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CHARACTERIZATION OF SOME NIGERIAN CLAYS AS REFRACTORY MATERIALS FOR FURNACE LINING

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ABSTRACT
The suitability of some Nigerian clays as refractory raw materials was investigated. The clay samples were first analysed to determine their chemical compositions. Fireclay bricks test specimens were prepared by standard method. They were then tested for properties such as apparent porosity, bulk density, thermal shock resistance, fired shrinkage, refractoriness and cold crushing strength. The result obtained showed that both test samples qualify as high melting fireclays. The refractory properties measured revealed them as being usable as refractory bricks when blended.

KEYWORDS: Nigerian clays, Refractory materials.

INTRODUCTION
Refractory materials are inorganic materials which can withstand high temperatures (usually above $1500^{O}C$) under the physical and chemical action of molten metal, slag and gases in the furnace.
Refractory products are required for various processes in chemical, ceramic, petrochemical, oil, foundry and iron and steel industries. Unfortunately there is no refractory industry in Nigeria despite the fact that there are abundant deposits of clay and other raw materials needed for the production of refractory products.

The raw materials for the production of various refractory products include kaolinite ($Al_2O_3.2SiO_2.2H_2O$), chromite ($FeOCr_2O_3$), magnesite ($MgCO_3$) and various types of clays. Other additives such as sawdust and binders are also available locally. Alumino silicate and magnesite refractory products are the major types of refractories used in Nigerian manufacturing industries. Though metallurgical industries are the major consumers of refractory products, other demands come from chemical, glass, boiler and petrochemical industries. The refractory needs of these industries were well over 300,000 tonnes as at the year 2000 (Omowumi 2001).

In the last few years, there have been tremendous works towards developing refractory products from local clay deposits. Various research works have found that our local refractory clays are suitable for use in furnace lining and steel industries.

Agha (1998) showed that some local clay have better refractory and physical properties than imported ones. Nnuka and Agbo (2000) studied the characteristics of Nigerian clays and discovered that the Otukpo clay has refractoriness of $1710^{O}C$, which compares well with imported refractories. Omowumi (2001) also discovered the close relationship of the clays studied with properties of known refractory materials. Another recent research by John (2003) has indicated the suitability of producing refractories for base plates of stoves, bricks for furnace lining and other industrial uses from Nigerian clay deposits.

In this work, various properties of two local clays were investigated to determine their suitability for producing refractory bricks for furnace lining.

EXPERIMENTAL METHODS
The clay samples were collected from deposits in Gur and Yamarkumi in Biu Local Government area of Borno state. The samples were air dried and sieved through 100 meshes. Test pieces for various experiments were mixed with 8 percent water and stirred to form a homogeneous plastic paste.8 percent water was determine as the optimum percentage necessary for optimum plasticity in the work of Nnuka and Agbo(2000).A plastic mass(10cm

Table 1: Chemical composition of the clay samples (%)

Sample location	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	L.O.I
Gur	59.20	21.25	15.70	0.98	1.92	11.05
Yamarkumi	41.80	19.68	8.89	0.98	1.51	14.05
*Refractory clay	46 - 62	25 – 39	0.4 - 2.7	0.2 - 1.0	0.2 - 1.0	8 - 18
*High melting clay	53 - 73	16 – 29	1 - 9	0.5 - 2.6	0.5 - 2.6	4 - 12
*Ceramics	67.50	26.50	0.5 - 1.2	0.18 - 0.3	0.1 - 0.19	-

N.D = Not Detected, * Nnuka and Agbo (2000)

by 5cm) was then moulded for each test piece, dried at temperature of $110^{O}C$ and fired in a muffle furnace at intervals of $100^{O}C$ for every 10 minutes till the temperature of $1110^{O}C$ was attained before determination of different parameters.

Equipments Used

The names of equipments used are as follows mechanical vibratory shaker, standard rammer, hydraulic press, strength tester, permeability meter, muffle furnace.

Chemical Analysis

The chemical analysis of the samples was carried out using atomic absorption spectrophotometry (AAS) method. The percentage composition of the various constituents is recorded in Table1.

Permeability to Air

Fired samples were completely sealed on the sides and the lower surface was exposed to an orifice. The cylinder was filled with $2000cm^3$ of water and a bell jar was put in place. The orifice was opened and the time taken for the $2000cm^3$ of water to displace equal volume of air through the specimen was taken. The pressure difference between the surfaces was measured by a manometer. Permeability was calculated from the equation below.

$$P_A = \frac{Vh}{Apt} \quad \text{-- (1)}$$

Bulk Density

Representative samples of each measuring (6cm ×6cm × 1.5cm) were cut from the fired test samples. The specimens were air dried for 24 hours and then dried at $110^{O}C$, cooled in a desiccators and weighed to the accuracy of 0.008(dried weight) after which the specimens were transferred to a beaker and heated for 30 minutes to assist in releasing the trapped air. The specimens were cooled and soaked weight (W) taken. The specimens were then suspended in water using beaker placed on a balance. The suspended weight (S) was taken. The bulk density was calculated from equation (2).

$$BulkDensity = \frac{D\rho_w}{W - S} \quad \text{--- (2)}$$

Apparent Porosity

The fired pieces were cooled and then transferred into a desiccator and weight to nearest 0.019 (Dried weight).The specimens was then transferred to into a 250ml beaker in an empty vacuum desiccator. Water was then introduced into the beaker until the tested pieces were completely immersed. The specimens were allowed to soak in boiled water for 30 minutes being agitated from time to time to assist to release trapped air bubbles. The specimens were transferred into an empty vacuum desiccator to cool. The soaked weights (W) were recorded. The specimens were

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Table 2; Physical properties of test samples compared with standard clay.

Sample location	Bulk density (g/cm^3)	Apparent porosity (%)	Permeability	Linear shrinkage (%)	Thermal shock resistance (cycles)	Cold crushing strength (KN/M^2)	Refractoriness (^{O}C)
Gur	2.11	19.50	215	1.11	7	15438	1370
Yamarkumi	2.06	22.26	489	1	5	27008	1400
#Fireclay	2.30	20 - 30	25 – 90	4 - 10	20 - 30	15000	1500 - 1700
**Siliceous Fireclay	2.0	23.7	-	-	1	15000	1500 - 16000

#Misra (1975), **Omowumi, 2001

then weighed suspended in water using beaker place on balance. This gave suspended weights (S). The apparent porosity was calculated using equation – (3).

$$ApparentPorosity = \frac{W - D}{W - S} \times 100\% \quad \text{-- (3)}$$

Thermal Shock Resistance

The prepared samples were inserted in a furnace which has been maintained at 900^{O}C. This temperature was maintained for 10 minutes. The specimens were removed with a pair of tongs from the furnace one after the other and then cooled for 10 minutes on firebricks. The specimens were returned to the furnace for further 10 minutes.

The process was continued until the test pieces were cracked. The number of cycles of heating and cooling before cracking for each specimen was recorded as its thermal shock resistance.

Cold Crushing Strength

The test pieces were fired in a furnace at 1100^{O}C and the temperature maintained for 6 hours. The samples were then cooled to room temperature. The specimens were placed each on a compressive tester and the load was applied axially by turning the hand wheel at a uniform rate until failure occurs. The manometer readings were recorded. Cold crushing strength (CCS) was calculated from equation (4).

$$CCS = \frac{MaximumLoad(KN)}{Crosssec tionalArea(m^2)} \quad \text{-- (4)}$$

Linear Shrinkage

The rectangular 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 slab was measured with vernier calliper. The samples were air dried for 24 hours and oven dried at 110^{O}C for another 24 hours. They were then fired for 6 hours. The test pieces were cooled to room temperature and measurements taken. The fired shrinkage linear shrinkage was calculated from equation (5).

$$FiredShrinkage = \frac{D_L - F_L}{D_L} \quad \text{-- (5)}$$

Refractoriness
The test piece was mounted on a refractory plague along with some standard cone whose melting point is slightly below that expected of the test cone. The plague was then put inside the furnace and the temperature was raised at a rate of $100^{O}C$ per minute. The test was continued until the tip of the test cone has bent over level with the base. The plague bearing the test piece was removed and the test cone examined when cooled to room temperature under microscope.

Loss on Ignition
50 gram of each sample was dried at $110^{O}C$ and cooled in the desiccator. A porcelain crucible was cleaned, dried and weighed (M_1) to nearest 0.001gram. The dried sample was introduced into the crucible and the crucible together with the clay sample ions weighed(M_2) to an accuracy of 0.001gram.The crucible containing the sample was placed in a muffle furnace and heated to a temperature of $900^{O}C$ for 3 hours. The crucible and its contents were cooled in a desiccator and then weighed (M_3) to nearest 0.001gram. The loss on ignition was calculated from equation (6).

$$LOI = \frac{M_2 - M_1}{M_2 - M_3} \quad \text{--} (6)$$

RESULTS AND DISCUSSION
Table 1 shows the result of chemical composition test while Table 2 is the physical properties of the test samples.

DISCUSSION
The chemical composition of both the clay samples tested showed that the alumina (Al_2O_3) content for Gur was 21.25% while that for Yamarkumi was 19.68%. Both of them were found to qualify as high melting clays but not as refractory clays. This is because the values of their alumina content lie within the recommended range for high melting clay as shown in Table 1. (Nnuka and Agbo 2000).

The silica (SiO_2) content of Gur 59.20% meets the standard for refractory clay (46-62%) while that for Yamarkumi, 41.80% is short of the standard. Gur clay can be used for lining of heat treatment furnaces, melting furnaces for low melting point metals, liquid metal ladles and portions of blast furnaces.

The iron oxide content of both clay samples is high and such level of oxide usually imparts a reddish colour to clay when fired, so making it attractive as a ceramic raw material. Yamarkumi, with 8.89% iron oxide meets the iron oxide requirement for both refractory clays and high melting clays. The high iron oxide content of Gur (15.70%) strongly supports the reddish colour when fired to $1200^{O}C$ and affects high temperature characteristics such as fired strength. This makes the clay attractive and suitable for structural engineering works (Nnuka and Agbo, 2000).

Loss on ignition (L.O.I)
This is the combustion of volatile matter present in the clay. They are often required to be low. As shown in Table 1, losses on ignition for samples are lower than 18% specified upper limit for refractory clays.

Apparent Porosity
The values for both samples fall within the standard values of 20-30% according to Omowumi as shown in Table 2.

Bulk Density
The average bulk density of the clay samples was within the range of 2.06-2.11 g/cm^3. This makes it suitable for siliceous fireclays as reported by Omowumi (2001); and fireclays as reported by Misra (1975). Bulk density is an important property of a steel work silica brick.

Cold Crushing Strength
The essence of undertaking this test is to determine the ability of bricks to withstand stresses in service. The values obtained for Gur and Yamarkumi clay samples were 15438 and 27008KN/M^2 respectively. These values are higher than the standard which is very satisfactory.

Permeability
The permeability numbers of both clay samples were found to be above the internationally accepted range of 25-90. Permeability is a function of gases or liquids passing through the brick. Refractories under the influence of liquids and gases should be impervious, that would help eliminate leakages of gases and penetration of liquids through the walls of the furnace.

Linear Shrinkage
The average linear shrinkage for both Yamarkumi and Gur are lower than the recommended range of 4-10% for fireclay as reported by Omowumi (2001).This is more desirable. Higher shrinkage values may result in warping and cracking of the brick and this may cause loss of heat in the furnace.

Thermal Shock Resistance
The thermal shock of the two sample are short of the acceptable values of 25-30 cycles as compared in Table 2.The practical implication of this is that their use is restricted to lining of ladles and slag pots which are early mended at shock intervals.

Refractoriness
The highest temperature reached was $1400^{O}C$ for Yamarkumi while Gur had $1370^{O}C$.These are lower than the recommended range for fireclay refractories of $1500\text{-}1700^{O}C$ as reported by Misra (1975). These low values of refractoriness are as a result of the high silica content of the clays. This means that their use is restricted to the processing of materials whose melting points do not exceed $1400^{O}C$ or non ferrous materials.

CONCLUSION
The investigations on the properties of the samples show that their values compare favourably with imported fireclay bricks. The following can also be inferred.

1. Based on the percentage of Al_2O_3 content, both tested clays qualify as high melting clays.
2. The clay samples produced firebricks with apparent porosity values which are lower than the normal 20-30% specified for fireclay bricks.
3. The loss on ignition (L.O.I) values for both bricks are between 11.05 and 14.05% which were less than the upper limit of 18% for fireclays.
4. The refractoriness of Yamarkumi is $1400^{O}C$ and Gur $1370^{O}C$.This implies that they can only be used to melt metals not exceeding these temperatures.
5. Both Yamarkumi and Gur clays has a good cold crushing strength of 27008 KN/m^2 and 15438 KN/m^2 respectively which are higher than the standard of 15000 KN/m^2.

More research work is being undertaken to improve the properties of these clays through the use of suitable additives and/or blending.

NOMENCLATURE AND SYMBOLS
A= cross section area of specimens (cm^2).
CCS =Cold Crushing Strength.
D = Dried weight.
D_L =Dried length.
F_L = Fired length.
h = Height of specimen.
M_1 =Mass of Porcelain crucible.
M_2 =Mass of sample and porcelain crucible.
M_3=Mass of fired clay sample and porcelain crucible.
P = Pressure of air in cm of water.
P_A= permeability number.

S= Suspended weight.
t = Time in minutes.
V=volume of air (cm^3).
W=Soaked weight.
ρ_w= Density of water.

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