International Journal of Scientific Research in Engineering
www.ijsrejournal.com
Volume 3 Issue 1ǁ Jan.- June 2018 ǁ PP. 28-32

Characterization of Opi Clay for Refractory Applications

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Abstract: *Opi clay deposits in Nsukka, Enugu state of Nigeria was characterized for refractory and other applications. The chemical analysis was done using X-ray florescence spectrometery. The physical properties tests like shrinkage, porosity, bulk density, water absorption, thermal shock resistance, making moisture,plastic limit, cold crushing strength were done following ASTM standards. Results obtained showed that Opi clay is chemically composed mainly of silica 55.64% alumina 26.98%, magnesia 3.88% and iron oxide 1.19%. The physical tests results show that the clay has linearshrinkage of 6.51%, apparent porosity of 22.36%, bulk density of 1.78g/cm^3, plastic limit of 26%,thermal shock resistance of 28cycles, refractoriness of 1696^{o}C. These physical and refractory characteristics investigated suggest that the clay belongs to the fire day class and can be employed in refractory and ceramic applications.*

Keywords: *characterization, Opi clay, refractory applications.*

Date of Submission: 25-06-2018 Date of acceptance: 10-07-2018

I. INTRODUCTION

Refractories are inorganic, non-metallic materials originating mainly as oxide mixtures capable of withstanding high temperature conditions without degrading their chemical, physical and mechanical integrity during application. They are usefully applied in high temperature environments, such as lining of kilns, furnaces, reactors, vessels in industries, as well as crucibles, tubes in electric furnaces, refractory cements, thermocouple sheaths, among others. Clay, from mineralogical and chemical view point is a complex aluminosilicate compounds with attached water molecules, originating from the chemical and mechanical degradation of rocks, like granites **[1]**. Clay with high silica and alumina contents are aluminosilicate minerals. The alumina content in such clay normally determine their potential usage as raw materials for developing aluminosilicate refractories. Among all clay minerals, the kaolinites possess the highest content of alumina (about 39.50% by weight), as indicated by the chemical formular, $Al_20_3.2Si.0_2.2H_2O$. Refractory materials, when classified by their chemical nature fall into, basic, acid and neutral refractories which are extensively discussed in literature **[2]**. The most important properties required of refractories are:

i. High strength at operating temperatures like low porosity and permeability, low thermal shrinkage, high thermal shock resistance, high refractoriness under local etc.
ii. Chemical inertness such as resistance to chemical attack in the operating environment, like high resistance to slag corrosion and liquid metal penetration.
iii. High refractoriness i.e. thermochemical stability such that it does to deform and melt at the operating high temperatures.

Most advanced countries in the world developed their refractories and industries over a century ago and their products dominate the world refractory market. Nigerian industries source their refractory needs from the international market despite abundant deposits of refractory raw materials all over the country. Several research work have been done on refractory clay in Nigeria **[3-8]**but there are many more clays in abundance throughout Nigeria not yet researched on. This work on Opi clay deposit aims at characterizing it and finding its suitability in refractory applications.

II. EXPERIMENTAL METHOD

2.1 Materials Preparation

A stratified random sampling was used to obtain the clay samples for this work. Sample area of 100m^2 was divided into ten smaller units and from each unit five samples were collected randomly at a depth of 50cm, using digger and shovel. The samples were mixed, crushed and soaked in water to enable non-clay materials float and clay material sediment at the bottom of the container. The water is decanted and the sedimented clayair-dried for moisture to evaporate. The dried clay is sieved through 1.8mm mesh. Cone and quartered method was employed to obtain a representative sample. The two alternate quarters are mixed again for further quartering. The final representative fractions for the sample was later ground to pass 60mesh (B5) sieve. The ground sample was used for the analysis.

2.2 Chemically Analysis

X-ray florescent (XRF) was used to determine the chemical composition of the clay. Clay sample, 1gwas added to10gof anhydrous lithium tetraborate ($Li_2B_4O_7$) acting as a fluxing agent. This constituent mixed inside a crucible was heated in a furnace up to 500^oC for 8 minutes and furnace cooled to room temperature to obtain a fused sample used for the analysis. Table 1 shows the result of the analysis of the sample's chemical composition.

Table 1. Chemical Analysis of Clay Material Collected

Sample Location	**Chemical Analysis (%)**									
	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	K_2O	Na_2O	MnO	TiO_2	LO1
Opi Clay(%)	55.64	26.98	1.19	3.88	0.69	0.02	0.08	0.011	0.03	11.419
Standard [2]	57.0	26.70	1.60	0.70	0.20	2.0	0.10	_	1.10	12.15

2.3 Loss on Ignition test (L.O.I)

The chemically combined water (H_2O) and sometimes organic matter content in inorganic materials is the loss on ignition [9]. A clay sample of 50g was put in a crucible, dried in an oven at 110^{0}C and cooled in adesiccators. A cleaned and dried porcelain crucible is weighed (M_1) to the nearest 0.001g in a chemical balance. The already dried clay sample was placed into the crucible and are weighed together (M_2) to an accuracy of 0.001g. The crucible with the sample is heated in a muffle furnace to a temperature of 900^{0}C,held for 3hrs, then cooled in a desiccator and weighed (M_3). Using the equation (1) below the loss on ignition was calculated:

$$\text{LO1} = \frac{\text{M2} - \text{M1}}{\text{M2} - \text{M3}} \,(1)$$

2.4 Bulk density test

The mass per unit volume of the clay ignoring the volume occupied by pores is the bulk density [10]. A brick specimen moulded to 5x5x4 cm dimensions was prepared from the clay, air dried for 24hrs, then dried in oven at 110^{o}C and cooled in a desiccators. It is then weighed (dried weight), then transferred into a beaker containing water and later heated on hot plate for 30minutes to assist in releasing air. The specimen was cooled and soaked weight (W) recorded. A beaker containing water was placed on a balance, then the specimen was suspended in the water and the suspended weight(s) was recorded. Using Equation [2], the bulk density was calculated thus:

$$\text{Bulk Density } = \frac{\text{Pw}}{\text{W} - \text{S}} \qquad (2)$$

Where P^W =Density of water

2.5 Apparent porosity test

The apparent porosity is a measure of the effective open pore space in a refractory into which molten metal, slag, fluxes, vapours, etc can penetrate and thereby contribute to eventual degradation of the structure. The porosity of any product is expressed as the average percentage of open pore space in the overall refractory volume. A brick specimen measuring 5x5x4cm was prepared from the clay, air dried and later dried in an oven at the 110^{o}C. The brick sample was gradually furnace fired at the rate of 10^{0}C/mm to the temperature of 900^{o}C, then cooled, stored in a desiccators and the dried weight (D) was recorded. The specimen was then put in a 250ml beaker, water was poured into the beaker until it is completely immersed in the water. It was then allowed to soak in boiled water for 30mins while being agitated intermittently to enable trapped air bubbles escape. The specimen was cooled in a desiccator and later the soaked weight (W) was recorded. A beaker of water was placed on a balance and the specimen is weighed while suspended in the water. This gave the suspended weight(s), and Equation (3) below was used in calculating the apparent porosity thus:

$$\text{Apparent Porosity } = \frac{\text{W} - \text{D}}{\text{W} - \text{S}} \times 100\% \qquad (3)$$

2.6 Linear Shrinkage test

Linear shrinkage is a property of claythat allows it to experience least structural changes and disintegration while being heated [**11**]. The clay sample was moistened with water to the wedging point (i.e. the moistened clay materials remain packed into a ball in hand until intentionally applied vibration causes the moisture to flow). The wedge sample was cast into standard slabs using wooden moulds. The clay bar test pieces were marked along a line in order to maintain the same position after heat treatment. The distance between the two ends of the slabs was measured with Vernier Caliper, and initial/ original length was marked on the samples. The bars were air dried for 24hrs, oven dried at 110^oC and fired in a furnace at the rate of 10^oC /min up to the temperature of 900^oC and held for 3hrs. The final length after firing was taken and the linear shrinkage was then calculated using Equation (4):

$$\text{Linear shrinkage } = \frac{A - B}{B} \qquad (4)$$

Where A= initial or original length, B = final length

2.7 Cold Crushing Strength

The cold crushing strength is the capacity of a refractory to provide resistance to a compressive load at room temperature. It is the load in kilograms persquare centimeter, at which the refractory breaks or fails. Cube specimen measuring 50mm^3 were made from the clay sample as refractory test pieces. They were dried in air for 24hrs, oven dried at 110^O C, fried in furnace at 900^oC and held for 5hrs at this temperature. The test pieces were later cooled to room temperature. The test sample was placed on a compressive tester and the load was applied axially by turning the hand wheel at a uniform rate until failure occurred. The reading of the manometer were recorded and cold crushing strength (CCS) was calculated using Equation (5):

$$\text{CCS } = \frac{\text{Maximum load (kN)}}{\text{Cross sectional area (M2)}} \qquad (5)$$

2.8 Thermal shock resistance test

This is the ability of the clay materials to withstand heating cycle (heating and cooling) several times before a deep crack appears [11]. Standard brick test pieces were moulded from the clay, air dried for 24hrs. and oven dried at 110^oC for 3hrs. the test pieces were fired in the furnace at 900^oC, held for 3hrs and allowed to cool. The test pieces werethen inserted in a furnace whose temperature has been constantly maintained at 900^oC. After 10minutes resident time, the test pieces were removed placed on firebrick, cooled in air for 10minutes and returned back to the furnace for another 10minutes holding. This process was continued until the test pieces cracked. The number of heating and cooling cycles before cracking of the test pieces was recorded as thermal shock resistance.

2.9 Refractoriness test

Refractoriness is the resistance of the clay material to fusion, softening and deformation at high working temperatures. It is the maximum temperature a clay material can endure without applied load before being deformed. A clay sample was prepared into cones of base diameter 1.27cm and height 3.81cm. The cone with some other standard cones of the same dimensions were mounted on a refractory platen and then placed inside a kiln. The cones were heated at a rate of 5^oC /min until the test cones bent over its own weight. They were then cooled and the test cones was compared with the standard cones. The test clay material was assumed to have the pyrometric cone equivalent (PCE) of the standard cone whose behaviour most resembled that of the test cone.

2.10Pfefferkorn Plasticity

50g of clay sample was put into a container ad a little amount of water added to it, the moistened clay was moulded into cylindrical shape using a cylinderical mould. The moulded clay was then deformed by dropping onto it a flat-headed plunger of known weight from a fixed height. The distance traveled was read from the graduated scale, and the modulus of plasticity (MOP) for the clay sample was obtained from the expression:

$$\text{MOP } = \frac{\text{Original Height}}{\text{Deformed Height}} \qquad (6)$$

2.11 Moisture content of the clay sample

The air dried sample was weighed (W), then placed in an oven and heated to a costant temperature of 110^oC for 24 hrs. The sample was taken out cooled in desiccator and re-weighed (W_1). The loss in weight gives the amount of moisture content (M.C) which can be expressed as percentage of initial clay sample, using the expression in equation (7):

$$\text{M.C } = \frac{W - W1}{W} \times 100 \qquad (7)$$

Where,
M.C is the moisture content (%),
W is the weight of the sample before drying (gm);
$W_l$is the weight of the sample after drying (gm)

III. RESULTS AND DISCUSSION

The chemical composition of Opi clay deposit is shown in Table 1. The silica and alumina contents of Opi clay, 55.6% and 26.98% respectively as shown in Table 1, are high. Silica content above 46.5% [12], indicates free silica, and this shows that the clay sample is rich in silica. This has contributed to a high compressive strength for Opi clay. The alumina content of 26.9% is within the standard range of25-45% and this with magnesia content of 3.88% contributed to good refractories of the clay. The presence of high value ofimpurities such as Fe_20_3in aluminosilicate refractory, lower the refractoriness and service limit of the clay bricks. Table 1 also compares the chemical compositions of the clay sample and the standard value. The major phases of the clay material collected are aluminosilicate and is acidic refractory in nature [13]. The moistures content of the Opi clay sample is 9.4%, showing that much water will not be needed for proper mixing of the material. The bulk density of the clay sample material is 2.15g/cm^3 as shown in Table 2, and is within the recommended value of 1.7-2.1g/cm^3 [14]. The apparent porosity of Opi clay sample which is 22.336% falls within the standard value of 20-30% [13,15]. Low apparent porosity is known to enhance gas entrapment in the material during operation. This will always affect adversely the life span of the refractory material during operation [16] the fired linear shrinkage of the clay is within the recommended value of 4-10%[13,15]. The cold crushing strength (compressive strength) of the Opi clay is close to the standard value of 15KN/m^2[17]. The thermal shock resistance of the clay sample is 28cycles which is within the acceptance values of 20-30cycles**[17].** The refractoriness value obtained for Opiclay 1696^{o}C, is within the range of 1500-1700^{o}C for the fireclay [14], see Table2.

Table 2: Physical Properties of Opi Clay with Standard Clay for Industrial applications [13-15]

Sample description	Bulk density (g/cm^3)	Apparent porosity (%)	Linear Shrinkage (%)	Cold Crushing Strength MN/M^2	Thermal shock resistance (cycles)	Refractoriness °C	Plasticity index	Making Moisture (%)
Opi Clay	2.15	22.36	8.40	14.90	28	1696	26	25.03
Fire Clay	2.30	20.30	4-10	15-00	20-30	1500-1700	-	-
Siliceouse fire clay	2.00	23.70	7-10	15.00	1	1500-1600	-	-
Ceramics	2.30	10-30	-	15.00	20-30	1430-1717	10-60	-
Refractory brick	2.30	10.30	-	15.00	20-30	1430-1717	-	-

IV. CONCLUSION

The investigation carried out on the Opi clay deposit showed that the service properties have favourable results such that when bricks are produced from it they can be used for living nonferrous and cast iron furnaces as well as kilus for cement industries. The result of the chemical analysis shows that Opi clay contains alumina (Al_2O_3) and silica (SiO_2) as major constituents. The physical properties of the clay are all within the standard range for refractory production for medium duty applications. Therefore Opi clay is found to be a source of raw material for the production of refractories are ceramics.

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