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Characterization of Inyi Clay Deposit for Its Ceramics Potentials

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ABSTRACT: *Inyi clay deposit in Enugu State, Nigeria has been evaluated for its potentials in ceramics applications. The chemical composition determined using x-ray florescence spectrophotometer showed that the clay contains 45.26% SiO_2, 35.19% Al_2O_3, 0.24%Fe_2O_3, 0.15%CaO,0.062MgO, 0.0554% Na_2O, 0.04% K_2O, 0.002% MnO, 2.460% moisture and 8.68% L.O.I. This indicates that the clay contents are more than 80% of SiO_2 and Al_2O_3. The physical and mechanical properties tests suggest that the clay possess the following after firing between 1000 to 1400^oC. Apparent porosity 20.1 to 22.03%; thermal shock resistance 28cycles; refractoriness 1660^oC; bulk density 2.43, cold crushing strength368.74 kg/cm^2.The clay as mined is light brown lateritic silt clay with heterogeneous structure of 350 micron sieved particle size. On firing, the colour changed from red brown at 900-1100^oC to dark brown at 1200 -1400^oC.*

Keywords: *Characterization, clay Deposits, Refractory Production*

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I. Introduction

Refractories are materials which withstand high temperature operations and resist fusion at such temperature regime. According to ASTMC71, they are "nonmetallic materials having those chemical and physical properties that make them applicable for structures or as components of systems that are exposed to environments above 538^oC [1]. At high temperatures, refractory materials possess the ability to retain their strength. Most refractory materials are developed from the oxides such as alumina, silica, magnesia, chromite, zirconia, beryllia etc. Also fireclay has been widely used in developing refractories [2].

Refractories are classified in different ways [3] as follows:

A. Based on refractoriness such as low heat duty (LHD), pyrometric cone equivalent (PCE) value (sugar cones) 19-28, and refractoriness / fusion point 1520 -1630^oC. Medium /intermediate heat duty refractories (MHD), PCE value (sugar cones) 28-30, and refractoriness/fusion point 1630-1730^oC. High heat duty refractories (HHD), PCE value (sugar cones) 30-33, refractoriness/fusion point 1670 -1730^oC.Super duty refractoriness (SD), PCE value (sugar cones) > 33, refractoriness/fusion point > 1730^oC.
B. Based on their chemical nature either as acidic,basic or neutral refractory. Acidic refractories are fire clay, silica, quartz, ganister sand;semi-silica etc. Basic refractories are basic oxides without silica such as alumina, magnesia etc. while neutral refractories are chromite, graphite, carbon refractory, carbide, mullite, kyanite etc.
C. Based on method of manufacture; (i) dry pressed (Fusion cast or electorcast), hand moulded(ii) formed (Normal, fire/burned or chemically bonded) (iii) unformed (monolithic –plastics, ramming masses, gunning, castable, spraying)

   Refractory materials are evaluated by determining their chemistry, physical as well as mechanical properties. Some important physical and mechanical properties of interest include: porosity and slag permeability, refractoriness, bulk density, linear shrinkage, cold crushing strength, thermal shock resistance.

II. Experimental Technique

2.1 Materials and Equipment

The clay sample used in this study was collected from Inyi in Enugu State, Nigeria. The sample materials were collected at a depth of 1.0 meter using pick, digger and shovel. A 50kg clay sample materials were weighed in a balance crushed with hammer mill and ground to powder using laboratory grinder. They were soaked in water, to allow non clay materials to float and decant with water. The clay materials are later air dried and sieved through 1.8 mm mesh. Representative sample was taken from the bulk for chemical analysis using x-ray florescence spectrophotometer. Some test samples were rammed into cylindrical standard size moulds of (50.0mm x 50.0mm) and (25mm x 25mm) using Ridsdale & company serial No. 891 standard laboratory rammer. The prepared test pieces were dried and fired in oven accordance with A5TM (1989) code {3}.

2.2.1 Compositional Analysis

The result of the compositional analysis of the sample mineral from Inyi clay deposit as determined using x-ray florescence spectrophotometer is presented in table 1.

Table 1: Chemical Composition of Inyi Clay (%)

Types of oxide (raw material)	Percentage (%)
SiO_2	45.26
Al_2O_3	35.19
Fe_2O_3	0.24
CaO	1.50
M_gO	0.062
Na_2O_3	0.054
K_2O_3	0.049
M_nO	0.002
Moisture	2.460
L.O.I.	8.68

The physical and mechanical properties evaluation of bulk density, apparent density, porosity, water absorption, changes in firing colour, thermal conductivity, thermal shock resistance, refractoriness and shrinkage tests were determined.

2.3 Physical Properties Evaluation

2.3.1 Bulk density determination

Some test pieces prepared from the clay body were air dried for 24 hours on a table, oven dried at 110°C for 6 hours, and fired at temperatures of 1000, 1100, 1200, 1300, 1400 °C respectively.

After cooling to room temperature each sample was weighed to an accuracy of 0.008 gm (dried weight, D). The specimens were then individually transferred into a beaker, heated for 30 minutes to enable evacuation of trapped air and then cooled to room temperature. Each of the specimens is then soaked in water to obtain its soaked weight (Ws) and later suspended to get its suspended weight (Ss). The bulk density (BD) was thus, evaluated using equation (1) [4].

$$BD = \frac{D \cdot \ell_w}{W_s - S_s} \quad g/cm^3 \qquad \text{\_\_\_\_\_\_\_\_\_\_\_\_ (1)}$$

Where BD is the bulk density
D is the dried weight
W_s is the soaked weight
S_s is the suspended weight
ℓ_w is the density of water

2.3.2 Apparent porosity

Some representative test sample pieces of bricks were prepared, air dried for 24 hours and subsequently oven dried at 100°C for another 24 hours. These samples were later dried at a temperature of 110°C, cooled to room temperature and moved into a dedicator. They were subsequently weighed on scientific balance to the nearest 1.01 gm to obtain the dry weight. The samples were transferred into 250ml beakers in empty vacuum desiccators. The beakers are filled with boiled water until the test samples were completely immersed, and then allowed to soak for 30 minutes while being intermittently agitated to enable the release of trapped air bubbles. The specimens were then cooled in empty vacuum desiccators and the soaked weight taken. Beaker of water is subsequently placed on a weighing balance and the specimens were weighed suspended in water. The suspended weights were recorded, the apparent porosity (A_p) was evaluated from the equation (2)

$$AP = \left(\frac{W - D}{W - S} \right) x100 \ \% \qquad \text{\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ (2)}$$

Where W = Soaked weight
D = Dried weight
S = Suspended weight

2.3.3Fired linear shrinkage

Some sample test pieces produced in standard slab forms were marked original /initial length along a line in order to maintain the same position after heat treatment. Vanier caliper was used to measure the distance and the samples were air dried for 24 hours, then dried at 110^{o}C for 6 hours in oven. Subsequently they were fired at different temperatures of 1000, 1100, 1200, 1300, 1400^{o}C respectively and furnace cooled to room temperature. The fired linear shrinkage at each temperature was calculated using equation (3).

$$F.S = \left(\frac{D_1 - F_1}{D_1} \right) x100 \ \% \qquad (3)$$

Where F.S = Fired linear shrinkage
D_1 = Initial length
F_1 = Final length

2.2.4 Thermal shock Resistance

Some test pieces of bricks measuring 50mm x 50 mm x 75mm were employed for this test. The tests were conducted using box type electric resistance heating furnace at temperature of 1400^{o}C. For the test temperature, three brick samples were placed in the furnace preset and maintained at that temperature. The specimens were allowed to soak at the set temperature for 10minutes, then removed from the furnace, cooled in the air for ten minutes and observed for cracks or fracture. Where none of these occurred, the bricks were returned to the furnace for a repeat cycle. This cycle of heating, cooling and observation for cracks or fracture was repeated continually at the test temperature until cracks or fracture was observed. The number of complete cycles required to produce visible cracks in each specimen was noted, and this constitute the thermal shock (spalling) resistance (2).

2.2.6 Refractoriness

It is the resistance of the clay to fusion and softening at high working temperatures. Refractoriness is the maximum temperature clay can withstand without any applied load. Some conical test pieces were prepared to the dimensions of standard cones whose melting points are higher than that of the clay samples. The test pieces were mounted on a refractory plaque along with some standard cones and placed inside a furnace. The cones were then heated at a rate of 5^{o}C/min until the test cones bend over their own weight. The test cones were furnace cooled and later compared with the standard cones. The test cones were taken to have the pyrometric cone equivalent (PCE) of the standard cone whose behavior is closely similar to that of the test cones.

2.2.7 Moisture Content of the Clay Sample

The air dried sample was measured to obtain its weight (W), and then placed in an electric resistance furnace and heated to a constant temperature of 110^{o}C for 24 hours. The sample was taken out and cooled in desicator and reweighed (W_1). The loss in weight gives the amount of moisture content (MC) which is expressed as percentage of the initial clay sample. The following expression in equation (4) was used to determine the moisture content.

$$MC = \frac{W - W_1}{W} x100 \ \% \qquad (4)$$

2.2.8 Plastic Index Tests

These tests were carried out in accordance with ASTM D423 – 66 (Re – approved 1972) or BS1377:1975, Test 2B) and ASTM 424 -59 (Re- approved 1971) or (Bs 1377:1975, Test 3) for the determination of atterberg limits using casangrade Apparatus and accessories. The plastic index, PI was computed using the moisture content determination for liquid limit (LL) and plastic limit (PL), giving the PI of the clay, to the nearest whole number.

2.2.9 Cold crushing strength test

Cold crushing strength is the clay's ability to withstand loading and abrasion. Some cubic samples measuring 50cm^3 was formulated from the clay body, dried, fired in a furnace at 900^{o}C and soaked at the prevailing temperature for 5 hours. The test piece was cooled to room temperature. The test piece was mounted on a compressive tester and the load was applied axially by turning the hand wheel at a uniform rate were recorded while the cold crushing strength (CCS) was calculated using equation (5)

$$CCS = \frac{Maximun\ \ Load\ (kg)}{Cross\ Section\ \ Area\ (cm^2)} \qquad (5)$$

III. Results And Discussion

The chemical analysis results of Inyi clay sample show high silica (SiO_2) and Alumina (Al_2O_3) contents of 45.26% and 35.19% respectively. It is also low in iron oxide (F_2O_3) 0.24%, calcium oxide (CaO), 1.50% and some other minerals appear very low. These values satisfy the standard for the manufacture of refractory bricks and some other ceramic materials, 46-62% $Si0_2$ and 25-44% Al_20_3 [2] Thus, it can be used in manufacturing refractory bricks for lining melting furnaces for aluminum alloys and copper alloys as well as other low temperature operating furnaces and kilns. During firing the colour of the clay samples changed from light brown at 1000^0C to dark brown at 1400^0 C. This was due to the presence of Iron oxide and other impurities in the clay.

The moisture content of Inyi clay sample is 2.69% which is within the range for standard reported by Chester 2.6 – 2.7% [1].

The loss on ignition (LOI) of Inyi clay sample is 8.68% which is within the range of 8- 18% for ceramics and refractory materials as well as 5 – 14% for high melting clay [1]. It is important for loss on ignition values to be low because of its effect on porosity of refractory materials.

The physical and mechanical properties of Inyi clay are shown in table 2.

Table 2: Physical and Mechanical Properties of Inyi Clay

Measured properties	**Fired temperatures ^{0}C**				
	1000	1100	1200	1300	1400
Bulk density (g/cm^3)	1.92	1.96	2.02	2.14	2.43
Apparent density (%)$^?$	1.76	1.80	1.88	1.98	2.27
Apparent porosity (%)	20.10	20.77	21.61	21.32	22.03
Water absorption (%)	19.56	17.14	15.02	11.16	9.08
Dry shrinkage (%)	5.84	7.45	9.36	12.11	14.21
Fired shrinkage (%)	1.81	1.92	2.32	3.23	3.94
Total shrinkage (%)	7.65	9.37	11.68	15.34	18.15
Firing colour	Red brown	Red brown	Dark brown	Dark brown	Dark brown
Cold crushing strength (kg/cm^2)	223.45	246.51	304.10	343.54	368.74

Table 3: Other Physical Properties Of Inyi Clay

Property	**Value**
Moisture content %	2.69
Green strength (K_gf/cm^2)	27.15
Plastic limit (%)	17.5
Refractoriness °C	166O °C
Conductivity	0.000667
True specific gravity (g/Cm^3)	2.68
Thermal shock resistance	28 cycles

The bulk density of Inyi clay sample increased on firing from 1.92 gm/cm^3 at 1000^0C to 2.43 gm/cm^3 at 1400^0 C. This is within the acceptable standard value range for refractory production, 2.2 – 2.8g/cm^3, [2]. The fired shrinkage of this clay varied from 1.81% at 1000^0 C to 3.94% at 1400^0C, which is also within the acceptable standard [1]. Consequently, based on the fired shrinkage the material will possess a better interlocking of grains, thus enhancing the strength of the refractory during operational service. The thermal shock resistance of Inyi clay is 28 cycles thus falling within the accepted value of 25-30cycles as reported by De Buss [5]. The apparent porosity, percentage shrinkage and the cold crushing strength increased with increase in the firing temperature. The water absorption of formed bricks decreased as the firing temperature increased.

The refractoriness of Inyi clay sample is 1660^0C which is within the acceptable standard range for fireclay refractory material of 1500 – 1700^0 C [6, 7]

IV. Conclusion

There are large quantities of clay minerals reserves in Nigeria, yet no refractory industrial is processing them for industrial application. Most industries requiring refractories in Nigeria import them. The current economic recession facing Nigeria and scarcity of foreign exchange is motivating some industries to look inwards in solving their raw materials need. Thus the need to produce quality refractory materials to take care of local indusial demands as well as for export market has become very imperative. The studies carried on Inyi clay sample has shown that it met all the parameters required of clay to be used as raw material for refractory production. The silica ($Si0_2$) content of the clay is high which positively affects its crushing strength. The presence of higher percentage of alumina in Inyi clay is responsible for its high refractoriness. These high percentages of both silica and alumina in Inyi clay show that it belongs to the aluminosilicate group. The clay is good for formulating acid refractories and also good for lining crucible furnaces, preheating and heat treatment furnaces, cement kilns, rotary kilns, cupola furnaces and direct reduction furnaces.

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