# Compositional Features and Industrial Application of Afuze Clay, Southsouth Nigeria

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ABSTRACT: Afuze clay deposits in Owan East L.G.A of Edo State, Nigeria were characterized for refractory and other applications. The characteristics investigated were chemical composition, plasticity index, liquid limit, plastic limit, particle size distribution, surface characteristics, water absorption, dry shrinkage, fired shrinkage, total shrinkage, Firing colour, compressive strength, void ratio, linear shrinkage, bulk density, cold crushing strength, thermal shock resistance, thermal conductivity, and refractoriness (softening point). Chemical analysis was conducted using an atomic absorption spectrophotometer (AAS). The physical property tests were done following ASTM standards. Results obtained showed that the Afuze clay is largely kaolinite and chemically composed of mainly silica (46.09%) and alumina (35.35% respectively). These oxides appreciated to 45.09% and 33.35% (silica), 31.8% and 21.30% (alumina) respectively in the fired product. The physical/refractory characteristics examined confirmed that the clays belong to the fireclay class and are useful for refractory and ceramic applications.

**Keywords**— Compositional, Refractory Applications, Bulk Density, Cold Crushing Strength, Compressive Strength, And Density, Dry Shrinkage, Firing Colour, Fired Shrinkage, Linear Shrinkage, Liquid Limit, Particle Size Distribution, Plasticity Index, Plastic Limit, Refractoriness (Softening Point), Surface Characteristics, Thermal Shock Resistance, Thermal Conductivity, Total Shrinkage, Void Ratio And Water Absorption.

#### INTRODUCTION

Refractory are materials, mostly non-metallic minerals that have enormous heat capacities and can withstand high temperatures, as well as other strains exerted on them such as abrasion, impact, thermal shock, chemical attack and high level loads at elevated temperatures. They are commonly applied in lining of furnaces.

Refractory are classified based on composition, (such as alumina content) and physical or industrial characteristics like thermal stability. They are commonly classified into "Basic and Acidic" types with a third and intermediate group. Basic refractory include magnesia-chromates or chromate-magnesia, forsterite and dolomite. Acid refractory include the alumino-silicates e.g. The clays, kyanites, silimanite, andeladsite anpolymsphis, and Alumina refractory. The third categories, which usually have relatively limited application than the major categories, include Zircon and Zirconia, graphite, siliconcarbide and carbon.

Refractory are required to satisfy primarily some or all of the following characteristics depending upon the service condition.

Refractories are indispensable materials in high temperature processes such extraction/refining, sanitary ware, table ware, wall and tiles, construction materials, electric porcelains insulator, for materials glazing and metallurgical heat treatments, foundry melting practices, and power generation. The refractories need of Nigeria -a developing industrial nation -is potentially enormous. It was estimated that the Ajaokuta Steel Company and Delta Steel Company will, at full capacity, respectively require 43,503 and 25,000 tonnes/year of fireclay refractories for their activities; and these products are sourced from abroad (Adondua, 1988). Onyemaobi (2002) noted that small-scale industries in Nnewi and elsewhere in the country have recently embarked on the fabrication of spare parts. These spare parts are fabricated using high temperature furnaces [foundry melting furnaces and heat-treatment furnaces] that require refractories as linings. He observed that most of the refractories consumed in this country are sourced from abroad whereas there are many clay deposits in Nigeria that could be used as refractories. Obadinma (2003) reported that in 1987 alone, Nigeria imported 27 million metric tones of refractories. The country expends a lot of foreign exchange importing refractories. Yet, a lot of clay deposits abound in the country, which can be developed to meet our local needs. Earlier works on various Nigerian clay deposits have shown many of them to be rather high in silica content and low in alumina (NMDC, 1999; Hassan, 2001). However, a number of deposits were found suitable for use as refractory raw materials; that is, if properly processed. Therefore, the development of our local materials for the production of to industrial refractories meet our technological requirements is not only justified but imperative.

The evaluation of some refractory characteristics of kaolinite clay deposits in Edo State is the focus of the present study. The properties covered are plasticity index, liquid limit, plastic limit, particle size distribution, surface characteristics, water absorption, dry shrinkage, fired shrinkage, total shrinkage, Firing colour, compressive strength, void ratio, linear shrinkage, apparent porosity, bulk density, cold crushing strength, thermal shock resistance, thermal conductivity, and refractoriness (softening point); in addition to chemical analysis of the clays

TABLE 1: Elemental Composition Analysis of the clay

S/N	Parameter	Level of Detection
		(%)
1	SiO <sub>2</sub>	46.09
2	Al <sub>2</sub> O <sub>3</sub>	35.38
3	Fe <sub>2</sub> O <sub>3</sub>	0.200
4	CaO	0.126
5	MgO	0.055
6	Na <sub>2</sub> O	0.050
7	K <sub>2</sub> O	0.044
8	MnO	0.005
9	Moisture	2.341
10	L.O.I	0.002

#### **Raw Material Preparation**

The raw clay from the mines was soaked in water for five days and then sieved on wet with a sieve of 350µm dried in open air for one week, to remove alkalis and some dead organic matters. The dried clay was crushed and ground into powder form, using jaw crushers and milling machine. The ground clay was sieved to pass through a sieve of 250 aperture size.

The granulated powder mixtures were uniaxially compacted using hydraulic press under a pressure of 50 MPa for the circular-shaped specimens (3 cm ×7 cm). The pressed specimens were held overnight and then dried at 90±100°C for 48hrs in an oven. Dried specimens were fired in a laboratory-type electrical furnace (Thermolyne 46200) at the rate of 5 °C/min). Technological parameters values were measured after firing steps. The firing procedure used involved heating and soaking the samples at various temperatures as shown below:

- (i) At 1000°C for 6 hours
- (ii) At 1100°C for 4hours
- (iii) At 1200°C for 3hours
- (iv) At 1300°C for 8hours
- (v) At 1400°C for 3hours

After firing, the bricks were then allowed to cool in the furnace at a cooling rate of about 1°C/min.

#### TEST PROCEDURE

#### 1. LINEAR SHRINKAGE

The bricks were pressed in green condition in a rotary disc of size (3 cm ×7 cm). The linear shrinkage was calculated using the formula below: Linear shrinkage = percentage of dried shrinkage + percentage of fired shrinkage.

#### 2. COMPRESSIVE STRENGTH

The compressive strength test was done using the Tensometric Machine. The samples of diameter 3cm was subjected to compressive force, loaded continuously until failure occurred. The load at which failure occurred was then recorded.

#### 3. BULK DENSITY:

The bulk density (D<sub>b</sub>) was calculated for each test specimen using the results of the apparent porosity test, thus:

**Db** =**True** eightBulk Volume = (Wda/Wsa-Wsw)......[]

## 4. ELECTRICAL INSULATION TEST (THERMAL CONDUCTIVITY)

Electrical Insulation resistance measuring device "The Megger" (model No:AM-5KV) was used to measure the insulation resistance of the composite materials. Copper plates with terminal heads were placed on both side of the clay product and voltage

of 250V passed through the composite material. The insulating resistance was then read from the instrument in  $M\Omega$ . The same process was repeated with voltages of 500V, 1000V, 2500V and 5000V through all the clay materials and their corresponding insulating resistance values read from the instrument. The leakage current was then determined by.

Where V is voltage, R is resistance, I is leakage current.

## 5. DETERMINATION OF COLD CRUSHING STRENGTH:

For each of the clay product samples were measured in height 7cm and diameter 3cm were tested for compressive strength.

The strength value of one brick was tested in the direction of forming pressure, while the other was tested in the direction normal to the direction of forming. The average strength value in MPa or (MN/m²) was noted.

## 6. DETERMINATION OF THERMAL SHOCK RESISTANCE:

The cylindrical bricks measuring in (7x3) cm were used for this test. The test samples were placed in a box- resistance - heating 1200°C Electric Furnace, preset and maintained at 1000°C, and soaked for 10 minutes. The samples were then removed one after the other using a pair of tongs, and cooled in air (outside the furnace) for 10 minutes, and observed for c racks. In the absence of cracks [or fracture], the bricks were put back into the furnace and reheated for a further period of 10 minutes and then cooled for another 10 minutes. This process or cycle of heating, cooling and observing for cracks was repeated until cracks were observed. The number of complete cycles required to produce visible cracks in each specimen was noted, and this constitutes the thermal shock (spalling) resistance.

## 7. DETERMINATION OF REFRACTORINESS (PCE):

The refractoriness or softening point was determined using the method of pyrometric cone equivalence (PCE) in accordance with ASTM C 24 – 79 [Standard test method for pyrometric cone equivalent of refractory materials]. The refractoriness for each test cone is the number of the standard pyrometric cone that has bent over to a similar extent as the test cone. The temperature corresponding to the cone number was read off from the ASTM Orton Series.

#### 8. COLOUR.

The colour of soil samples should be assessed in a freshly excavated condition. This colour may be different from a dried sample, and from the sample mixed with water. Under some circumstances it may be important to know these differences.

It is preferable to use a standard colour chart such as that developed by Munsell. If standard colour charts are not available, use colour descriptions which are readily understood, e.g. red, brown, green, yellow, white black, pink etc. These can be supplemented by the use of words like: light, dark etc. Soils can also be one colour mottled with another, or one colour blotched or veined with another.

#### 9. STRUCTURE.

The soil being sampled may have a distinct structure and if so this should be recorded in the description. Tables1. Present guidelines for the descriptive terms to be used. Coarse and very coarse soils. Boulders, cobbles, gravels and sands.

## 10. LIQUID LIMIT (CASAGRANDE METHOD)

Apparatus: Equipment for the determination of moisture content (weighing to 0.01 g), Soil mixing equipment (glass plate, spatulas, and distilled water), Timer clock, Casagrande liquid limit device, Grooving tool, height gauge and Calibration of apparatus

- a) Mix about 300 g of the prepared soil (after 24 hours maturing) with a little distilled water if necessary, using two spatulas, for at least 10 minutes. At this point the first blow count should be about 50 blows. If a plastic limit test is required it is convenient to set aside a portion of soil for this purpose.
- b) With the cup resting on the base, press soil into the cup being careful to avoid trapping air. Form a smooth level surface parallel to the base giving a maximum thickness of 10 mm.
- c) Beginning at the hinge, and with the chamfered edge of the tool facing the direction of movement, make a smooth groove with a firm stroke of the grooving tool, dividing the sample into two equal parts. The tip of the grooving tool should lightly scrape the inside of the bowl, but do not press hard.

When using the tool, apply a circular motion so that it is always normal to the surface of the cup

- d) Rotate the handle at a speed of two turns per second check with a seconds timer.
- Stop turning when the bottom of the groove closes along a continuous length of 13 mm (use the back of the grooving tool as a gauge). Record the number of blows.
- e) Add a little more soil from the mixture on the glass plate to the cup and mix in the cup. Repeat stages (b) to (d) stated above until two consecutive runs give the same number of blows for closer. Record the number of blows.
- f) Remove a portion of about 10 g of the soil adjacent to the closed gap with a clean spatula, transfer to a weighed container and fit the lid immediately. Record the container number and determine the moisture content.
- g) Repeat steps (b) to (f) stated above after adding increments of distilled water, mixing the water well in. At least two determinations should give more than 25 blows, and two less than 25, in the range of about 10 to 50 blows. Do not add dry soil to the soil paste. Protect the soil on the glass plate from drying

out at all times. Each time the soil is removed from the cup for the addition of water, wash and dry the cup and grooving tool.

#### 11. PLASTIC LIMIT

Apparatus: Equipment for the determination of moisture content (weighing to 0.01 g), Soil mixing equipment (glass plate, spatulas, distilled water), Smooth glass plate free from scratches, for rolling threads on, A length of rod, 3 mm in diameter and about 100 mm long.

- a) Prepare and mature the test sample using wet or dry preparation method or take the sample previously set aside from the liquid limit test.
- b) Take about 20 g of the soil and allow it to lose moisture until it is plastic enough to be shaped into a ball without sticking to the fingers. Mould into a ball between the fingers and roll between the palms of the hands until slight cracks appear on the surface. Moulding and kneading is necessary throughout the test to preserve a uniform distribution of moisture and to prevent excessive drying of the surface only.
- c) Divide the sample into two roughly equal portions and carry out a separate test on each portion.

- d) Divide the first portion into four pieces. Mould one piece into a cylinder about 6 mm diameter between the first finger and thumb.
- e) Roll the cylinder under the fingers of one hand on a smooth glass surface, applying enough pressure to reduce the diameter to about 3 mm in about 5 to 10 complete forward and backward movements. Maintain a uniform pressure. Do not reduce pressure as the 3 mm diameter is approached. Use a metal
- rod of 3 mm diameter to judge the thread diameter.
- f) Pick up the soil thread, mould further and repeat the above. Repeat until the thread shears both longitudinally and transversely at a diameter of 3mm. Depending on the nature of the soil.
- g) Crumbling can usually be felt by the fingers. The crumbling condition must be achieved, even if greater than 3 mm diameter. If smooth threads of 3 mm diameter (like noodles) are formed, the soil is not dry enough. The first crumbling point is the plastic limit, do not attempt to continue reforming and rolling beyond this point.
- h) Gather the crumbled soil quickly, place in a small weighed container, and fit the lid immediately. Repeat the above process on second, third and fourth pieces of soil and place all fragments in the same container. Weigh it as soon as possible.

## 12. DETERMINATION OF THE PLASTICITY INDEX

The Procedure is a simple calculation and requires the determination of the liquid and plastic limits for the soil. The Casagrande method is to be used to determine the liquid limit.

The plasticity index of a soil is the numerical difference between the liquid limit and the plastic limit:

PI = LL – PL Results and Discussion

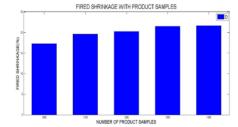
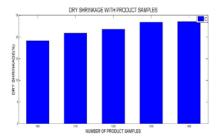
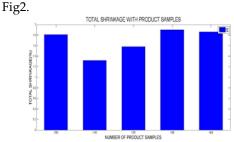
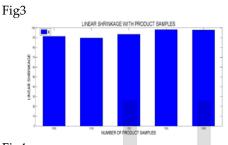


Fig 1.







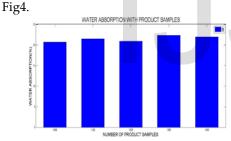


Fig5
When Figure 1, 2, 3 and 4 are compared with
Figure 5, there seems to be a correlation between
fired shrinkage, dry shrinkage, total shrinkage,
linear shrinkage and water absorption. High
shrinkage resulted to reduced water absorption as
a result of the closure of internal pores.

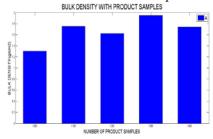
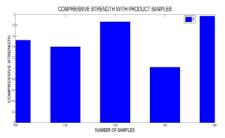


Fig6.

Figure 5 and 6. Displays the results obtained. It can be observed that the water absorption varies inversely with the bulk density. The water absorption values of the product samples clays are found within the acceptable ranges between 10 – 30% for fireclay refractories as recommended in (Chester, 1973). Also, the bulk density values fall approximately within the range of 1.7 to 2.1g/cm³ for dense firebricks (Hassan, 2001).



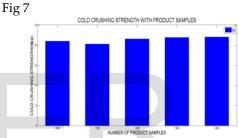


Fig 8
Figure 12. For site B products

The average cold crushing strength values are given in Figures 7 and 8. The stress at peak values of 1.5211MPa and 1.968MPa respectively fall within the ranges between 1.000 to 68.8MPa recommended for fireclay bricks (Krivandin and Markov, 1980) and are higher than the recommended minimum of 5MPa (Chester, 1973). Cold crushing strength is a useful indicator of the ability of a refractory to withstand handling and impact at low temperatures. The higher the efficiency of firing or degree of vitrification, the denser the product samples is and the higher the cold crushing strength.

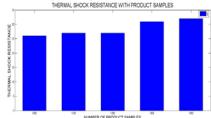


Fig9.

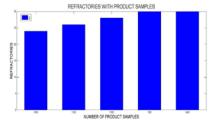


Fig 10 From the analysis in fig 10 the clay materials, the thermal shock resistances of both clays fall within the accepted value of 25-30 cycles as reported by De Bussy (1972).

It was observed from fig 9 analysis that, the refractoriness of the clay can fall within the standard value (1500oC-1750Oc) for fireclays. (Grimshaw, 1971).

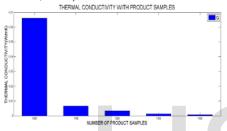


Fig10.

Fig 10 above shows that, the thermal conductivity of the clay samples ranges from (0.066 to 0.00066) W/mk. It means that, the insulation resistance of the clay materials experienced an increase in voltage of the current which is directly proportional to the thermal conductivity of the clay sample products.

The same trend was observed at all voltage. It was observed that the insulation resistance of clay sample products experienced a slight increase as the voltage increased, but thermal conductivity drastically decreased when the applied voltage was decreased.

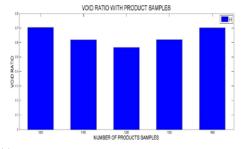


Fig 11. Fig 11 above shows that, the void ratio have a slight difference of 0.2 ranges from 0.566 to 0.702 which is normal for good clay materials.

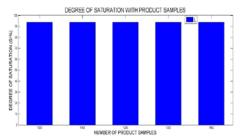


Fig 13.

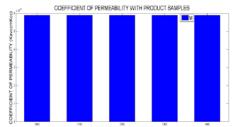


Fig 13.
Fig 12 and 13 above shows that, the degree of saturation and coefficient of permeability remained constant in the clay materials without any variation.

## Data from Liquid Limit Test

Table 2				
Description	clay			
Test number	45	33	22	18
Can identification	1	5	7	15
Number of bumps, N				
Can + wet clay, m <sub>2 (g)</sub>	32.6	32	31.6	31
Can +dry clay, m <sub>3 (g)</sub>				
can, m, (g)	27.6	27	26.6	26
Dry clay, m <sub>3</sub> -m <sub>1 (g)</sub>	16	16	16	16
moisture loss, m <sub>2</sub> - m <sub>3 (g)</sub>				
moisture content, w=	11.6	11	10.6	10
$\begin{pmatrix} m2 - m3 \\ m3 - m1 \end{pmatrix} \times 100$	5	5	5	5
	43.1	<b>4</b> 5. 5	47.2	50
Liquid Limit = Average W%	46.45	ı	1	1

## **Data from plastic limit test** Table 3

Description	1	2
Test number	P.L	P.L
Can identification	22	20
Can + wet clay, M2	22.8	22.9
Can +dry clay, m3-m1 (g)	21.8	21.9
Can m1	16	16
Dry clay, M3-M1 (g)	5.8	5.9

Moisture loss m2-m3	1	1
moisture content, $w = \begin{pmatrix} m2 - m3 \\ m3 - m1 \end{pmatrix} x$ 100	17.2	16.9
Plastic limit= Average W%	17.1%	

Plasticity Index = Liquid Limit - Plastic limit = 29.35%

The measurement is based on the moisture content at which the material has some arbitrarily defined consistency. In these tests, high moisture contents are associated with high plasticity and vice versa.

The liquid limit, plastic limit in this study, liquid limit ranges between 43.1-50%, plastic limit ranges between 16.9-17.2%, with plasticity index of 29.35%. Liquid limit and plasticity index are used to classify fine-grained soils. All liquid limit values in this study are <100%, classifying the clay as inorganic clay. Plasticity index of 29.35% indicates the clay is of high plasticity. This observation was further supported using Casagrande, (1948) plasticity chart to further support this assertion.

### Sieving Result

Table 4

1 able 4	
Description	clay
Steve aperture size or	350
particle size X (um)	
Initial mass on 350 um	10,000
sieve, mi (g)	
Mass retained on sieve, m	1400
(g)	
Mass retained= <u>m_x100</u>	14
mi	
Cumulative mass %	14
retained (oversize) R	
Commlative mass%	86
passing (undersize ) p=	
100R	

Table 4 shown the particles size distributions of Afuze clay, revealed a moderate percentage of clay and silt size particles passed 60 to  $100\mu m$  sieve). Grain size is important in assessing clay material for characteristics required for various industrial applications. The grain size composition of clay controls ceramic strength, shrinkage, paper filling and paper coating and its glossiness (Aref, 2002). Forbus et al, (1993), Veglio et al, (1993)

Ryan, (1979) specified that fine-grained minerals such as kaolinite are very industrially, in making paper, pulp filler, coating and also as an essential material in pharmaceutical and ceramic Industries. The grain size of kaolinite for example is said to range from below 1µm - 50µm (Heinskanen, 1996). Afuze kaolinite deposit falls in this category and could usefull especially in ceramic and paper industries. However, beneficiation of Afuze kaolinite through intercalation with urea can cause reduction in grain size that can facilitate its requirement in other industrial applications (Al-Shameri & Rong, 2009).

#### **CONCLUSION**

Compositional features and industrial application of Afuze kaolinite was evaluated based on chemical composition and physical characteristics of the deposit with a view to determining its suitability as an industrial raw material. From the study, it is obtained that Afuze clay deposit is predominantly kaolinite with a very low Fe<sub>2</sub>O<sub>3</sub> and CaO contents. It is rare for any single clay deposit to completely satisfy all the manufacturing requirement of its derivative product. Obviously, different sets of criteria are important to the production of any specific blend for a specific industry. Therefore, Afuze kaolinite can find application in refractory, pottery, paper and paint industry with beneficiation of the deposit where necessary.

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