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Industrial Analysis of Iba Oku Clay Deposit in Akwa Ibom State, Nigeria

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Abstract: In this work, clay sample excavated from Iba Oku community along Uyo Village road, Akwa Ibom State Nigeria was taken to the laboratory and analyzed using standard physico-chemical tests, for its industrial potentials. In the last thirty years or more, there have not been visible activities demanding clay in its various forms as input material in manufacturing activities in Akwa Ibom or even the adjoining States. The reason for this state of affairs may not be unconnected to either complete lack of information or research data on the abundant clays in Akwa Ibom State, particularly the Iba Oku clay. It is therefore in pursuant of the need to stimulate entrepreneurial interest, besides accumulating academic research data, that the current research became pertinent. The sample was first crushed, soaked and decanted, dried in an oven and then sieved. Thereafter, the plasticity, particle texture, grain volume, linear shrinkage, mineralogy and elemental composition were studied using various techniques. The clay sample is composed of the two minerals quartz (80%) and kaolinite (20%) and two dominant elements, silicon at 45.93 weight % and aluminum at 18.90 weight % both comprised approximately 65% weight content. Furthermore, particle textural distribution classifies the sample as inorganic clay soil, its determined plasticity index is 33.1%, porosity is 51.93%, linear shrinkage is 13% and grain volume is 12.82cm^3. The findings show that clay sample from the study area is plastic, whitish to light pink colored, free of accessory mineral impurities, kaolinitic but with very high non-clay quartz content. This makes it suitable as raw material to obtain a triaxial ceramic when mixed with feldspar for industrial production of ceramic floor tiles.

Keywords: Kaolinite, Clay, Quartz, Plasticity, X-ray Diffraction (XRD), Energy Dispersive X-ray Spectroscopy (EDX)

1. Introduction

Akwa Ibom State (Figure 1) occupies a total land area of 2,734 square miles. The state sits on an appreciable quantity of mineral resources, some of which have been harnessed and given Akwa Ibom a name. Akwa Ibom State is the largest producer of Crude Oil in Nigeria with a turnout of 31.4% total national production [2]. However a great portion of her solid mineral resources remain untapped. In the year 2016, she identified only with laterite mining with produced capacity of 90,691tons [2]. The other potential solid minerals yet commercially untapped recorded in the State include clay, glass sand, salt, silica sand, granite, coal, petroleum, natural gas, kaolin, limestone, lignite [2]. Confirmed deposits of clay minerals are found in Eket, Etinan, Ikot Abasi, Ikot Ekpene, Ini, Itu, Onna, Oruk Anam and Nsit Ibom Local Government Areas [3-6]. Geologically speaking, the soil of Akwa Ibom state is categorized as coastal plain sand physiography with characteristic dominance of sandy textured grains [7-9]. Uyo town located at the heart of Akwa Ibom State is the state capital described as low lying to gentle rolling typography with an average elevation of 52 meters above sea level [10]. A lot of research has been conducted on Akwa Ibom soil potentials for mineral exploitation and for agricultural purposes [4-6, 10-12] while, other research has examined the load bearing and other construction characteristics [7, 8]. Currently, Iba Oku clay is utilized as raw material for sculpture and pottery making by the indigenous community and the close by Fine and Applied Arts Studio of the University of Uyo.

Clay is a component of the earth soil formed by the weathering of primary igneous rocks through the mechanical and chemical action of water, wind, carbon dioxide, humic acids and earth movements working together [13]. Clay soils are composed of different mineral species combined together to impart the unique physical appearance and properties.

Kaolin, a typical example of clay with the formula $2SiO_2.Al_2O_3.2H_2O$ is named after Kao Ling in China, the location of its first major utilization. Kaolin clay consists of minerals kaolinite, quartz and feldspar [13] with each mineral possessing inherent properties thereby resulting to the clay exhibiting a behavior to vary as one or the other of these constituents tend to increase. Kaolinite is plastic with high values of shrinkage and refractoriness, quartz is non-plastic with very low values of shrinkage and tensile strength while feldspar is fusible but with low plasticity. The building block of these minerals is a network of crystalline ionic compound, silicon-oxygen tetrahedron $[SiO^4]^{4-}$, bonded covalently to a network of closely packed hydroxyl ions in an octahedral formation surrounding a trivalent aluminium cation. The elements silicon and aluminum constitute a bulk of its molecular weight. Quartz and Feldspar minerals which remain unchanged by weathering are composed of silicon, oxygen and potassium. In addition to these minerals, clay in the natural form is found to be combined with several independent metallic carbonates and oxides most particularly iron. The presence of iron oxide characteristically will produce certain definite colors dependent on the amount of iron content present. Reference [14] states that the various manifested tints which are exhibited in many types of clay are due to a mixture of carbonaceous matter and iron. Because their presence takes away from the plastic nature of clay minerals, these associated oxides, carbonates as well as the non-plastic mineral quartz inherent in clay minerals are termed impurities which are beneficiated to enhance the plastic inducing mineral specie present. Other clay plastic inducing minerals aside kaolinite include montmorillonite, illite, vermiculite and palygorskite. This category of clay minerals has the orthogonal hydroxyl sheet sandwiched in between two tetrahedral sheets. Because the sheets are stacked on top each other, clay minerals owe their plasticity to their fine grained and flat shaped nature, and the lubricating movement of water molecules between the sheets. As applied to clay, plasticity defines the behavior of clay to take up water of given quantity and become amenable to deform without rupture under applied pressure, thus retaining the new shape when pressure is released. Clays have been of interest to the industry due to their fine texture, plasticity, absorption and ion exchange capacity as well as the robustness and durability of their fired finish. This makes clay the constituting backbone material to the ceramic wares industry. Their stability and transformation with accompanying sintering gives rise to porcelain highly utilized in high voltage installations for the electricity industry. Clay is a well applied material in the petroleum field for drilling mud due to the adsorption swelling nature of bentonitic clays. Polymer and paper industries take advantage of the fine grains, negative charged particles and absorptivity of clays to form materials of clay and organic compounds where the clay charge adds polymeric strength to the final plastic product. For paper production the clay acts as filler, taking up the space between the cellulose fibres of the paper matrix. In the chemical industries, clays are considered useful as catalysts in promoting organic reactions. Clays present two main properties of use in catalysis: a large particle surface area due to their small grain size; and a larger internal, chemically active surface area due to their absorptive properties.

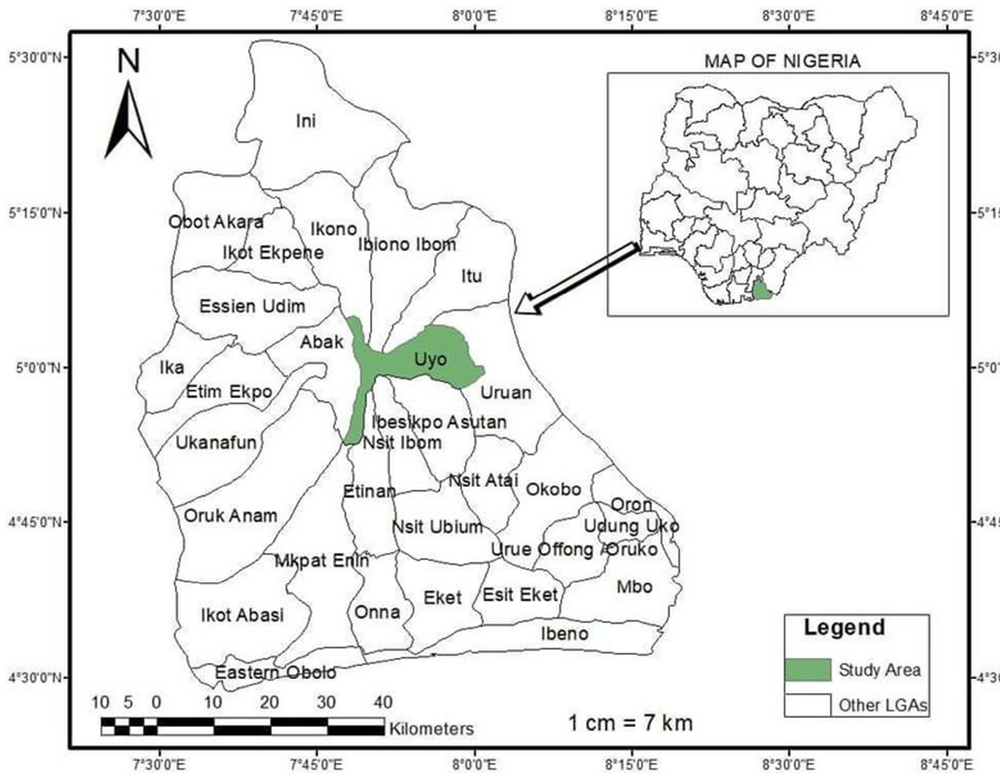

Figure 1. *Map of Akwa Ibom State showing Uyo [1].*

The objective of this study was to carry out the mineralogical, physical and chemical characterization of Iba Oku clay deposits for the purpose of evaluating its industrial potential and thus bring attention to the economic benefits in its exploitation to the State Government.

2. Materials and Methods

2.1. Materials

Equipment used in this study include Empryean X-ray diffractometer model PW 4030, ASTM 152H Bouyoucos hydrometer, OFITE Core Nitrogen Gas Porosimeter model 350 (V_1=54.03, V_2=155.14), ASTM D4318 Cassangrande device model 1900, ASTM E11 500, 425 and 125 sieve sizes, Vernier Calipers and Spatula. Material list included Iba Oku clay sample, distilled water, sodium hexametaphosphate solution.

2.2. Sample Collection from Clay Deposit

Clay soil sample weighing 3 kg was collected at Iba Oku quarry, Figure 2 which lies within latitude 5.03 N and 7.55 E along Uyo Village road.

Figure 2. *Raw clay in situ at Iba Oku.*

Clays were collected at 8 meters depth using a clean spade to hack out chunks which were deposited into sack bags and conveyed to the laboratory for further analysis. The color of clay was pinkish white.

2.3. Processing of Clay Soils

The sample obtained from Iba Oku quarry was taken to the laboratory for processing. The obtained 3kg sample was weighed on an electronic weighing balance (Camry model no ACS-30-ZC41). Furthermore, the clay chunk was processed into powder for physical beneficiation and subsequent characterization. The sample was crushed in a ceramic mortar with the aid of a pestle and then subsequently soaked in water for 8 hours. The organic impurities were separated by decantation. Slaked clay was stirred into a homogeneous solution, poured unto a steel pan, Figure 3, and dried out in an oven at 110°C for 24 hours to reduce moisture content. Thereafter, the dried clay samples were collected and sieved through 500 micron sieve, while sample for XRD was sieved through 125 micron sieve.

Figure 3. *Processed clay ready for drying.*

2.4. X-ray Diffractometry Analysis

The qualitative and semi-quantitative determination of the mineralogical properties of the clay samples by X-ray diffraction (XRD) method as well as the elemental composition of the clay sample using energy dispersive X-ray spectroscopy (EDX) technique were carried out on an automated Empyrean pan-analytical X-ray diffractometer model PW 4030. The diffractometer consists of three basic elements: an X-ray (Copper K α- radiation) tube, a sample holder, and an X-ray detector. 125 micrometer sieved sample was prepared by compressing in a sample holder to create a flat and smooth surface and then loaded into the chamber of the diffractometer. X-rays were generated in a cathode ray tube by heating a filament to produce electrons. These X-rays were collimated and directed onto the sample. As the sample and detector were rotated, the intensity of the reflected X-rays was recorded. The EDX technique detected X-rays emitted from the sample during bombardment by an electron beam to characterize its elemental composition. The tests were carried out at the National Geological Survey Agency, Barnawa, Kaduna State.

2.5. Clay Soil Mechanics Analysis

2.5.1. Liquid Limit Test by Atterberg

Liquid Limit (LL) is the percent water content of clay at the arbitrarily defined boundary between the liquid and plastic states. 200g of sample were measured out of 500 microns sieved clay and further sieved using prescribed 425 microns sieve size. 12ml of distilled water was added per 50g of sample and mixed into a smooth uniform paste. Four empty moisture cans were labeled and weighed and their weights were recorded on a data sheet. Then a portion of previously mixed soil was added to the Cassangrande cup and spread out to a depth of 10 mm at deepest point using a spatula to squeeze and eliminate air pockets. The grooving tool was carefully used to cut a clean straight groove down the center of the cup by holding tool perpendicular to the surface of cup. Then the handle of the Cassangrande device was turned and number of blows was counted till two halves of the soil came into contact at the bottom of the groove. The number of blows was recorded. Then a sample out of the cup was taken and placed in the labeled and weighed empty moisture can, then weighed immediately. Afterwards, the cans were placed in an oven set at 105°C for 24 hours. Finally they were brought out and reweighed. Results were reported as:

$$Percent\ Moisture\ Content = \frac{Mass\ of\ Moisture}{Mass\ of\ Dry\ Soil} \times 100 \quad (1)$$

The value for Liquid Limit plotted against 25 blows using value for percent moisture content for four cans tested per sample to obtain the percent liquid limit.

2.5.2. Plastic Limit

Plastic limit (PL) is the minimum moisture content at which the soil can be rolled into thread of 3mm diameter without crumbling. Additional equipment used for this procedure included glass plate. Sieved sample was prepared first into an ellipsoidal mass by addition of distilled water in turns, until the consistency became non-stick to touch. The mass was then shaped to threads of 3mm diameter by a stroke of 90 rolls per minute. Afterwards the thread was broken to smaller bits and reformed into ellipse and re-rolled to smaller threads till finally thickness could not be maintained at 3mm. Then the crumbled threads were gathered and placed into labeled and weighed empty moisture can, reweighed and placed in an oven set at 105°C for 24 hours, and reweighed. Moisture content was subsequently determined using equation 1.

2.5.3. Plasticity Index

Plasticity index (IP) is the calculated difference between obtained liquid and plastic limits.

2.5.4. Shrinkage

Left-over sieved sample was prepared to wet lump and cast in two oil-painted steel frames of dimensions 70 x 25 x 20mm. The wet clays were allowed to air dry for 24 h till it obtained sufficient green strength. Two points of 40mm spacing were marked on surface of each of the clay sample using vernier caliper and recorded as wet length. Subsequently samples were dried in an oven at 11°C for 6 hours and the spacing measured as dry length. Wet-to-Dry linear shrinkage (wet basis) was computed as

$$Dry\ linear\ shrinkage\ (wet\ basis) = \frac{Wet\ length - Dry\ length}{Wet\ length} \times 100 \quad (2)$$

2.5.5. Particle Size Distribution

Particle size distribution was determined by the Bouyoucos hydrometer method as described by [15].

2.6. Bulk Volume, Grain Volume and Porosity Analysis

Porosity is a dimensionless parameter, defined as a ratio of the void space volume to the bulk volume [16]. The working principle of OFITE Core Nitrogen Gas Porosimeter model 350 is based on Boyles Law which states that at isothermal conditions, the pressure of an expanding gas correlates proportionally to volume of contained space. Since volume for porosimeter is constant, the gas pressure after equilibrium can be measured.

The clay sample was shaped into cylindrical forms called 'cores' using 40 mm diameter PVC pipes cut to lengths of 50 mm. The 500 microns sieved clay samples were mixed with just enough quantity of water to turn clay soil wet and plastic. Thereafter, while placing the cut sections of pipes perpendicular upon a flat surface with one end opened and the other end sealed with a stopper, the plastic clay was scooped and filled into the internal space and leveled out with a spatula in order to compact the clay and remove air pockets. The filled PVC pipe was set out to dry in the sun till they hardened sufficiently to cut out the plastic pipe and then further oven dried for 24 hours at 110°C. The diameter and length of the cores were measured with calipers, recorded before being inserted into the sample holder of the Porosimeter. The regulator was rotated clockwise until the pressure read approximately 180psi and with P_1 lock-in valve turned to off position the P_1 value was recorded from the Pressure Gauge. Then the gas was released and expanded into the test chamber by turning the "P_2 Test" valve to the vertical position. Subsequently the P_2 value was recorded. Results were reported as Percent Porosity, which are derivatives of Core Bulk Volume and Core Grain Volume (see Equations 3 to 6).

Core Bulk Volume,

$$V_B = \frac{\pi D^2 L}{4} \quad (3)$$

Where: V_B --- Bulk Volume, D --- Diameter and L --- Length

Core Grain Volume,

$$V_G = V_2 - V_3 \quad (4)$$

Where: V_G --- Core Grain Volume, V_2 --- Constant of the Porosimeter

$$V_3 = \frac{P_1 V_1}{P_2} \quad (5)$$

Porosity,

$$\phi = \frac{V_B - V_G}{V_B} \times 100\% \quad (6)$$

3. Results and Discussion

The results of the qualitative and semi-quantitative diffraction analysis of Iba Oku clay sample are presented as combined X-ray diffraction spectrum–Pie chart in Figure 4. The mineral characterization of Iba Oku clay sample clearly indicates the presence of two mineral species and their weight percentages, which are quartz at 80%, and kaolinite at 20%. The availability of clay mineral kaolinite in the sample was indicated by the X-ray peak reflections at 12.37°, 20.85°, 26.63°, 35.89°, 36.5° and 62.19 2θ angles. Quartz on the other hand was indicated by reflections at 20.85°, 36.52°, 50.10° and 67.70° 2θ angles. The high content of quartz observed in the clay is attributed to the raw sample possessing a gritty feel to it. The presence of clay mineral kaolinite authenticates the argillaceous nature of the soil at Iba Oku quarry, and with a content of 20% kaolinite mined at 8 meters depth, provides evidence to the economic viability of excavating and processing clays for industrial purposes.

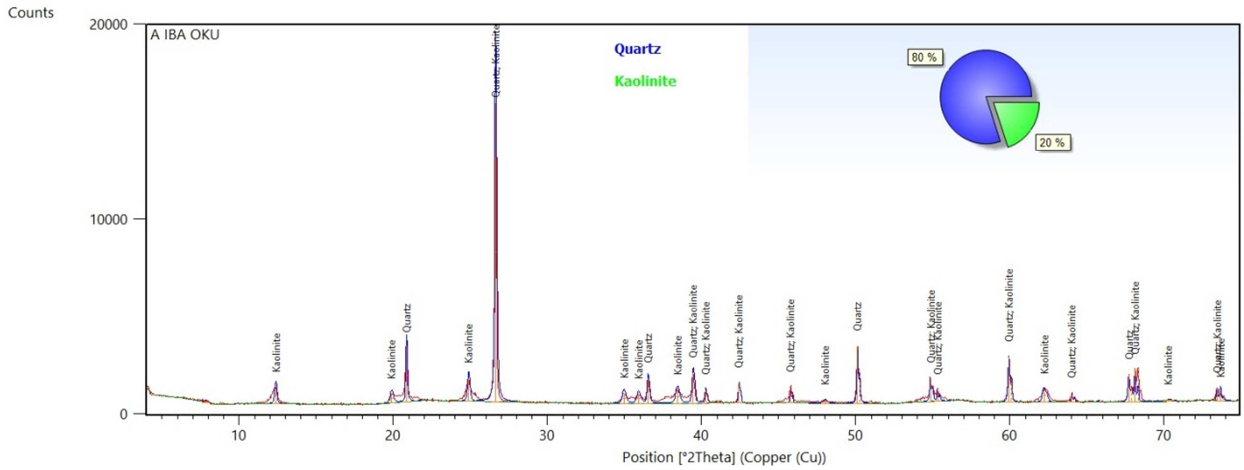

Figure 4. *XRD Characterization of Iba Oku Clay.*

Table 1. *Physical Properties of Iba Oku Clay.*

Atterberg Limits				Textural Class				Porosimeter Values		
Liquid Limit (%)	**Plastic Limit (%)**	**Plastic Index (%)**	**Linear Shrinkage (%)**	**% Clay**	**% Sand**	**% Silt**	**Class**	**Bulk Volume cm^3**	**Grain Volume cm^3**	**Porosity %**
62.00	28.9	33.1	13.04	50.7	25.9	23.5	clay	26.67	12.82	51.93%

Table 1 presents result for Atterberg limits, particle texture and index properties of analyzed sample from Iba Oku. The sample recorded a high liquid and plastic limit of 62% and 28.9% giving a plasticity index of 33.1%. The Atterberg limit values are consistent with kaolinite presence in clay as compared with results published from [17] swamp clay in Uganda with plastic limit of 29%, liquid limit of 66% and plastic index of 37%. Obtained plasticity index is also close to results published for processed china clay in Jos, Nigeria [17]. The line plot shown in Figure 5 plot indicates a close range of the tested average moisture content and correlation with data for applied number of blows using the Cassangrande device. Recorded linear shrinkage for the sample at 13.04% shows the evident effect of quartz mineral contained in the clay giving a low shrinkage value. The texture examined under Bouyoucos hydrometer soil testing by analyzing the changes in suspension density dispersed using sodium hexametaphosphate solution indicated that sample is 50.7% clay, 25.9% sand and 23.5% silt which is deduced as inorganic clay, from the textural triangle chart [18].

Table 2. *Elemental Composition of Iba Oku Clay.*

Element	Si	Al	Fe	Sb	Ag	K	Nb	Ca	Yt	P	Cl	S	Mg	Na
Atomic Concentration %	54.04	23.15	5.89	4.58	1.40	3.21	0.84	1.71	0.53	1.51	1.16	1.10	0.49	0.40
Weight Concentration %	45.93	18.90	9.96	6.63	4.56	3.80	2.35	2.07	1.43	1.41	1.24	1.07	0.40	0.28

Further results obtained for porosity of sample was 51.93% with grain volume per centimeter cube given at 12.82 and bulk volume at 26.67.

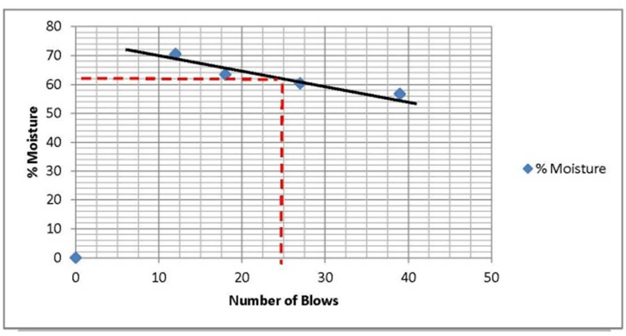

Figure 5. *Plot of Moisture Content (%) vs Number of Blows.*

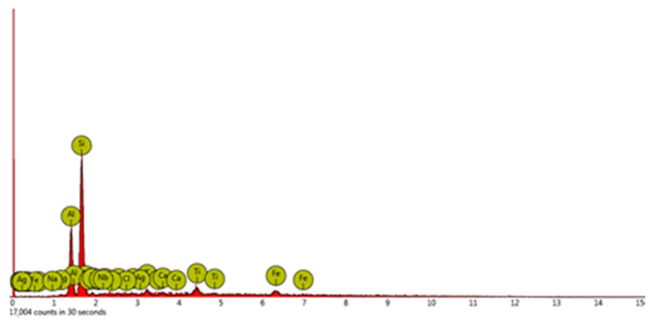

Figure 6. *EDX Spectrum for Iba Oku Clay.*

The result of EDX technique presents the elements and their atomic and weight concentration percentages in Table 2 and Figure 6. It shows the major elemental constituent silicon and aluminium, backbone of clay molecular structure dominating the tested clay sample, with silicon and aluminum as high as 45.93 and 18.9 weight percentages, respectively. Also detected are the presence of iron at 9.96%, antimony at 6.63% and silver at 4.56%. These metallic elements present in the clay form oxides and hydroxides with the oxygen and hydroxyl anions found in clay and constitute varying adverse effects to its physical properties and, therefore, by industrial practices, are reduced to the barest minimum using beneficiation techniques.

4. Conclusion

Physico-chemical tests to establish the mineralogy and properties of Iba Oku clay samples were successfully conducted. The results have shown that Iba Oku clay has 50.7% clay and 25.9% sand content and also contains 20% kaolinite and 80% quartz. Its elemental composition shows silicon and aluminum making up 65 weight percent with presence of trace metallic element iron. Other trace elements indicated are titanium, antimony, silver and potassium. The physical properties collaborates the behavior of kaolinite with determined porosity levels to be 51.93%, plasticity index at 33.10% and shrinkage at 13.04%. Due to the high presence of quartz and low kaolinite content, Iba Oku clay is not suitable on its own for whiteware or refractory production. However, the overall properties have shown its suitability as clay and silica component in a triaxial ceramic composition which in combination with feldspar, a high temperature flux, can be used for industrial production of ceramic floor tiles.

5. Recommendations

The industrial analysis of Iba Oku clay deposit in Akwa Ibom State Nigeria has the potential of stimulating diverse research interests in other aspects of the clay to fully position it for industrial application. It is therefore, in line with this objective, that the following recommendations are proffered:

1. A study of the sintering behavior of Iba Oku clay under elevated temperature, through differential thermal techniques will increase knowledge on its industrial application.
2. Further structural analytical study on the physical and mechanical properties of shaped and fired Iba Oku clay will further enhance its application.
3. Rheological behavioral study of the slip properties will facilitate the predictability of the flow nature and castability of Iba Oku clay slip, as well as the determination of required deflocculant to maximize the industrial potential of Iba Oku clay.

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