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Compositional Characteristics and Industrial Assessment of the Cretaceous Clay Deposits within Northern Anambra Basin, Nigeria

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Abstract
Compositional and physical characteristics of the Createaceous clay deposits within Mamu Formation of Northern Anambra Basin outcropped at Aloji, Udane Biomi, Ofe jiji and Agbenema of Kogi Sate, Northern Nigeria were assessed to evaluate their potentials as raw materials. The deposits are of varying thicknesses and are capped by thin lateritic soil. The clay varies from white to dirty-white and greyish/brownish in colour as a result of the decolouration from the lateritic overburden. The physical parameters of the clay specimens include specific gravity (2.11 – 2.40), liquid limit (17.38 – 35.80), plasticity index (14.05 – 25.43) and loss on ignition (2.70 – 11.10) values. The mineralogical determination by x-ray diffraction (XRD) of the clay, indicate kaolinite as the major clay mineral with quartz and microcline as the non-clay mineral. Chemical analysis of the samples reveals the predominance of SiO_2 (69.67- 90.28%) and Al_2O_3 (6.32- 19.22%) with a silica/alumina ratio of 3.50 – 14.73%.The clay is also characterized by significant Fe_2O_3 (0.32 – 2.06%), TiO_2 (0.79 – 1.63), K_2O (0.04 – 1.87%) values, while CaO, MgO, Na2O, MnO and P_2O_5 have individual values < 1%. Physical parameters, Mineralogical and chemical compositions suggest that the clay deposit could be exploited for ceramics, paint, pottery and refractory industries.
Keywords: Clay minerlas, Cretaceous, Northern Anambra Basin, Industrial Assesment, Mineralogy, Geochemistry.

Introduction

Clays are naturally occurring hydrous alluminosillicates which are composed of mixtures of ultra fine grained clay minerals, crystals of other minerals and metal oxides (Guggenheim and Martin, 1995). The different minerals present in clay account for its different properties, plasticity being one of these (Wikipedia, 2011). The various clay properties in turn account for their wide range of industrial and domestic applications.

Plasticity, adsorptivity, cation exchange and shrink-swell capacity are perhaps the most important properties of clay as they are the most frequently exploited for industrial purposes; plasticity in pottery (Amethyst Galleries, 2011), adsorptivity and ion exchange in water purification (Mockoviakova and Orolinova, 2009) as well as the removal of heavy metal pollutants and other unwanted substances from the environment., industrial waste effluents and other media in which they are present. One study (David, 1976) which investigated the interaction of petroleum heavy-end with montmorillonite reported that the adsorption of asphaltenes onto montmorillonite occurred rapidly, and to a large extent, irreversibly under near anhydrous laboratory conditions and that the adsorption was influenced by exchangeable ions on the clay, neighbouring molecules and the solvent. In similar studies (especially with cationic sorbates), clays seem to be receiving tremendous attention as the preferred adsorbent. Studies abound where clays or their modified forms have been used for metal removal from aqueous solutions. Illite has been used for the removal of Cd^{2+} (Echeverria *et al*, 2002), natural and Na-exchanged bentonites have been used for Cr^{3+}, Zn^{2+} and Cu^{2+} adsorption (Alvarez and Garcia, 2003). Sepiolite has been used for Co^{2+} (Kara *et al*, 2003)], and kaolinite for Mn^{2+}, Co^{2+}, Ni^{2+} and Cu^{2+} removal (Bhattacharyya and Sengupta, 2006).

The largest application of kaolinite is in the manufacture of glossy paper. This is because Kaolinite is soft, earthy and usually white dioctahedral clay (Agbende, 2010). It is also used as food additive, in white incandescent light as well as in cosmetics (Bear, 1978). Smectites on the other hand are known for their ability to absorb significant amounts of water molecules between their tetrahedral and octahedral sheets thus causing a significant expansion of their crystal lattice than observed for other clay types and as such they have found application in the oil industry as a component of drilling mud (Bear, 1976). Montmorillonite has also been used to hold soil water in drought–prone soils (Mukherge and Biswas, 1974).

Several clay occurrences have been investigated and reported in Nigeria. These occurrences vary from the lateritic and residual profiles derived from the weathering of basement rocks (Elueze and Bolarinwa, 1995, 2001; Nton and Elueze, 2005) to those of the sedimentary units and alluvial bodies within the various depositional basins and along the major fluvial channels (Emofurieta et al, 1994; Elueze et al, 1999; Imeokparia and Onyeobi, 2007; Obrike et al, 2007, Idakwo et al, 2013, Alege et al, 2014). The interest in these studies is hinged on the effective exploitation of the local raw materials for sustainable economic growth and creation of employment opportunities (Elueze, 1998)

Because the chemical composition of given clay accounts for its chemical and physical properties and by extension its application, it has become necessary to characterize clay samples from different locations with a view to sourcing and identifying the most suitable clay for different applications, therefore this study aimed at reporting the compositional and geotechnical properties of the Cretaceous Clay deposits in Kogi State, Northern Anambra Basin of Nigeria, compares these with properties of clay deposits elsewhere and known standards, with view to ascertaining its industrial potentials and thereby sourcing efficient low-cost materials for informed industrial applications.

Geological Description

Clay mineral and geochemical distribution was studied in major clay units within the age of Createaceous (Campanian-Maastrichtian) (Figures 1a and b) in different geological situation that occurred in the Northern Anambra, North Central Nigeria (Figures 1a and b). This unit is described briefly.

Sedimentation in the Anambra Basin commenced with the Campanian-Maastrichtian marine and paralic shales of the Enugu and Nkporo Formation (Figure 2). The fluviodeltaic sandstones of the Ajali and Owelli Formations lie on the Mamu Formation and constitute its lateral equivalents in most places. The coal-bearing Mamu Formation and the Ajali Sandstone accumulated during this epoch of overall regression of the Nkporo cycle. The Mamu Formation occurs as a narrow strip trending north–south from the Calabar Flank, swinging west around the Ankpa plateau and terminating at Idah near the River Niger. The Nsukka Formation and the Imo Shale mark the onset of another transgression in the Anambra Basin during the Paleocene. The shales contain significant amount of organic matter and may be a potential source for the hydrocarbons in the northern part of the Niger Delta (Reijers and Nwajide, 1998). In the Anambra Basin, they are only locally expected to reach maturity levels for hydrocarbon expulsion. The Eocene Nanka Sands mark the return to regressive conditions. The Nanka Formation offers an excellent opportunity to study tidal deposits. Well-exposed, strongly assymetrical sandwaves suggest the predominance of flood-tidal currents over weak ebbreverse currents. The presence of the latter are only suggested by the bundling of lamine separated from each other by mud drapes reflecting neap tides. A good outcrop of the Nanka Formation is the Umunya section, 18 km from the Niger Bridge at Onitsha on the Enugu – Onitsha Expressway.

Sampling and Methods

Fifteen (15) representative samples were collected from different parts of the clay deposits as exposed along the road in Aloji, Udane Biomi, Ofe-Jiji and Agbenema in Kogi State (Fig. 2). The number of samples was determined by the outcropping pattern of the clay deposit.

The physiochemical properties determined for the clays were: Specific gravity, moisture content and plastic limit.

A quantitative determination of the mineralogical property of the clay samples using X-ray diffraction were carried out at ACME Analytical Laboratories Ltd, Vancouver, Canada.

To study clay mineral assemblage, the selected samples were subjected to different processes: grinding and homogenization. Grind the dried sample thoroughly with the mortar and pestle. The particles should be much finer than 0.062mm to avoid fractionation of the minerals. The finer the powder the greater the opportunity for obtaining an adequate number of particles with random orientation and less likely the surface roughness will reduce low angle intensities. The powdered sample was weighed and tested using a PW 1840 automated powder diffraction equipped with a Cu – Kα radiation source, inbuilt standards, Peak/width and a detector.

The angles and intensities of diffractions for each minerals are recorded electronically using a detector, electronics and specialized MDI Jade 5.0 software resulting in a plot of 2θ (horizontal axis) vs. intensity (vertical axis) for the specimen was used, from the angles recorded electronically and which also gives us the area of the peak under d-spacing for each mineral, the mineralogical composition in percentages were done. The interpretation of the diffractograms of clay samples were based on the comparison of the peaks obtained with those of the standard minerals established by Carrol (1971) and JCPDS (1980).

Major and trace element composition of the clay samples were determined using Inductively Coupled Plasma-Emission Spectrometry (ICP-ES) and Inductively Coupled Plasma-Mass Spectrometry (ICPMS) at ACME Analytical Laboratories Ltd, Vancouver, Canada.

Results and Discussion

The result of the physical test is presented in Table 1. The cretaceous clay of the Northern Anambra Basin is generally light grey to brownish grey in hand specimen. Other physical properties exhibited are specific gravity (2.12-2.40), moisture content (11.11-38.89), plasticity (14.05-28.57%), liquid limit (17.38-35.80) and LOI (2.70-11.10%). They are essentially fine grained <2μ and they plot in the region of inorganic clays of medium compressibility on the Casagrande plasticity chart (Figure 3). As shown in Table 1, the moisture contents of clay samples signifies that clay samples from kogi are expected to have higher degree of inter-layer hydration and swelling capacity thus allowing a much freer movement of the clay structure than in the latter. The effect would be an enhancement in adsorption and ion exchange capacity.

Typical difractogram of the Kogi clay (Figure 4) shows prominent quartz, kaolinite and microcline. Prominent basal reflections, strong and sharp peaks are indicators of moderate to well crystalline mineral components (Jubril and Amajor 1991; Obrike et al, 2007).

The clay mineral assemblage as evident by the diffractogram comprises of kaolinite with quartz as the main non clay mineral (Table 2).

Major and trace elements concentration of the analyzed kogi clay samples are presented in Table 3 The low alumina to silica ratio (0.12) suggests a high quantity of free quartz in the clay samples. The analyzed samples also show a silica-sesquioxide ratio of 3.50-12.75, a predominance of SiO_2, (82.88%), Al_2O_3 (9.66%) and LOI (4.99) clearly defines the clay as hydrated alumino-silicates, contaminated by free silty quartz.

Table 4 compares the plasticity test results of the Kogi clay with other reference clay/shale samples. The studied samples when compared with the reference samples has medium plasticity except with the Udubu clays of the Kerri-Kerri Formation in the southern part of Udubu, all others are highly plastic (LL>50%).

The comparison between the average chemical composition of the Kogi Clay with chemical composition of some notable clay samples and specification of some industrial clay are presented in Tables 5 and 6. The Kogi clay is siliceous, with generally low MgO and CaO content, indicative of no associated carbonates or dolomitization process in the study area. The relatively low values of K_2O in the Kogi clay are an indication of the absence of illite (Akpokodje et al, 1991). More also, the low values of Na_2O are probably consequent upon the relatively low occurrence of sodic feldspar and soda-rich clays in the analysed samples. Comparartively, the average silica and alumina values of the Kogi clay are not similar to the plastic fire clay of St. Louise, (Huber, 1985). The chemical composition of the Kogi clay is also similar to the average concentration of the Florida active kaolinite (Huber, 1985) with the major difference being a lower Alumina value in the latter and higher silica. In comparison with the chemical specification of kaolin requirements in the ceramic industry (Singer and Sonja, 1971) and refractory bricks (Parker, 1967), the average chemical composition of the Kogi caly falls within the stipulated specifications (Table 6).

Industrial assessment

Physical properties, mineralogical and chemical composition are the parameters used in assessing the suitability of the studied samples for use in various industrial specifications (Table 6). The Mamu Cretaceous clay at Aloji, Agbenema, Udane Biomi in Kogi State could be utilized as raw materials for the production of ceramics (Singer and Sonja, 1971), refractory bricks (Parker, 1967) and pottery production (Arua and Onyeoku, 1978). The considerable level of FeO in the Kogi clay samples may constitute a challenge on their industrial use for paper and rubber production. Iron oxides will produce colouration effect on finished products. Then appreciable amount of sodium, calcium and magnesium will also lower the vitrification of the clays. The high alumina iron ratio value (19.43) in the Kogi clay renders it less suitable for the production of good quality cement. Abatan et al (1993) advocates a silica-sesquioxide ratio range of 1.5 to 4.0 and alumina-iron ratio range of 1.71 to 2.45 for clay suitable for utilization in the manufacture of good quality cement.

There is a considerable amount of silt in the fines (<65μm) as evidence by the SiO_2 values (82.88%). The loss on ignition (LOI) of 2.7 to 11.1% is an indicative of low porosity and that the finished products would show no cracks or damages on firing, thus, rendering them suitable for ceramic wares. Medium plasticity coupled with the general fineness of clays is an important consideration for the use of clays as fillers and coating materials in the paint and cosmectic industries (Elueze et al, 1999). Samples from the study area are essentially made up of clay fractions (<65μm) above 70% and are moderately compressible (Table 1 and Figure 3).

Conclusion

Results of investigation conducted on the Kogi clay as outcropped in Aloji, Agbenema, Ofe-jiji, and Udane Biomi, show that mineralogically, the clay samples have kaolinite as the predominant clay mineral with quartz as the main non clay mineral.

Chemically, the clay show major element concentration of the following oxides: SiO_2, Al_2O_3, Fe_2O_3, K_2O and TiO constitute more than 85% while Na_2O, MnO, CaO, MgO have relatively low values. The Kogi clay possesses moderate plasticity and compressibility, in addition to a medium loss on ignition values. The Mamu clay in Kogi has higher SiO_2 content than the Florida active, Afam, Plastic fine Clay of St. Louis, Isan (Brown), Isan (Red) Ara-Ijero (Grey) clay, in addition to the lower Al_2O_3 values. In comparison with reported industrial specifications, results of the cretaceous Mamu clay at Kogi, show that it could be employed as raw materials in the production pottery, bricks, ceramics, refractories and in paints manufacturing. The high alumina-iron ratio value in the kogi clay renders it less suitable for the production of good quality cement, a suitable that can be remedied by proper blending, appropriate beneficiation with lime and refining processes.

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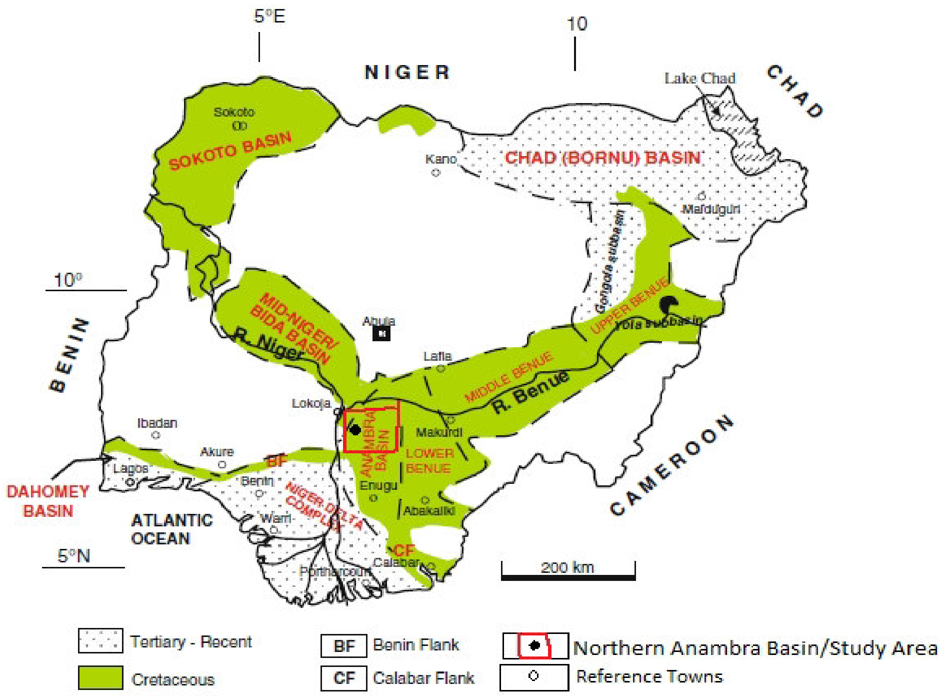
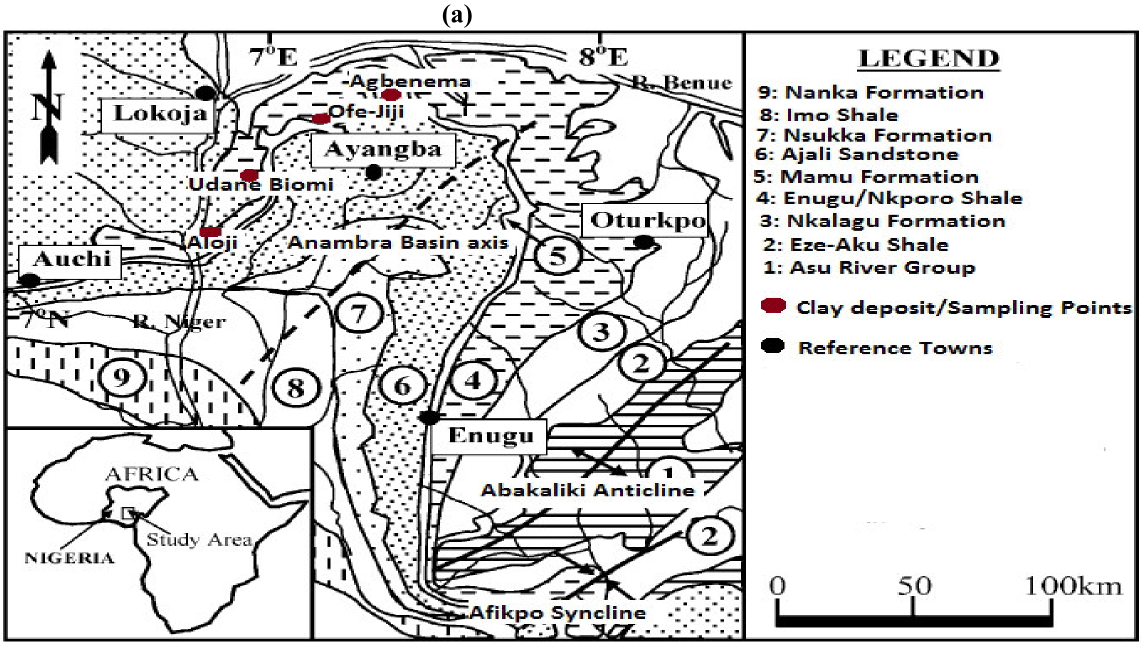


(a)



(b)

Figure 1: a) Map of Nigeria showing the Anambra Basin and other Sedimentary Basins of Nigeria; b) Geological Map of Anambra Basin showing the different locations of Clay Deposit/Sampling Points at the Northern Part of the Basin and different Sedimentary Units (Inset: Showing Location of Nigeria and the Anambra Basin)

(Modified after Obaje et al, 2009)

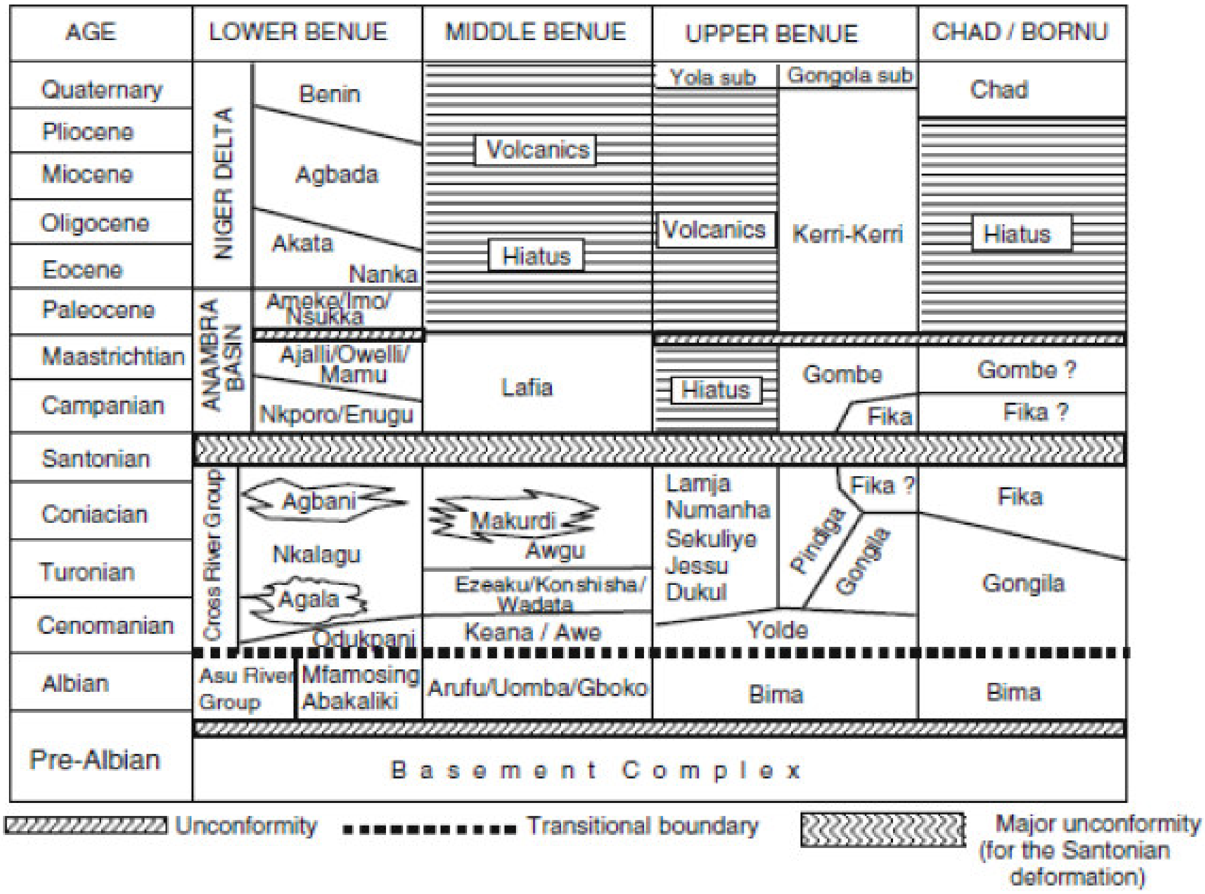


Figure 2: Mesozoic-Cenozoic Stratigraphic correlation chart of the Benue Trough showing the Anambra Basin

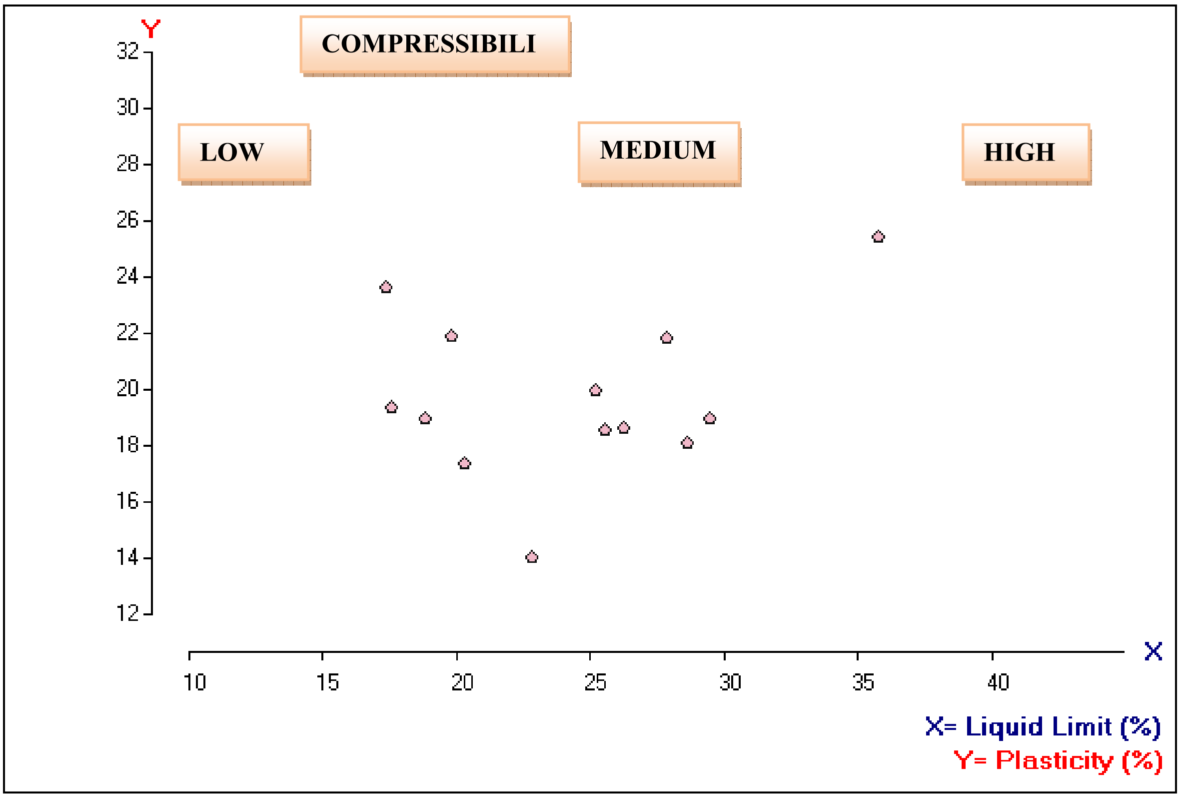


Figure 3: Plots of the Kogi clay samples on the Casagrande plasticity chart

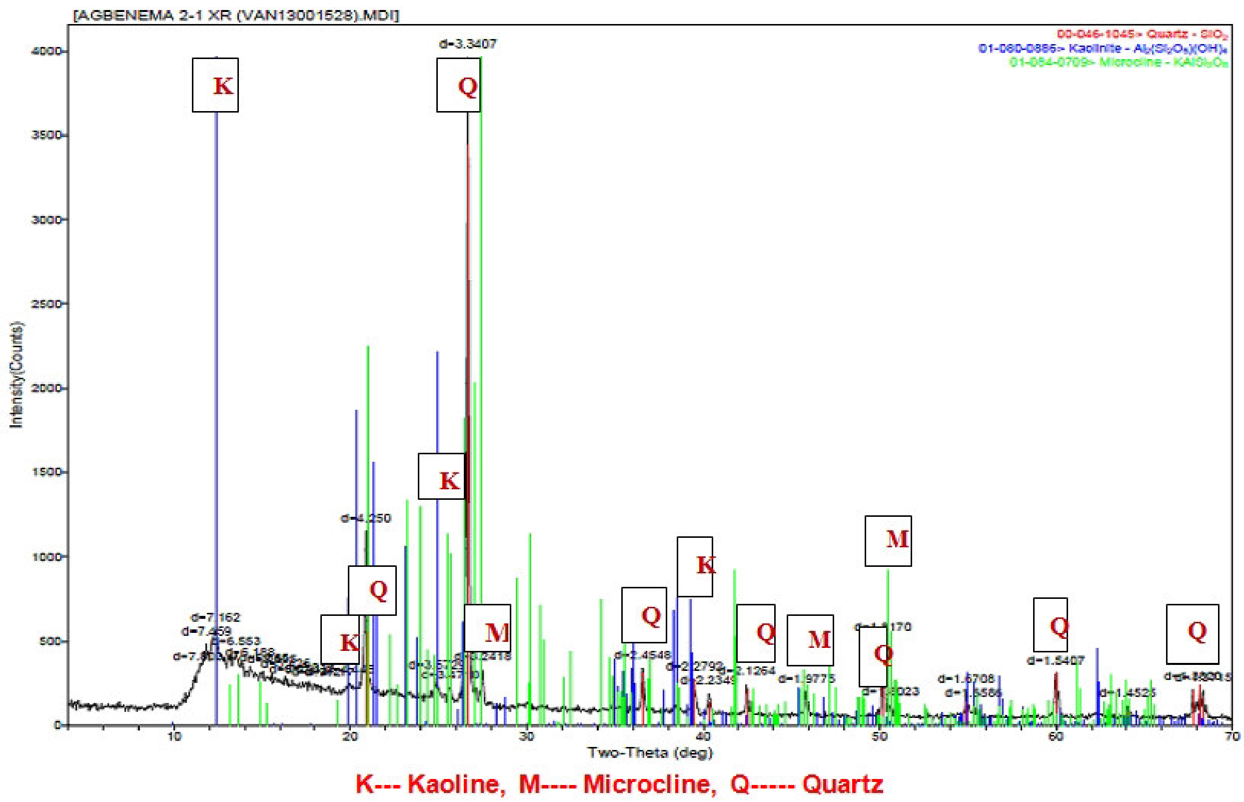


Figure 4: X-Ray diffraction result of Agbenema clay deposit in Kogi State, Nothern Anambra Basin

Table 1: Results of the geotechnical tests of the studied samples

Sample	Specific gravity	Moisture Content (%)	Liquid Limit (%)	Plasticity Index (%)	LOI (%)
Al. 1.1	2.37	26.66	29.50	18.98	8.10
Al. 1.2	2.40	27.64	20.29	17.35	5.20
Al. 1.3	2.22	16.25	26.29	18.63	6.50
Al. 2.1	2.11	35.80	35.80	25.42	5.50
Al. 2.2	2.12	11.11	17.56	19.35	4.00
Al. 2.3	2.22	23.69	17.38	23.63	4.10
Al. 2.4	2.24	19.52	19.80	21.91	3.30
Al. 2.5	2.38	25.44	18.88	18.94	3.60
Of. 2.1	2.16	24.65	29.42	28.57	5.20
Of. 2.2	2.12	22.33	27.88	21.83	11.10
Ud. 1.1	2.24	38.89	22.81	14.05	3.30
Ud. 1.2	2.30	28.25	25.60	18.55	2.70
Ud. 1.3	2.23	28.25	25.23	20.00	3.40
Ag. 2.1	2.12	31.55	28.62	18.13	4.70

Table 2: Mineralogical composition (in percentages) of clay deposits from different locations in Kogi State, Northern Anambra Basin

Mineral %	Ag. 2.1	Al. 1.1	Of. 1.1	Ud. 1.1
Kaolinte	22	26	25	25
Quartz	70	74	70	75
Microcline	08	-	5	-

Table 3: Chemical analysis of the clay samples in percentage from Kogi State

Oxides	Al	Al.1.1	Al. 1.2	Al.1.3	Al.2.1	Al.2.2	Al.2.3	Al.2.4	Al.2.5	Of. 2.1	Of.2.2	Ud.1.1	Ud.1.2	Ud.1.3	Ag.2.1	Av.
SiO_2	83.62	69.67	82.62	75.84	82.81	87.32	85.49	88.77	87.75	84.65	71.39	86.53	90.28	86.43	79.99	82.88
TiO_2	0.98	1.63	1.00	1.41	0.90	0.79	1.08	0.85	0.85	0.97	1.22	1.29	0.66	1.00	1.31	1.06
Al_2O_3	10.05	19.22	10.53	15.24	10.32	7.34	8.73	6.65	7.24	6.32	12.91	7.95	5.10	7.28	9.57	9.66
Fe_2O_3	0.37	0.70	0.32	0.55	0.28	0.33	0.33	0.31	0.39	0.76	1.46	0.41	1.02	1.63	2.06	0.73
MnO	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
MgO	0.03	0.07	0.03	0.05	0.02	0.02	0.02	0.02	0.02	0.04	0.12	0.03	0.02	0.03	0.09	0.04
CaO	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
Na_2O	0.01	0.02	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.06	0.05	0.01	0.01	0.01	0.07	0.02
K_2O	0.10	0.33	0.14	0.22	0.07	0.04	0.07	0.04	0.04	1.63	1.19	0.09	0.07	0.07	1.87	0.40
P_2O_5	0.02	0.04	0.02	0.03	0.01	0.01	0.02	0.01	0.01	0.06	0.16	0.04	0.02	0.01	0.03	0.03
LOI	4.7	8.1	5.2	6.5	5.5	4.0	4.1	3.3	3.6	5.2	11.1	2.7	2.7	3.4	4.7	4.99
Sum	99.9	99.8	99.89	99.87	99.94	99.88	99.87	99.98	99.93	99.71	99.62	99.07	99.9	99.88	99.71	
S.R	8.02	3.50	7.61	4.80	7.81	11.38	9.45	12.75	11.50	10.17	4.97	10.35	14.73	9.70	6.88	8.91
A.R.	27.16	27.46	32.91	27.71	36.86	22.24	26.45	21.45	18.56	8.32	8.84	19.39	5.00	4.47	4.65	19.43
MgO + CaO	0.04	0.08	0.04	0.06	0.03	0.03	0.03	0.03	0.03	0.05	0.13	0.04	0.03	0.04	0.10	0.05
Na_2O+K_2O	0.11	0.35	0.15	0.23	0.08	0.05	0.08	0.05	0.05	1.69	0.06	0.02	0.02	0.02	1.94	0.42

Table 4: Plasticity test results of studied samples compared with other clays and shales

	*Average for study area	A	B	C	D	E	F
Liquid Limit (%)	**24.65**	**55.83**	**54.00**	**66.00**	**146.00**	**29.37**	**53.50**
Plasticity Index (%)	**20.38**	**31.17**	**26.00**	**36.00**	**78.00**	**15.83**	**24.50**

***Average of 14 samples**
A) Mpu Shale (Obrike et al, 2012)
B) Auchi Shale (Emofurieta, 1994)
C) Gombe Shale (Emofurieta, 1994)
D) Okada Shale (Obrike et al, 2007)
E) Udobo Clays (Omada et al, 2007)
F) Ewekoro Shale (Nton and Elueze,2005)

Table 5 : Comparison of the average chemical composition of Kogi clay with the average chemical composition of some clay samples

Oxides(%)	Kogi Clay	Afam Clay	Plastic fine Clay of St. Louis	Florida active Kaolinite	Isan (Brown)	Isan (Red)	Ara-Ijero (Grey)
SiO_2	82.88	42.20	57.67	52.92	55.49	60.47	60.70
Al_2O_3	9.66	26.20	24.00	9.42	18.63	17.77	17.75
Fe_2O_3	0.73	5.10	3.23	3.65	9.67	8.18	6.04
MgO	0.04	0.70	0.30	0.08	1.25	1.26	1.22
CaO	0.01	1.60	0.70	1.91	0.77	0.47	0.83
Na_2O	0.02	2.90	0.20	0.03	0.46	0.44	0.23
K_2O	0.40	8.30	0.50	0.98	1.84	1.17	1.40
TiO_2	1.06	-	-	1.18	-	-	-
P_2O_5	0.03	-	-	0.02	-	-	-
MnO	0.01	0.03	-	-	0.04	0.03	0.03
H_2O^+	-----	--	10.50	10.19	10.18	9.72	10.71

Kogi Clay (Present study)
Afam Clay (Jubril and Amajor, 1991)
Plastic fire Clay of St. Louis (Huber, 1985)
Florida active Kaolinite (Huber, 1985)
Isan (Brown) Clay (Elueze and Bolarinwa 1995)
Isan (Red) Clay (Elueze and Bolarinwa 1995)
Ara-Ijero (Grey) Clay (Elueze and Bolarinwa 1995)

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Table 6: Comparison of average clay values in the study area with industrial specification

Oxides (%)	Kogi Clay	Reference Samples				
		A	B	C	D	E
SiO_2	82.88	51.70	67.50	44.90	45.90-48.90	38.67
Al_2O_3	9.66	25.44	26.50	32.35	33.50-36.10	9.45
Fe_2O_3	0.73	0.50 – 1.20	0.50-1.20	0.43	0.30-0.60	2.70
MgO	0.04	0.20-0.70	0.10-0.19	Tr.	-	8.50
CaO	0.01	0.10-0.20	0.18-0.30	Tr.	0.00-0.50	15.84
Na_2O	0.02	0.80-3.50	0.20-1.50	0.14	0.00-1.60	2.76
K_2O	0.40	-	1.10-3.10	0.28	0.00-1.60	2.76
TiO_2	1.06	1.00-2.80	0.10-1.00	1.80	0.00-1.70	-
P_2O_5	0.03	-	-	-	-	-
MnO	0.01	-	-	-	-	-
H_2O^+	-----	-	12.04-12.50	-	-	3.04

A) Refractory bricks (Parker, 1967)
B) Ceramics (Singer and Sonja, 1971)
C) Rubber (Keller, 1964)
D) Paper (Keller, 1964)
E) Brick clay (Murray, 1960)