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Characterization Of Iboko And Nduage-Echara Clay Deposits In Ebonyi State, Nigeria For Refractory Applications.

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ABSTRACT:*This investigation is directed towards harnessing local potentials in refractory material sourcing for use in Metallurgical and other industries. Clay deposits from Iboko and Nduage Echara in Ebonyi State, South-East Nigeria were characterized for their refractory properties. The results showed that the clay samples have the needed refractory properties for developing medium duty fire clay refractories. The samples collected belong to the family of alminosilicate and acidic type refractory material. The chemical analysis shows that the clay contain silica (SiO_2) and alumina (Al_2O_3) as the major constituents with 57.41%SiO_2 and 23.91% Al_2O_3 for Iboko Clay, 53.74% SiO_2 and27.12% Al_2O_3 for Nduage Echara clay. The refractoriness of Iboko clay is 1.50 while that of Nduage Echara is 1670˚C. These are all within the standard value range of 1500-1700˚C. The thermal shock resistance of the materials were 26 and 28 cycles for Iboko and Nduage Echara respectively which are also within the standard value range of 20-30 cycles for refractories. The cold crushing strength of Iboko and Nduage Echara clays are 23.4MPa and 21.2MPa respectively, also within the standard value range of 9.8-68.8MPa for refractories. The linear shrinkage, bulk density as well as the porosity of the two clays are within permissible limits for refractory clays, for metallurgical and industrial applications.*
KEYWORDS: *Characterization, Iboko, Nduage-Echara clays, Refractory.*

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I. INTRODUCTION

Refractory materials are materials normally employed in high temperature environments especially in industries for lining kilns, furnaces, reactors, vessels, etc. These materials are normally non-metallic, inorganic in nature, mainly mixtures of oxides which are capable of withstanding very high temperature regions without losing their chemical and mechanical stability. Silicon and aluminum oxides are commonly found in refractory mixtures and they are the main constituents of all aluminosilicate minerals, especially the clay mineral. The potentials of a clay mineral to be employed as an aluminosilicate refractory raw material is determined by the amount of alumina in it [1]. Kaolinite with up to 39.50wt% alumina as indicated by the chemical formular, $Al_20_32SiO_2.2H_20$ has been found to possess the highest amount of alumina. Fire- clay are also extensively employed in developing refractories. The indispensability of refractory materials in applications where high temperatures are inevitable such as ceramics production, power generation, metallurgical and cement industries as well as reactors e.t.c are well known [1,2]

Refractory materials are very expensive and their sudden failures during service result in very great loss in economy, energy, time, products, and equipment and in some cases human accidents. Some basic properties of clay such as the chemical composition, plasticity, permeability, refractoriness, strength, shrinkage are highly required in refractory materials formulation. Omowumi [3], stated that the most basic property of clay for refractory production is its resistance to high temperature.

Chesti [4], classified refractories using their service temperature ranges, as low refractory (below 1770^0C); medium refractory (1770 - 2000^0C) and high refractory materials (above 2000^0). Gupta [5], used chemistry of refractories to classify them into three types, acidic, basic and neutral refractories. These show that appropriate materials selection is very much required for the production of refractory materials of specified properties.

A lot of studies have been done on some local refractory clays and their applications in Nigeria [2, 3, 6-10]. There are potentials of high requirement for refractory products in Nigeria as industrial development struggle advances. Currently most industries that require refractory materials import them at very high cost and this translates to exorbitant prices of the products of such industries. Therefore, not minding the amount ofresearch works onour local clays for industrial utilization, enormous works are still to be done on harnessing the abundant clay deposits spread all over Nigeria. Thus, this study is aimed at characterizing the Iboko and Nduage Echara clays to determine their suitability for refractory applications in metallurgical and other industries.

II. EXPERIMENTAL METHOD

2.1. Materials and sample preparation

The clay samples employed for this work were collected from Iboko and Nduage Echara in Ebonyi state Nigeria. These clay samples were dug from a depth of 1.2meter using shovel and digger, later crushed using hammer mill and roll crusher. The pulverized sample was then soaked in water for clay to settle and organic matter to float and be decanted. The samples were dried in air for 48hours and sieved through 63 microns aperture. The bulk sample was coned and quartered to obtain the required representative sample which was used for chemical analysis. Also the samples for other tests were obtained.

2.2. Chemical Analysis

X-ray florescent was employed in determining the chemical composition of the clays. For each of the clay sample, 10g of anhydrous lithium tetraborate ($Li_2B_4O_7$), a fluxing agent, was mixed with 1g of clay samples in a clean crucible. The crucible was placed in a furnace and heated up to 500^0C for 8 minutes and cooled to room temperature to obtain a fused sample used for the chemical analysis.

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

This is the quantity of chemically combined water (H_2O) as well as organic matter content in inorganic materials. A 50g of clay sample was dried in oven at 110^0C and cooled in a desiccator. A cleaned porcelain crucible was dried and weighed (M_1), to the two nearest 0.001g. The cooled clay sample was put into the crucible and the two were weighed together (M_2) to 0.001g accuracy. The crucible with its content was transferred into a furnace, heated to a temperature of 900^0C for 3 hours, later cooled, stored in a desiccator and then weighed (M_3). From equation (1), the loss on ignition was calculated thus:

$$\mathrm{L.O.I} = \frac{\mathrm{M_2 - M_1}}{\mathrm{M_2 - M_3}} \qquad (1)$$

2.4. Fired Linear Shrinkage Test

The linear shrinkage is a property of the clay that makes it undergo least structural changes and disintegration when heated. Test sample pieces were prepared in conformity with ASTM standards into slabs [12]. Marks were made on each test piece along a line in order to maintain the same position after heat treatment. Vernier caliper was used to measure the distance between the two ends of the slab. The test pieces were air-dried for 24 hours, then oven-fired at 110^0C for 6 hours, cooled to room temperature and measurements taken. Equation (2) was used in calculating the fired linear shrinkage:

$$\mathrm{FS} = \frac{(\mathrm{D_1 - F_1})}{\mathrm{D_1}} \times 100\% \qquad (2)$$

Where D_1 is the dried length
F_1 is the fired length

2.5. Bulk density test

The mass per unit volume of the clay neglecting the volume occupied by pores is the bulk density. Some clay samples were moulded into standard brick test pieces of 50 x 50 x40mm. These test pieces were air-dried for 24hours and then oven-dried at 110^0C, cooled in a desiccator and weighed (dried weight). The test piece was put into a beaker of water, heated for 30 minutes for air to be released, then cooled and soaked, weight (W)recorded. The suspended weight (S) was also recorded and from equation (3), the bulk density calculated as:

$$\text{Bulk density} = \frac{\ell^{w}}{\mathrm{W - S}} \qquad (3)$$

Where, ℓ^{w} is the density of water.

2.6. Apparent Porosity Test

The ability of the clay material to be impervious to liquids and gases is a measure of apparent porosity. During firing of a clay body, pores are formed as water, gases and other organic matter are given off. Test samples measuring 50 x 50 x 40mm were prepared, air-dried for 24 hours, and later oven-dried at 110^oC. They were transferred into a kiln and heated at the rate of 10^oC/min till the temperature of 900^oC was attained. The samples were cooled and stored in vacuum desiccator. The dried weight (D) of the samples were measured to the nearest 0.01g. Each of the test piece was soaked in 250ml beaker containing water until the sample is completely immersed. The test pieces were allowed to soak in boiled water for 30 minutes while being agitated intermittently to enable trapped air bubbles to escape. The specimen were cooled in a vacuum desiccator and the soaked weight (W) was measured. Each specimen was then weighed suspended in a beaker of water placed on a balance and the suspended weights (S) were measured. Equation (4) was used in determining the apparent porosity:

$$\text{Apparent Porosity} = \frac{W - D}{W - S} \times 100\% \qquad (4)$$

Where, W is the soaked weight
D is the dried weight
S is the suspended weight

2.7. Moisture Content Test

The test specimens for moisture content determination were weighed and weight (G) recorded for each. The samples were then transferred into an oven, heated to a temperature of 110^{o}C, cooled and weighed to obtain the weight (G_1). The amount of moisture content in percentage (W) is the loss in weight which is determined using Equation (5):

$$W = \frac{G - G_1}{G} \times 100\% \qquad (5)$$

Where, W is the moisture content, (%)
G is sample weight before drying, (g)
G_1 is sample weight after drying, (g)

2.8. Thermal Shock Resistance

The ability of the clay material to resist deformation during cyclic heating for many times before deep cracks occur is the thermal shock resistance of the clay. Four test pieces of slabs were moulded, air-dried for 24hours and oven-dried at 110^{o}Cfor 3hours. They were then transferred into a kiln, heated to 900^{o}C, and held for 10 minutes. They were removed with a pair of tongs, placed on brick platen and cooled to room temperature. The specimens were returned back to the Kiln maintained at 900^{o}C, held for another 10 minutes, later removed and cooled to room temperature. This heating, holding and cooling cycle continued until crack developed on the specimens. The number of the cycles endured by the test pieces before cracking developed was recorded as the thermal shock resistance.

2.9. Cold Crushing Strength Test

This is the ability of the clay materials to withstand abrasion and loading without deforming or shattering into pieces. Standard brick cubes measuring 50mm^3 were subjected to strength tests using universal strength testing machine. The strength values obtained both in the direction of forming and normal to the direction of forming of the bricks were recorded.

2.10. Permeability Test

The measure of the rate at which fluids (gases, liquids) can pass through a porous material determines its permeability. Refractories as materials working under fluid environment should be impervious to liquids and gases. Gas leakages and liquid penetration through refractory walls must be avoided always. The pores sizes, their uniform distribution as well as the internal surface area and capillary effects greatly affect the permeability. Test samples were prepared to 50mm diameter and 50mm height specification using a standard rammer. The test pieces were air-dried for 24 hours and oven-dried at 110^{o}C for 12 hours. Air of 200cm^3 was passed through each sample from the jar containing water. A permeability meter, by Ridsdale& Co. serial no 894 was used to measure the pressure difference between the surfaces. The equation (6) was used to calculate the permeability [5]:

$$PA = \frac{VH}{APT} \qquad (6)$$

Where, PA is permeability no;
V is volume of air (cm^3)
H is Height of the specimen (mm);
A is the cross sectional area of the specimen (MM^2);
P is the air Pressure in mm of water

T is the time (in minutes).

2.11. Refractoriness Test

The resistance of the clay body to fusion or softening at the high operating temperature is a measure of its refractoriness. Clay samples were produced into cones of base diameter 12.7mm and 38.1mm height. The cones were mounted on a refractory plague along with some other standard cones of the same dimensions and standard compositions. This arrangement was transferred inside a kiln, heated at the rate of 10^{o}C/min up to the value of 1200^{o}C, then the rate was reduced to 5^{o}C/min, and the heating continue until each of the test cone bent over its own weight and the final temperature recorded. The plague was removed from the kiln and after cooling, the test cones were compared with the standard cones. The test clay material was taken to have the pyrometric cone equivalent (PCE) of the standard cone whose behavior most resemble that of the test cone.

2.12. Modulus of Rupture (MOR) Test

The Modulus of Rupture (MOR) is the flexural breaking strength of a refractory. It is measured at room temperature and expressed in kilograms per square centimeter. Eight long rectangular test pieces were moulded and air-dried for seven days, followed by oven-drying at 110^{o}C until a constant weight was attained. Six test pieces were fired to their respective temperatures of 900, 1000, 1100, 1200, 1300 and 1400^{o}C in a laboratory kiln and later cooled. Each of the test piece is placed in electrical transversal strength machine to determine the breaking load, P (kg), of the specimen. A vernier caliper was used to measure the distance between support L (cm) of the transversal machine. The height, H (cm) and the width, B (cm) of the broken pieces were determined and the average value obtained from the two broken parts was recorded. The modulus of rupture was thus calculated using Equation (7):

$$\text{Modulus of Rupture}(\text{Kg}/\text{cm}^3) = \frac{3PL}{2BH^2} \qquad (7)$$

III. RESULTS AND DISCUSSION

The chemical composition of the clay deposits is shown in Table 1. The Table shows that silica and alumina are the major constituents of the clay. Iboko clay has 57.41% silica and 23.91% alumina, while Nduage Echara has 53. 74% silica and 27.12% alumina. The relative proportions of silica and alumina in the refractory clays are relevant. The higher the proportion of alumina, the higher the refractoriness needed to form the verification of the material which characterizes ceramic products [1]. This explains why the Nduage Echara clay has higher refractoriness value than Iboko clay. The combined silica and alumina percentages in both clays are very close, with Iboko clay having 81.32% and Nduage Echara clay having 80.86%. Other oxides constituents of both clays are in small quantities. The silica content in the clay samples investigated is high. Silica content above 46.5% indicates free silica [13], which showed that the clay samples are rich in silica and this contributed to their high crushing strength.

Table 1, shows the chemical composition of the Iboko and Nduage Echara clays compared with standard clay for industrial applications [1, 11]

Table 1: Chemical Composition of Iboko and Nduage Echara Clays Compared with Standard Clay for Industrial Applications [1, 11]

Composition	Iboko	Nduage Echara	ceramics	Refractory brick	High melting clay	Glass	paper	Paint
SiO_2	57.49	53.74	60.50	51.70	53 – 73	80-95	45.0-45.8	45.3-47.9
AL_2O_3	23.91	27.12	26.50	25 -44	16 -29	12-17	33.5	37.9-38.4
Fe_2O_3	1.85	1.75	0.5-1.2	0.5 – 2.4	1 -9	2 -3	03-0.6	13.4-13.7
CaO	0.93	0.14	0.18-3	0.10 – 20	0.5 -2.6	4 – 5	0.03-0.	0.03 -0.60
MgO	2.89	3.40	-	-	-	-	-	-
T_iO_2	1.46	1.15	-	-	-	-	-	-
Na_2O	0.67	0.23	-	-	-	-	-	-
K_2O	0.46	0.75	-	-	-	-	-	-
M_NO	0.04	0.20	-	-	-	-	-	-
LOI	12:00	13:00	8 -18	8-18	5-14	-	-	-

The impurities in aluminosilicate refractory, such as, Fe_2O_3, Na_2O etc. when present in high values lower the refractoriness and service limit of the bricks. From Table 1, Iboko clay can serve as high melting clay. While Nduage Echara clay can be used in producing refractory brick when compared to the standard value. The loss on ignition for Iboko and Nduage Echara clays are 12%, and 13% respectively which are within the standard values of 5-14% for high melting clay and 8-18% for refractory brick. The values of loss on ignition are required to be low [3], because of its effect on the porosity of material, more especially refractory bricks. The physical properties of the clays are shown in table 2.

Table 2: The physical properties of Iboko and Nduage Echara Clays compared with Clay for Industrial Applications (1, 3, 15)

clay	Linear Shrinkage %	Bulk Density g/cm^3	Apparent Porosity %	Moisture Content %	Thermal Shock Resistance Cycles	cold crushing strength MPa	Permeability No	Refractoriness ^oC	Modulus Of rupture Kg/cm^2
Iboko	7.69	2.21	20.40	10.2	26	23.4	39	1650	23.80
Nduage Echara	7.30	2.26	17.38	8.6	28	21.2	36	1670	23.15
Standard	2-10	22-2.8	10-30	8-12	20-30	9.8-68.8	25-90	1500-1700	-
Fire clay siliceous fire clay	4-10 7-1	2.3 2.0	20.30 23.7	- -	- -	- -	- -	1500-1700 1500-1600	- - - -
Ceramic	-	2.30	10-30	-	20-30	-	-	1430 1717	-
Refractory brick	-	2.30	10-30	-	20-30	-	-	1430-1717	-

The linear shrinkages is an indicator of the firing efficiency of the clay samples. The values for Iboko and Nduage Echara clays are 7.69% and 7.30% respectively. This is within the standards as quoted by omowuni [3] which is 4-10%. Abolarin et al [14] were of the view that lower value ranges were more desirable as this implies the clay is less susceptible to volume change. Chester [1], recommended that refractory clays can have liner shrinkage range of 7-10%. It implies that both clays being studied can be classified as refractory clays based on this range.

The bulk densities of the two clays are 2.21g/cm^3 and 2.26gm/cm^3 for Iboko and Nduage Echara respectively as shown in Table 2. These values are within the standard value ranges of 2.2-2.8 g/cm^3 for refractory clays [1]. The apparent porosity of a refractory raw material, especially fireclays is affected by factors like clay composition, size and shapes of particles, ramming pressure, as well as reactions occurring during firing. The porosity values of the bricks developed from Iboko and Nduage Echara clays, Table 2 are within the acceptable range of 10-30% for fireclay refractories as recommended by chester [1].The moisture content of Iboko clay, 10.2% and Nduage Echara 8.6% are all within the standard value ranges of 8-12% quoted in literature [3].

The thermal shock resistance of the two clays are 26 cycles for Iboko clay and 28 cycles for Nduage Echara clay. These are within the standard value ranges of 20-30 cycles recommend by chester [1]. The cold crushing strength (compressive strength) values of 23.4 MPa for Iboko and 21.2 MPa for NduageEchara are within the range of 9.8-68.8MPa recommended for fireclay refractory bricks by krivandin and Markor [15], as well as higher than the minimum of 5 MPa recommended by Chester [1].The permeability numbers for the two clay samples as shown in Table 2 are low with Iboko clay having the highest. There is correlation between permeability and porosity, since the more porous a material is, the higher its permeability. Iboko clay being more porous also has higher permeability while Nduage Echara clay has more resistance to molten metal, slag and flue gases penetration. The refractoriness is the ability of a material to withstand high firing temperatures without the deterioration of their physical and mechanical properties. Nduage Echara clay withstood up to 1670^{o}C while Iboko withstood 1650^{o}C. The alumina content of clay determines its refractoriness [4], and the presence of alkali metals in the clay normally lowers its fusion temperature. In terms of chemical composition Nduage Echara clay has higher alumina content than Iboko clay. Thus, the refractories of both clays fall within the standard of 1500-1700^{o}C, which is the recommended range for fireclay refractories [13]. Therefore, these clays can be used for developing medium duty fireclay refractories.

IV. CONCLUSION

The investigation carried out on the two clay deposits, Iboko and NguageEchara revealed that their service properties met the standards reported in many literatures [1,3,15] for refractory and ceramic production. The chemical analysis shows that the clay samples contain silica (SiO2) and alumina (Al_2O_3) as major constituents. These make them suitable as raw material for developing aluminosilicate refractory. Generally they are refractory clays as their acceptable values for linear shrinkage, bulk density, refractoriness, thermal shock resistance and their other mechanical properties show.

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