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Characterization of Amaiyi Edda Clay Deposit as Refractory Material for Furnace Lining

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Abstract

The performance evaluation of the refractory properties of Amaiyi Edda clay in Afikpo South local Government Area (Edda) of Ebonyi State Nigeria was investigated. The clay sample collected from the deposit was crushed, soaked, sieved, dried and ground. The chemical and mineralogical analyses were done using X-ray fluoresce and X-ray diffraction while the micro structural examination was done with scanning electron microscope. The processed sample was molded, dried and fired at 1200^0C. The refractory properties were tested based on ASTM standards. Results obtained show that Amaiyi clay has 22.9% alumina and 48.9% silica, hence qualify for high melting clay. The clay has linear shrinkage of 4%, bulk density of 2.15g/cm^3, apparent porosity of 11.56%, thermal shock resistance of 29 cycles, modulus of rupture of 33.5 N/mm^2, thermal conductivity of 3.97W/m^{0}C and refractoriness of 1520^0C. Hence, the results showed that the clay can be used for lining of heat treatment and melting furnace for non ferrous metal, ceramic kiln and oven ladle.

Keywords: Characterization, refractory clay, furnace lining, alumina, silica.

1.1 Introduction

Refractories are materials which can withstand high temperature (usually above 1580^0C) under the physical and chemical action of molten metal and hot gas in the furnace. (Nnuka and Agbo, 2000). The ability of a refractory to withstand high temperature in service is known as refractoriness and its degree in any particular refractory depend on the amount of alumina (Al_2O_3). (Agbo, Idenyi and Mbah; 2015). It is those properties which enable the refractory clay to withstand high temperature and resist physical and chemical reactions that determine the suitability of such clay for use as furnace lining. (Nuhu and Abdullahi, 2008).

The chemical composition of clay mineral affects the properties of refractory product. Clay refractories are mainly produced from clay that has alumina and silica contents between 18-44% and 50-70% respectively which are the principal constituents. (Odo et al, 2010). Aliyu et al,2014 noted that the percentage of the mineral oxides like Fe_2O_3, MgO, CaO, etc in the clay determine the area of application like bricks floor tile and paper while alkali metal oxides like Na_2O, K_2O, etc determine its suitability for making ceramic product. Aremu and Ibrahim, (2013) reported that the iron oxide (Fe_2O_3) content in a refractory brick should be within the range of (0.5 – 2.4)%, else it will affect the strength and such high level of iron oxide,(Fe_2O_3) content makes the brick reddish in colour.

Refractories find application mostly in the metallurgical, chemical, cement, ceramics, glass and petrochemical industries. Nigeria has many industries which use refractory materials in the production process and also many clay deposits which are raw material for refractory products. In spite of the deposits, the country keeps importing refractory materials at high cost to the detriment of the national economy. Mark U, (2010) and Aderibigbe, (1989).

Therefore, encouraging production of refractory products with our local raw materials does not only improve our economy but also enhances the development of our industrial sector and provides employment for our teeming youths. Hence, the objective of this study is to characterize and evaluate the performance of refractory properties of Amaiyi Edda- Nigerian local clay and determine its suitability for use as furnace lining.

It is in the light of the above mentioned problems that many research studies have been directed toward developing refractory products from local clay. Studies have found clays in some parts of Nigeria useful for furnace construction as a result of their high thermal shock resistance, crushing strength, bulk density and refractoriness values. (Simeon and Oluwaseun, 2014) and (Abolarin, Olugboji and Ugwoke, 2005). Nnuka and Agbo, (2000) studied the characteristics of Nigerian clay and showed that Otukpo clay has refractoriness of 1710^0C, which compares well with imported refractories. The performance of refractory bricks produced from locally sourced clay materials from Ogbara, Ekpan, Ubeji and Jeddo communities in Delta state, Nigeria was evaluated and was found to compare favourably with imported refractory bricks. (Osarenmwinda, Abel and Chukwuemeka, 2014). The refractory characteristics of Kwi clay deposit in Plateau State, Nigeria was examined by Nnuka and Adekwu, (1997) and it was found that the clay could be utilized for ferrous and non ferrous refractory application based on the properties. Nnuka and Apeh, (1991) found that Ukpo clay is a fire clay with adequate strength that could be ultilized for cupola furnace lining. Yami, (2007) observed that Yamar clay and Gur clay have refractoriness of 1400^0C and 1370^0C respectively which are lower than the recommended range of fire clay refractoriness of 1500^0C - 1700^0C. Aliyu et al, (2012) studied and compared the refractory properties of selected clays in North Central Nigeria and observed that Maikunkele clay had an impressive refractoriness value of 1710^0C which makes it excellent for ferrous metal foundry. According to Nnuka, Utin and Adegoye, (1998), high refractoriness value is due to the relatively high quantity of alumina which controls refractoriness. Other properties like shrinkage, porosity and bulk density are interrelated. The more a material shrink, the less porous it becomes and the density rises. (Nnuka and Okunoye,1991) and (Nnuka, Ogo and Elechukwu, 1992).

2.0 Material and methods

2.1Materials

The clay material used in this research was obtained from Amaiyi Edda in Afikpo South local Government Area of Ebonyi State, Nigeria. The equipment used include Empyrean X-ray diffractometer made by panalytical of Netherland, minipal 4 ED- X-ray fluorescence made by panalytical of Netherland, scanning electron microscope (SEM) Zeiss, model EV010, electric furnace, (Thermodyne 46200), ceramic kiln, model 88FC2468, Electrical transversal strength machine, model 235 (salter), digital weighing machine, spiral weighing balance wooden mortar and pestles, moulds, pair of tongs, heat conduction testing machine, model H9406/02877PA Hilton, pyrometric cone, meter rule and venire caliper.

2.2 Methods

2.2.1 Oxides and mineralogical phase analysis

The chemical composition of the clay was determined using x-ray fluorescence while the x-ray diffraction technique was used to determine the mineralogical constitution of the clay.

2.2.2 Processing of sample

The samples were ground and soaked for two days to dissolve and remove soluble oxides that could affect mullite development. The beneficiated clay was sieved, dried and ground to fine particles before moulding the different test samples. The samples were further dried at 110^0C in the kiln and thereafter fired at 1200^0C before testing for the refractory properties.

2.2.3 Determination of the refractory properties of the materials

The refractory properties tested in this research study were grouped into three categories:

Physical and service properties

Mechanical properties

Thermal properties

The physical and service properties included apparent porosity, bulk density, and refractoriness. The mechanical property that was investigated was the modulus of rupture

(MOR) while the thermal properties included linear dry-fired shrinkage, thermal shock (spalling) resistance and thermal conductivity.

Determination of linear shrinkage

This test was done to determine dimensional stability of the sample after a given period of time and temperature change. The method of test used is in line with ASTM C-326 standard for measuring linear shrinkage. Measured 50 mm points were marked on the surface of the test green sample. The green test sample which was given a 50 mm mark (L_o) on the surface was dried in the kiln to a temperature of 110^0C and thereafter the sample was brought out. The same previous 50 mm mark was measured to get the new length of the points after drying (L_d). The same specified length on the test sample was used when the sample was fired and the new dimension of the length was taken as fired length L_f. The dry-fired shrinkage was calculated as the linear shrinkage represented as:

$$\left(\frac{L_d - L_f}{L_d}\right) \text{x } 100 \qquad (1)$$

Determination of bulk density and apparent porosity

This test was done according to ASTM C 20-80a standard test for apparent porosity, water absorption and bulk density. The long rectangular shaped test sample measuring 9.5cm length, 2cm width and 5cm height was used for these two experiments. The specimens were fired to the required temperatures in preparation for the test. The dry weight (W_a) in air was taken using digital weighing balance. They were transferred into a vessel of boiling water for 30 minutes after which the boiling was discontinued. The specimens were allowed to cool to room temperature in the vessel of water for four hours. After 30 minutes in cold water, the specimens were tied to a string on a spiral balance suspended in a beaker of water, to get the suspended weight (W_{sp}). The specimens were removed from the water and gently cleaned before weighing it again to get the soaked weight (W_{so}).

From the above data, the physical parameters stated above were calculated:

$$\text{Bulk density} = \left(\frac{\text{Weight in Air }(\text{W}_\text{a})}{\text{Soaked Weight}(\text{W}_\text{so}) - \text{Suspended Weight }(\text{W}_\text{sp})}\right) \qquad (2)$$

$$\text{Apparent porosity} = \left(\frac{\text{Soaked Weight}(\text{W}_\text{so}) - \text{Weight in Air }(\text{W}_\text{a})}{\text{Soaked Weight}(\text{W}_\text{so}) - \text{Suspended Weight }(\text{W}_\text{sp})}\right) \text{x } 100 \qquad (3)$$

Determination of refractoriness

The refractoriness or softening point was determined using the method of pyrometric cone equivalence (PCE) in accordance with ASTM C24-79. The test pieces were mounted on the refractory plaque along with some standard cones whose softening points are slightly above or below those expected of the test cones. The plaque was then inserted into the electric furnace. The temperature was raised at the rate of 5^0C per minute during which softening of Orton cone occurred along with the specimen test cone. The temperature was further raised up to 1400^0C and the samples with-stood the temperature, hence were soaked in the furnace at that same temperature for a period of 4 hours until the tips of the test cones had bent over the level with the base. Then the plaque bearing the specimens was removed from the furnace and the test cones examined when cold. The test cones were then compared with the standard cones and the test materials were said to have the pyrometric cone equivalent (PCE) of the standard cone that it resembled most in bending behaviour.

The refractoriness of each test cone is the number of the standard pyrometric cone that has bent over to a similar extent as the test cone. The temperature corresponding to the cone number was read off from the ASTM Orton series. The material was able to withstand the temperature of 1400^0C for 4 hours soaking time before it yielded.

The soaked time in minutes was converted to equivalent temperature value by dividing with a factor of two.

Determination of thermal shock (spalling) resistance

This test was carried out with the help of an electrical furnace (Thermodyne 46200) heated at the rate of 5^0C/min in line with ASTM C-484 standard for measuring spalling resistance of refractory brick. The test pieces of the refractory bricks were thoroughly dried and placed in the cold furnace and heated at the rate of 5^0C/min until the furnace temperature got to 1200^0C. The samples were then removed one after the other using a pair of tongs and cooled in air for 10 minutes, and then observed for cracks. In the absence of cracks (or fracture), the bricks were put back into the furnace and reheated for a further period of 10 minutes and then cooled for another 10 minutes. This process or cycle of heating, cooling and observing for cracks was repeated until cracks were observed. The number of complete cycles that produced visible cracks in each specimen was noted. This constituted the thermal shock (spalling) resistance.

Determination of thermal conductivity

The thermal conductivity was determined under steady state condition at room temperature. The test was conducted using heat conduction equipment. Circular test specimen measuring 40 mm diameter and 4mm thickness were used for the test. The specimens were inserted and clamped in between the heater and cooler faces of the equipment.

5Watts power input was selected and maintained for 30 minutes until steady state conditions were achieved. The temperature T at all six sensor points (three on the heater section and three on the cooler section) were recorded. The thermal conductivity was calculated using Fourier's law as:

$$K = \frac{Q\ dx}{A.dT} \quad (4)$$

Where Q is quantity of heat supplied, X is the specimen thickness; A is the cross sectional area of the specimen sample and dT is the temperature difference between the two circular faces.

Determination of mechanical property

The mechanical property tested in this work is the modulus of rupture (MOR) using 3 point bend tester. The long rectangular test pieces were dried at 110^0C until a constant weight was obtained. They were fired to their firing temperature of 1200^0C in the kiln.

This test is utilized for two reasons namely;

To determine the correctness of the production process in relation to having obtained the desired degree of compactness and consistency.

To determine whether or not the brick will be capable of supporting the load that will be imposed on it.

The standard used for this test is the ASTM C- 648 standard method for measuring modulus of rupture. The electrical transversal strength machine was used to determine the breaking load, P (kg). A venier caliper was used to determine the distance between supports L (cm) of the transversal machine. The height H (cm) and the width B (cm) of the broken pieces were determined. The modulus of rupture was then calculated as:

Modulus of rupture $Kg/cm^2 = \frac{3PL}{2BH^2}$ (5)

Where P = Load applied when the specimen failed

L = Distance between the centre line of the lower bearing edges of the equipment.

B = the width of the broken specimen

H = Height of specimen (cm).

3.0 Results and Discussions

The results obtained in this study are presented in Tables 1and 2 and in Plates 1 and 2.

3.1 Chemical Composition

The chemical composition of the clay used in this study showed that the alumina content was 22.9% while the silica content was 48.9%. The alumina content qualified it as high melting clay (16-29) % while the silica content met the standard for refractory clay (46-62) % according to Nnuka and Agbo, (2000). The iron oxide Fe_2O_3 content was 10.51% which is higher than the recommended standard for both refractory and high melting clay as reported by Nnuka and Agbo, (2000), Omowomi, (2007) and Aremu and Ibrahim, (2013). This suggests that the high iron oxide content could be reduced since such high content can affect the clay strength. (Aremu and Ibrahim, 2013). It was also observed that such high value of iron oxide in the result collaborated the reddish colour of the fired bricks obtained. The percentage composition of calcium oxide was 2.79. This oxide, a low temperature fussing agent must therefore have reduced the sintering temperature. Potassium oxide has composition of 5.15% which was high and so, assisted the effect of calcium oxide. In view of the negative consequence on the refractoriness and other related properties of both oxides, there is need for beneficiation of the clay during processing.

Oxide	Al_2O_3	SiO_3	K_2O	CaO	TiO_2	V_2O_5	Cr_2O_3	Fe_2O_3
Composition	22.9	48.9	5.15	2.79	1.63	0.098	0.037	10.51
Oxide/Element	CuO	ZnO	Ga_2O_3	MoO_3	Ag_2O	Eu_2O_3	Au	HgO
Composition	0.019	0.02	0.018	3.9	1.98	0.14	0.072	0.12

3.2 Mineralogical analysis

The mineralogical phase analyses done with the aid of XRD and results interpreted using International Centre for Diffraction Data (ICDD) software revealed the main mineralogical phases in Amaiyi clay to be kaolinte($Al_2Si_2O_5(OH)_4$), xonotlite($Ca_6Si_6O_{17}(OH)_2$), chrysotile($Mg_3[Si_2\text{-}xO_5](OH)_{4\text{-}4x}$), quartz($SiO_2$), anhydrite ($CaSO_4$), os ($KMg_2Al_3(Si_{10}Al_2)O_{30}$) and clinochryso ($Mg_3Si_2O_5(OH)_4$).

3.3 Refractory properties of the clay material

3.3.1 Linear shrinkage

The linear shrinkage of 4% fell within the lower range of the recommended value of (4-10%) for fire clay as reported by Omowumi, (2001). This indicates that the stability of the clay material is good at high temperature. This could be traceable to the fine particle size of the clay material and the chemical composition which are the controlling factors for thermal expansion. The particle size of the clay used for this study was 0.18mm size which was very fine, hence had direct effect on the linear shrinkage value.

When compared with Gwarmi and Mubi clay as reported by Nnuka and Agbo, (2000), Omowomi,(2001) and Aremu and Ibrahim, (2013), it is found Amaiyi clay has excellent material stability that qualifies it for high temperature application.

3.3.2 Bulk density and apparent porosity

The value of the bulk density was 2.15g/cm^3 while that of the apparent porosity was 11.56%. These values are within the internationally accepted standard values for the properties investigated as reported by Grimshaw,(1977), Aremu and Ibrahim, (2013) and Agbo, Idenyi and Mbah, (2015). The low value of apparent porosity and linear shrinkage observed yielded high density value. This is in agreement with the result of Nnuka and

Okunoye, (1999) which stated that shrinkage, porosity and density are interrelated. The more a material shrinks, the less porous it becomes and the density rises.

3.3.3 Thermal shock resistance

The value of the thermal shock resistance was 29 cycles. This value is within the recommended range of 25-30cycles. The result showed that Amaiyi clay is of good tolerance to thermal fluctuations and as such qualifies for use in supporting high temperature structures. This tolerance could be traced to the low thermal coefficient of expansion due to fine particle size with strong inter particle bonding which yielded high thermal stability and resistance to sudden change in temperature. These qualities qualify it for use as refractory lining for furnace.

3.3.4 Modulus of rupture

The value of modulus of rupture for the clay was $33.5N/mm^2$. This is a good indication of the ability of the material to support the load that will be imposed on it. The result also show that the clay material will not fracture easily during use due to the degree of compactness obtained during processing.

3.3.5 Refractoriness

The refractoriness of Amaiyi clay was 1520^0c. The value is within the range of 1500^0c-1700^0c and 1500^0c-1600^0c recommended for fire clay and siliceous fire clay as reported by Aremu and Ibrahim, (2013) and Grimshaw,(1977). The moderate value of refractoriness is due to average value of alumina content. This oxide enhances the refractoriness and strength development in clay material. Hence, it indicates that Amaiyi clay could be used for furnace lining for nonferrous metal.

3.3.6 Thermal conductivity

The thermal conductivity value of the material was $3.97W/m^0c$. This high value correlates with high value of bulk density and low value of apparent porosity indicated in the SEM results shown in plate 1 and 2. Hence, the clay is considered as dense refractory material.

Table 2 Refractory properties of Amaiyi clay and internationally accepted standard.

Sample	Linear Shrinkage (%)	Apparent Porosity (%)	Bulk Density g/cm^3	Modulus of rupture N/mm^2	Thermal shock resistance (cycles)	Refractoriness (^{0}c)	Thermal conductivity W/m^{0}c
Amaiyi	4	11.56	2.15	33.5	29	1520	3.97
Fire clay	4-10	2-30	1.17-2.1	-	25-30	1500-1700	-

Source: Grimshaw R.W, (1971)

3.5 Microstructural Examination.

The micro structural images shown by SEM in plates 1 and 2 indicated a homogeneous uniform structure in the clay with limited small pores shown by dark spots in the structures. The small pore sizes in the structure gave credence to low value of apparent porosity of 11.56% observed in the result. The image also revealed the presence of mullite shown by whitish needle like shapes. This phase is responsible for good value of refractoriness and other related properties.

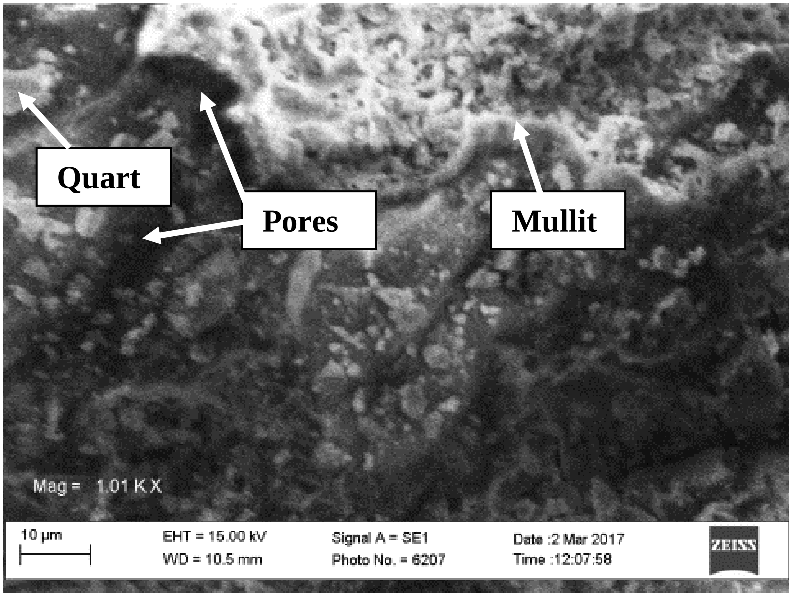

Plate 1 SEM of Amaiyi clay

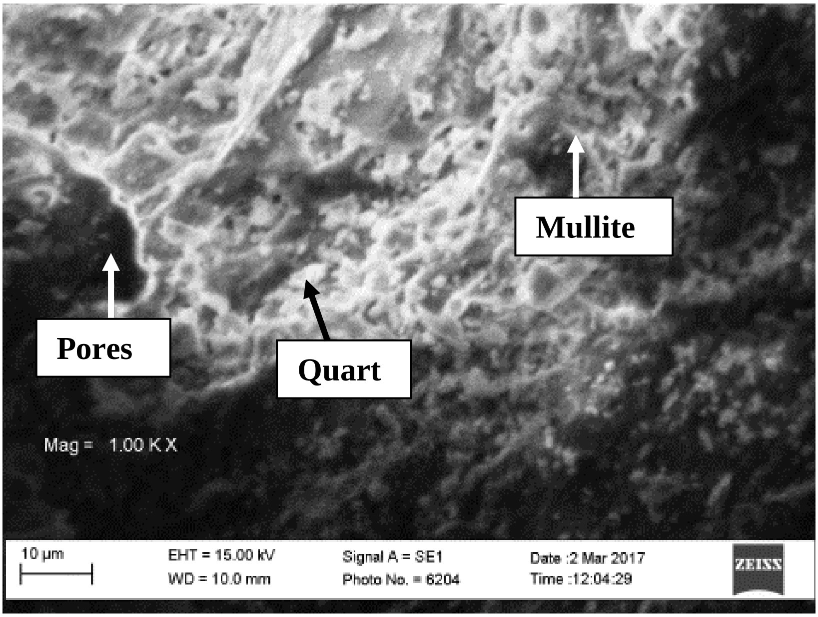

Plate 2 SEM of Amaiyi clay

4.0. Conclusion

Based on the results obtained in this study, the following conclusions were made:

-The clay material understudied belongs to high melting clay considering the alumina content.

- The clay has good stability for high temperature application.

-The apparent porosity of the clay material was moderate but not high compared to 2-30% values specified for fireclay and this led to high value of thermal conductivity. Hence the material could be appropriate for dense refractory and not for insulating refractory.

-The clay material has good inter particle bonding capable of withstanding high temperature fluctuations.

-The refractoriness value which is 1520^{0}C limits its use for lining in non ferrous metals.

-The clay material compares well with internationally accepted standard and as such should be exploited for lining of furnaces.

5.0 Recommendation

The characterized clay materials should be exploited as raw material for refractory production for melting of non ferrous metal.

Mineralogical survey should be done on the deposit to investigate the extent of the clay deposit in order to determine the refractory producing industry that will be sited in the area.

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