



Performance Evaluation of Refractory Bricks produced from locally sourced Clay Materials

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ABSTRACT: The performance evaluation of refractory bricks produced from some local clay deposits in Delta State, Nigeria was investigated. Four major sites in Delta State renowned for abundant clay deposits were selected, namely; Oghara, Ekpan, Ubeji and Jeddo. The clay samples were crushed, milled (pulverised) and sieved to produce very fine grains of less than 250 μm particle size distribution for all samples. They were then tested for shrinkage, bulk density, Loss on ignition, cold compression strength, apparent porosity, and thermal shock resistance. Also, their mineralogical composition and fusion temperatures were examined. The results shows that kaolin deposit at Oghara was found to be the best material suitable for the lining of the walls of most high thermally operated equipment as the expected operational temperatures of most of them are below 1200°C. It had a bulk density, porosity, cold compressive strength, shrinkage, loss of ignition, slag resistance of 1.74g/cm³, 31.44%, 100kg/cm², 5% and 12.18% respectively. Oghara clay was found to be less dense, contains more porosity, and possesses low iron content and higher refractoriness with fusion temperature above 1200°C. Ekpan clay is also suitable for medium thermal application in furnaces, kilns and stoves, while the rest could be benefited to improve on their insulating properties. © JASEM

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Introduction: Refractories are inorganic, non-metallic, porous and heterogeneous materials composed of thermally stable mineral aggregates, a binder phase and additives. They have the property of forming a coherent, sticky mass when mixed with water, being readily mouldable when wet but if dried retains its shape (Gerhard, 1986, Grim, 1968)). They provide linings for high temperature furnaces and other processing units. Refractories must be able to withstand physical wear, high temperatures (above 538°C), and corrosion by chemical agents. (Encyclopaedia Britannica, 2008).

Sayel et al, 2012, studied the possibilities of using locally available raw materials for the production of refractory bricks. The basic raw material was kaolinitic clay from Dewechla deposit; it was possible to produce Chamotte refractory bricks with the initial softening point of 1180°C. Olokode et al, 2012 investigated the suitability of using clay – cow dung to produce insulating firebrick and to determine the optimal ratio of the constituent. Two clay samples were collected from Ilese Awo clay deposit. Preliminary test were carried out to determine the chemical constituents of clay samples and cow dung. The brick samples were fired at a temperature of 1200°C and some of the samples gave

the following limits of results: - linear shrinkage: 6 – 8%, bulk density: 1.97 – 2.35g/cm³, porosity: 22.54 – 30.62%, permeability: 88 – 112%, cold crushing strength: 64 -152.5Kg/cm², thermal shock resistance: 12 – 30 cycle, and thermal conductivity: 0.156 – 0.21W/mK. The results showed that all the brick samples had good insulating characteristics, suggesting its suitability for production of insulating fire- bricks.

Refractory materials play useful and very crucial roles in the industrial development of any nation. Aderibigbe, (1989), reported that virtually all the refractory requirements in all the metallurgical industries in Nigeria are imported. The Nigerian metallurgical industries are struggling today because of many factors which include short supply of refractory materials. Adondua (1988), reported that Ajaokuta Steel Complex requires about 43,503 tonnes per year of fireclay refractories for its operations; and these refractories are sourced abroad.

Despite having extensive clay mineral deposits in Nigeria, Nigeria continues to depend on the importation of refractory materials for many of her industries. Nigeria imported about 27 million metric tonnes of refractory materials in 1987 (Obadinma,

2003). Since Nigeria has some sample reserve of Kaolin and Ball Clay which could be exploited for economic purposes as reported by Aderibigbe, 1989, this work therefore is focused on the performance evaluation of refractory bricks produced from locally sourced clay materials from Oghara, Ekpan, Ubeji and Jeddo communities in Delta State, Nigeria; to serve as good alternative to the imported refractory bricks.

MATERIALS AND METHODS

Materials: The materials for investigation are clay samples from Oghara, Ekpan, Ubeji and Jeddo communities of Delta State, Nigeria respectively; and also a sample of imported refractory castable. The various clay samples were denoted as: (A) for Oghara clay sample, (B) for Ekpan clay sample, (C) for Ubeji Clay sample and (D) for Jeddo clay sample respectively; and (E) for imported refractory castable.

Method: The clay samples were dug from each of the Communities by the use of a shovel and the refractory castable was collected from Warri Refining and Petrochemical Company (WRPC) Warehouse. The samples were sun dried to reduce the moisture content and enhance grinding. 3600g of the sun dried samples were ground to powder with pestle and mortar. 3000g of the samples were taken for the physical property tests, while the remainder was further ground to finer particles and then used for the chemical composition tests. The samples were weighed and dried in an oven at 110°C for one hour to ensure complete evaporation of the moisture.

The samples were then mixed with 8% water and stirred to form homogeneous plastic paste. 8% water was determined as the optimum percentage necessary for optimum plasticity of clay from the work of Nnuka and Agbo (2000). Five (5) cubicle moulds (10.0 x 10.0 x 10.0 cm) of steel plates, were constructed. The plastic paste were then poured into the mould and rammed using a 3kg rammer. A suitable plunger was used to extrude the moulded samples from the oil lubricated mould. Moulded samples and metallic moulds are shown in Fig 1.

The prepared samples were dried in the Memmert oven at a temperature of 110°C to ensure evaporation of moisture. There were further fired in a Roto Lab Carbolite furnace at intervals of 100°C for every 10 minutes till the temperature of 1200°C was reached. The samples were then soaked at 1200°C for 8 hours and allowed to cool in the furnace for 24 hours.

Equipment used : The equipment used in this investigation include: Shovel, Pestle, Mortar, Cubicle Mould (10.0 x 10.0 x 10.0 cm), Rectangular Mould (10.0 x 5.0 x 5.0 cm), Sand Rammer, Sand Mixer, Rotary Sieve Shaker (Chauvin), Roto Lab Carbolite Furnace, Memmert Oven, Electric Balance, Cold Crushing Strength Machine, Disc Die, Hydraulic Press, Perkins Elmer Atomic Absorption Spectrometric (AAS-3110) machine.



Fig. 1: Moulded samples and metallic moulds

Test Procedure: Chemical Analysis : The chemical analysis of the clay samples from Oghara, Ekpan, Ubeji and Jeddo communities and the refractory castable was carried out on the Atomic Absorption Spectrometric (AAS) machine. This technique was used to identify and determine concentrations of elements present in solids, powdered and liquid samples.

The clay and refractory castable samples were ground with mortar and pestle and sieved for 10 minutes with Chauvin sieve shaker in a 120-mesh size to produce 95µm particle size of clay specimen. 5grams of each of the sieved samples was intimately mixed with 20-ml of 0.1N HCl solution (extracting solution) in a 300-ml beaker and placed in a mechanical shaker for 15 minutes. This mixed solution was filtered through Whatman #42 filter paper into a 50-ml volumetric flask and diluted to 50 ml with the extracting solution. The Spectrometer was switched on and allowed to warm up in order to stabilize the optics and the x-ray tube. It was then calibrated to determine the expected elements present in the samples. The samples were run using the prepared programs (calibrations) and the elemental

concentrations present in the samples were calculated and displayed after applying automatic statistics to the results from the AAS spectrometer.

Apparent porosity: Test : The clays and refractory castables were made into bricks in cubicle form (10.0 x 10.0 x 10.0 cm) and oven dried at 110 °C until a constant weight (W_D) is obtained (with an accuracy of 0.1grams). The dried specimen was suspended in 5000ml beaker containing distilled water and boiled for two hours, in this position. It was later allowed to cool down to room temperature and its new weight(s) determined (W_S). The specimen was removed from water, and again reweighed in air to obtain (W_W).

Apparent Porosity (P) and water absorbed (WA) was determined using equation (1) and (2) respectively:

$$P = \frac{W_W - W_D}{W_W - W_S} \times 100 \text{ ---- (1)}$$

$$\% \text{ Water Absorbed (WA)} = \frac{W_W - W_D}{W_D} \text{ ---- (2)}$$

Where W_D = Weight of fired specimen, W_S = Weight of fired specimen in water,

W_W = Weight of soaked specimen suspended in air

Cold crushing strength: Cold crushing strength is the amount of load that the clay/refractory material could withstand after it has been fired to a temperature of 1200°C. In determining the cold crushing strength (CCS) of the clay/refractory samples, cubicle specimens were made from the clay/refractory samples. The dimensions of the test pieces were taken before they were fired to the required temperature and allowed to cool to the room temperature before the tests were carried out. A cardboard sheet not exceeding 0.63 cm in thickness was then placed between the platens of the hydraulic press and the breaking faces of the test piece, which was placed centrally on the platen. Hydraulic load was applied on the test piece until the test piece failed to support the load. The maximum recorded load was taken as the crushing load. The CCS was calculated using equation (3)

$$CCS = \frac{\text{Load}}{\text{Area}} \text{ ---- (3)}$$

Bulk density: Representative samples of each measuring (10.0 x 10.0 x 10.0 cm) were cut from the fired test samples. The specimens were air dried for 24 hours and then dried at 110°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 (4).

$$\text{Bulk Density} = \frac{DP_w}{W-S} \text{ ---- (4)}$$

Where D = Dried weight, P_w = Density of water, W = Soaked weight,

S = Suspended weight

Refractoriness Test: This is the measure of the fusibility of a material and indicates the temperature at which the material softens. The refractoriness of a clay sample is directly related to its softening temperature and is expressed as its Pyrometric Cone Equivalent (PCE). Pyrometric cone equivalent is the number which represents the softening temperature of a refractory specimen of standard dimension (38 mm vertical height and 19mm triangular base) and composition. Refractory clays are classified into different grades in respect of their softening temperatures, typified by the number of the standard pyrometric cone which deform under heat treatment. Test cones were prepared from the refractory clays, having the same dimensions with segar cones (standard cones). Then the test cone is placed in an electric furnace along with segar cones, and heated. The furnace is heated at a standard heating rate of 10°C per minute during which softening of segar cone occur along with the specimen test cone. The temperature at which the apex of the cone touches the base is the softening temperature. The test cones are then compared with the standard cones and the test material is said to have the pyrometric cone equivalent (PCE) of the standard cone that it resembled most in bending behavior. The minimum PCE for low, intermediate, high, and super duty are 19, 29, 31/32 and 33 respectively; which corresponds to the following fusing temperatures: 1500°C, 1650°C, 1690°C and 1750°C respectively. .

Thermal Shock Resistance Test: Test piece of refractory bricks were thoroughly dried and placed in the cold furnace and heated at the rate of 5°C/min until the furnace temperature read 1200°C. This temperature was kept for 30 minutes after which the test pieces were removed with a pair of tong previously warmed in the furnace for a short time. The test pieces were then placed on cold fire bricks in the environment free of draught and allowed to cool down for about ten minutes. The test pieces were returned to the furnace for a further 10 minutes. The

cycle was repeated 30 times before thermal crack could occur.

Slag Attack Resistance Test: The refractory material was made into a brick with a hole drilled into it. The hole is packed with the sample of iron slag (from Delta Steel Company) that it might likely encounter when put to service. The refractory brick was then heated to a standard temperature of about 1300°C and maintained at this temperature for a period of one hour. The brick was then cooled, sectioned and examined to observe the degree of attack and penetration of the slag.

The loss on ignition: The loss on ignition (LOI) is the weight reduction on the total weight of the prepared clay samples, in percentage. Hence, the loss in weight by each clay sample was determined to be the difference in their weights before and after firing and consequently, the loss on ignition at that temperature is determined as shown in equation (5).

$$LOI = \frac{W_1 - W_2}{W_1} \times 100 \text{ -----(5)}$$

Where: W_1 = Initial Weight of clay sample before firing, W_2 = Final Weight of clay sample after firing

Shrinkage Test: The test pieces of the refractory clay materials were made into rectangular shapes of dimension 10.0 x 5.0 x 5.0 cm in a mould and compacted under a hydraulic pressure of 350kN/m². A slanted line of length 10cm was inserted diagonally on each piece and recorded as (L_1). The test pieces were then placed inside the furnace and fired up to 1000°C and the line drawn across the diagonal axis of the pieces was measured to determine its final length (L_2) after firing. The linear shrinkage of the materials was determined with equation (6).

$$\text{Linear Shrinkage (\%)} = \frac{L_2 - L_1}{L_1} \times 100 \text{ ---- (6)}$$

RESULTS AND DISCUSSION

The particle size distribution for the clay samples was determined with the aid of sieve analysis and was found to be less than 250µm for all samples. The results of the chemical, physical analysis and Refractoriness of Refractory samples of the samples are presented in Tables 1, 2 and 3 respectively.

Table 1: Chemical composition (wt%) of selected Clays/Refractory samples

Samples	Al ₂ O ₃ (%)	SiO ₂ (%)	CaO (%)	MgO (%)	Fe ₂ O ₃ (%)	K ₂ O (%)	ZnO (%)	MnO (%)	Na ₂ O (%)	Cr ₂ O ₃ (%)
Oghara	30.46	50.92	0.19	0.50	2.07	0.33	0.10	0.01	0.70	0.02
Ekpan	17.75	53.90	0.72	0.60	11.80	3.30	0.90	0.03	0.80	0.04
Ubeji	12.48	40.68	1.12	0.60	25.55	2.88	0.40	0.15	1.40	0.06
Jeddo	10.92	56.90	1.90	0.62	11.40	3.25	0.10	0.19	0.60	0.04
Imported	36.96	48.90	3.34	0.70	2.24	4.0	0.30	0.21	2.00	0.03

Table 2: Physical Properties of selected Clays/Refractory samples

Sample	Bulk Density (g/cm ³)	Porosity (%)	C.C.S. (Kg/cm ²)	Shrinkage (%)	LOI (%)	Slag Resistance
Oghara	1.74	31.44	100	5.00	12.18	Good
Ekpan	2.00	20.69	140	2.00	7.85	Good
Ubeji	2.00	19.10	227	1.50	10.78	Poor
Jeddo	1.99	17.31	83	1.90	7.98	Poor
Imported	2.60	35.21	325	7.74	5.53	Very Good

Table 3: Refractoriness of selected Clays/Refractory samples

Sample No	Sample Name	Refractoriness			Pyrometric Cone E equivalent (PCE)
		PCE No	Range/ Limit	Temperature	
A	Oghara	Seger cone 29	>	1500°C	High PCE
B	Ekpan	Seger cone 16	<	1500°C	Intermediate PCE
C	Ubeji	Seger cone 10	<	1200°C	Low duty PCE
D	Jeddo	Seger cone 10	<	1200°C	Low duty PCE
E	Imported	Seger cone 32	>	1600°C	High duty PCE

Chemical Analysis: It is observed that Oghara clay sample as reported in Table 1 is more kaolinitic than all other clay samples obtained from the four different sites in Delta State with 30.46% of alumina to 50.92% of silica which could apparently be taken as ratio 1:2 of alumina to silica. This agrees with the standard as reported by Sanni (2005). The clay samples from Ekpan, Ubeji and Jeddo locations contain higher impurities of mineral oxides and Iron (III) Oxide which makes them more plastic (Ball Clay) than Oghara clay. Consequently, Table 1 shows Oghara clay contains the least amount of Iron (III) Oxide (Fe_2O_3), which is 2.07% wt/wt as against Ubeji clay sample with 25.55%; Ekpan and Jeddo with 11.8% and 11.40% of Fe_2O_3 respectively; while the imported refractory castable has 2.24% of Fe_2O_3 (Table 1). Nevertheless, the proportion of elemental composition of iron in clays determines the thermal conductivity potential of such materials; hence Sanni (2005) suggested that any fire clay to be used in refractories should have at least 30% Al_2O_3 and less than 1.8% Fe_2O_3 . The proportional increase in the ratio of Al_2O_3 in samples will undoubtedly improve clay refractoriness, whereas a progressive reduction in Fe_2O_3 content of samples will perhaps lower their thermal conductivity in that order. This probably suggests why Oghara clay would be regarded as having better insulating property than the samples from other locations. By implication therefore, thermal conductivity will be highest in Ubeji, moderate in both Ekpan and Jeddo clay, but least in Oghara clay, making Oghara clay the material of choice. Other associated impurities in the various clay samples include K_2O , CaO , ZnO , MnO , MgO , Na_2O and Cr_2O_3 which are present in various proportions by weight as shown in Table 1, but the impurities are least in Oghara clay.

*The Loss on Ignition :*The Loss on Ignition of Refractory clays (LOI) was determined as the percentage of moisture loss to ignition on firing the prepared clay samples. This is highest in Oghara clay with 12.18% and moderate in Ubeji clay with 10.78%, but least in Ekpan and Jeddo clay samples with 7.85% and 7.98% respectively (Table 1). This represents the amount of moisture the clay materials could hold or percentage weight reduction of samples which may probably be a reflection of their grain structure and fineness. This suggests that Oghara clay is of finer grains than others and more compact than them.

*The bulk density :*The bulk density of Oghara clay which was 1.7 g/cm^3 is least among the four samples studied. While that of Ekpan and Ubeji are both 2 g/cm^3 and that of Jeddo is 1.99 g/cm^3 (Table 2). The bulk density of clay (kaolin) commonly plays an important role in its economic value when fired (as a refractory, filler, coater, absorbent, etc.). High-density is commonly desired or demanded for clay refractories because high fired-density usually confers high physical strength at high service temperatures and high resistance to service corrosion, slag penetration, and abrasion. The high-density clays in the natural, lump state which also are high in P.C.E. (Pyrometric Cone Equivalence a measurement of fusion) may typically yield refractory material in the high density range, and may necessarily do so (Bauman and Keller, 1975). This property is very vital in the handling and transportation of the refractory materials.

The densities of the materials were considered in this work as a function of the major constituents of the clay samples. The principal elemental contributor to the overall density of the clay materials remain Aluminum Oxides (Al_2O_3), Silicon Oxide (SiO_2) and Iron(III) Oxide (Fe_2O_3), all other impurities being negligible.

Porosity: The value of the porosity of the clay samples in percentages was also determined as shown in Table 2. Though the imported refractory castable has the highest porosity of 35.21%, Oghara clay has the highest porosity of 31.44% against 20.69% for Ekpan, 19.10% for Ubeji and 17.31% for Jeddo clays respectively. The porosity of refractory clay material is directly related to the air pockets contained in it, hence, the higher the porosity of the clay material, the higher its insulating properties ((Hassan,et al., 1993). High porosity in refractory materials translates to increased air pockets and improved thermal insulation particularly in a pyrolysis plant where linings are not exposed to fumes and vapour. This suggests that, Oghara clay has better capability to minimize thermal losses in the furnace of a pyrolysis plant than other selected samples. In other words Oghara clay which has the fineness grain has a better insulating property than other samples, except the imported refractory castable.

Cold compressive strength: The cold compressive strength (C.C.S.) of the clay samples were determined as reported in Table 2. In all the imported

refractory clay has the highest compressive strength of 325kg/cm^2 (as should be expected), however, Ubeji clay specimen has the highest compressive strength of 227kg/cm^2 and a better advantage over others with respect to rigidity and load bearing capability. Oghara clay also has a load bearing strength of 100kg/cm^2 , while Ekpan and Jeddo clays have the compressive strength of 140kg/cm^2 and 83kg/cm^2 respectively.

Shrinkage: The shrinkage of the clay samples were considered as indicated in Table 2. Shrinkage is highest in Oghara clay (compared with other clay samples) with 5% reduction in lateral size, while Ekpan and Jeddo shrunk by 2% and 1.9% respectively. Consequently, Ubeji clay has the least shrinkage of 1.5%; while the imported refractory castable has the highest overall shrinkage of 7.74%. This indicates that the moisture content of Oghara clay sample is higher than others, leading to Oghara clay having finer grains than others.

Slag resistance : The slag resistance of Oghara clay and Ekpan clay samples are good, but that of Ubeji and Jeddo clays are poor; and that of the imported refractory clay very poor as revealed in Table 2. Good slag resistance of Oghara and Ekpan clay samples suggest that they could resist the penetration of corrosive vapours and fumes without deformation if used as furnace lining. Ubeji and Jeddo clay samples have bad slag resistance properties and therefore expected to fail in service if employed as furnace lining materials.

Refractoriness of the clay: The refractoriness of the clay samples was determined as shown in Table 3. Oghara clay sample has the highest Pyrometric Cone Equivalent (PCE) of seger cone 29, as it can withstand the deformation temperature of about 1600°C before fusing or bend under its own weight. Ekpan clay has an intermediate Pyrometric Cone Equivalent (PCE) of seger cone 16 as it only withstand temperatures below 1500°C . Ubeji and Jeddo clays have low PCE with fusion temperature under 1200°C . However, the imported refractory castable has the highest overall PCE (as supposed) of seger cone 32, as it can withstand the deformation temperature of about 1800°C . Hence, Oghara clay has the best refractory property among the four samples investigated. This is due to the fact that it can withstand higher temperature than others because of its higher fusion temperature. The thermal conductivity of refractory clays depends on the chemical properties, mineralogical composition,

silica content of the refractory and on the application temperature (UNEP, 2006). Oghara could therefore be used for the lining of furnaces that could operate at temperature well above 1500°C without fear of thermal deformation of the furnace wall.

The test pieces were subjected to several thermal shock resistant cycles of heating and cooling and all the test pieces were able to survive about 10 cycles without any crack. This indicates that the clay samples could withstand abrupt changes in temperature.

Conclusion : The performance evaluation of refractory bricks produced from locally sourced clay materials from Oghara, Ekpan, Ubeji and Jeddo communities in Delta State, Nigeria have been studied. The results showed that these local clays investigated are suitable to produce locally available refractory bricks which was found to compare favourably with the imported refractory bricks. Refractory material produced from Oghara clay was found to be the one with the best properties of all the clay investigated from Delta State, Nigeria. It had a bulk density, porosity, cold compressive strength, shrinkage, loss of ignition, slag resistance of 1.74g/cm^3 , 31.44%, 100kg/cm^2 , 5% and 12.18% respectively. Its properties were found to be comparable with the imported castable refractory brick with bulk density, porosity, cold compressive strength, shrinkage, loss of ignition, slag resistance of 2.60g/cm^3 , 35.21%, 325kg/cm^2 , 7.74% and 5-53% respectively. We therefore conclude that the local clay deposit at Oghara is considered suitable for exploitation and would be used for the wall linings of our high temperature equipment in Warri Refining and Petrochemical Company (WRPC) and other local industries in Nigeria, where such equipment are used for operations.

Acknowledgment: The clay and refractory samples were subjected to chemical and thermal tests at the Warri Refining and Petrochemical Company (WRPC) Instruments and Carbon Black (CB) Plant Laboratories, while the physical tests were done at Nigercat Laboratory, Ekpan; to determine their suitability in lining furnaces.

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