

Clay Minerals Assessment from Maastrichtian Syclical AFIKPO, Nigeria

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Abstract- The Maastrichtian Mamu and Nsukka Formations in the Anambra Basin (SE Nigeria) consists of a cyclic succession of coals, carbonaceous shales, silty shales and siltstones interpreted as deltaic deposits. The Anambra Basin was formed at the NW of the anticlinorium and the Afikpo syncline at the SE part of the anticlinorium, making both basins a depocenter during the Campanian to Maastrichtian. Clay fractions of shale samples obtained from the Cretaceous Mamu and Nsukka Formations in the Afikpo Basin, south eastern Nigeria through the process of sedimentation technique were air – dried and analyzed using the empyrean diffractometer manufactured by Panalytical to determine the paleoclimate and depositional environments of the shales. The result shows that the bulk mineral composition of the shales comprises of quartz, clay minerals, carbonates and iron rich minerals, while the dominant clay mineral is kaolinite (70 – 80%) with minor amounts of illite (4 – 7 %) and smectite (10 – 20). This shows that both Formations were deposited under warm and humid conditions (typical of tropical to sub – tropical climate), while the depositional environment with evidence from biostratigraphy is suggested to be a near shore estuarine environment.

Index Terms- Clay mineralization, biostratigraphy, depositional environment, Maastrichtian, Afikpo Basin.

I. INTRODUCTION

The area is within the Anambra Basin, it is sandwiched between the lower Benue Trough and the Niger Delta basin, its origin and structure is derived from the Santonian compressional movement along NE-SW trend, a second structural cycle from the three major tectonic cycles (Murat, 1972). This resulted in the folding and uplift of the southern Benue trough. All pre-Santonian deposits were folded, faulted and uplifted, thereby forming the Abakaliki anticlinorium. The Anambra basin was formed at the NW of the anticlinorium and the Afikpo syncline at the SE part of the anticlinorium, making both basins a depocenter during the Campanian to Maastrichtian. (Reyment, 1965).

Clay minerals refer to sheet-structured alumino-silicate minerals that primarily occur in the clay-sized (2 μ m) fraction of soils, sediments, sedimentary rocks, and weathered or altered rocks (Nesse, 2000). They are formed as a result of chemical weathering of pre-existing rocks, through hydrothermal alteration of granitic rocks and as a result of diagenesis.

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). 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).

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. 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.

II. GEOLOGY OF THE AREA

The infilling of the Anambra and Afikpo basins started during the early Campanian to the early Paleocene (Danian) under two major eustatic cycles; the more pronounced Nkporo transgression and the less active Nsukka transgression with the Anambra basin showing the most complete stratigraphic sections (Fig. 1). These cycles are also found in the Afikpo syncline SE of the Abakaliki anticlinorium and the Dahomey embayment, west of the Ilesha basement spur, although both are incomplete (Murat, 1972).

The first cycle which took place during the Lower Campanian to the Maastrichtian started with the deposition of the Nkporo shale whose lateral (age) equivalents are the Enugu shale and Owelli sandstone (Fig. 2). This is the basal unit of the Campano-Maastrichtian transgression and comprises of dark

mudstone, gray, fissile friable shales with thin beds of marl, sandy shale and limestone overlying an angular unconformity (Reyment, 1965). The regressive phase was marked with the development of a large offlap complex, starting with the paralic sequence of the Mamu formation (Lower coal measure) overlying the Nkporo shale (Reyment, 1965). It is thought to be lower Maastrichian in age with a basal part that contains thin marine intercalations, while the coal bearing part consist of fresh water and low salinity sandstones, shale, mudstone and sandy shales with coal seams occurring at several levels (Simpson, 1955).

The Mamu formation is overlain by the continental sequence of the Ajali sandstone. This sandstone unit has received several names such as false bedded sandstone (Tattam, 1944), basal sandstone (Simpson, 1955) etc. its present name was given by Reyment (1965) after establishing its type locality at the Ajali river. Virtually all exposures of the formation are characterized by a lateritic profile at the top. It was deposited during the regressive phase of the Campano-Maastrichian transgression and is age Maastrichian.

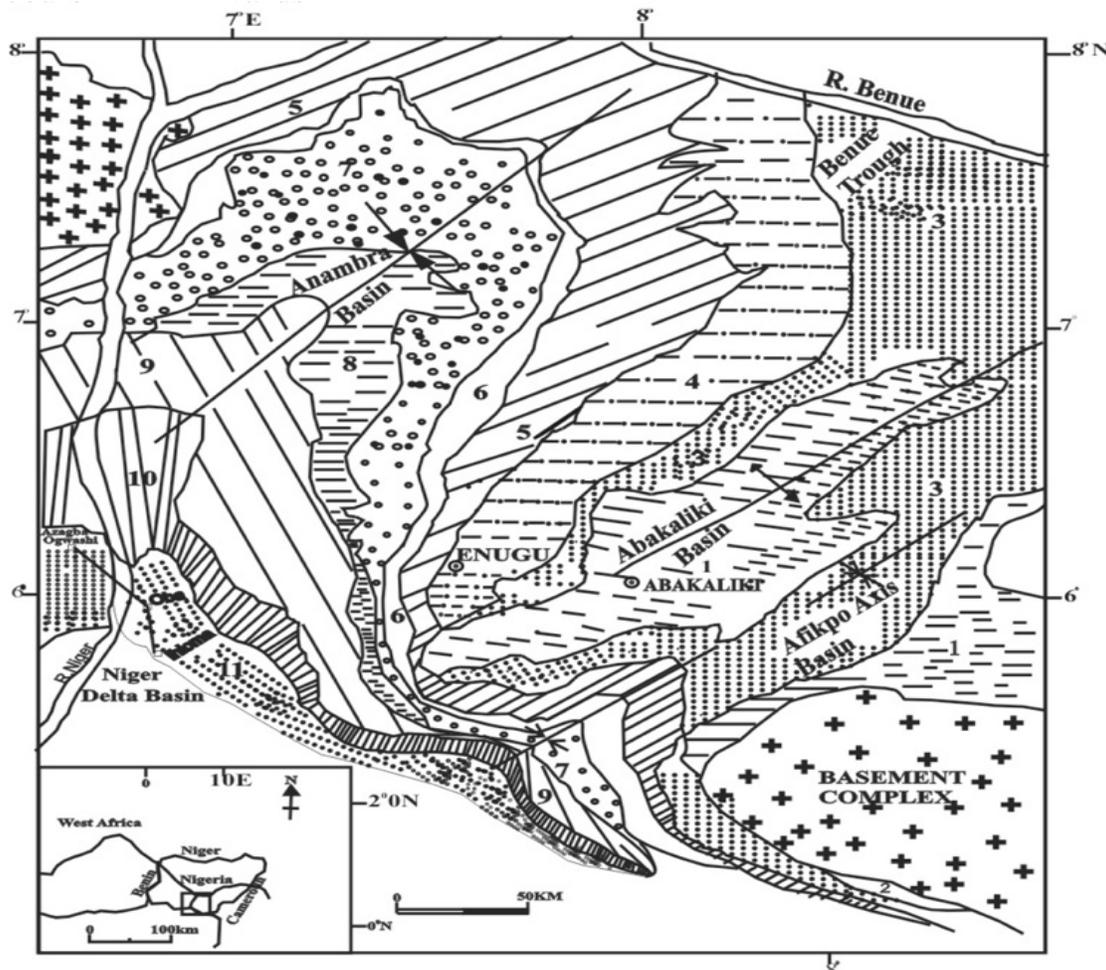


Figure 1: Generalized geological map of southeastern Nigeria (boxed area of inset) showing the location of the Cretaceous to Tertiary sequences north of the Niger Delta Basin. Numbers of the formations as follows: 1. Asu River Group; 2. Odukpani Formation; 3. Eze-Aku Shale; 4. Awgu Shale; 5. Enugu/Nkporo Shale; 6. Mamu Formation; 7. Ajali Sandstone; 8. Nsukka Formation; 9. Imo Shale; 10. Ameki Formation; 11. Ogwashi-Asaba Formation (After Akande et al., 2007).

The Ajali sandstone is overlain conformably by the Nsukka formation (Upper coal measures), and it consists of alternating succession of gray sandy shales, sandstones, plant bearing beds

and thin beds of coal (Reyment, 1965). Thin bands of marine limestone heralded the return of marine sedimentation at the top of the formation. These dark shales and the intensely bioturbated sandstones are well exposed at Ihube, along the Enugu – Port Harcourt expressway. The age range of the formation is late Maastrichian to Danian based on the fossil record. This formation bears the K/T boundary which is described by Reyment (1965) as a period of transition in Nigeria. Mbuk et al. (1985) identified this boundary in the Nsukka Formation in Ozu Abam area of Abia state.

AGE	SEDIMENTARY SEQUENCE	LITHOLOGY	DESCRIPTION	DEPOSITIONAL ENVIRONMENT	REMARKS		
					Coal Rank	ANKPA SUB-BASIN	ONITSHA SUB-BASIN
MIOCENE OLIGOCENE	OGWASHI-ASABA FM.		Lignites, peats, Intercalations of Sandstones & shales	Estuarine (off shore bars; Intertidal flats)	Liginites	NO DEPOSITION	REGRESSION
EOCENE	AMEKE/NANKA FM. SAND		Clays, shales, Sandstones & beds of grits	Subtidal, intertidal flats, shallow marine	Unconformity		(Continued Transgression Due to geoidal Sea level rise)
PALEOCENE	IMO SHALE		Clays, shales & siltstones	Marine		(? MINOR REGRESSION)	
MAASTRICHTIAN	NSUKKA FM.		Clays, shales, thin sandstones & coal seams	? Estuarine	Sub-bituminous	TRANSGRESSION (Geoidal sea level Rise plus crustal Movement)	
	AJALI SST.		Coarse sandstones, Lenticular shales, beds of grits & Pebbles.	Subtidal, shallow marine	Sub-bituminous		
	MAMU FM.		Clays, shales, carbonaceous shale, sandy shale & coal seams	Estuarine/ off-shore bars/ tidal flats/ chemier ridges	Sub-bituminous		
CAMPANIAN	ENUGU/ NKPORO SHALE		Clays & shales	Marine	3 rd Marine cycle	Unconformity	
CONIACIAN-SANTONIAN	AWGU SHALE		Clays & shales	Marine	2 nd Marine cycle		
TURONIAN	EZEAKU SHALE		Clays & shales	Marine	1 st Marine cycle	Unconformity	
CENOMANIAN	ODUKPANI FM.					Unconformity	
ALBIAN	ASU RIVER GP.					Unconformity	
L. PALEOZOIC	B A S E M E N T C O M P L E X						

Figure 2: The Stratigraphy of the Anambra Basin Southeastern Nigeria (After, Ladipo, 1988 and Akande et al, 1992; Modified in Uzoegbu et al., 2013b).

MATERIAL AND METHOD

The field analysis involved the collection of shale and clay samples from hand dug wells in the Nsukka and Mamu Formations from the Afikpo Basin (Fig. 3). Shale samples of the Nsukka Formation were collected at Amuvi, Obotme, and Okobo

while those of Mamu Formation were collected at Nkana and Amakofia, all within the Afikpo Basin.

Clay fractions (<2µm) was extracted from the samples through sedimentation technique. These samples were air-dried for about 24 hours and mounted on glass slides. The air-dried oriented

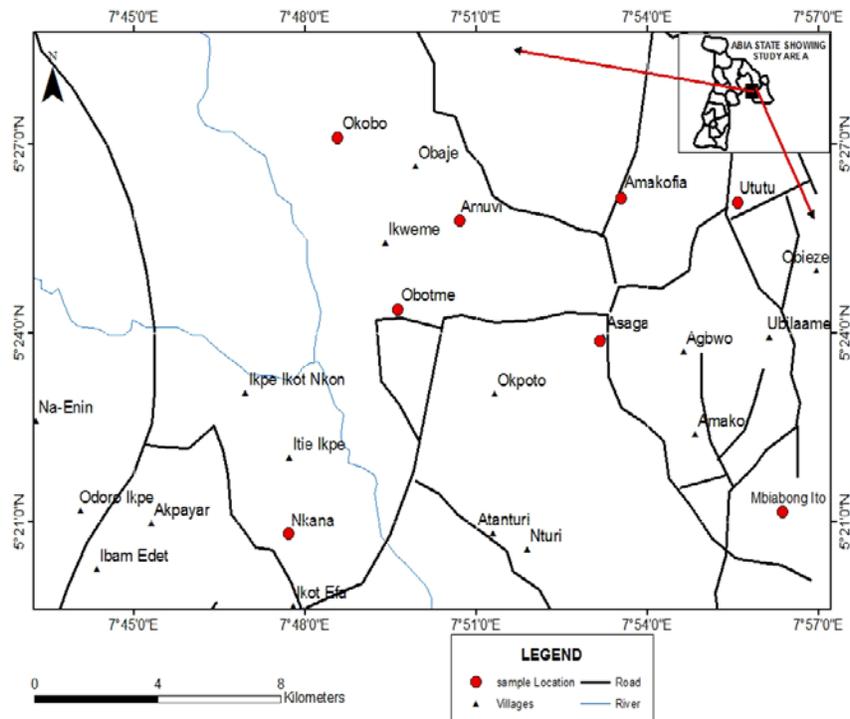


Figure 3: Location map of the sampled area

samples were analyzed using X-Ray Diffractometer (XRD) with copper K alpha radiation running at a rate of 2θ (Copper K alpha radiation was preferred because of its relatively high pick energy, intensity, low peak to background ratio, convenient wavelength, the high heat conductivity of the target material and the symmetry of the K alpha 1 and K alpha 2 composite peaks).

Identification of the clay minerals was based on the basal reflection patterns on the diffractograms (Figs 4 and 5). The diffraction angles (2θ) produced the reflection peaks which correspond to the basal spacing (d-spacing) of the mineral.

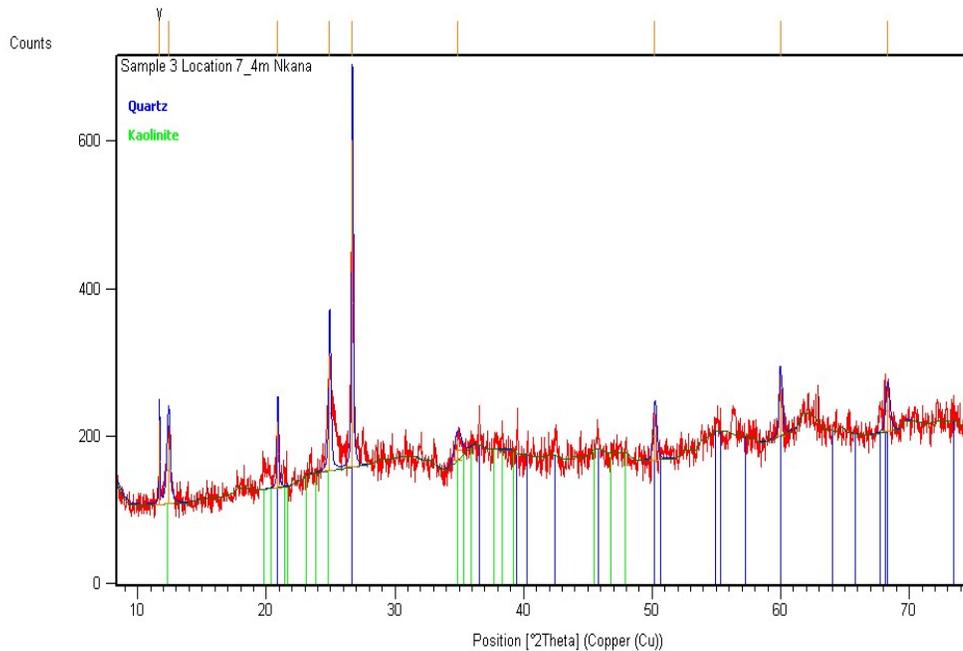


Figure 4: XRD pattern for Mamu Formation

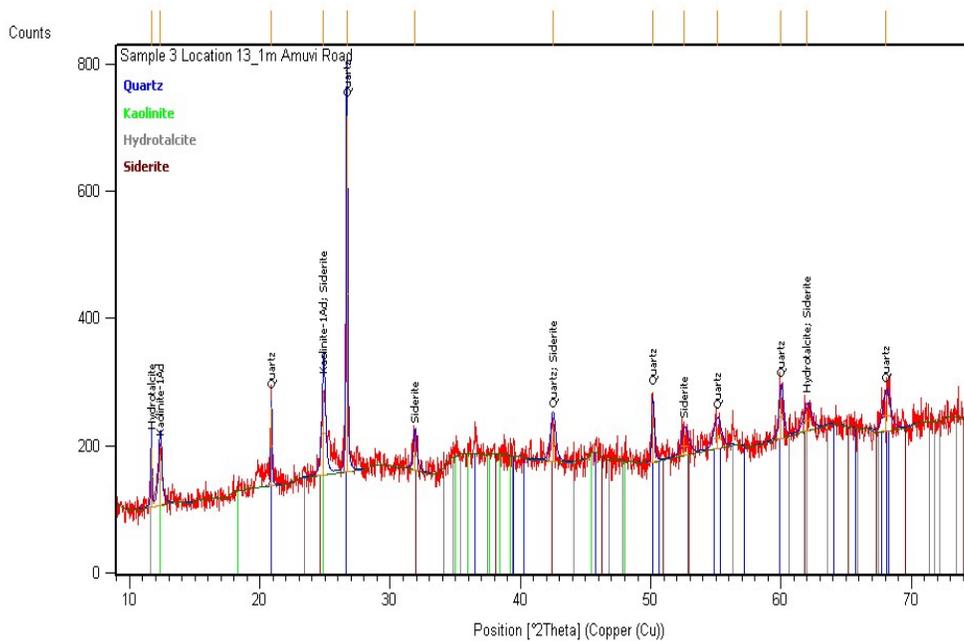


Figure 5: XRD pattern for Nsukka Formation

Comparison of the reflection peaks with those from established standard minerals such as the Joint Committee on Powder Diffraction Standard (JCPDS) alongside other references helped in the identification of the clay mineral assemblage.

The data was analyzed at the Nigerian Geological Research Laboratory (NGRL), Kaduna using an Empryan Diffractometer with a copper anode material manufactured by Panalytical.

The samples obtained were finely grounded to pass through 63µm, homogenized and average bulk composition was determined. Powdered samples were then prepared using the sample preparation block and compressed in a flat sample holder to create a flat, smooth surface that was later mounted on the sample stage in the XRD cabinet.

Mounted samples were analyzed using the reflection – transmission spinner stage with 2 theta settings. 2 theta starting position was 0.00483 and ends at 75.000 with a 2 theta step of 0.026 at 37s per step. Tube current was 40Ma and the tension was 45VA. Fixed divergent slit size of 1° was used and the Goniometer radius was 240mm.

The intensities of diffracted x-rays were continuously recorded as the sample and detector rotate through their respective angles. A peak in intensity usually occurs when a mineral contains lattice planes with d-spacing appropriate to diffract x-rays at the value of θ . Also, each peak consists of two separate reflections ($K\alpha_1$ and $K\alpha_2$), at small values of 2θ , the peak locations overlap with $K\alpha_2$ appearing as a hump on the side of $K\alpha_1$. Greater separations occurs at higher values of θ .

Although these combined peaks were treated as one. The 2λ position of the diffracted peak is typically measured as the centre of the peak at 80% height.

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.

III. RESULT AND DISCUSSION

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 figures 4 and 5 below are results from the XRD analysis.

Table 1: Bulk mineral composition of the shales

Location	Qtz	Clay	Carbonate	Iron Mineral	Al ₂ O ₃	SiO	TiO
Amuvi	20.00	33.34	13.33	nd	9.99	13.32	3.33
Amuvi	18.18	30.30	13.12	6.06	12.12	6.06	6.06
Obotme	13.33	33.34	16.67	6.66	10.00	9.99	3.33
Obotme	10.00	28.00	22.00	16.00	14.00	2.00	2.00
Okobo	13.79	37.59	20.70	6.70	10.35	10.35	3.45
Nkana	15.15	24.24	27.27	6.06	12.12	3.03	6.06
Nkana	22.73	31.82	18.19	nd	18.20	nd	4.55
Nkana	17.86	36.71	10.71	7.14	10.71	7.14	3.57

nd = Not Determine

Clay Mineral Distribution

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. Kaolinite 65 -80% 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 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 Rao and Srikari (1980), under such conditions, smectite and marine type clay lose their characteristic ions (K⁺, Na⁺, Ca²⁺, and Fe²⁺) 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 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

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. 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 is within 7-15%, a clay mineral that commonly results from the weathering of basic rocks, favoured by poor drainage, occurs in the presence of calcium at the expense of potassium and mostly associated with mildly alkaline conditions (marine environment). They occur sporadically in the basin. While kaolinite is mainly concentrated in continental to marginal marine environments, smectite is usually known for its abundance in normal marine sediments. Also, during size sorting, while kaolinite and illite

tends to be concentrated in near shore environments due to their grain sizes and their ability to flocculate, smectites usually settles as finer particles in deep offshore environments.

Depositional Environment

The clay minerals of the Mamu and Nsukka Formations are largely dominated by kaolinites with minor amounts of illite and smectite as shown on Table 2. This shows that both formations have higher percentages of continental to marginal related clay mineral (kaolinite) than marine type clays such as illite and smectites. Parham (1966) stated that kaolinite is also known to be concentrated in near shore sediments and decrease in abundance with distance from the shoreline as other clay minerals (illites and smectites) increases.

According to Ideozu (2014), the depositional environment of Mamu and Nsukka Formations were interpreted as shallow inner neritic to outer neritic based on the occurrence of ostracods, pelecypods and gastropods along with foraminifera. An overall increase in the abundance of pollen/spores in the investigated area suggests proximity to the shore line.

Base on the dominant clay mineral reported, and the evidence from biostratigraphic studies, a near shore estuarine environment is been reported as the depositional environment for the two formations. Also, the presence of non-clay minerals such as siderite and periclase in the Nsukka Formation, hematite and rutile in the Mamu Formation also shows that both formations must have being deposited under low ph conditions with low oxygen.

Significance of Clay Minerals in Paleoclimate Determination

Climate is one major factor that has control over the type of clay minerals that will be deposited in a particular location at a particular point in time; therefore, it can be used in the determination of paleoclimates. According to Churchman (2000), the two-layer/three-layer clay mineral ratio is mainly controlled by climate. Therefore, it is easy to recognize between warm and humid conditions typical for kaolinite formation, or dry seasons, specific for illite or smectite formation. Furthermore, the formation of kaolinite is favoured by acidic (pH ~3) conditions, and high leaching environments. While relatively low or no leaching environment and conditions under neutral to low alkaline pH favour the formation of montmorillonite and other three layer clay minerals.

The dominant clay mineral in both formations is kaolinite and is formed under warm, humid, acidic conditions and high leaching environment. Under such climatic conditions, minerals such as smectite and illite lose their characteristic ions (K^+ , Na^+ , Ca^{2+} and Fe^{2+}), while H^+ is added to produce kaolinite (Rao and Srikari, 1980). Also, detrital kaolinite formation is highly favoured under tropical to sub-tropical humid climatic conditions (Chamley, 1989), although kaolinite can also be formed by diagenetic processes and such processes may hinder its use for paleoclimatic studies. During diagenesis, the morphology of kaolinite changes from aggregates of books (its bookshelves shape) to individual blockier crystals. However, previous studies have shown that there is no evidence of the role of diagenesis in the formation of the clay minerals in the study area. Smectites being one of the least clay minerals identified, on the other hand, is favoured by conditions of prolonged dry seasons alternating with less pronounced wet seasons. Other factors that favour its formation are mildly alkaline marine environments, gently sloppy terrains that are poorly drained and the availability of calcium at the expense of potassium. Also the presence of coal seams and leaf impressions occurring in both formations confirms the existence of such humid conditions during their deposition.

Based on the dominant clay mineral reported, it shows that the Mamu and Nsukka Formations must have being deposited under warm and humid conditions (tropical to sub – tropical climate).

IV. CONCLUSION

Clay mineral assessments of shales from Mamu and Nsukka Formations within the Afikpo Basin have being used to evaluate the depositional environments and paleoclimatic conditions during their formations.

Mineralogical analysis carried out using X-Ray Diffractometer revealed that the bulk mineral composition comprises of silica, clay minerals, carbonates and iron rich minerals. The clay minerals are dominated by kaolinites with minute amount of illites and smectites; suggested to be as a result of provenance, climatic factors, size, sorting and depositional environments. The dominance of kaolinites and evidence from biostratigraphic analysis suggests that the shales must have being deposited in a near shore environment (estuarine). Also, the dominance of kaolinite in the shales reflects that they must have being subjected to strong chemical weathering with very good leaching, favoured by tropical to sub-tropical humid climates. This evidence is further strengthened by the presence of coal seams and leaf impressions in the Mamu and Nsukka Formations. This assessment has shown that despite that both Formations are in different stratigraphic positions. They were both deposited under similar climatic conditions and depositional environments though slightly different lithostratigraphic units (Uzoegbu et al., 2013a).

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