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CHARACTERISTICS OF CLAY DEPOSITS FROM AKPOHA AREA, SOUTHEASTERN NIGERIA: IT'S SUITABILITY AS A RAW MATERIAL IN CERAMICS INDUSTRIES

O.M. Nweke[1*], A.I. Omeokachie[2], H.N. Eze[3], C.O. Okogbue[4], Nwaejigh V.O.[5] and C.G. Nwanekezi-phil[6]

[1,5,6]Department of Geology and Geophysics, Alex Ekwueme Federal University, Ndufu Alike Ikwo, Nigeria
[2]Department of Geological Sciences, Nnamdi Azikwe University, Awka
[3]Department of Geology, University of Nigeria, Nsukka, Nigeria
[4]Department of Geology and Exploration Geophysics, Ebonyi State University, Abakaliki

**Corresponding author; Email: nwaekeoby@yahoo.com, Tel: +234 (0)803 262 4349*

Abstract
In this research, the clays widely deposited around Akpoha region, were sampled and evaluated to determine their suitability for use as raw materials in ceramic applications. The clay samples were first characterized by evaluating their physical properties identified by grain size distribution and Atterberg limits. Winkler diagram and Casagrande clay workability chart were employed for technological classification of clay products. The bulk and clay (< 2 μm) fractions of the samples were further analyzed by X-ray diffraction to identify the mineralogical compositions, whereas their chemical compositions were determined using Philips PW 1600 X-ray spectrometer. To determine their ceramic applications, technological properties such as bulk density, linear firing shrinkage, weight loss, water absorption and flexural strength were evaluated by firing the clay samples at high temperatures ranging from 800°C to 1200°C. From the results, the clays consist of fine particle with high plasticity and were composed predominately of illite, chlorite-vermiculite and kaolinite. The oxides were dominated by SiO_2, Al_2O_3 and Fe_2O_3. The feldspar contents and the alkali elements revealed their potential as stating materials for earthenware production. The results of the fired properties revealed significant development of densification of ceramic behavior noticed at the firing temperature above 800°C. The results also revealed an increase in linear firing shrinkage and flexural strength. From the mineralogy, grain size distribution, plasticity and fired properties of the studied clays, they are suitable as raw material for ceramic production. They utilization can boost the nation's economy in terms of gross domestic product and foreign exchange earnings.
Keywords: *Ceramic, Clay, Extrusion, Industry, Plasticity, Quartz*

Introduction

Clays are very important in process industries, agriculture, engineering, environmental and miscellaneous applications. For centuries, clays served globally as major raw materials in industrial applications such as ceramics, earthenware products, tires, papers as well as in petroleum industries (Diko-Makia and Ligege 2020; Revuelta 2021). They are generally considered to be < 0.002 mm in size (Semiz 2017). Presently, demands for clays as raw materials have risen sharply which could be attributed to the rapid increase in construction purposes owing to globalization and urbanization (Akintola *et al*. 2020). To continue in manufacturing ceramic products, and with the fact that clays as raw materials may become scarce in future (Terrones-Saeta *et al*. 2020) and considering that major countries like China, are already limiting their productions due to insufficient raw materials, there is an urgent need to source for cheap, locally available and sustainable materials that can meet the requirements of the raw materials (either raw or when beneficiated) to serve as alternative to the depleting natural resources.

In Nigeria, ceramic sector is considered one of the most important economic markets that have been growing rapidly for years now. Therefore the position of ceramic technology in Nigeria is considered a veritable tool in nation development but has been hindered by inadequate funding of ceramic industries resulting in existence of moribund industries, and poor researches on available raw materials for sustainability as well as non-existence of statutory

regulatory support. Akpoha region (Southeastern Nigeria) is widely endowed with large deposit of clays. The need to source for low-cost, readily available and sustainable raw materials, as alternatives to depleting natural resources have led to comprehensive evaluations of the potentials of the clay as raw material in the manufacturing of structural ceramics and earthenware products. For this context, mineralogy, geochemical and physical properties of the clays samples were determined. Good numbers of veteran diagrams were employed to establish their suitability for possible use as alternative raw materials in ceramic industries as the data generated are crucial for evaluating their potentialities in ceramics applications.

Geographical and Geological Setting

Geographical Setting

This study area covered the Akpoha areas (Fig. 1 a) and its suburbs in Afikpo South Local Government Area of Ebonyi State, Nigeria. The study area is geographically bounded by longitude $7^0$49' E to $7^0$54' E and latitude $5^0$45' N to $5^0$50' N covering an area extent of about 62 km^2. Tropical rainforest with distinct wet and dry seasons is the typical climate in the area (Okogbue and Nweke 2018).

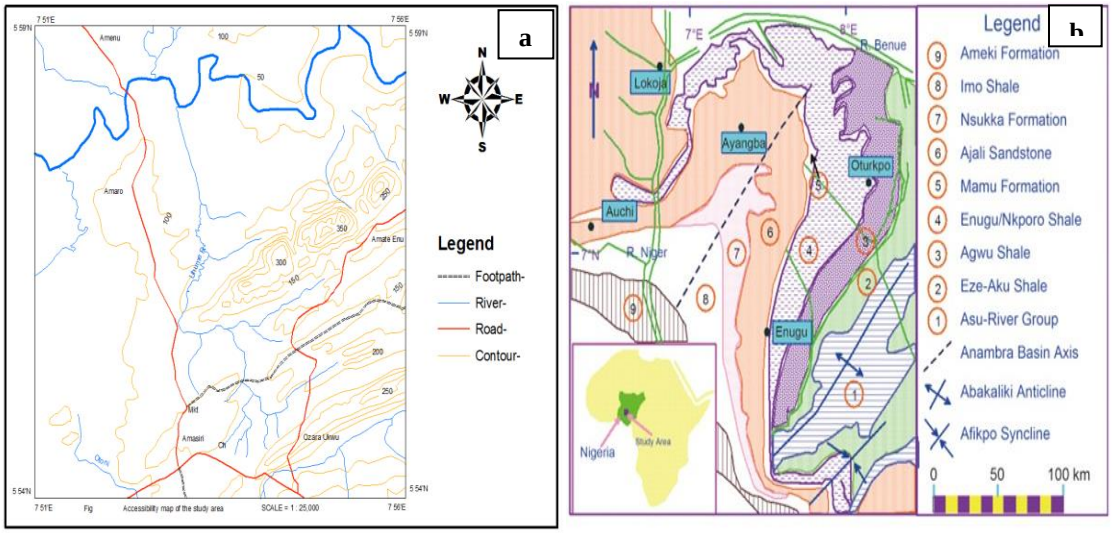

Fig. 1: a Map of Akpoha and its environs, b Regional Stratigraphic map of the southern Benue Trough, Nigeria (adapted from Nwajide, 1990) showing the Eze-Aku Shale where the study was carried out

The dry season which lasts from November to March is characterized by a period of dry hot weather, while the rainy season which usually begins in April and ends in October is characterized by a prolonged period of rainfall with a short period of reduced rains in August. The average monthly rainfall ranges from 31 mm in January to 270 mm in July, with the dry season experiencing much reduced volume of rainfall. The hydro geologic network is made of perennial streams with dendritic network.

Geological setting

The study area is situated in Afikpo, lying within Anambra Basin (Fig. 1 b) whose genesis has been linked with the development of the Niger Delta Miogeosyncline and the opening of the Benue Trough. The stratigraphy comprises of cyclic sedimentary sequence that started in the early Cretaceous time, Agumanu (1989), Marine and fluviatile sediments comprising friable to poorly cemented sands, shales, clays and limestone were deposited, with occasional coal, peat and thin discontinuous seams of lignite. The sediments have been affected by the major Santonian folding and a minor Cenomanian folding and uplift, Murat (1972). The Nkporo Shales consists of soft shales grey to dark grey in colour with intercalations of sandstone and

ironstone. The Mamu formation is made up of coal, shale and sandstone. The clay samples were obtained from Eze-Aku Formation and its environs.

Materials and Methods

Sampling

Three clay samples were collected from three different points, for laboratory tests. The different bags full of clay samples were packaged in different polyethylene-sacks, labeled, coded CL1 to CL3 and the bags full of the samples were later transported to the laboratory at National Steel Raw Materials Exploration Agency, Kaduna Nigeria where sample preparations and laboratory testing on each of the clay samples commenced within 48 h of sampling. The samples were finally analyzed for various basic laboratory tests while the specimen briquettes were molded for technological tests.

Preparation and laboratory testing

The grain size distribution analyses of the samples were performed by sedimentometry for particles of size ≤ 80 μm in accordance with D-422 ASTM standards. The fines fractions of the clay samples were characterized using hydrometer analysis that was based on Stroke law of sedimentation. The degree of plasticity of samples were achieved through the determination of Atterberg limits (liquid limit, LL and plastic limit, PL) using Casagrande apparatus in accordance with the ASTM-D4318 (2010) standards earlier described by Casagrande (1947). The mineralogical analyses on the powdered samples were carried out using the X-ray diffraction (XRD) and in accordance with that earlier described by Moore Duane and Reynolds Robert (1989). X-ray diffractograms were run under 3 treatments (natural, glycol and heated in 500°C for 4 h) ranging from 2° to 70° 2θ for bulk-sample diffractograms and from 2° to 40° 2θ for air-dried, ethylene-glycolated for 24 h, and heated to 550°C for 4 h for the clay proportions. Mineral identification from the diffractogram and a semi-quantitative mineralogical composition were carried out using EVA software. X-ray fluorescence (XRF) spectrometer was used to determine the oxide of elements on the powered form of the samples. The Philips PW 1600 X-ray spectrometer equipped with an end window 4 kW Rh-anode X-ray tube was used. In order to study the evolution of firing properties of the samples, cylindrical samples were prepared. The samples were oven dried at 105^{o}C for 24 h and grounded to a fine powder, then sieved using a mesh-size of 100 μm and were mixed with 26-28% water content to enhance particle binding in order to produce cylindrical shape. Then the wetted powered samples were pressed under 150 kg/cm^2 pressure to obtain 100 × 50 × 8 mm prismatic samples. Then the samples were air dried for 24 h and oven dried at 105^{o}C for 24 h to ensure that absorbed moistures were eliminated. After drying, the specimens were cooled in a drying room maintained at a temperature of 24^{o}C, with a relative humidity between 30% and 70%. The specimens were therefore stored in the drying room at the required temperature and humidity until they were tested. Using a laboratory kiln, the dried samples were fired at different temperatures of 800^{o}C, 900^{o}C and 1000^{o}C (in 100^{o}C intervals) for a period of 5 h at a heating rate of 5^{o}C/min using electrically powered laboratory furnace. A total of four tests were conducted per each property and the average results were tabulated.

Linear firing shrinkage (LFS) was calculated using the expression shown as follows

$$LFS\ (\%) = [(L0-L)/L0] \times 100 \quad \ldots\ldots 1$$

This expression was according to the relative variation length of the briquette, where L0 represents the length of the briquette before firing and L represents briquette after firing.

Water absorption capacity (WA) was measured by weighing the fired briquette (M1) and the wet briquette (M2) after immersion in water for 24 h and was indicated with an expression as follows

$$WA\ (\%) = [M2- M1)/M1] \times 100 \quad \ldots\ldots 2$$

Weight loss (WL) was calculated through the expression below
$WL\ (\%) = [(M_d - M_f)/M_d] \times 100$……..3
Where M_d represents the dry mass measured in g at 105°C and M_f represents the fired mass measure in g at each final firing temperature.

Bulk density (BD) of the briquette was represented in the equation (4) as
$BD\ (g/cm^3) = M_f/V$……..4
Where Mf is the fired briquette weight measured in g and V represents the measured volume (cm^3) of the briquette.

The flexural strength (FS) of the samples evaluated using three point bending test method in accordance with ASTM F417-96 procedure. The value of modulus of FS for each of specimen was computed and recorded to the nearest 0.01 MPa with the expression represented as follows;
$FS\ (MPa) = 3FL/2bd^2$……..5
Where F = maximum load, L = distance between the supports (mm), b = net width of the specimen at the plane of failure (mm), and d = depth of the specimen at the plane of failure (mm).
The data derived from the clays were fired at varying temperatures and the results were compared with standard specifications.

Results and Discussion
Compositions
The representative matched XRD patterns for the identified minerals are shown in Figures 2a-b. The prominent peak intensities at 2θ=25.4°, 2θ= 27.6° and 2θ= 35.6° (2.5 Å and 3.4 Å) are characteristics of quartz. The presence of illites were indicated by the visible peak intensities at 2θ = 14.7 Å while kaolinite at 2θ = 7.48 Å and feldspars were identified at 2θ = 3.19 Å and 3.24 Å.

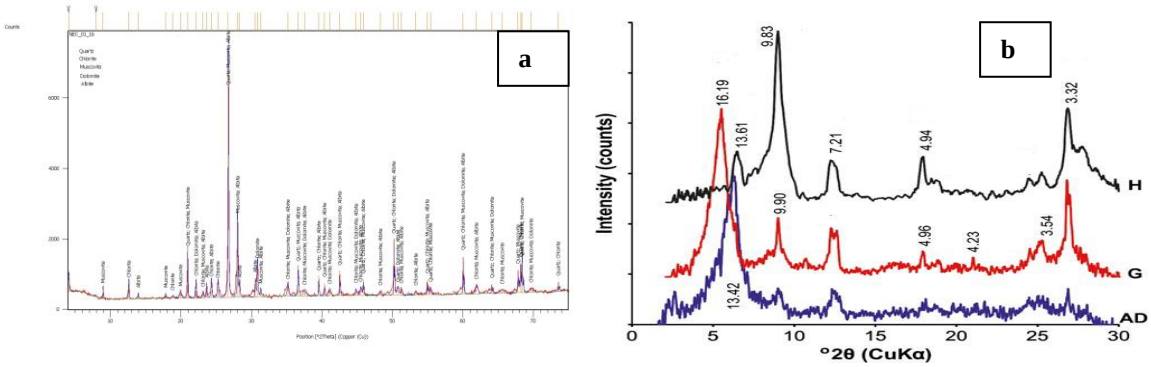

Fig. 2: a XRD analysis of the clays from Akpoha, southeastern Nigeria, **b.** Clay fraction of representative samples. Illite dominated in the left-hand patterns. Normal (H), glycolated (G), and heated at 550°C (AD).

From the semi-quantitative abundance of the identified minerals in the samples, the mineralogical compositions are summarized in Table 1. The XRD results revealed the predominance of clay minerals (illite-chlorite-kaolinite-vermiculite) (48.4%) with high quantities of quartz content (30%) as well as considerable percentages of carbonate and feldspar.

Table 1: Mineralogical composition of Akpoha clays in weight percent

Whole rock					**< 0. 002 mm fraction**					
Sample ID	Quartz	Feldspar	Carbonate	Chlorite	Clay	Kaolinite	Smectite	Illite	Chlorite	Vermiculite
CL1	30	7.5	9.1	4.9	48.4	10.2	5.3	74.0	9.4	1.4

Using the compositional ternary diagram which was based mainly on the total clay mineral-carbonate-quartz + feldspar diagram (Fig. 3a), the mineralogical classification of the samples revealed that majority of the tested samples fell within roofing tiles region and structural clay products. .

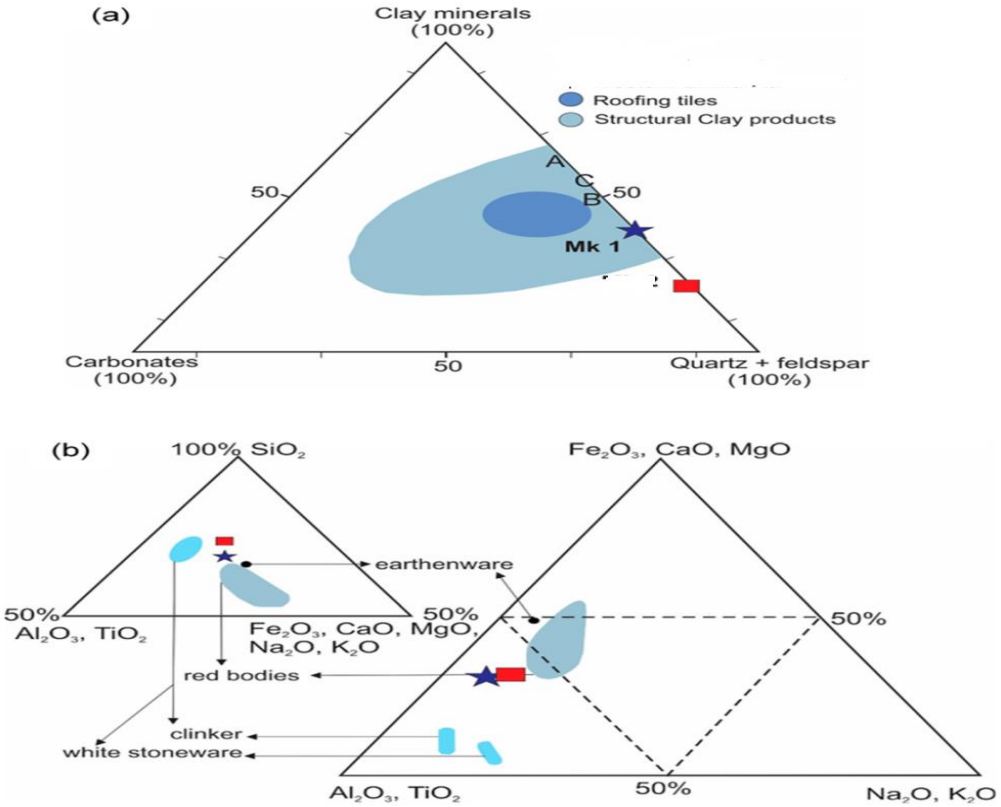

Fig. 3: Classification of raw materials based on Mineralogy and chemical composition: ***a*** *clay minerals, carbonate, and quartz + feldspar ternary diagram (Diko-Makia & Ligege, 2020);* ***b*** *major oxide contents (Diko-Makia & Ligege, 2020).*

The results of the chemical compositions as presented in Table 2 revealed the presence of major oxides with varying amounts of SiO_2 (57.1-60.1%) > Al_2O_3 (16.7-17.8%) > CaO (4.30-6.20%) > K_2O (4.05- 6.05%) and > Na_2O (0.93-1.36%). The SiO_2/Al_2O_3 ratios ranging from 3.38 to 3.58 are higher than the values found in both pure kaolinite (SiO_2/Al_2O_3:1.18) and smectite (SiO_2/Al_2O_3:2.36) (Tsozue *et al*. 2017; Semiz and Celik 2020); a situation partly related to the high amount of free silica (quartz). According to Galan (2003), Na_2O and K_2O are regarded essential in production of roofing tiles as they act as fluxing agents thereby lowering the melting point of ceramic mixtures.

Table 2: Results of the chemical analyses (in wt %) of Akpoha clays.

Sample Code	SiO_2	Al_2O_3	SO_3	Na_2O	K_2O	CaO	MgO	TiO_2	Fe_2O_3	MnO	LOI	Total	SiO_2/Al_2O_3
CL 1	60.1	17.8	0.04	1.36	5.05	5.2	0.48	0.75	1.94	0.09	7.21	99.2	3.38
CL 2	59.0	16.7	0.32	1.30	5.09	6.2	0.50	0.76	3.39	0.11	6.43	100	3.53
CL 3	58.1	17.2	0.80	0.93	4.05	4.3	0.48	0.72	2.60	0.09	10.4	99.9	3.38
Min	57.1	16.7	0.04	0.93	4.05	4.3	0.48	0.75	1.94	0.09	6.43		
Max	60.1	17.8	0.90	1.36	6.05	6.2	0.50	1.76	3.39	0.11	10.4		

The fluxing agents such as alkali oxide (Na_2O and K_2O; 4.98-7.41%) and alkaline earth oxides (CaO and MgO; 4.78-6.70%) were noted to be generally > 4.0%, an indication of possible densification at high temperature. According to Dondi *et al*. (2002) and Semiz and Celik (2020), classifications based on Fe_2O_3 concentration of clay rich raw materials are as follows: (i) red firing clays, Fe_2O_3 concentration > 5%, (ii) tan-burning clays, Fe_2O_3 concentration between 1 and 5%, and (iii) white firing clays, Fe_2O_3 concentration < 1%. The Fe_2O_3 concentrations of the studied materials fell within the class of tan-burning clay material. Based on chemical specifications, the concentrations of Al_2O_3, MgO and Fe_2O_3 for the studied clays are within the allowable parameters for ceramics formulations.

Grain size distribution and Atterberg limit

The results of the grain size distribution tests as presented in Table 3 revealed that the clay fraction (< 2 μm) ranged from 24% to 28%, the silt fraction (2 - 20 μm) ranged from 44% to 48% while the sand fraction (> 20 μm) ranged from 24% to 30%; an indication of the predominance of clay-silt materials.

Table 3: Results of the grain size distribution and Atterberg limit tests of Akpoha clays

S/N	Sample ID	Grain size distribution			Atterberg limit		
		Clay (%) < 2 μm	Silt (%) 20-2 μm	Sand (%) > 20 μm	Liquid Limit %	Plastic Limit %	Plasticity Index %
1	CL 1	24	48	28	44	16	28
2	CL 2	28	46	24	50	16	34
3	CL 3	26	44	30	47	26	21
	Min	24	44	24	44	16	21
	Max	28	48	30	50	26	34

High amounts of finer fractions of <2 μm, according to Ekosse (1994), accounted for excessive shrinkage during firing which may not likely be the case of the study clays. The results of finer fractions of < 2 μm (22% to 28%) obtained can result in very low cracking due to shrinkage when fired for ceramic applications. To formulate ceramics bodies, Winkler Diagram was the tool used with the objective of obtaining products with good technological properties (Semiz 2017; Diko-Makia and Ligege 2020).With regards to Winkers diagram, the studied samples behaviors as they fell within vertical corrugated bricks and perforated products (Fig. 4). From the diagram, three samples plotted within the region of roofing tiles region while the other two samples fell between the perforated bricks region and the region of not suitable material (Fig. 5).

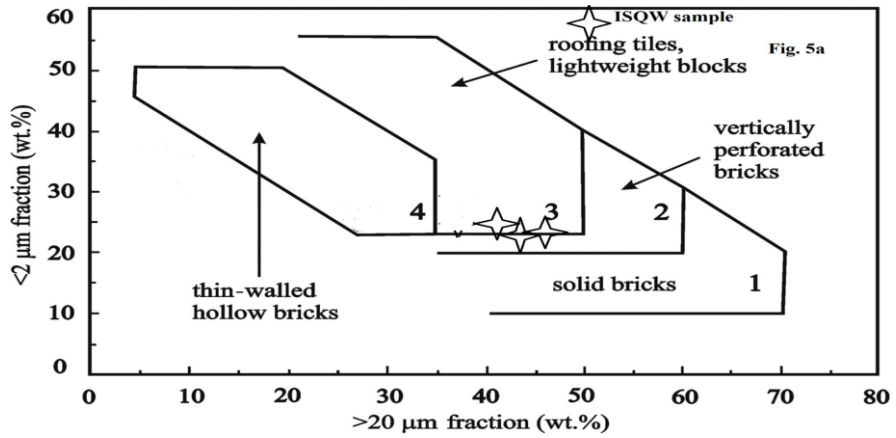

Fig. 4: Grain-size distribution classification based on Winkler diagram for the technological classification of clay products (Winkler, 1954) and Diko-Makia & Ligege (2020). Fields are defined as: (1) common bricks, (2) vertically perforated/corrugated bricks, (3) roofing tiles and masonry bricks, and (4) perforated/hollow products.

The results of the Atterberg limits tests of the studied clays as presented in Table 3 revealed that liquid limit (LL) ranged from 44% to 50%, plastic limit (PL) ranged from 16% to 26% while the plasticity index (PI) ranged from 21% to 34%. The LL and PI values for the samples plotted on the Holtz and Kovacs (1981) diagram (Fig. 5a) revealed that all the tested samples plotted in the medium to high plasticity region. From the diagram, the medium plasticity displayed by the studied samples showed consistency with predominant clay mineral being illite. The scattered plots in the Holtz and Kovacs diagram revealed that the clays fall within illite and smectite region with medium plasticity and these can be attributed to the abundance of fines fractions. The Casagrande workability chart (Fig. 5b) revealed that the clays are generally of medium plasticity region, as they plot within the region of acceptable properties suitable for ceramic production.

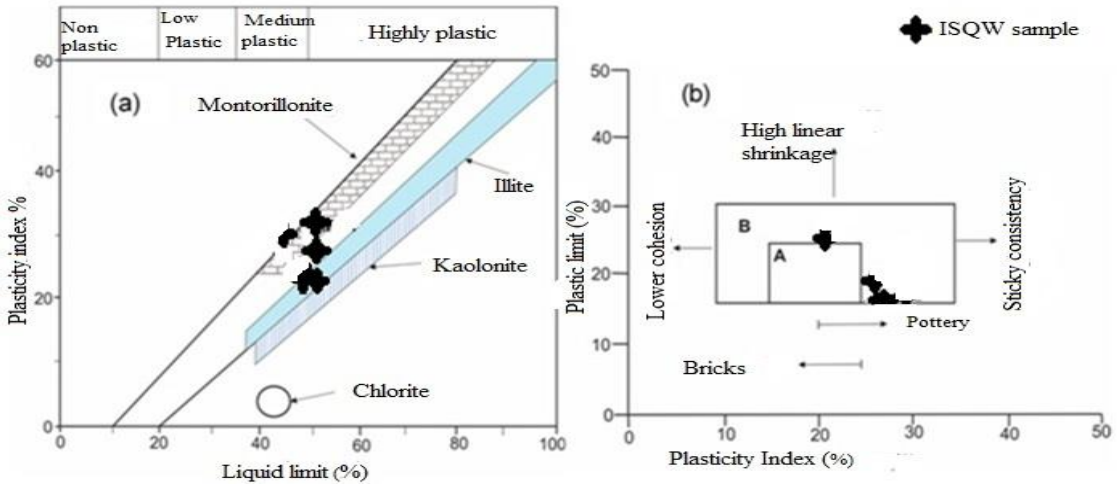

Fig. 5: Atterberg limits of the clays: ***a*** *according to the Holtz and Kovacs (1981) diagram,* ***b*** *according to the Casagrande (1947) clay workability chart*

Technological properties of Akpoha clays

The results generated for linear firing shrinkage (LS) tests indicate that at 800^{o}C, the linear firing shrinkage varied from 9.23% to 12.2% while at increased temperature of 1000^{O}C, the LS varied from 14.8% to 18.3% (Table 4); an indication of increment with increased temperature. Linear shrinkage, according to Semiz and Celik (2020) gives an indication of the

efficiency of firing and the acceptable values for aluminium silicates, kaolin and fired clays ranged from 7 to 10% (Diko-Makia and Ligege 2020).

Table 4: Results of technological properties tests at varying temperature.

Sample Code	Fired Temperature	Linear firing shrinkage %	Water absorption %	Bulk density g/cm^3	Weight loss %	Flexural strength (MPa)
CL 1	800 °C	9.23	14.4	2.01	5.2	2.45
	900 °C	12.7	12.7	2.09	6.2	2.61
	1000 °C	17.8	9.80	2.19	8.3	3.22
CL 2	800 °C	12.2	16.0	1.89	5.0	2.99
	900 °C	14.7	12.7	1.98	6.2	3.09
	1000 °C	18.3	10.6	2.00	7.3	3.21
CL 3	800 °C	9.28	14.9	2.13	4.9	2.40
	900 °C	12.1	12.9	2.13	6.2	2.89
	1000 °C	14.8	9.12	2.23	6.3	3.23

The values of LFS ranging from 14.8% to 18.3% were not within the internationally acceptable range of 7 to 10% (Semiz 2017) for possible use as raw materials in ceramic production. The water absorption (WA) tests as carried out on the specimens indicated that at 800^{o}C, the WA varied from 12.4% to 15.4% while at firing temperature of 1000^{o}C, the WA varied from 8.33% to 10.7%. From the results, WA capacity of the samples decreased significantly as the fired temperature increased from 800^{o}C to 1000^{o}C. The decrease in the WA for the fired clays could be translated to the dehydration reactions, decarbonation and combustion of organic matter (Ngun *et al*. 2011) which will influence greatly the mechanical properties and durability of the raw materials thereby transforming them into more resistant and more durable materials. The specified values for WA of Brazilian clay-based products according to Souza *et al*. (2002) are WA< 25% and WA< 20% for dense bricks and roofing tiles, respectively. The WA of the studied materials with values $\leq$ 10.7% at firing temperature 1000^{o}C suggest that they are suitable for use in massive brick (WA $\leq$ 25%), ceramic blocks (WA $\leq$ 25%), and roofing tiles (WA $\leq$ 20%) productions. The values obtained for bulk density (BD) as the firing temperature increased are presented in Table 4. The BD at 800^{o}C ranged from 1.89 g/cm^3 to 2.13 g/cm^3 while at increased firing temperature of about 1000^{o}C, the BD ranged from 2.0 g/cm^3 to 2.23 g/cm^3. In ceramic industry, strength variation with increasing temperature in firing process is very important (Semiz and Çelik 2020). From the results, strength variations that were observed in bulk density values in all the five tested samples were not significant as the temperature increased. The strength gain obtained at 1000^{o}C confirmed that mineralogical transformations may have likely occurred (Tsozue *et al*. 2017). The weight loss (WL) at 800^{o}C, ranged from 4.9% to 5.2% while at higher temperature of about 1000^{o}C, the WL ranged from 6.3% to 8.5%. The presence of higher quartz content in the samples may have contributed in the lower average WL of $\leq$ 7.0% (Garcia-Valles *et al*. 2015). El Gamouz *et al* (2007) suggest that WL < 5.0% in the weight of fired products is generally acceptable for raw materials as that serves as an added advantage in making it cost effective materials in ceramics industries. The flexural strength (FS) ranged from 2.40 to 2.56 MPa at 800^{o}C while at fired temperature of 1000^{o}C, the FS values increased from 3.21 MPa to 3.99 MPa; an indication that the technological property showed great dependence on the firing temperature. Therefore, the FS values (3.21-3.99 MPa) obtained from this study at 1000^{o}C are significantly lower than those FS values earlier reported by Dondi *et al*. (2002) for Italian brick clays as well as Kagonbe *et al*. (2021) for roofing tiles except for production of massive bricks.

Suitability of Akpoha clays as raw materials

The predominance of clayey-silt type materials favored them as good raw materials in ceramic bodies (Dondi 2001). The proportion of the clay fraction in a structural ceramic raw material is an indicator of plasticity and workability (Diko *et al*. 2011). The LL (44-50%), are in agreement with the range defined by Baccoura *et al*. (2009) (30-60%) to the composition of raw materials used for ceramic production. From the Winkler's diagram and McNally diagram, the studied samples showed heterogeneous behaviors as they plot within vertical corrugated bricks and perforated products and bricks and tiles production, respectively. From the chemical and mineralogical studies, the chemical results are consistent with mineralogical compositions which revealed the predominance of quartz, illite and chlorite with lesser amounts of feldspar and carbonate. Chemically, the major oxides present are SiO_2, Al_2O_3 and K_2O with higher CaO contents, and smaller quantities of MgO, MnO, Fe_2O_3 and TiO_2. The low WA recorded from the fired samples suggest low porosity which may likely results in no cracking of the finished products, thereby rendering them suitable for use in ceramic industry. For ceramic application, after firing to the temperature of about 1000^{o}C, raw materials with FS $\geq$ 5.7 MPa (Onana *et al*. 2019) while WA < 20% and WA < 25% (Onana *et al*. 2019) are considered valuable for roofing tile and dense bricks and ceramic blocks, respectively. Therefore, the fired temperature at about 1000^{o}C can serve as most suitable condition for the beginning of liquid phase sintering, increased material densification and strength development of the raw materials. Considering the fact that with high linear firing shrinkage values, deformation and micro-cracking are possible during the production of bricks.

Conclusions

Characterization of clays as raw materials in ceramic industry enabled the following conclusions to be drawn;

1. The clays classified predominantly as clayey-silt texture qualifies them for industrial ceramic applications. The mineralogy revealed the predominance of illite, chlorite and vermiculite with high contents of quartz and feldspar and they greatly influence their development as ceramic materials.
2. During firing, at 800^{o}C and above, results revealed an increase in the firing shrinkage and flexural strength with a decrease in WA values. Based on technological specifications, the materials revealed acceptable weight loss, linear firing shrinkage and water absorption with unsatisfactory performances for bulk density and flexural strength ($\leq$ 3.99 MPa) at fired temperature of 1000^{o}C in ceramic applications.
3. Better knowledge of the technological properties of the clay samples revealed their possible utilization as raw material in industrial production of ceramic products and this boost the nation's economy in terms of its gross domestic product and foreign exchange earnings.

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