



CHARACTERIZATION OF BARUTEN LOCAL GOVERNMENT AREA OF KWARA STATE (NIGERIA) FIRECLAYS AS SUITABLE REFRACTORY MATERIALS

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ABSTRACT

Studies have shown that adequate attention needs to be paid on processing of solid minerals that are potentially available in Nigeria to address its economic problem. Clays from five major towns in Baruten Local Government Area, Kwara State, Nigeria were examined using ASTM guidelines to determine their suitability for refractory applications. The clay samples were classified as Alumino-Silicate refractories due to high values of Al₂O₃ and SiO₂. The results showed apparent porosity (19.4-25.6%), bulk density (1.83-1.90 g/cm³), cold crushing strength (38.7-56.1 N/mm²), linear shrinkage (4.4 – 9.3%), clay contents (52.71-67.83%), moisture content (17.0-23.6%), permeability (68-82 cmsec⁻¹), plasticity (16.7-30.4%), refractoriness (>1300°C) and Thermal Shock Resistance (23-25 cycles) for the clay samples, which were measurable with the established standards for fireclays, refractory clays/brick lining or alumina-silicates and kaolin. Hence, the natural clays could suitably replace imported clays in some refractory applications. Appropriate use of information from this study would improve Nigeria's industrialization and economic diversification.

Keywords: Apparent Porosity, Bulk Density, Clay, Shrinkage and Refractory

1. INTRODUCTION

Majority of the available natural resources in Nigeria have not been receiving sufficient attention [1, 2]. These resources, if adequately used could help the country in achieving its economic growth. Industrialization has been identified as a good channel to improve a country economic status [3, 4, 5, 6, 7, 8, 9]. To achieve this, effective and adequate attention needs to be paid on processing of solid minerals that are potentially available in Nigeria, most especially during the present economic recession that requires shifting attention from over dependency on oil and gas as main economic source. This requires development of industries that would process these minerals from the raw stage to the most useful state. These process industries would have to employ high temperature section likes foundry and equipment (such as furnace) to achieve this objective. Therefore refractory materials

become very important in this regard. Refractory materials are inorganic materials mainly mixture of oxides, which are capable of withstanding very high temperature conditions. They do this without an undue deformation, failure or change in composition [10].

Refractory materials are produced from clay refractory and non-clay refractory. Special refractories are used where there are very high temperature applications as in nuclear reactors such as thoria and beryllia [11]. The cost of fireclay is much lesser than that of non-clay refractory [12].

Refractories are known to be chemically and physically stable at high temperature. Refractory materials retain their shape, strength, chemical identity and are not deformed at high temperature. They need to be chemically inert and be resistant to thermal shock [13]. These properties make refractory materials suitable in production of high temperature devices and equipment

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like furnaces, incinerators, crucibles, insulation and other metallurgical furnaces linings where high resistant to high temperature is required [14]. The devices and equipment are very significant in processing of raw materials to finished goods in industries.

Refractories have been successfully used to improve performance and energy efficiency in heat engineering plants [13]. In basic metal industries (70%), refractories are mostly used [15]. About 80% of the total refractory materials are used in metallurgical industries for the construction and maintenance of furnaces, kilns, reactor vessels and boilers. The remaining 20% are being used in the non-metallurgical industries such as cement, glass and hardware industries [16]. Firebrick is the most common form of refractory material and is used extensively in iron and steel industry, nonferrous metallurgy, glass industry, pottery kilns, cement industry, and many others [17]. Virtually all refractory requirements in all metallurgical industries in Nigeria are imported [18].

With the privatization of Ajaokuta Steel Complex, and Delta Steel Company (DSC) Aladja and the development soft solid minerals in Nigeria, there is going to be an intensive increase in the use of refractory materials. For instance, the Ajaokuta steel complex on completion will require about 36,000 tonnes of refractory bricks worth over Twenty Million Naira, just for furnace lining purposes only [19]. Hassan and Adewara [16] also asserted that about 80% of the refractory bricks required at Ajaokuta annually will be fireclays and over 1.5 Million Naira worth of fireclays is consumed annually in the DSC as at 1994. Therefore research into the local materials for refractory purposes is justified in order to meet the technological requirements of the country (Nigeria) and to conserve the much-needed foreign exchange.

Earlier researches on some Nigerian clay deposits for refractory purposes showed the need for characterization of other clay deposits in the country for their suitability for intending purposes. The results revealed that Igbokoda clay-bonded sand has very good durability up to five times re-use [20]. The study conducted on the suitability of the natural sand from the Ahmadu Bello University, Zaria for foundry work also showed that the materials possessed properties that made them comparable with the imported moulding sand [21]. Sand from Kubami River and three indigenous clays from Yelwa, Battati and Kankara were investigated and found that they could be used for general casting purpose [22].

Chemical compositions of clays from different locations were collated from Technical Literatures and presented in Table 1 to be used as standards for smooth classification of the studied clay samples. The categories of the clay involved include refractory brick, ball clay, fireclay, refractory and ceramics. The main components of the clay samples as shown in Table 1 are silica, alumina and oxides of iron, calcium, magnesium, sodium and titanium.

1.1 Study Area

Baruten is one of the Local Government Areas (LGA) in Kwara State, Nigeria, with an area of 9,749 km² and a population of 209,459 at the 2006 census [28]. It shares a long border with the Republic of Benin. The location of the LGA is shown graphically in Figure 1.

In spite of abundant availability of natural resources, like natural clay, in this area, there is no presence of any industry in the area. Industrialization greatly depends on production capacity from availability of locally available raw materials used in development of technology and in the transformation of the raw materials to finished products [3].

Table 1: Chemical Composition of Clays collated from Technical Literatures

S/N	Clay Samples	SiO ₂	Al ₂ O ₃	TiO ₂	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	L.O.I	Classification
1	Refractory brick [23]	51.70	25.00 – 44.00	ND	0.50 – 2.40	0.10 – 0.20	0.20 – 0.70	0.80 – 3.50	ND	ND	Refractory
2	Ball clay [24]	40.00- 60.00	25-60	-	0.25 - 4.00	-	-	-	0.00 - 0.75	0.50 - 4.00	Siliceous (Ball clay)
3	Fire clay [25]	20-30	2.30	25-90	4-10	15000	20-30	-	-	1500-1700	
4	Siliceous fireclay [25]	23.7	2	-	-	15000	-	-	-	1500	
5	Fireclay[26]	15-25	1.9-2.3	-	7.0-10.0	≥150	20-30	-	-	1500 – 1700	
6	Fireclay[27]	46.00- 62.00	25-39	-	0.40 - 2.70	0.20 - 1.00	0.20 - 1.00	0.30 – 3.00	0.30 - 3.00	8.00 - 18.00	
7	Refractory Clay [27]	46-62	25-39	ND	0.4-2.7	0.20 – 1.00	0.2 – 1.0	ND	ND	8 – 18	Refractory

ND – Not determined

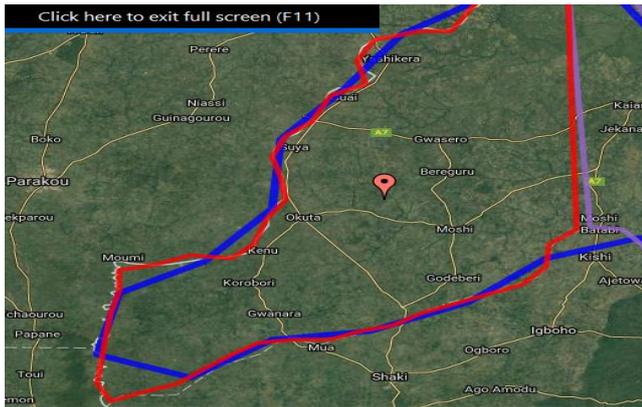


Figure1: Map of Baruten Local Government Area, Kwara State [29].



Figure 2: Hydraulic press used



Figure 3: Blended clay in the mould



Figure 4: Some of the bricks made from the clay samples [31]

Appropriate and adequate knowledge about the clays in this LGA will definitely foster the establishment of sustainable small and medium scale (SME) enterprises and medium industries, which will help in creating jobs

and at long run address many economic and social problems in the area. This is one of the bases for this study. The results of this work will equally provide information require to harness the clay deposit in the production of ceramic wares and refractories for improvement of the nation’s economy and its technological advancement.

2. MATERIALS AND METHODS

2.1 Materials

For this study, five (5) clay samples were collected using ASTM D4220/D4220M-14 guidelines [30] from different clay deposits in Baruten Local Government Area of Kwara State, Nigeria, labelled as: A – Okuta, B – Ilesha, C - Baruba, D – Gure and E - Yashikira.

2.2 Methods

2.2.1 Processing of Materials

The samples collected were air-dried, crushed, grinded and sieved through 75 micron (μ), 200 μ m and 600 μ m sieves. The specimens for various experiments for the refractory analysis were rolled and pressed, dried and kept in a crucible in a plastic form before different parameters were determined. The chemical and physico-mechanical properties of the clay samples were tested for easy characterization using ASTM guidelines. The tested properties include chemical composition, clay and moisture content, and grain distribution.

2.2.2 Brick Production

The plastic clays were moulded into block form with rectangular mould of dimensions 5 x 5 x 5 cm (cylindrical shape) using hydraulic press shown in Figure 2. The bricks were dried in hot air oven and fired. The refractory properties of the clay samples were then determined using the bricks produced. Blended clay in the mould and some samples of the fired bricks are shown in Figure 3 and 4 respectively. The brick samples in Figure 4 had also been previously used in other studies [31].

2.2.4 Determination of the Samples’ Chemical Composition

In line with the Chesti’s recommendation that x-ray fluorescence (XRF) or Atomic Absorption Spectrophotometer (AAS) can be used for that purpose [31], representative samples of the selected clay materials were analysed to determine their chemical

constituents using Atomic Absorption Spectrophotometer (AAS).

2.2.5 Grain Size analysis / Sieve analysis

For grain size analysis, 500g of each of the clay samples was taken and dried in the oven for 15 minutes at 110°C and later cooled in desiccators. Each sieve pan in a set of Indian Standard (I. S.) was weighed. The sieves were assembled and each sample was sieved through sieves using a mechanical sieve shaker machine. After shaking, each sieve and pan with clay sample retained in each of them were weighed and recorded. The percentages of clay retained and percentages of clay passed were determined.

2.2.6 Atterberg limit (Consistency tests)

Liquid limit, plastic limit and plastic index were determined in the course of carrying out refractory clay materials' consistency tests in accordance with ASTM guidelines as discussed in details elsewhere [32,33]. Liquid limit, plastic limit and plastic index were determined in the course of carrying out refractory clay materials' consistency tests.

Liquid limit

A 100g of the clay sample each was made to pass through 425 micron sieve. The sieved samples were thoroughly mixed with distilled water in evaporating dish to form a uniform paste. In the cup of the liquid limit device, a portion of the paste was placed and levelled to have a maximum depth of 1cm as shown **Figure 5**. The grooving tool was drawn through the sample along the symmetrical axis of the cup by holding the tool perpendicular to the cup. The handle of the device was then rotated till the two parts of the clay particle came into contact at the bottom of the groove. The numbers of blows were counted to achieve this. Oven drying method was then used to determine the clay's water content. The operations were repeated. Five readings were obtained in the range of 25 blows.

The liquid limit was determined using required Atterberg table and by plotting the graph on semi-logarithmic graph between the numbers of blow.

Plastic limit

A 15 g from each of the sieved clay sample was mixed thoroughly with distilled water until the clay mass became plastic to be easily moulded into a ball with fingers. To form clay mass of thread of uniform of 3 mm throughout its length, portion of the ball was taken and rolled with the palm (hand) on a glass plate. The clay was re-moulded again into a ball repeatedly until the thread started to crumble at the diameter of 3 mm. The crumbled thread was kept in desiccators for further experiment.

In determining the clay water content, a portion of the plastic clay was weighed, sieved through a set of sieve and then fired at 115°C for 30 minutes. It was then cooled in desiccators for 15 minutes and re-weighed. The different in the weight was then determined and recorded. Average plastic limit was determined from three different tests carried out. The mix levelled to have a maximum depth of 1cm during the test is as shown in Figures 5 in this study, which had also been previously used in other studies [31].

The plasticity index was calculated using the relationship in equation (1):

$$\text{Plastic Index (PI)} = \text{Liquid Limit} - \text{Plastic Limit} \quad (1)$$

2.2.7 Moisture content (Oven dried method)

The clay samples were air-dried and weighed. They were then heated in a furnace for 24 hours to a constant temperature of 110°C, cooled in desiccator and weighed. The samples' moisture contents (M.C.) were determined using equation (2)

$$M.C. = \frac{WBD - WAD}{WBD} \times 100 \quad (2)$$

Where WBD weight before drying and WAD is the weight after drying.



Figure 5: Determination of a clay sample liquid limit [31]

2.2.8 Clay content

A 50 g of clay was taken from each of the clay samples, dried in an oven at 110°C for four hours and introduced in a sand wash bottle. Distilled water (475 ml) and 25 ml of sodium hydroxide solution were added to the sample and thoroughly stirred for five minutes. Sufficient water was added to make up to the wash bottle. The mixture was left for ten minutes. The water was siphoned from the wash bottle. The wash bottle was refilled with water and stored once again with the glass rod. This was allowed to settle for ten minutes at the end of which it was siphoned. This operation was carried out repeatedly until clear water indicated in the wash bottle. This was carefully removed from the wash bottle and the rest content dried in an oven at 105°C for an hour. The percentage clay content was determined from the loss in weight.

2.2.9 Loss on ignition (LOI)

Samples of the clay were dried in oven at 110°C and cooled. The samples were placed in a clean and dried crucible, heated in muffle furnace to a temperature of 900°C for 3 hours. The LOI was determined using the relationships in equation (3).

$$LOI = \frac{WDC - WC_l + C_r}{WC + C_r - WC_r} \times 100 = \frac{MC}{WC_l} \times 100 \quad (3)$$

Where WDC is the weight of dried clay, WC_l is the weight of clay, C_r is the crucible, WC is weight of the clay, WC_r is the weight of the crucible, M_c is the moisture content.

2.3 Determination of Fired Refractory Properties

Some of the properties of the fired refractory were determined using brick samples produced from each of the selected clays and tested in accordance with ASTM standard test methods for plasticity, drying shrinkage, firing shrinkage, apparent porosity, water absorption, apparent porosity, specific gravity and bulk density [34 – 38] as described in sub-sections 2.31-2.37.

2.3.1 Apparent porosity (A.P)

Samples of the brick produced from all tested clays were air-dried for 24 hours and later oven-dried at 105°C for another 24 hours, cooled and kept in desiccators. The dried-air weight (W_a) of each specimen was obtained. Subsequently, the samples were fully immersed in boiling water for 30 minutes. The samples were agitated from time to time to ensure that the trapped air bubbles were released. It was allowed to cool in the water. The weight of the soaked sample (W_s) was obtained. It was then removed from water, mobbed up and re-weighed in air to determine

the saturated weight (W_{ss}). A.P. was then determined using equation (4).

$$A.P = \frac{V}{Brick\ volume} \times 100 = \frac{W_{ss}}{W_s} - \frac{W_a}{W_s} \times 100 \quad (4)$$

Where V is the volume of the water after boiling.

2.3.2 Bulk density

Fresh two (2) pairs of each sample of the clay bricks were selected to determine the bulk density of the clay samples. The samples were dried for 24 hours in air and later in oven at 110°C for 24 hours. The samples were cooled in desiccators. The dried weights of the samples were also measured nearest to 0.001 g. The weighed samples were transferred into separate beakers and soaked with water. They were heated for 30 minutes to release the trapped air in the samples. The samples were cooled and the weight of each sample was taken. Each sample was suspended in water in the beaker and the suspended weight was measured. The bulk density of each of the clay samples was determined using the relationship in equation (5).

$$Bulk\ Density \left(\frac{g}{cm^3} \right) = \frac{Dried\ Weight}{SW - DW} \quad (5)$$

Where SW is the soaked weight and DW is the density of water.

2.3.3 Refractoriness

The refractoriness of the samples was determined using Pyrometric Cone equivalent (PCE). Some standard cones were mounted on a refractory plaque and placed in kiln. The temperature was raised to 1300°C at the rate of 10°C per minute. The rate was gradually reduced till the tips of the test cones bent touching the refractory plaque. The refractory plaque was removed and allowed to cool prior to examining the test cones. The refractoriness of the clay being tested is the number of the standard that has bent to the same level as the tested cones. The temperature similar to the cone number was determined from the ASTM Orton series.

2.3.4 Thermal Shock Resistance (TSR)

Samples of the brick produced from the studied clays were placed in a resistance heating box of an electric furnace, heated up to 1300°C. The furnace temperature was maintained for 30 minutes for uniformity and homogeneity transformation. The specimens were then cooled in air and returned to the furnace for further heating for 10 minutes. Cracks were observed on them. The specimens without any observed crack were returned to the furnace to undergo the same process as earlier done. The process was repeated until the

samples cracked. The TSR for each specimen was noted, being the number of cycles required to cause conspicuous crack on the brick sample.

2.3.5 Resistance of Refractory Clays to Slag Attack

Hole was drilled into each of the refractory brick made from the studied clay samples. The hole was packed with the sample of iron slag. This iron slag was assumed to be expected slag the clay samples are likely to encounter in service. The brick were heated to 1300°C and maintained at the temperature for one hour. The refractory brick was then cooled and sectioned. Through examination, the degree of slag attack and penetration was observed.

2.3.6 Linear Shrinkage

The specimens were rolled in rods of 12cm and marked along a line to maintain the same position after heat treatment. The specimens were dried in still air for 24 hours and later fired at temperature of 110°C in oven for 6 hours, allowed to cool at room temperature and transferred into desiccators. The dried lengths of the specimens were recorded and their linear shrinkage values of the specimens were determined using the relationship in equation (6).

$$\text{Linear shrinkage} = \frac{\text{Dried Length (D)}}{\text{Fired Length (F)}} \times 100 \quad (6)$$

2.3.7 Cold Crushing Strength / Compressive Strength in Green Mould Sample

The samples were prepared from the clay samples materials, which were moulded into a flat shape size 7.62 cm³. The specimens were fired at 110°C for 6 hours in a furnace. The specimens were cooled to room

temperature. The compressive strengths of the cooled specimens were determined on a Testometric Materials Testing Machine (Model 0500-10080, Win test analysis; 100kN capacity, England made) showing in Figure 6.



Figure 6: Testometric Materials Testing Machine Used

3. RESULTS AND DISCUSSION

3.1 Experimental Results

The results showing various elemental chemical constituents of the clay samples obtained from various clay deposits in Baruten Local Government Area of Kwara State in Nigeria are as shown in Table 2. Table 3 presents summary of the clay samples' grain size distribution, while Table 4 also presents some refractory properties of Baruten (Nigeria) clay samples and some properties' values for refractory materials in literature. The refractory properties include plasticity, clay content, moisture content, porosity, bulk density, crushing strength, permeability, thermal shock resistance and refractoriness.

Table 2: Chemical composition of the clay samples

Clay Samples	Composition (Weight %)										
	SiO ₂	Al ₂ O ₃	CaO	Fe ₂ O ₃	MgO	K ₂ O	MnO ₂	NaO	TiO ₂	P ₂ O ₅	L.O.I
A	61.2	30.2	0.37	1.16	0.44	1.98	0.016	0.085	0.18	0.041	4.32
B	58.5	31.0	1.18	1.23	0.13	1.74	0.011	0.051	0.04	0.010	7.10
C	62.3	31.7	1.03	1.31	0.06	1.13	0.016	0.080	0.49	0.160	2.73
D	57.1	35.3	0.42	0.72	0.16	0.91	0.027	0.21	0.09	0.180	4.82
E	53.7	34.3	0.74	2.95	1.19	1.73	0.031	0.137	1.31	0.163	5.01

Table 3: Some refractory properties of the clay samples

Clay Samples	Particle Distribution (% clay contents)	Atterberg Limit			
		Liquid Limit (LL) %	Plastic Limit (PL) %	Plastic Index (PI) %	Linear Shrinkage (%)
A	64.40	77.7	47.3	30.4	9.3
B	58.77	77.7	52.6	25.1	7.8
C	52.71	35.6	18.9	16.7	4.4
D	67.83	68.4	39.4	29.0	9.0
E	64.20	58.0	33.7	24.3	6.3
Fireclay[26]					2 - 10

Table 4: Clay samples' refractory properties and some standard values in literature

Clay Samples	Apparent Porosity (%)	Bulk Density (g/Cm ³)	Permeability (X 10 ⁻⁵ cmsec ⁻¹)	Cold Crushing Strength (N/mm ²)	Moisture Content (%)	Thermal Shock Resistance (Cycles)	Slag Resistance	Refractoriness (°C)
A	25.6	1.90	78	38.7	23.6	23	Good	>1300
B	24.5	1.85	68	48.0	21.9	25	Good	>1300
C	19.4	1.83	71	56.1	17.0	25	Good	>1300
D	24.9	1.89	81	41.2	22.5	22	Good	>1300
E	21.9	1.83	82	51.0	19.7	23	Good	>1300
ASTM Standards for Fireclay [26]	15– 25	1.9 – 2.3	82	≥15.0		20–30		
Commercial Bricks[39]	22-26	1.9 - 2.0				>10		
Commercial Bricks [40,+41]	22-26	1.9 – 2.0*		>20.0		>10		
Commercial Bricks [42]		1.9 - 2.0						≈1230

3.2 Chemical Composition of the Clay Samples

It is shown in Table 2 that the clay samples belong to Alumino-Silicate refractories group having silica as the highest proportion of the elements present in the clays, ranging between 53.7 and 62.3%. Alumina (Al₂O₃) in the samples also ranges between 30.2 and 35.30%. According to ASTM C27-98, Hassan and Adewara [16,43,44], clay that contained Aluminum Oxide between 25 – 45% belongs to Alumino-Silicate Refractories. Other elements present in the clay samples include oxides of iron, calcium, magnesium, sodium, potassium and titanium (Fe₂O₃, CaO, MgO, Na₂O, K₂O, TiO₂) and loss on ignition (L.O.I). The elemental compositions of the clay samples in Table 2 are in agreement with the view that any fireclay to be used in refractories should have at least 30% Alumina (Al₂O₃) and less than 1.8% Iron (III) oxide (Fe₂O₃) [45]. This means that the clay samples are suitable as refractories chemically, except sample E that possesses 2.95% Fe₂O₃. This value is a bit above 1.2 – 2.5% Fe₂O₃ recommended range for a Standard Commercial brick [39]. Clay with high amounts of iron is not a suitable source of aluminium, since the effect of the iron tends to be deleterious on extraction process of aluminium [46]. But, the clay sample may be fit for the production of earthen wares and chalk.

The appearances of the raw clay samples were majorly reddish colour, except sample D with black/ash colour. The reddish colour can be attributed to the presence of iron oxide in accordance with the view of Huber [47] that sometimes iron containing minerals could be part of the kaolinite. The iron oxide concentration ranges from 0.72 to 2.95%. Sample E and D recorded the

highest and lowest value respectively. The Higher value of Fe₂O₃ (2.95%) in clay sample E is a reflection of its higher reddish colour, while other samples A, B, and C with relatively average Fe₂O₃ values exhibited lighter reddish colour of the clay. Sample D with 0.72% Fe₂O₃ value also displaced a slight blackish colouration, which can be attributed to the presence of carbonaceous (organic) matter (like vegetable) in the clay.

Other fluxing agents (impurities) in the clay such as CaO, MgO, K₂O and Na₂O, are within the acceptable ranges as shown in Tables 1 and 2. Impurities, such as CaO, MgO, Na₂O, CaSO₄, CaCO₃, MnO₂, P₂O₅, Fe₂O₃ and TiO₂ in an ideal kaolinite should be in small quantities [48].

The chemical compositions of samples presented in Table 2 are compared to that of clays from previous research works in Table 1. The values of the constituents of clay samples derived from the chemical analysis carried out are similar to that of local and international clays (refractory materials). Thus, the studied clay samples may be used as suitable major raw materials for different applications as ceramic, refractory and fireclay, if other major criteria (physico-mechanical properties) were met.

3.3 Analysis of the Distribution of the Grain Size Particle Distribution (Percentage of Clay Content)

Virtually, it was observed that most of the clay particles were retained within the sieve mesh 75 micron (μ). The results of the sieve analysis of the clay (as shown in Table 3) indicates that the clay samples fall within silt-clay materials as their clay contents were greater than 35% (52.71 – 67.83%) in line with American

Association of State Highway and Transportation Officials (AASHTO) classification of soils and Soil-Aggregate Mixtures [49].

3.3.1 Plasticity

Through sieve analysis of the clay samples, all the studied clays possess liquid limit greater than 35%. This reveals that studied clay samples belong to medium to high plasticity clay [47]. Medium to high plasticity clay usually possess liquid limit between 30 - 77.7% with compressibility shrinkage characteristics [49], higher value of liquid limit of clay indicates high compressibility of the clay. Though, clay sample C had the least plasticity with liquid limit of 35.6% and plastic index of 18.9 %, which is still within the acceptable range. Medium to high plasticity of the clay samples is an indication that the clays may belong to kaolinite or illite group [32]. The clay samples' plasticity values are in ascending order of sample A, D, B, E and C.

Reduction in the value of plastic index increases permeability and decreases compressibility [32], which is reflected in the results in Figure 8 and Tables 3 - 4. However, clay samples with low plasticity possess low clay contents (Figure 9). According to Cansagrande chart (Figure 10), clay with Plastic Index less than or equal to 10% and liquid limit less than or equal to 30% is of low plasticity [49]. Figure 10 illustrates of the relationship between plastic index, plasticity, compressibility and liquid.

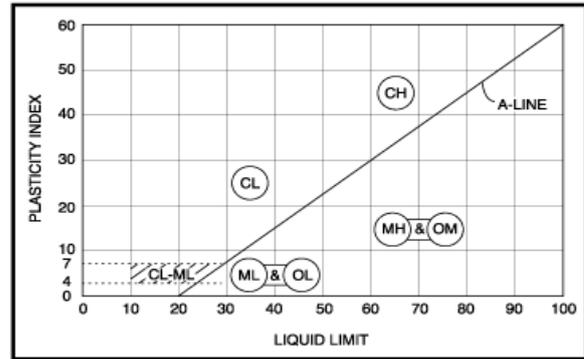


Fig.10: Cansagrande chart [49].

Linear shrinkage

The results in Table 3 show linear shrinkage values within 4.4 and 9.3% for the clay samples. Sample C has the least value of 4.4%, while samples A exhibited the highest value of 9.3%. This is an indication of good efficiency of firing since the values are within the recommended linear shrinkage value (2.0 - 10.0%) for alumina-silicates, kaolin and fire clays [26]. The result shows indication of better interlock of grains that enhances strength of refractory in operation. Too much shrinkage of refractories usually leads to spalling, warping and cracking of the brick [12]. Loss of heat in furnace is associated with cracking of brick. Clay samples A and D with value of 9.3% and 9.0% respectively are likely to possess higher finer grains and moisture content than other clay samples, while clay sample C (with value of 4.4%) likely possess the least finer grains and moisture content, since linear shrinkage of clay increases with moisture content and consequently finer grains [32]. The result of the experimental tests also reflected this trend as shown in Tables 3 and 4. That is, the higher the clay sample's linear shrinkage value, the higher the value of moisture in the clay samples. This trend is shown in Figure 11.

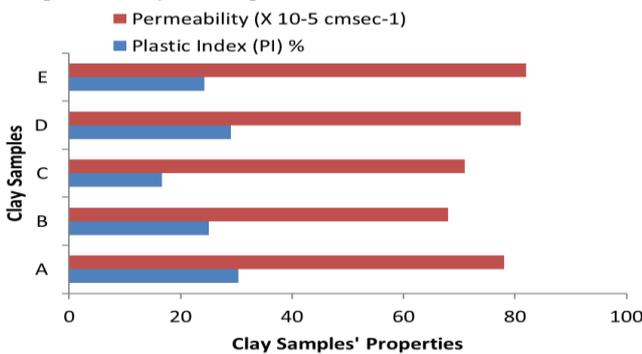


Figure 8: Relationship between the clay samples plastic index and permeability

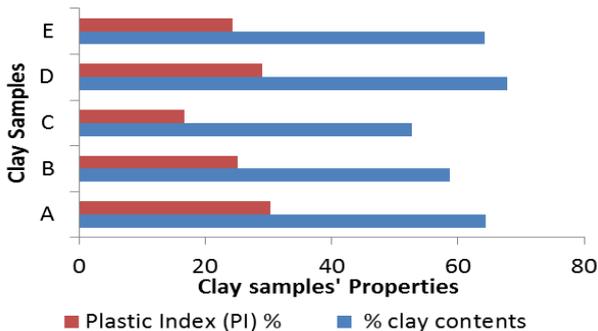


Figure 9: Relationship between the clay samples plastic index and percentage clay contents

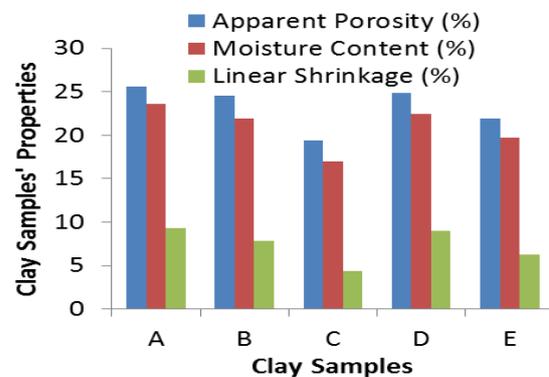


Figure 11: Relationship between the clay samples' apparent porosity, moisture content and linear shrinkage

3.4 Physico-Mechanical Properties of the Natural Moulding Sands (Refractory Properties)

The results in Table 4 are compared with different clay properties collated from literature to evaluate the studied clays' suitability as refractory materials for some applications.

3.4.1 Apparent Porosity (A.P.)

The Apparent porosity of the clay samples are within 19.4 and 25.6%, with clay sample A having the highest value, while sample C possess the lowest value. Sample D, B and E respectively possess A.P. value of 24.9, 24.5, 21.9 %. Most of these values fall within the standards recommended values of 22 – 30% for fired bricks [49], and 22-26% for commercial bricks [39,40], except for samples C and E with 19.4% and 21.9% respectively. Meanwhile, Chesters [50] also gave 10-30% as recommended apparent porosity values for refractory clays. The experimental result is an indication of likely good life span for the clay materials, if use as refractory materials in operation, since the values are within the recommended range. Meanwhile, low percentage of apparent porosity enhances the trapping of gases in the clay materials which has adverse effect on refractory material life [52]. The higher the value of porosity of clay material the higher the material insulating properties [33]. The relationship between the clay samples' apparent porosity, linear shrinkage and moisture content is shown in Figure 11. The clay samples' apparent porosities increase with the clay linear shrinkage and moisture contents.

3.4.2 Bulk Density

The clay samples' bulk density values are also between 1.83 and 1.90 g/cm³ as shown in Table 4. The clay samples' bulk density values are in ascending order of sample C / E, B, D and A, that is 1.83, 1.85, 1.89, and 1.9 g/cm³ respectively. Sample C and E possess equal density value 1.83 g/cm³. The bulk density values fall within acceptable range for refractory bricks (brick lining) of 1.7 – 2.4 g/cm³ [51]. Though, sample A and E respectively has highest and least value of 1.90 and 1.83 g/cm³. High bulk density impacts available water capacity. It is an indication of low porosity and compaction, which is reflected in the results in Table 4 that the higher the clay bulk density, the higher its porosity.

Bulk density of clay plays significant role in its economic value when fired as a refractory, filler, coater, and absorbent among others. It is desired that clay refractories possess high bulk density, due to the fact that high fired-density usually confers high physical

strength at high service temperature and high resistance to corrosion in service, slag penetration, and abrasion [33]. Thus, the clay samples have desirable values of bulk density for standard refractory materials.

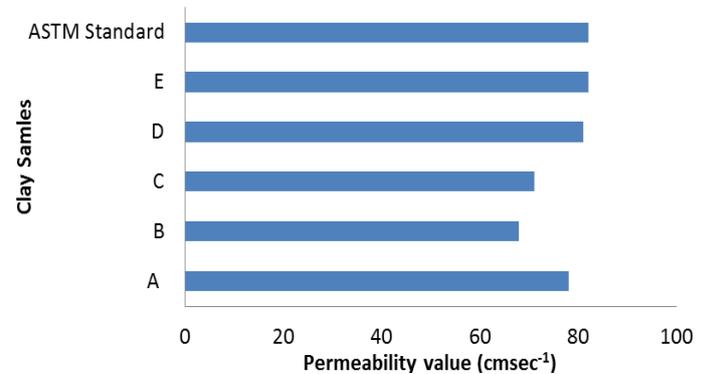


Figure 12: Clay samples' permeability value

3.4.3 Permeability

From the experimental results in Figure 12, the clay sample A, B, C, D and E respectively possess permeability values of 78, 68, 71, 81 and 82. According to De Bussy and Gupta [51,53], the fireclay's permeability values are between 25 and 90, but ASTM [26] recommended 82 as the fireclay's permeability. By De Bussy and Gupta [52,53] recommendations, the clay samples permeability are within the acceptable range, an indication that the clay materials were suitable insulating refractories. Putting ASTM [26] clay permeability value into consideration, samples A, B, C, and D possess permeability values that are lesser than the recommended value. The clay samples' permeability can be improved by appropriate application of organic solvents [54] or with addition of organic materials like sawdust, rice husk, among others [55].

3.4.4 Cold Crushing Strength

From the results in Table 4, the clay samples possess cold crushing strength between values of 38.7 and 56.1 N/mm², with sample A and C having the lowest and highest value respectively. That is, the strength of the clay samples increases in the order of sample A, D, B, E and C (38.7, 41.2, 48.0, 51.0 and 56.1 N/mm² respectively). The clay samples possess appreciable strength values, which are within the stipulated compression crushing strength for fireclay bricks (15 - 59 MPa (N/mm²) [34, 56, 57]. The recommended minimum suitable compressive strength for fireclay is 150 kg/cm² (14.71 N/mm²) [58]. Clay samples with low crushing strength usually have low resistance to load, tension and shear stresses than the clay samples

with high crushing strength [59]. The clay samples' crushing strengths analysed in this study are high enough for a refractory material, which accounts for good bonding and verification during firing [60].

3.4.5 Water Absorption (Moisture Content)

Table 4 reveals that the clay sample A has the highest percentage of moisture content of 23.6%, while clay sample C has lowest value of 17.0%. Other clay samples (sample D, B and E) respectively possess 22.5, 21.9 and 19.7%. Generally, the moisture in the clay samples is within the range of 17 – 23.6%. An indication that the clay samples contains water adequate for proper mixing, ensures simple handling and better moulding capacity into any desire shapes. This agrees with Hassan's view that good water contents make ease mouldability of the clay possible [61].

The results of the study show good relationship between rate of water (moisture content) in the clay samples, the clays' percentage linear shrinkage and apparent porosity (Figure 11). In the clay samples, the percentage of the linear shrinkage increases with that of moisture contents (Table 4 and Figure 11).

3.4.6 Thermal Shock Resistance (TSR)

The clay samples survived 23-25 cycles without any crack as presented in Table 4. This indicates that the clay samples could withstand abrupt changes in that temperature ranges. The clay samples fall into "Good class" going by Hassan [61]'s classification of TSR for fireclay brick that $>30 =$ excellent, $25-30 =$ good, $12-20 =$ fair, $10-15 =$ acceptable and $<10 =$ very poor. Alternatively, 20-30 numbers of cycles is being recommended as refractory fireclay's TSR standard values [51, 57].

3.4.7 Refractoriness

The clay samples' refractoriness values are found to be greater than 1300°C. This shows the ability of clay samples material to withstand the deformation temperature at that temperature before fusing or bend under its own weight [58]. It is the ability to use these clay materials without fear of thermal deformation of the furnace wall. At temperature above 1300°C (refractoriness), all the clay samples did not show any sign of failure. This is a sign an indication that the sintering levels of the samples were high; thus, they have good refractoriness. The samples can therefore be used for lining of furnaces and other high temperature devices for melting low and medium temperature metals. The peak operational temperature of the

furnace available for the study was a bit above 1300°C, which served as a constraint in the course of this study.

4. CONCLUSION AND RECOMMENDATIONS

4.1 Conclusions

The following conclusions are drawn from this study's experimental results:

The clay samples are found to belong to Alumino-Silicate class of refractories due to the presence of Alumina between the ranges of 30.2 – 35.3%. The studied clay samples also belong to medium to high plasticity clay. Thus, the class of clay samples can be used as fireclay refractory raw materials for furnace and kiln lining; and as suitable potential raw material for ceramics, paper, paints and other industries.

The clay samples exhibited reasonable physico-mechanical (good refractory properties), such as such as clay contents, linear shrinkage, bulk density, porosity slag resistance, crushing strength thermal shock resistance and refractoriness that are comparable with standards for fireclay. This makes them to be suitable for thermal insulation in many applications, such as in pyrolysis plant where linings are not exposed to fumes and vapour. An indication that the clay samples either in their raw conditions or at higher temperature were suitable as high melting and fireclay refractory and ceramic raw materials of acceptable standards.

The properties of the clay samples are comparable with that of similar clays in literatures and international standards. Thus, they are found to be good materials for refractory applications and could suitably replace imported clays in refractory applications, such as in production of earthen wares, chalk; as insulating refractories for casting and melting of low and medium temperature iron and steel. If these clays are carefully utilised, it will assist in addressing the nation's economic problem by reducing over dependence on foreign goods.

4.2 Recommendations

This study recommends the need to give adequate attention that will enhance proper utilization of available local clay materials for local production of ceramic wares and other refractory products instead of relying on imported ceramic products and over dependent on oil and gas as Nigeria major economic source. This will assist to boost the lean economic of the country through industrialization and help Nigeria to come out permanently from its present economic recession.

The government and individuals need to encourage elaborate research works on clay and other mineral resources and focus on establishment of small and medium scale refractory firms within the identified clay deposits (environments) to enhance local capacity building, reduce unemployment rate in Nigeria and possibly rates of crimes among the youths.

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