellent thermal and electrical properties with other properties including, high melting point, high hardness, poor conductivity, high moduli of elasticity, chemical resistance, and low ductility. Ceramic composites also include fiberglass and carbon fiber [8].

Some outstanding properties of clay are mostly mechanical, electrical, and optical. The mechanical

properties of ceramics are important in structural and building materials. Ceramic materials are bonded by both ionic and covalent bonds which are crystalline and amorphous [9]. The ice-plating technique is used to control the mechanical properties of ceramics to their desired application [10]. Some variables can be controlled during ice-plating to influence the morphology of the microstructure.

The electrical properties of ceramics include materials acting as semiconductors. The most widely known ceramic semiconductor is a varistor which exhibits the property that resistance drops sharply at a certain voltage threshold [11]. This device uses electricity to produce a mechanical motion, and the mechanical motion in turn is used to produce electricity through the generation of signals [12].

The development of translucent materials from ceramics has seen a wide range of applications, including heat-seeking missiles, windows for fighter aircraft's, and scintillation counters for computed tomography scanners [13].

### Clay Ceramics

The origin of clay deposits points to the complex weathering, transport, and deposition by sedimentation within a geological period [14].

The composition of common clay includes kaolin and feldspathic mineral that is anhydrous and not decomposed. Clays have different degrees of plasticity because of their moisture content with some being more or less malleable for molding. Plastic clays are used in the production of bricks, tiles, tobacco pipes, firebricks, and many other products [15]. Clay is the oldest ceramic material due to its plasticity as a result of the molecular film of water surrounding the clay particles. Most natural clays are white or light-colored while those with impurities are reddish or brownish as a result of iron oxide contents [16]. Clay bricks are prominent in the building industry. Clay, a naturally occurring deposit can be distinguished from other fine-grain soils by its grain particle size and mineralogy [8]. Clay's ability to absorb water and expand in volume during construction makes it a major challenge to civil engineers [7].

Clay is composed of hydrous aluminum phyllosilicate minerals that contain aluminum and silicon ions bonded into particles that are interconnected through oxygen and hydroxyl ions. These mineral compounds affect the physical properties of clay giving it a tough but flexible and moist clay appearance. These mineral compounds give clay an aggregate cohesion that forms its plasticity [17].

## 2.0 MATERIALS AND METHODS

### Materials

In undertaking the research work, lots of materials were used in the analysis of the clay samples. The materials used for moisture content include –

weighing balance, empty crucibles, and oven. For compaction test - Compaction mold, capacity 1000ml, Rammer, mass 2.6 kg, Detachable base plate, Collar, 60mm high, IS sieve, 4.75 mm, Contest Instrument 200 °C Oven, Desiccator, Atom A-120 Weighing balance, large mixing pan, Straight edge, Spatula, Graduated jar, Mixing tools, spoons, trowels, etc. For particle size analysis - Glass cylinders, 1000-ml capacity, ETI 2001 Thermometer, Hydrometer, Electric mixer with dispersing cup, Plunger, and Atom A-120 Weighing balance. X-Ray Diffraction (XRD) used GBC Enhance Mini-Material Analyzer (EMMA) which employed the XRD technology to conduct material analysis using standard XRD reference data. XRF (X-ray fluorescence) used SKYRAY INSTRUMENT, EDX3600B X-ray fluorescence spectrometer applies XRF technology to conduct a fast and accurate analysis of complex composition. Atterberg limits tests used Liquid limit device, ASTM "Casagrande" grooving tool, Ceramic bowl, Spatula, Wash bottle, distilled water, Atom A-120 Weighing balance, drying oven, No. 40 sieve, pan, and lid, Plastic limit glass plate, Aluminum moisture cans, 3 mm diameter rod, 10 cm long or calipers for measuring, and Beaker for mixing the sample with distilled water. The following materials were required for conducting Atterberg limits tests - Liquid limit device, ASTM "Casagrande" grooving tool, Ceramic bowl, Spatula, Wash bottle, distilled water, Atom A-120 Weighing balance, drying oven, No. 40 sieve, pan, and lid, Plastic limit glass plate, Aluminum moisture cans, 3 mm diameter rod, 10 cm long or calipers for measuring, and Beaker for mixing the sample with distilled water.

### Methods

5kg each of the clay was obtained from both Ikot Usan and Ikot Edet deposits. The clay was processed for analysis through grinding to powder form and drying before being used for the physical and chemical analyses.

### Moisture Content Analysis

This involved weighing and recording the weight of an empty crucible ( $W_1$ ). 100g of the processed clay sample was placed in the empty crucible and weighed again ( $W_2$ ). The sample with the container was placed in an oven at 110 °C for 24 hours. After 24 hours the clay with the container was removed, re-weighed, and weight recorded. Mathematically, moisture content analysis was calculated using the formula in Equation 2.1;

$$\text{Moisture Content (\%)} = \frac{W_2 - W_3}{W_2 - W_1} \times 100 \quad \text{Equation 2.1}$$

where,

$W_1$  - weight of the container with the lid;  $W_2$  - weight of the container with the lid and sample before drying; and  $W_3$  - weight of the container with the lid and sample after drying.

The moisture content analysis of the clay samples was carried out to standard specifications using ASTM D2216.

### Compaction Test

3 kg of the processed clay was obtained and passed through the No. 4 sieve. The weight of the container was taken and recorded, and the weight of the soil and mold without collar ( $W_m$ ) were equally taken and recorded. Water was added to the clay sample to achieve the desired moisture content ( $W$ ). The collar was lubricated to ensure its smooth functioning. The mixed soil was placed on the mold in 3 layers. The compaction was carried out with 27 blows per layer. The clay filled the mold and extended to the collar, not more than 1 cm. After the number of blows had been achieved, the collar was carefully removed with the soil trim above the mold to a sharpened straight edge. The weight of the mold and the clay ( $W$ ) were taken and recorded. The clay was extruded from the mold. The moisture content of the soil sample was taken. The clay was placed again on the mixer and water was added to achieve a higher moisture content ( $w$ ). The compaction test analysis was calculated as follows;

The dry unit weight ( $\gamma_d$ ) of the clay was calculated using Equation 2.2;

$$\gamma_d = \frac{W - W_m}{(1+w) \cdot V} \text{ Equation 2.2}$$

Where,

$W$  – the weight of the mold and the soil mass (kg),  
 $W_m$  – the water content of the clay (%),  $V$  – the mold volume, taken as  $0.033 \text{ m}^3$

The compaction test of the clay samples was carried out using standard specifications of ASTM D4718/D4718M.

### Particle Size Analysis

1kg of the processed clay samples was sieved to obtain the results. The analysis was carried out by placing the sieve on a mechanical shaker that shook the sieve vigorously for 20 mins. The weight of the aggregate retained on each sieve was measured and the percentage determined.

The particle size analysis of the clay samples was carried out using standard specifications of ASTM D3740

### X-Ray Diffractometer (XRD)

100g of processed clay was used for the X-Ray Diffraction (XRD) analysis which was aimed at ascertaining the molecular structure of crystalline material by diffracting x-rays through the sample. The analysis was carried out using an XRD analyzer which worked by obtaining interference patterns reflecting lattice structures by varying the angle of incidence of the X-Ray beam. This test was performed by directing an x-ray beam at a sample and measuring the scattered intensity as a function of the outgoing

direction. Once the beam was separated, the scatter, also called a diffraction pattern, indicated the sample's crystalline structure. The Rietveld refinement technique was then used to characterize the crystal structure which provided the observed pattern.

### X-Ray Fluoroscopy (Chemical Analysis, XRF)

100g of processed clay was used for the XRF (X-ray fluorescence) analysis. XRF is an analytical technique used to determine the elemental composition of materials through the use of XRF analyzers, which determine the chemistry of a sample by measuring the fluorescent (or secondary) X-ray emitted from a sample when it is excited by a primary X-ray source. Each of the elements present in a sample produces a set of characteristic fluorescent X-rays ("a fingerprint") that is unique for that specific element, which is why XRF spectroscopy is an excellent technology for qualitative and quantitative analysis of material composition. A SKYRAY Instrument, EDX3600B X-ray fluorescence spectrometer applies XRF technology to conduct a fast and accurate analysis of complex composition. The system detects elements between Sodium (Na,  $Z=11$ ) and Uranium (U,  $Z=92$ ) with high resolution and fast analysis.

### Atterberg Test

The Atterberg limits test was carried out using 500g of processed clay to determine the critical water content of the fine-grained clay. The test procedure conducted under the Atterberg limit test included; shrinkage limit, plastic limit, and liquid limit. Soil samples appear in four states depending on their water content level; solid, semi-solid, plastic, and liquid. Atterberg limit test takes the consistency and behavior of soil at each site into consideration as it affects its engineering properties. The water content at which the soils change from one state to the other is known as the consistency limits of Atterberg's limit. Atterberg test analysis of the clay samples was carried out using standard specifications of ASTM D4318/ D4943.

## 3.0 RESULTS AND DISCUSSION

### Results

The results of the various analyses carried out on the processed clay samples are shown below. The physical analysis of the processed clay samples captures the following analysis; Moisture Content Analysis, Compaction Test, Particle Size Analysis, and Atterberg Test while the chemical characterization of the clay samples was carried out using X-ray Diffraction (XRD) and X-ray fluoroscopy (XRF).

Table 3.1 presents the result of the physical properties of the clay samples.

**Table 3.1: Physical Properties Analysis of the Clay Samples**

Analysis	Units	Sample		Standards for Clay	ASTM Standard
		A (Ikot Usan)	B (Ikot Edet)		
Natural Moisture Content	%	20.14	22.15	21 – 37	D2216
Compaction Test	%	28.20	27.62	-	D4718/D4718
Particle Size Analysis	G	39.1	23.4	12 – 41	D3740

Table 3.2 presents the result of the physical properties of the clay samples using the Atterberg test.

**Table 3.2: Atterberg Test**

Analysis	Units	Sample A	Sample B	Standards for Clay	ASTM Standard
		(Ikot Usan)	(Ikot Edet)		
Liquid Limit Test	%	47.56	49.59	40 – 50	D4318
Plastic Limit Test	%	31.69	38.28	-	
Plastic Index Linear	%	17.3	23.7	18 – 25	
Shrinkage Test	%	15.96	16.07	10 – 30	

Table 3.3 presents the results obtained from X-ray Diffraction (XRD) analysis of the clay sample A. The result covers the names of the identified compounds and minerals, chemical and molecular formulae, and atomic and molecular weights of the compounds.

**Table 3.3: Chemical Properties Analysis of the Clay Sample A**

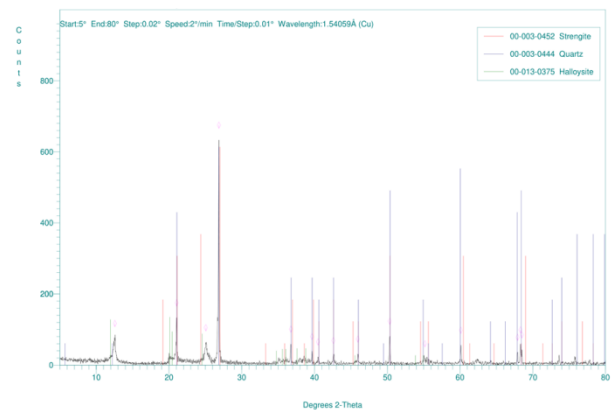
Description	Sample A		
<b>Chemical Formula</b>	Si O <sub>2</sub>	Al <sub>2</sub> Si <sub>2</sub> O <sub>5</sub> (OH) <sub>4</sub>	Fe P O <sub>4</sub> · 2 H <sub>2</sub> O
<b>Empirical Formula</b>	Si O <sub>2</sub>	Al <sub>2</sub> H <sub>4</sub> Si <sub>2</sub> O <sub>9</sub>	Fe H <sub>4</sub> O <sub>6</sub> P
<b>Weight (%)</b>	Si <sub>46.74</sub> O <sub>53.26</sub>	Al <sub>20.90</sub> H <sub>1.56</sub> Si <sub>21.76</sub> O <sub>55.78</sub>	Fe <sub>29.89</sub> H <sub>2.16</sub> O <sub>51.38</sub> P <sub>16.58</sub>
<b>Atomic (%)</b>	Si <sub>33.33</sub> O <sub>66.67</sub>	Al <sub>11.76</sub> H <sub>23.53</sub> Si <sub>11.76</sub> O <sub>52.94</sub>	Fe <sub>8.33</sub> H <sub>33.33</sub> O <sub>50.00</sub> P <sub>8.33</sub>
<b>Compound Name</b>	Silicon dioxide	Aluminosilicate	Hydrated Iron Phosphate
<b>Mineral Name</b>	Quartz	Halloysite	Strengite

Table 3.4 presents the result obtained from the X-ray Diffraction (XRD) analysis of the clay sample B. The result covers the names of the identified compound and mineral, the chemical and molecular formula, and the atomic and molecular weight of the compounds.

**Table 3.4: Chemical Properties Analysis of the Clay Sample B**

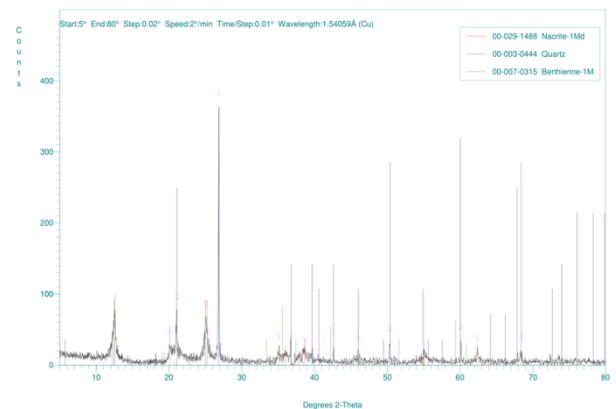
Description	Sample B		
<b>Chemical Formula</b>	Si O <sub>2</sub>	Al <sub>2</sub> Si <sub>2</sub> O <sub>5</sub> (OH) <sub>4</sub>	(Fe +2, Fe +3, Al) <sub>3</sub> (Si, Al) <sub>2</sub> O <sub>5</sub> (OH) <sub>4</sub>
<b>Empirical Formula</b>	Si O <sub>2</sub>	Al <sub>2</sub> H <sub>4</sub> Si <sub>2</sub> O <sub>9</sub>	Fe <sub>3</sub> H <sub>4</sub> Si <sub>2</sub> O <sub>9</sub>
<b>Weight (%)</b>	Si <sub>46.74</sub> O <sub>53.26</sub>	Al <sub>20.90</sub> H <sub>1.56</sub> Si <sub>21.76</sub> O <sub>55.78</sub>	Fe <sub>45.07</sub> H <sub>1.08</sub> Si <sub>15.11</sub> O <sub>38.73</sub>
<b>Atomic (%)</b>	Si <sub>33.33</sub> O <sub>66.67</sub>	Al <sub>11.76</sub> H <sub>23.53</sub> Si <sub>11.76</sub> O <sub>52.94</sub>	Fe <sub>16.67</sub> H <sub>22.22</sub> Si <sub>11.11</sub> O <sub>50.00</sub>
<b>Compound Name</b>	Silicon dioxide	Aluminum Silicate Hydroxide	Iron Aluminum Silicate Hydroxide
<b>Mineral Name</b>	Quartz	Nacrite-1Md	Berthierine-1M

Fig 3.1 presents the X-ray fluorescence (XRF) for clay sample A, showing that the clay contained; Strengite, Quartz, and Halloysite. Strengite from the resulting chart was the most prominent compound in the clay sample and recorded as 00.003 – 0452.



**Fig 3.1: XRF Analysis of Sample A**

Fig. 3.2 presents the X-ray fluorescence (XRF) for clay sample B, showing that the clay contained; Nacrite-1Md, Quartz, and Berthierine-1M. Nacrite-1Md was the major compound in the clay sample and its recorded as 00.029 – 1488.



**Fig 3.2: XRF Analysis of Sample B**

## DISCUSSION

The analyses of the Use Abat clay samples showed credible chemical and physical characteristics. Table 3.1 shows the physical properties of clay samples. The physical analysis conducted on the clay samples included moisture content analysis, particle size, and compaction test. The results from the analysis showed that the clay samples were within the international standards, with moisture content for sample A giving 20.14% and sample B 22.15%, falling within the standard range of 21-37%. The compaction test result showed 28.20% for sample A and 27.62% for sample B. The particle size analysis for sample A showed 39.1g while sample B showed 23.4 and were within the standard range of 12-41g.

Table 3.2 represents the Atterberg test for the clay samples, showing results for the liquid limit test, plastic limit test, plastic index, and linear shrinkage test. The liquid limit test showed 47.56% for sample A and 49.59% for B, which compared to the standard range of 40 – 50%. The plastic index of the clay samples showed 17.3% for sample A and 23.7% for sample B and were within the standard range of 18 – 25%. The result showed a linear shrinkage test for sample A as 15.96% and sample B as 16.07%, which agreed with the standard range of 10 – 30%. The physical characteristics of the clay samples portray the uniqueness of the clay materials obtained from Use Abat, thereby conveying its suitability for various industrial applications with the results comparing with the results of the works of Omotoyinbo, J. A. et. al [18].

Table 3.3 shows the chemical properties of clay sample A obtained from X-ray Diffraction (XRD) analysis. The result covers the names of the identified compounds and minerals, chemical and molecular formulae, and atomic and molecular weights of the compounds. The minerals in Sample A include Quartz (Silicon dioxide), Halloysite (Aluminosilicate), and Strengite (Hydrated Iron Phosphate). Table 3.4 shows the chemical properties of clay sample B obtained from X-ray Diffraction (XRD) analysis with the following mineral compounds identified, Quartz (Silicon dioxide), Nacrite-1Md (Aluminum Silicate Hydroxide), and Berthierine-1M (Iron Aluminum Silicate Hydroxide).

Fig. 3.1 presents the X-ray fluorescence (XRF) for clay sample A, showing that the clay contains; Strengite, Quartz, and Halloysite. Strengite from the resulting chart is the most prominent compound in the clay sample and it is recorded as 00.003 – 0452, while Quartz shows 00.003 – 0444, and Halloysite shows 00.013 – 0375.

Fig. 3.2 presents the X-ray fluorescence (XRF) for clay sample B, showing that the clay contains; Quartz, Nacrite-1Md, and Berthierine-1M. Nacrite-1Md from the resulting chart is the most prominent compound in the clay sample and it is recorded as 00.029 – 1488,

while Quartz shows 00.003 – 0444, and Berthierine-1M shows 00.007 – 0315.

Evaluating the industrial potential of each of the minerals as shown in Figs 3.1 and 3.2, the clay samples show the following:

**Quartz:** A hard crystalline mineral that is composed of silicon and oxygen atoms, quartz can be applied industrially in various areas. The industrial application of quartz includes jewelry, glass making, watches, foundry materials, the refractory industry, the ceramic industry, kitchen countertops, and laboratory crucibles.

**Halloysite:** Halloysites find their application in the ceramics industry, cement, and fertilizers.

**Strengite:** Strengite is used as a constituent of corrosion-resistant materials such as phosphate coatings.

**Nacrite-1Md:** Nacrite is traded in China as a gemstone and is used for carvings.

**Berthierine-1M:** A kaolin element that can industrially be applied in coating paper and for use as stable components in many industrial products.

The clay samples from Use Abat are a unique clay material that has enormous industrial applications and could be applied industrially in various sectors that are economically viable. Two areas of industrial application for consideration are ceramics and paper production.

**Ceramics:** The clay materials possess the required qualities for the industrial production of ceramic products such as porcelain, fine ceramics, coarse ceramics, cement, electro-ceramics, tiles, and refractories.

**Paper Production:** Clay with kaolin is the most extensively used particulate mineral in the filling and coating of paper. It improves paper appearance, which is characterized by gloss, smoothness, brightness, and opacity, and of greatest significance, it improves printability. Paper is also filled with kaolin to extend fiber.

## 4.0 CONCLUSION

The clay deposit in Use Abat, Ibiono Ibom, Nigeria showed on analyses that it portends huge economic benefits in the production of ceramics and papers and its attendant economic benefits accruable to the host communities, if the resources are explored and harnessed. Quartz, Halloysite, Strengite, Nacrite-1Md, and Berthierine-1M are mineral compounds that were found to be contained in the clay samples after analyses, and there could significantly contribute to the economic development of Akwa Ibom and Nigeria if the resources are effectively harnessed and utilized.

The application of clay minerals is determined by their structural, physical, and chemical characteristics. The physical and chemical properties of the clay minerals dictate their utilization in the process

industries and the beneficiation required before usage.

This research work which aimed at the physicochemical and mineralogical characterization of Use Abat, clay for industrial application was successfully carried out and the huge data collated will contribute immensely towards updating the Nigeria's mineral database.

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