

EVALUATION OF KAOLIN DEPOSITS IN MAASTRICHTIAN FORMATIONS OF THE AFIKPO BASIN, SOUTHEASTERN NIGERIA

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ABSTRACT

The study area lies within latitude 5°20' to 6°00'N and longitude 7°40' to 7°60'E. Kaolin or china clay, a pure, soft, white clay of variable but usually low plasticity that retains its white color when fired. The chief constituent of kaolin is the mineral kaolinite, a hydrous aluminum silicate, $Al_2Si_2O_5(OH)_4$, formed by the decomposition of aluminum silicates, particularly feldspar. Kaolinite is the dominant clay mineral in both Formations followed by minor amounts of illite and montmorillonite. The plasticity values show samples no. (AMV2, NKA2 and NKA 3) were medium plastic, samples no. (AMV1, OBO1, OBO2, OKO, NKA1 and AMA) are low plastic, clays with plasticity, permits it to be shaped or moulded when mixed with water, and have sufficient wet and air dried tensile strength to maintain their shape after forming. Kaolinite values ranges from 70.40 to 80.00% as the major clay mineral present in both Formations. It mainly occurs in open environments with high acidic content. Under acidic, warm, tropical conditions, smectite and marine type clay lose their characteristic ions (K^+ , Na^+ , Ca^{2+} , and Fe^{2+}) while H^+ 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.

Keywords: Kaolin, Mineral, Evaluation, XRD, Maastrichtian, Afikpo.

INTRODUCTION

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.

Clay, earth or soil that is plastic and tenacious when moist and that becomes permanently hard when baked or fired. Of widespread importance in industry, clays consist 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. 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 decomposed. Clays vary in plasticity, all being more or less malleable and capable of being molded into any form when moistened with water. The plastic clays are used for making pottery of all kinds, bricks and tiles, tobacco pipes, firebricks, and other products. The commoner varieties of clay and clay rocks are china clay, or kaolin; pipe clay, similar to kaolin, but containing a larger percentage of silica; potter's clay, not

as pure as pipe clay; sculptor's clay, or modeling clay, a fine potter's clay, sometimes mixed with fine sand; brick clay, an admixture of clay and sand with some ferruginous (iron-containing) matter; fire clay, containing little or no lime, alkaline earth, or iron (which act as fluxes), and hence infusible or highly refractory; shale; loam; and marl.

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).

Diagenetical processes of clay minerals based on grain sizes under marine conditions shows that clay mineral grain

decreases with depth offshore as a result of water current (Porrenga, 1967), therefore kaolinite will be associated with continental to shallow marine environments while montmorillonite, illites and chlorites will suggest deposition in outer shelf (deep marine) environment.

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. According to Churchman (2000), the two-layer/three-layer clay mineral ratio is mainly controlled by climate.

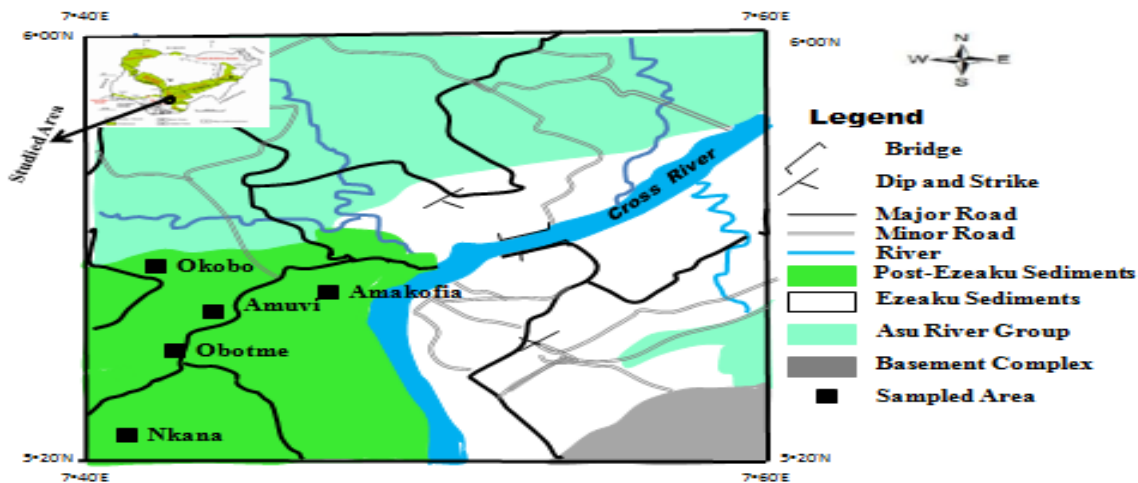


Fig. 1: Geologic map of Afikpo sub-Basin showing the study locations (after Omontese *et al.*, 2019).

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. Conversely, relatively low or no leaching environment and conditions under neutral to low alkaline pH favor the formation of montmorillonite and three layer clay minerals.

Kaolin or china clay, a pure, soft, white clay of variable but usually low plasticity that retains its white color when fired. The material was first obtained from a hill called Kaoling and was sent to Europe in the early 18th century. Pure kaolin is used in the manufacture of fine porcelain and china; impure varieties are used in making pottery, stoneware, and bricks; as filler for pigments; and in the manufacture of paper. The chief constituent of kaolin is the mineral kaolinite, a hydrous aluminum

silicate, $Al_2Si_2O_5(OH)_4$, formed by the decomposition of aluminum silicates, particularly feldspar. The term *kaolin* is often extended to include other porcelain clays not discolored by firing.

Geology of the Area

The Santonian deformational process resulted in the fragmentation of the lower Benue trough (Fig. 2) 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. 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 elobiceras and monticeras ammonites (Ojo, 1999).

AGE		ANAMBRA BASIN	AFIKPO BASIN	CALABAR FLANK
Tertiary	Oligocene	Ogwashi-Asaba Fm	Ogwashi-asaba Fm	
	Eocene Paleocene	Ameki Group Imo Shale Nsukka Formation	Ameki Group Imo Shale Nsukka Formation	
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			Awl Formation
Precambrian		Basement Complex		

Fig. 2: Stratigraphic units of the lower Benue trough (after Ojo, 1999).

MATERIAL AND METHODS

The shale and clay samples from hand dug wells 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 soils are given in Table 1. 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_2O_2 treatment, the separates were Ca-saturated, washed free of excess salt, and dried. Clay samples were also separated from fragments of weathered granitic 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 (DCN) (Mehra and Jackson, 1960). Noncrystalline material was dissolved by boiling DCB-treated samples in 0.5 NaOH (Hashimoto and Jackson, 1960). The Fe 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 $BaCl_2$ 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).

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 3-7).

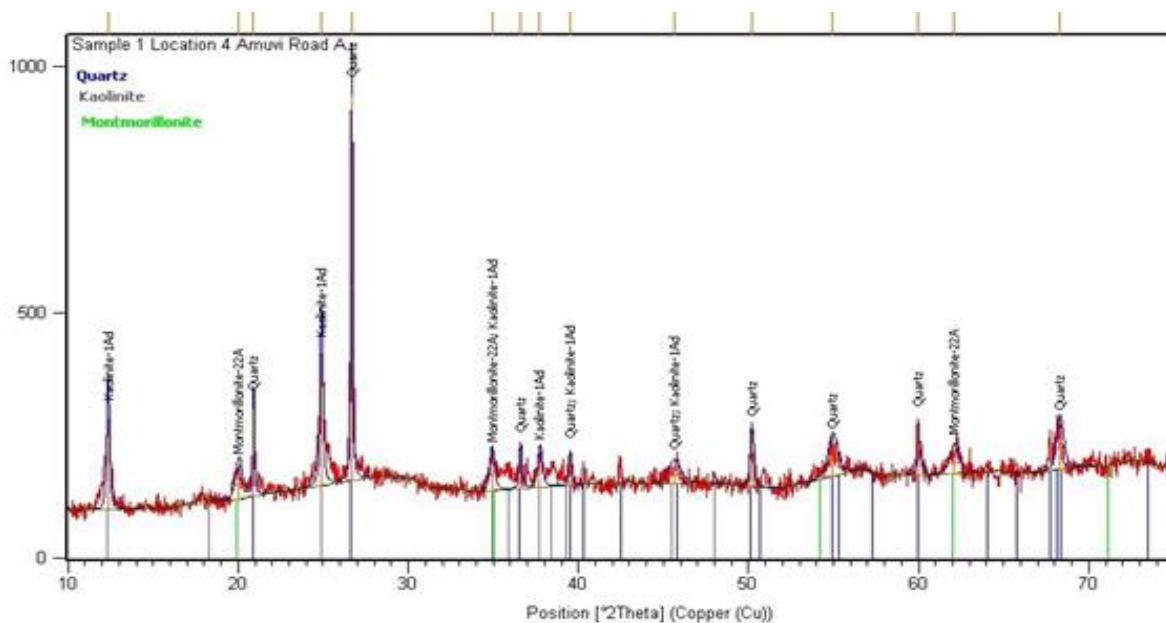


Fig. 3: XRD pattern for Amuvi sample in the Nsukka Formation.

The sands were prepared as random powders, whereas the silts were sedimented onto glass slides. The clays were divided into coarse (2-0.2 μm) and fine (<0.2 μm) fractions by centrifugation. Subsamples were saturated with K^+ , 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 μm , Ca-saturated), equilibrated for at least 4 days in a desiccator over a saturated $\text{Mg}(\text{NO}_3)_2$ solution, using a Stanton-Redcroft 673-4 instrument at a heating rate of 20°C/min in a nitrogen atmosphere. Al_2O_3 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 50 ml of 1 HCl on a water bath at 80°C for 24 hr.

The residue was washed with 25 ml 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^{2+} 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 2θ 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.

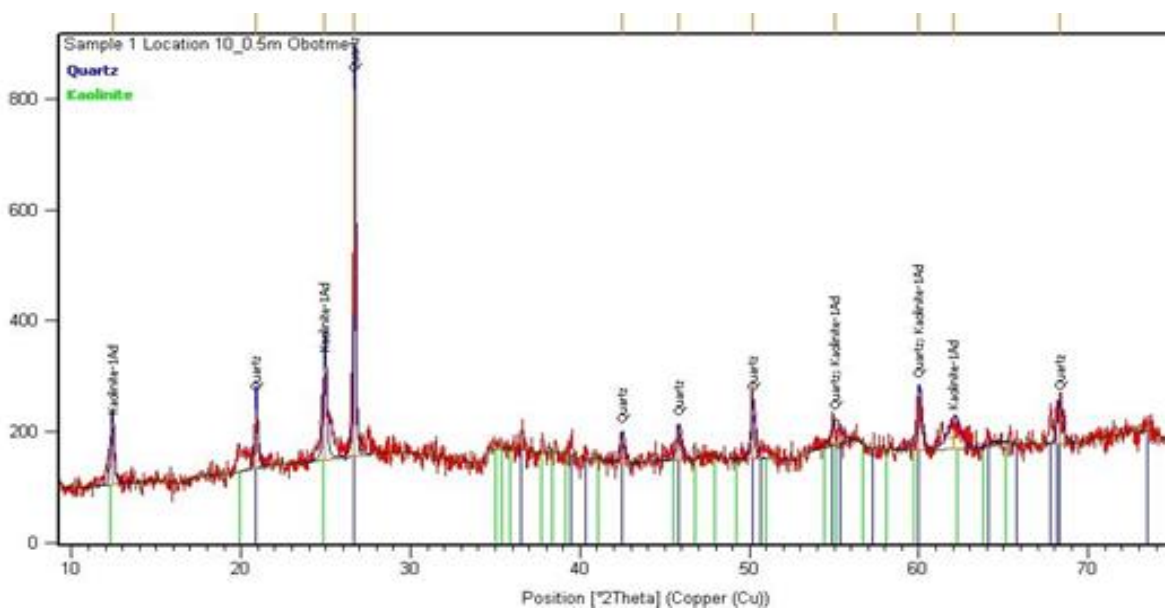


Fig. 4: XRD pattern for Obotme sample in the Nsukka Formation.

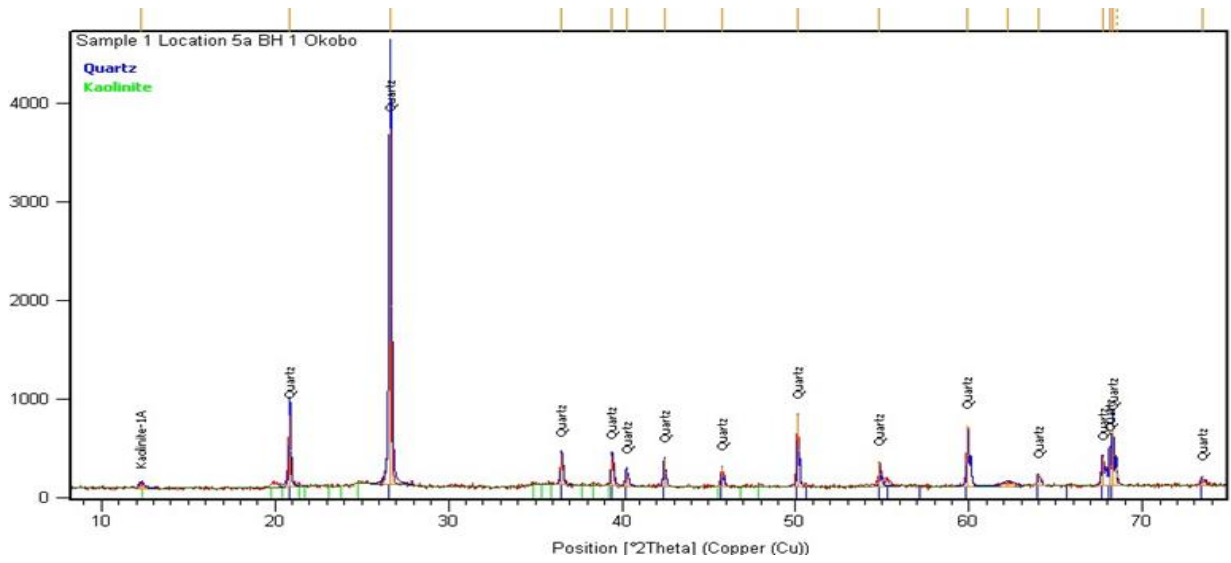


Fig. 5: XRD pattern for Okobo sample in the Nsukka Formation.

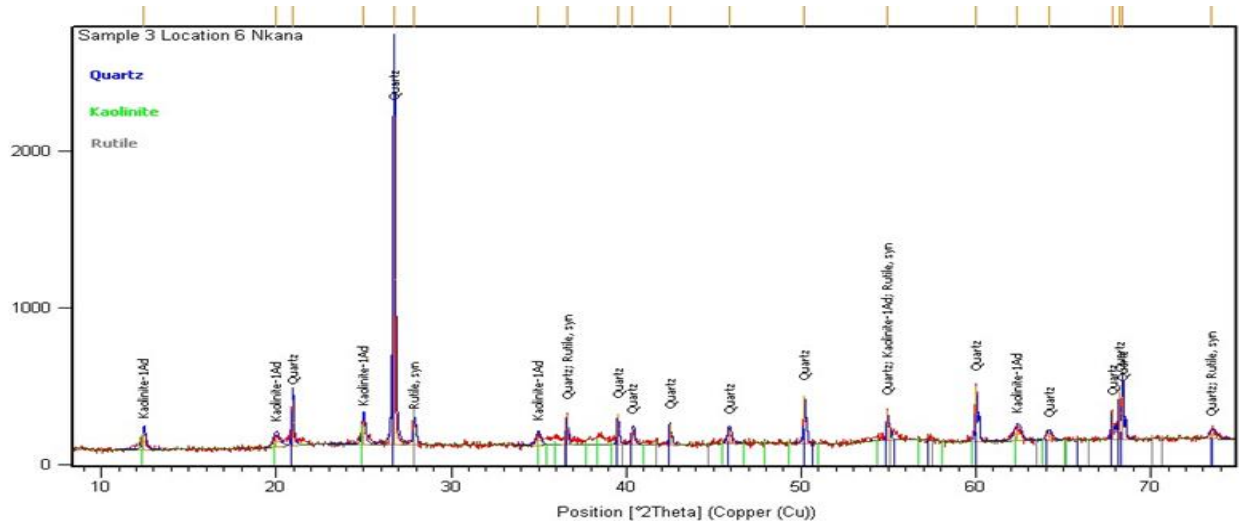


Fig. 6: XRD pattern for Nkana sample in the Mamu Formation.

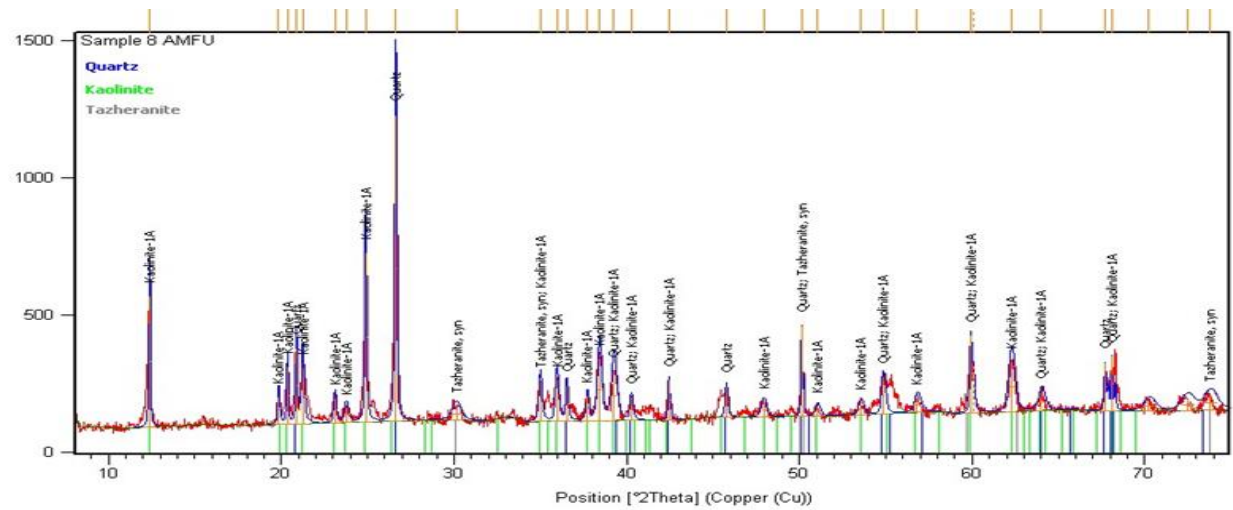


Fig. 7: XRD pattern for Amakofia sample in the Mamu Formation.

RESULTS AND DISCUSSIONS

A proper understanding and evaluation of Kaolin from the Maastrichtian formations of the Afikpo Basin is thus the need for this study. Kaolin is a white, soft, plastic clay composed mainly of kaolinite, $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$, and other related clay minerals such as nacrite and dickite (Baker and Uren, 1982; Abou-El-Sherbini *et al.*, 2017; Jikan *et al.*, 2017; Schroeder, 2018; Omang *et al.*, 2019). Kaolin deposits are classified as primary or secondary according to their genesis; primary deposits originating in situ by alteration, whereas secondary deposits are of sedimentary origin (Murray, 1988; Abou-El-Sherbini *et al.*, 2017; Jikan *et al.*, 2017; Schroeder, 2018; Omang *et al.*, 2019). Primary deposits are formed directly from hydrothermal alteration of volcanic and granitic rocks with examples as hydrothermal and volcanic clay types. Secondary clay deposits are products of weathering of pre-existing rocks, and subsequent alteration of the alumino-silicates into clays such as residual and sedimentary clays. However, clay can further be classified on the basis of genetic and industrial characteristics.

The formation and localization of sedimentary clay are controlled by the location of the sedimentary basin and the presence of weathered feldspar-rich rocks such as granite, syenite or gneiss adjacent to the basin, particularly rapidly eroding paleo-topographic highs. Ideal conditions necessary to produce kaolinitic clays by means of chemical weathering are high rainfall, warm temperatures, lush vegetation, low relief and high groundwater table (Cravero and Dominguez, 1999; Abou-El-Sherbini *et al.*, 2017; Jikan *et al.*, 2017; Schroeder, 2018; Omang *et al.*,

2019). Thus, kaolin is eroded and transported by streams to a quiet, fresh or brackish water environment. Post depositional leaching, oxidation, and diagenesis can significantly modify the original clay mineralogy with improvement of kaolin quality (Hassan, 2014; Abou-El-Sherbini *et al.*, 2017; Jikan *et al.*, 2017; Schroeder, 2018; Omang *et al.*, 2019). Kaolin has a wide range of application among which is the ceramic industry. Each specific use, to which kaolin is put, requires that it possess certain specific properties.

Kaolin is an important raw material with widespread industrial applications such as water treatment, as porcelain, cement and ceramics production (Abou-El-Sherbini *et al.*, 2017; Lima *et al.*, 2017; Jikan *et al.*, 2017; Schroeder, 2018; Omang *et al.*, 2019) and equally as fillers for polymer, pigment in paint/inks and plastics/rubber (Kotal and Bhowmick, 2015; Abou-El-Sherbini *et al.*, 2017; Jikan *et al.*, 2017; Liu *et al.*, 2017; Schroeder, 2018).

Some Pigments from kaolin offer opacity and brightness because they have the ability to scatter light. The degree to which the minerals scatter light depends on their intrinsic index of refraction and also on the structure they form in the coating. Titanium dioxide has high refractive index, strong absorption in the UV region of light spectrum and strong reflectance in the visible spectrum thereby giving it a very high light scattering properties which, when combined with its particle size creates an effective pigment for brightness and opacity (Imerys, 2013; Omang *et al.*, 2019). Also, some organic pigment form from kaolin absorbs UV light thereby preventing it from damaging the binder in paint formation (Clark, 2013; Abou-El-Sherbini *et al.*,

2017; Jikan *et al.*, 2017; Schroeder, 2018; Omang *et al.*, 2019).

The results obtained from unheated, air dried and unglycolated shale samples shows that the bulk mineral composition of the shales from both Mamu and Nsukka Formations as in the Table 1 comprise of quartz, clay minerals, carbonates, iron rich minerals such as 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). Secondary minerals were difficult to identify due to the greater peaks of kaolinite and quartz. The shales are dominated by quartz, clay minerals and minor amounts of carbonates and iron rich minerals (Table 2). Kaolinite is the dominant clay mineral in both Formations followed by minor amounts of illite and montmorillonite. The figures 6 and 7 below are results from the XRD analysis.

Table 1: Bulk mineral composition of the Maastrichtian sediments

Location	Qtz	Clay	Carbonate	Iron Mineral	Al₂O₃	SiO	TiO
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

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. Illite, kaolinite, montmorillonite, smectite were the main clay minerals in the raw material samples (Abou-El-Sherbini *et al.*, 2017; Jikan *et al.*, 2017; Ahmed *et al.*, 2018; Schroeder, 2018).

Table 2, kaolinite (70.40 - 80.00%), which is continental related clay mineral specie, is the major clay mineral present in both

Formations. it mainly occurs in open environments with high acidic content. The low content of potassium, sodium, calcium and magnesium ions in the shales of the Nsukka and Mamu 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 Rao and Srikari (1980), under such conditions, smectite and marine type clay lose their characteristic ions (K^+ , Na^+ , Ca^{2+} , and Fe^{2+}) while H^+ 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.

Table 2: Clay mineral composition of the Maastrichtian sediments

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

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; Abou-El-Sherbini *et al.*, 2017; Jikan *et al.*, 2017; Schroeder, 2018). 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).

CONCLUSION

Based on the results of this study the following conclusions have been reached out; the plasticity values show samples no. (AMV2, NKA2 and NKA 3) were medium plastic, samples no. (AMV1, OBO1, OBO2, OKO, NKA1 and AMA) are low plastic, clays with plasticity, permits it to be shaped or moulded when mixed with water, and have sufficient wet and air dried tensile strength to maintain their shape after forming. Kaolinite values ranges from 70.40 to 80.00% and is continental related clay mineral specie. It is the major clay mineral present in both Formations. It mainly occurs in open environments with high acidic content. The low content of potassium, sodium, calcium and magnesium ions in the shales of the Nsukka and Mamu Formations also suggests

exhaustive leaching under acidic, warm, tropical conditions.

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