

## SPATIAL DISTRIBUTION AND MINERALOGICAL COMPOSITIONAL OF CLAYS IN OZUABAM AREA, SOUTHEAST NIGERIA

<sup>1</sup>Ndukwe O.S., <sup>2</sup>Uzoegbu M.U., <sup>3</sup>Ndukwe V.A. and <sup>4</sup>Agibe A.N.

<sup>1,4</sup>Department of Geology, Federal University, Oye-Ekiti, Ekiti State, Nigeria.

<sup>2,3</sup>Department of Geology, University of Port Harcourt, Port Harcourt, Nigeria.

E-mail: uche.uzoegbu@uniport.edu.ng

GSM: 08030715958

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### ABSTRACT

*Clay materials are often imported for the manufacturing of ceramic, pottery, drugs and paints. This has subjected our currency to pressure. Consequently result in a high cost of production. The objective of this study is to determine the mineralogical composition of Ozuabam clays for suitability of the manufacturing of the above mentioned items in Nigeria. Clay sediments collected were subjected to X-ray diffraction for identification of the mineralogical composition. The results revealed that Amuvi1 (AMV1), Amuvi2 (AMV2), Obotme1 (OBO1), Obotme2 (OBO2), Okobo (OKO), Nkana1 (NKA1), Nkana2 (NKA2), Nkana3 (NKA3) and Amakaofia (AMA) were quartz, calcite, dolomite, illite and montmorillonite whereas quartz, calcite dolomite, kaolinite, illite and montmorillonite were recorded at OBO2 and NKA3. On the obtainable result NKA3 and OBO2 contains the clay material suitable for the manufacturing of the products mentioned above. Clay mineralogical and environmental studies at the study area proved that crafter and other manufacturers can depend on Ozuabam clay better than importing it from abroad.*

**Keywords:** Ozuabam, Clay deposits, X-Ray diffraction, Maastrichtian sediments, Afikpo Basin.

### INTRODUCTION

Clay mineral compositions in shallow marine and deep marine environments are largely controlled by detrital clay derived from the continents. Also the mineralogical composition of clay minerals depends on the environment of deposition (Porrenga, 1967; Shichi and Takagi, 2000; Dogan et al., 2002; Murray, 2007). Therefore, they

can be used in determining the depositional environments of sediments (Mamman et al., 2010).

Research has shown that clays in open marine environment contains mainly of illites, montmorillonite and chlorites with illites and chlorites increasing seawards and kaolinites decreasing seawards (Keller, 1970; Ahmed et al., 2018).

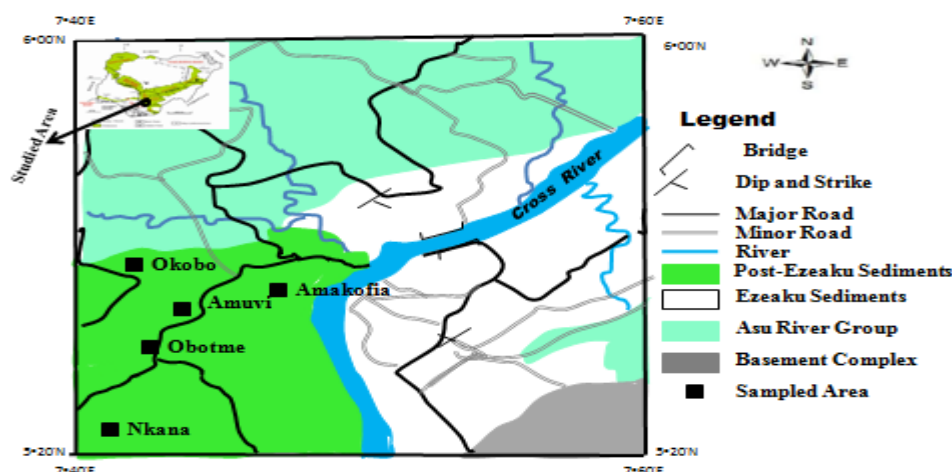


Figure 1: Geology of Afikpo sub-Basin and study locations (after Omontese et al., 2019).

The most abundant, ubiquitous, and accessible material on the earth crust is clay (Rado, 1988; Nayak and Singh, 2007; Nweke and Ugwu, 2007; Ahmed et al., 2018). Clay is simply defined as earth or soil that is plastic and tenacious when moist and becomes permanently hard when baked or fired. It consists of a group of hydrous aluminosilicate minerals formed by the weathering of feldspathic rocks, such as granite. Individual mineral grains are microscopic in size and shaped like flakes. This makes their aggregate surface area much greater than their thickness and allows them to take up large amounts of water by adhesion, giving them plasticity and causing some varieties to swell (expandable clay). Common clay is a mixture of kaolin, or china clay (hydrated clay), and the fine powder of some feldspathic mineral that is anhydrous (without water) and not decompose.

Using clayey raw minerals in art craft, clay bricks and ceramic production have been extensively studied, particularly in the fields of industrial ceramic applications production of drugs, paint manufacturing, etc, around the World (Baccour et al., 2008;

Vieira et al., 2008; Boussem et al., 2016; Ahmed et al., 2018) and archaeological ceramics (Hein et al., 2004; Adelabu, 2012; Semiz, 2017; Ahmed et al., 2018).

Indeed, clay minerals are used widely for different scientific purposes such as interpreting and understanding such problems as tectonics, provenance, facies, boundaries, correlation, zonation, age, metamorphism, oil exploration with its latest application in paleoclimate determination.

Therefore, it is easy to recognize between warm and humid conditions typical for kaolinite or halloysite formation, or dry seasons, specific for illite or smectite formation. Furthermore, the formation of kaolinite and halloysite is favored by an acidic (pH ~3) conditions, and high leaching environments (Burhan and Emin, 2010; Kabeto et al., 2012). Conversely, relatively low or no leaching environment and conditions under neutral to medium alkaline pH favor the formation of montmorillonite and three layer clay minerals. The objective of this study is to determine the spatial distribution and compositional variation of clay minerals in

the Maastrichtian sediments of the Afikpo Basin vis-à-vis the Mamu and Nsukka formations.

### Geologic Setting

The study area falls within latitude 5°20' to 6°00' N and longitude 7°40' to 7°60' E (Fig. 1). This region constitutes part of southern extension in Anambra Basin, referred to as Afikpo sub-basin. It is situated within Okigwe – Afikpo areas of southeastern Nigeria. The Maastrichtian sediment in the Afikpo Basin form part of the Mamu and Nsukka formations. Accessibility to the study area is through the Port-harcourt - Enugu express way, via Umuahia - Bende to Ohafia - and Okigwe - Amaseri to Afikpo (Fig. 1). An access route to the study area also includes - a network of major, secondary, minor roads and footpaths which help in interconnecting the inhabitants of these areas.

The Santonian deformational process resulted in the fragmentation of the lower

Benue trough (Table 1) into the Abakiliki syncline (Kogbe, 1976). The predominantly Albian-Cenomanian marine depositional cycles which terminated by a phase of folding (Nwachukwu, 1972; Olade, 1975) affected the Asu River Group in the area.

A second transgressive – regressive of deposition in the Turonian to Santonian was again terminated by a phase of folding and faulting in the early Santonian times which affected all the sediments deposited before the tectonism and this gave rise to the Afikpo (Abakiliki) syncline (Fig. 2). The Afikpo Basin is part of the Southern Benue Trough, an Inland intra-continental sedimentary basin originating from the lower Benue Trough (Obaje et al., 2004) with NE-SW trending towards the Niger Delta. According to Kulke (1995), petroleum exploration in this basin was provoked as a result of existence of surface seeps dating back to the early 1930s.

Table 1: Stratigraphic units of the lower Benue trough ( after Nyong and Ramanathan, 1985).

AGE		ANAMBRA BASIN	AFIKPO BASIN	CALABAR FLANK
Tertiary	Oligocene	Ogwashi-Asaba Fm Ameki Group Imo Shale Nsukka Formation	Ogwashi-asaba Fm Ameki Group Imo Shale Nsukka Formation	
	Eocene Paleocene			
Cretaceous	Maastrichtian	Ajali Sandstone Mamu Shale	Ajali Sandstone Mamu Formation Nkporo Shale Enugu Shale Afikpo Sandstone	Nkporo Shale
	Campanian	Nkporo Shale Enugu Shale		
	Santonian			
	Coniacian	Agwu Shale	Agwu Shale	
	Turonian	Eze-Aku Group	Eze-Aku Group	New Netim Marl
	Cenomanian			Ekenkpon Shales
	Albian		Asu-River Group	Mfamosing Limestone
	Aptian			Awi Formation
<b>Precambrian</b>		Basement Complex		

Nevertheless, due to the Santonian inversion and prevalence of continental sediments, including discoveries in the prolific Niger-Delta, the exploration and exploitation for petroleum has been ongoing in this area (Ekweozor and Gormly, 1983; Doust and Omatsola, 1990; Haack et al., 2000; Ugwueze, 2015).

The first marine transgression in Nigeria occurred during the middle Albian. Albian sediments not mentioned and unvarying comprise Asu River Group and its equivalents (Ojoh, 1990). Ukaegbu and Akpabio (2009) have differentiated the Albian sediments, northeast Afikpo Basin as consisting of alternating shale, siltstone with occurrence of sandstone, maximum thickness of 1000m rich in ammonites as well as foraminifera, radiolarian and pollens. Also present in the shales are traces of elobicerias and monticeras ammonites (Ojo, 1999).

## **MATERIAL AND METHODS**

The shale and clay samples from hand dug wells of between 30 and 60cm depth were derived from Maastrichtian sediment at Amuvi (AMV), Obotme (OBO), Okobo

(OKO), Nkana (NKA) and Amakaofia (AMA) from the Mamu and Nsukka formations in the Afikpo Basin, southeast Nigeria, were analyzed. The Amuvi sample showed some mottling in the AMV2 indicative of drainage impedence, whereas OBO1 samples appeared to be freely drained. Some properties of these clays are given in Table 2. Sand, silt and clay fractions were separated by the usual sieving and sedimentation procedures following dispersion of the soils using a Rapidis 150 ultrasonic vibrator, according to method of Genrich and Bremner (1972).

After destruction of organic matter (OM) by H<sub>2</sub>O<sub>2</sub> treatment, the separates were Ca-saturated, washed free of excess salt, and dried. Clay samples were also separated from fragments of weathered granite by first crushing them in a mortar and then dispersing them ultrasonically.

Free Fe and Al in the size fractions were extracted using dithionite-citrate-bicarbonate (DCB) (Mehra and Jackson, 1960). Noncrystalline material was dissolved by boiling DCB-treated samples in 0.5 NaOH (Hashimoto and Jackson, 1960).

Table 2: Some properties of clay materials from Ozuabam (after Ahmed et al., 2018).

<b>Sample number</b>	<b>Clay, flexibility characteristics and the forming.</b>	<b>Clay in the air</b>	<b>Clay after firing under the 1031 degree</b>
<b>AMV1</b>	Suitable for throwing use, soft, but it has a smell	Does not change the shape form	Suitable for art craft
<b>AMV2</b>	Suitable for throwing but it is so soft	The clay does not show a change the shape form	Suitable but sometimes showed cracks at the surface
<b>OBO1</b>	Suitable and easy forming.	The clay does not change the shape form	Suitable after firing
<b>OBO2</b>	Suitable and easy forming and has a big of flexibility	The clay didn't change the shape form	Suitable after the fire
<b>OKO</b>	Suitable and easy forming and building.	Not suitable and it caused cracked within the body surface after drying	The form changed and the surface bend and it caused cracked with the body of the pottery
<b>NKA1</b>	Suitable and easy forming and has a big flexibility.	Suitable and the form didn't change its design.	Suitable after firing
<b>NKA2</b>	Suitable and easy forming	Suitable and the form didn't change its design.	Suitable after firing
<b>NKA3</b>	Suitable and easy forming and has a big of flexibility	The clay didn't change the shape form	Suitable after the fire
<b>AMA</b>	Suitable and easy forming.	The clay does not change the shape form	Suitable but sometimes showed cracks at the surface

Application of wheel for the clay samples was used. This stage is the most important one for the potters and Ceramists designer, to design and make a cups and bowls or vases. In the beginning of this step, the clay of each sample purified from grains Table 2, stones and pseudo-matrix by using strainer size 100 ml, then preparing the clays to be use for throwing. Throwing on the wheel start with centering the clay; opening, expanding and rise the clay up, then forming the bowl or the cup. Trimming started at the back side of the shape to get

the final form. These processes were done for all samples of the Ozuabam clay. In the final step cups and bowls were fired in the electrical kiln under temperature 1031 Celsius.

For XRD, Fe was released by this treatment was extracted by an additional DCB treatment. A cation-exchange capacity (CEC) was determined on deferrated, NaOH-treated samples using a BaCl<sub>2</sub> solution buffered at pH 8.2 with triethanolamine. Potassium fixation was estimated by the method of Alexiades and

Jackson (1965). Removal of interlayer Al was attempted on selected samples using the sequential sodium citrate extraction procedure of Frink (1965).

The sands were prepared as random powders, whereas the silts were sedimented onto glass slides. The clays were divided into coarse (2-0.2 $\mu$ m) and fine (< 0.2 $\mu$ m) fractions by centrifugation. Subsamples were saturated with K<sup>+</sup>, pipette onto glass slides, and air dried. The K-saturated samples were also heated at 300° and 550°C in a muffle furnace for 2 hours. A Mg-glycerol samples was prepared by suspending a Mg-saturated samples in 1ml of 10% glycerol in water. Differential thermal analysis (DTA) was performed on samples clay (< 0.2 $\mu$ m, Ca-saturated), equilibrated for at least 4 days in a desiccator over a saturated Mg(NO<sub>3</sub>)<sub>2</sub> solution, using a Stanton-Redcroft 673-4 instrument at a heating rate of 20°C/min in a nitrogen atmosphere. Al<sub>2</sub>O<sub>3</sub> was used as reference. Acid dissolution of samples, previously treated with DCB and NaOH to remove free oxides and amorphous silicates, was carried out by digesting 100 mg of sample (Ba-saturated) in 50ml of 1 HCl on a water bath at 80°C for 24hr.

The residue was washed with 25ml of 1 HCl. The wash solutions were combined with the supernatants and brought up to volume. The residue were treated with boiling 0.5 NaOH for 2.5 min. Si and Fe<sup>2+</sup> in the extracts were determined colorimetrically (Pruden and King, 1969). Al, total Fe, Mg, Ca, K and Na were

determined by atomic absorption spectroscopy. The effect of time on acid dissolution of selected silts was assessed by subjecting the samples to acid treatment from 2 to 30 hr. The residues were boiled in 0.5 NaOH as before.

Results were presented as peak positions at 2 $\theta$  and x-ray counts in the form of a table or an x-y plot. Intensity (*I*) is reported as peak height intensity (intensity above background). The relative intensity is recorded as the ratio of the absolute intensity of every peak to the absolute intensity of the most intense peak, and then converts to a percentage.

## RESULTS

X-ray powder diffraction (XRD) analysis was carried out on deferrated, NaOH-treated samples of sands, silts and clay using a Philips X-ray diffractometer and Ni-filtered CuK $\alpha$  Radiation at 40 kV and 20 ma. Identification of the clay minerals was based on the basal reflection patterns on the diffractograms (Figs 2-6).

The results obtained from unheated, air dried and unglycolated clay samples shows that the bulk mineral composition of the clays from both Mamu and Nsukka Formations as in the Table 3 comprise of quartz, clay minerals, carbonates, hematite, etc. Kaolinite is the major clay mineral present in both Formations with minor amounts of illite and smectite (with the smectite mainly occurring in the Nsukka Formation along Amuvi).

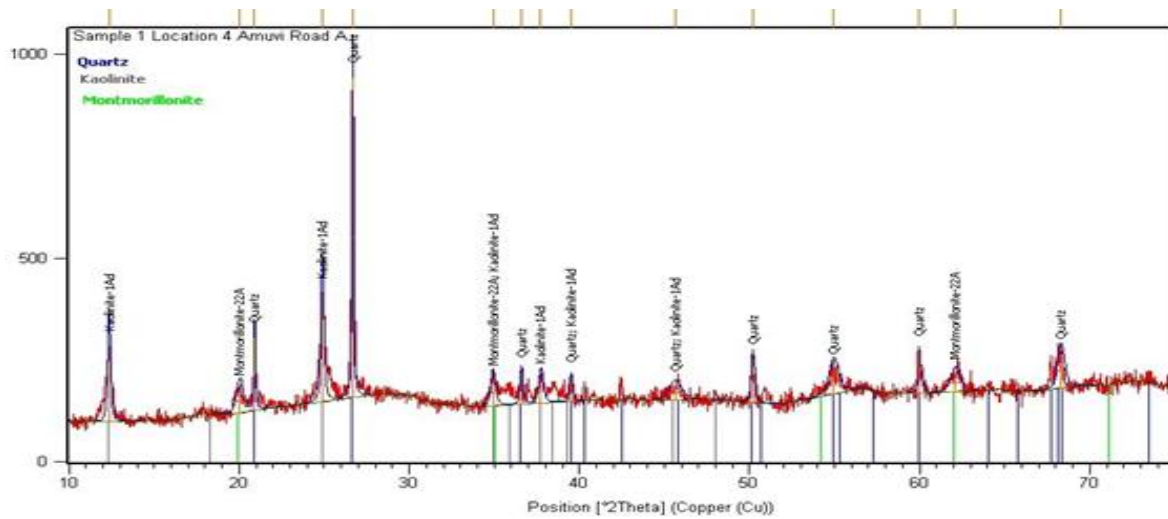


Figure 2: X-Ray Diffraction pattern for Amuvi sample in the Nsukka Formation.

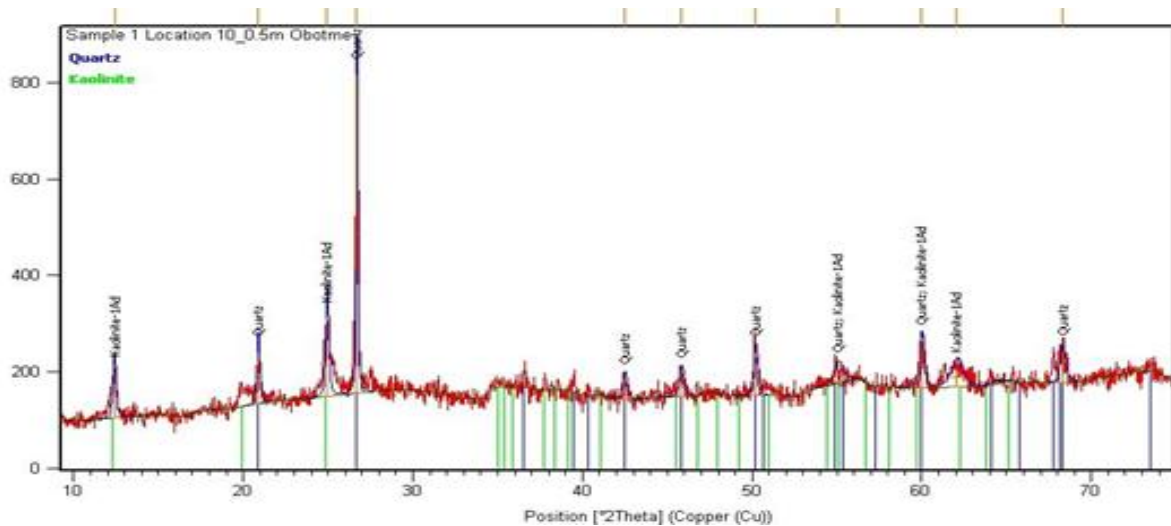


Figure 3: X-Ray Diffraction pattern for Obotme sample in the Nsukka Formation.

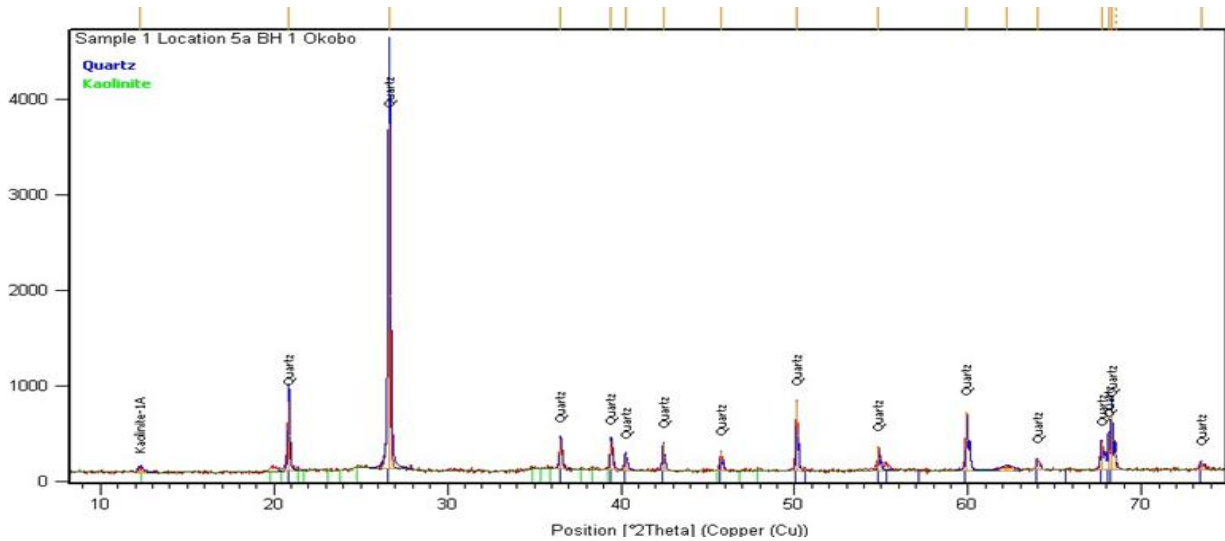


Figure 4: X-Ray Diffraction pattern for Okobo sample in the Nsukka Formation.







is the dominant clay mineral in both Formations followed by minor amounts of illite and montmorillonite. Kaolinite 65 - 80% is continental related clay mineral

specie, is the major clay mineral present in both Formations (Onyeogu et al., 2016).

Table 3: Bulk mineral composition of the Maastrichtian shales

Location	Qtz	Clay (%)	Carbonate (%)	Iron	Al <sub>2</sub> O <sub>3</sub> (%)	SiO (%)	TiO (%)
				Mineral (%)			
AMV1	20.00	33.34	13.33	Nd	9.99	13.32	3.33
AMV2	18.18	30.30	13.12	6.06	12.12	6.06	6.06
OBO1	13.33	33.34	16.67	6.66	10.00	9.99	3.33
OBO2	10.00	28.00	22.00	16.00	14.00	2.00	2.00
OKO	13.79	37.59	20.70	6.70	10.35	10.35	3.45
NKA1	15.15	24.24	27.27	6.06	12.12	3.03	6.06
NKA2	22.73	31.82	18.19	Nd	18.20	nd	4.55
NKA3	17.86	36.71	10.71	7.14	10.71	7.14	3.57
AMA	14.62	34.31	18.13	5.22	11.10	8.21	4.15

nd = Not Determine

Table 4: Clay mineral composition of the Maastrichtian shales

Location	Kaolinite (%)	Illite (%)	Smectite (%)	Others (%)
Amuvi	79.00	6.00	12.00	3.00
Okobo	76.80	9.70	12.30	nd
Obotme	70.40	10.80	15.58	3.22
Nkana	82.00	5.00	13.00	nd
Amakaofia	80.00	6.00	14.00	nd

nd = Not Determine

It mainly occurs in open environments with high acidic content. The low content of potassium, sodium, calcium and magnesium ions in the clays of the Mamu and Nsukka Formations also suggests exhaustive leaching under acidic, warm, tropical conditions. Such conditions are further strengthened by the occurrence of

coal seams and leaf impressions in both Formations. According to Parham (1966), under such conditions, smectite and marine type clay lose their characteristic ions (K<sup>+</sup>, Na<sup>+</sup>, Ca<sup>2+</sup>, and Fe<sup>2+</sup>) while H<sup>+</sup> is added to produce kaolinite. Due to its stability in low pH waters, it may be converted to illite

during diagenesis in the presence of alkaline connate water.

Illite 5-10% is the most common clay mineral found in marine deposits. Formation of illites is generally favoured by alkaline conditions with high concentration of aluminium and potassium. Its presence in the Mamu and Nsukka Formation is minimal (Onyeogu et al., 2016). The low amount of illite from both formations might be as a result of erosion on the rock bearing minerals and the acidic conditions that prevailed during the time of deposition. Smectite composes of 7-15%, clay mineral that commonly results from the weathering of basic rocks. This is favoured by poor drainage which occurs in the presence of calcium at the expense of potassium and mostly associated with mildly alkaline conditions (marine environment). It occurs sporadically in the basin. Smectite is usually known for its abundance in normal marine sediments. In deep offshore, smectite usually settles as finer particles. Kaolinite is mainly concentrated in continental to marginal marine environments. Also during size sorting, kaolinite and illite tends to be concentrated in nearshore environments due to their ability to flocculate.

The clays after purification exhibited the potential for application in the production of refractories. Authors concluded the present of kaolinite mineral in all samples are detrital, transported mostly by SE winds comes from Cross River, and equatorial climate. The clay deposits studied correspond to highly plastic clays with high contents of quartz (Ahmed et al., 2018).

All studied samples were considerably plastic raw materials explaining their

excellent aptitude for pressing except sample no. NKA2, which show less plasticity due to high carbonate and gypsum content. The sample mineralogy indicates quartz as a primary mineral, followed by plagioclase (Onyeogu et al., 2016). Illite, kaolinite, montmorillonite were the main clay minerals in the raw material sampled which is in accordance with Ahmed et al. (2018). The composition of illite, kaolinite and montmorillonite made the clay minerals from OBO2 and NKA3 the most suitable for manufacturing of those products mentioned above.

From the industrial application for clays, originally, compositionally, they are an excellent raw material for the formulation of low porosity ceramic stoneware (Dondi et al., 2014; Folorunso et al., 2014). The presence of illite in all studied samples was influenced by burial diagenesis leads to alteration of smectite to illite (Hoffman and Hower, 1979; Chamley, 1989; Ghandour et al., 2004; Ahmed et al., 2018).

From this experiment of applying samples inside the mold wheel, the best clay can be seen in sample OBO2, this because the clay body kept the same form of the mould cup. Also, it gave a strong body pottery after firing. The next best sample was sample number NKA3, then sample NKA1, NKA2, AMV1 this because of firming of the silicate and less grains. Finally, came sample AMV2, OBO1 and AMA, but this sample was bind during shrinking and change the pottery form of the cup. Sample OKO is not suitable for clay forming in the mould. This because of containing more sands and grains or stones or it has a less silicate. In general, some clay could add

more materials towards occurring or improving the body of the clay samples.

## CONCLUSION

X-Ray diffraction showed that the clay samples from Ozuabam studied consist of illite, montmorillonite and kaolinite and non-clay minerals (quartz, calcite and dolomite). The physical and mechanical properties that concern with the use of clay mineral for clay brick are low volume and linear shrinkage during both drying and firing with higher temperature at 1000°C. The raw clay materials taken from Ozuabam at Southeastern Nigeria were observed to contain these properties and are suitable for producing high quality clay brick. Ozuabam clay is valid for use in artistic and production pottery because of its mineralogical composition, physical and mechanical properties it possessed. The most purify clay from grinds and stones were sample OBO2. The best sample that can be suitable for throwing clay in the wheel is sample (AMV1), (OBO1), (OKO), (NKA1) and finally (OBO2). For press mould of clay samples, the best sample was (OBO2), (AMV1), and sample (NKA2) but with a little bit binding. In other hand the invalid samples were sample (AMV2), (NKA3), and sample (AMA) which crashed after firing. In conclusion, sample (OBO2) was show a good behavior for mold, throwing and less grinding than (OBO1) and (OKO) samples.

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