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CHARACTERIZATION OF UMULUMGBE CLAY DEPOSITS FOR REFRACTORY APPLICATIONS

Agbo A. O[1]., Idogwu S.[2] and Diyoke C.[2]

1. Department of Metallurgical and Materials Engineering, Enugu State University of Science and Technology
2. Department of Mechanical and Production, Engineering, Enugu State University of Science and Technology

Abstract

Umulumgbe clay deposit has been characterized with a view of finding its usefulness in furnace refractory production and other industrial needs. The chemical analysis was conducted using X-ray Flourescence (XRF) techniques. The result of the chemical analysis showed that the clay has SiO_2 (51.0%) and Al_2O_3 (26.0%) as its predominant oxides with La_2O_3 (0.011%) and MoO_3 (0.04%) as its minor oxides. The physical properties test conducted at firing temperatures of 900^0C, 1000^0C, 1100^0C and 1200^0C respectively showed that porosity has (18.38%), shrinkage has (7.20%), bulk density has (1.84g/cm^3), apparent density has (2.16g/cm^3), and refractoriness has (1654^0C) at firing temperatures of 1200^0C. Most of the properties tested were compared favourably with the standard values for fireclay refractories. The clay sample can be effectively used in the production of ceramics products and refractory bricks for lining of furnances for ferrous and non-ferrous metals.

Keywords: Characterization, Umulumgbeclay, Deposits, Refractory applications

1.0 Introduction

Refractory materials are inorganic non-metallic substances, mainly mixtures of oxides, which can withstand very high-temperatures, the most common are those of aluminium (Al_2O_3) and silicon (SiO_2), high alumina and silicon (SiO_2) are the chief constituents of all aluminosilicate minerals, particularly the clays[1]. The potential use of aluminosilicate refractory raw material depends on the amount of alumina clay materials present. One of the clay minerals, the kaolinites has the highest content of alumina (up to 39.50% by weight), as revealed by the chemical formular $Al_2O_3.2SiO_2.2H_2O$ [1].
The development of heavy industries such as iron and steel, non-ferrous industry cement, ceramic industries have been impossible without development of refractories [2].
Fire clay refractories are the most important types from the view point of turn over they are used in: Boiler furnaces, blast furnaces for melting, reheating and heat treatment of iron, steel and non ferrous metals [3]. Various researches have been carried out on Nigeria clay in order to determine their potentials for ceramic, industrial and refractory applications. Omoumi (2001) reported in his investigation and concluded that the properties of refractory samples from Onibode, Ara-Ekiti, Ibamajo and Ijoko clays, complete excellently with the imported fire clay refractories[1].Nnukaand Ejiofor (2010) studied the characteristics of Nigerian clays and discovered that the Otukpo clay can withstand a furnace temperature of about $1710^)$C which compared favourably with imported refractories[4]. Amuda (2005), in an earlier research on the characterization and evaluation of refractory properties of some clay deposits in Southwest Nigeria, reported that the clays displayed reasonable refractory properties that compared favourably with standard values and recommended a blend of these clays for furnace lining[5]. Ovat F. A. and Bisong M.K.A. (2017) studied the industrial potential of some Nigerian Kaolinitic clay

deposits, the results of the research showed that both Idere and Ito clay samples could be used for bricks making, floor tiles and stoneware production[6].

1.1 Types of Refractories

1. Firebricks:– modular burned refractory shapes, laid in an overlapping half bond with each other and usually but not always, bonding with thin refractory mortar. They are also referred to as fireclay bricks. Most firebricks are made from blends of two or more clays; some bricks, especially those of the low-duty class are made of a single clay. The mixes for superduty and high-duty brick commonly contain raw flint and bond clays, with or without calcined clay.
2. Refractory castables:– refractory concretes, poured in place. They belong to a larger group of monolithic refractories. These are special mixes or blends of dry granular or stiffy plastic refractory materials with which virtually joint-free linings are formed.
3. Plastic refractories:- unburned blocks of refractories in a plastic state, usually piled up and pounded with a hammer into a monolithic wall, being burnt in place when the furnace is lit.[7]

2.0 Materials and Method

2.1 Materials

The clay sample used for this research work was collected from Umulumgbe in Udi Local Government Area in Enugu State-Nigeria. The clay materials were collected at depth intervals of 10cm with the aid of a shovel, digger and handpicked to reduce the possibility of contamination. 15kg of clay materials were collected and air-dried for three days. The clay materials were then finely crushed to achieve homogeneity of the particle size. The ground and sieved samples were mixed with some quantity of water to a pastry state. The past sample was moulded into bricks and compacted with hydraulic press according to ISO standards. The clay samples were subjected to both physical and chemical tests.

2.2 Method

The chemical analysis of the clay was carried out using X-ray florescence (XRF) techniques. The physical properties tests carried out include plasticity and plasticity ratio, moisture content, total shrinkage, modular of rapture, water absorption, apparent porosity, apparent density, bulk density, cold crushing strength, thermal shock resistance, loss on ignition and refractoriness. For each of the tests, five (5) specimens were used and the average taken and recorded.

Cold crushing strength (C.C.S.): Test pieces measuring 60 x 40 x 40mm were prepared and air-dried for 48 hours after which they were transferred to a furnace and heated for a period of 5 hours and at a temperature of 1200^{0}C. After the heating process, samples were removed and allowed to cool at room temperature and each piece was placed in a crusher. During test, the pressure adding surface was adequately aligned to the centre of the spherical seat of the equipment. Load was applied axially and continually until the test piece fractured. The procedure was repeated for other test pieces. The respective loads at which each test piece fractured were recorded. The cold crushing strength (CSS) was calculated from the equation.

$$CCS = \frac{P}{A} \dots\dots\dots\dots\dots (1)$$

Where A = Area of test specimen

P = Applied load

Plasticity and plasticity ratio

Five (5) specimen were produced using cylindrical mould and a plastomer was used in deforming them.

$$Modulus\ of\ plasticity = \frac{m_1}{y_1}$$

$$Plasticity\ ratio = \frac{m_2}{y_2}$$

Where m_1 = original height of deforming load
y_1 = deformed height of load
m_2 = original height of sample
y_2 = deformed height of sample

Moisture content: Five cylindrical specimens (1.5cm dia. x 10cm length) were weighed green and their weight noted. They were then dried in air for two weeks and later oven dried at a temperature of 110^0C for twenty four hours. The dry weight of the specimens was measured and recorded.

$$Moisture\ content = \frac{W_w - d_w}{W_w} \times \frac{100}{1}$$

Where W_w = wet weight
d_w = dry weight

Green, Dry and Fired Strength; five rectangular specimens (1.5 x 3.5cm) were taken and recorded. They were oven dried for two days and a rupture testing machine was used to test for the strength at given, dry and fired state. The fired strength was tested after firing the specimens to temperatures of 900^0C, 1000^0C, 110^0C and 1200^0C respectively.

$$modulus\ of\ repture: \frac{3pl}{2bh}\ or\ \frac{8pl}{\pi d^2}$$

Where: p = applied load (kg)
I = distance between supports constant (cm)
b = width of the specimen at the point of rupture (cm)
h = height of the specimen at the point of rupture
d = diameter of the cylindrical specimen

Apparent porosity, water absorption, apparent and bulk density tests. Rectangular specimens (5cm x 7cm) were prepared and their weight recorded. They were dried for two weeks, oven dried and their new weight recorded. They were fired to temperatures of 900^0C, 1000^0C, 110^0C and 1200^0C respectively. Their weights at each stage of firing were recorded. The fired specimens were soaked in water for twenty-four hours and the weight taken and recorded. They were calculated as follows:

$$Apparent\ porosity = \frac{S_w - F_w}{S_w - S_{sw}} \times \frac{100}{1}$$

$$Water\ absorption = \frac{S_{sw} - F_w}{F_w} \times \frac{100}{1}$$

$$Apparent\ density = \frac{F_w}{F_w - S_{sw}} \times \frac{100}{1}$$

$$Bulk\ density = \frac{F_w - dl}{S_w - S_{sw}} \times \frac{100}{1}$$

Where S_w = soaked weight
F_w = Fired weight
S_{sw} = Suspended weight
dl = Density of water

Linear dry and fired shrinkage: Rectangular specimen (5cm x 7cm) were produced and marked 5cm lengths. Temperatures of heating ranges was inscribed on the specimens to be fired. They were dried in air for two weeks and oven dried for forty hours. The change in the 5cm length mark was measured. They were then fired to temperatures of 900^0C, 1000^0C, 1100^0C and 1200^0C respectively.

$$a.\ Dry\ Shrinkage\ (\%) = \frac{wl - dl}{wl} \times \frac{100}{1}$$

$$b.\ Fired\ Shrinkage\ (\%) = \frac{wl - fl}{dl} \times \frac{100}{1}$$

Where wl = wet length (cm)
dl = dry length (cm)
fl = fired length (cm)

Permeability: Five specimens were prepared using standard specifications of 5.08cm diameter and 5.08cm length/height. They were dried in air for two twenty hours and oven dried for ten hours. Permeability meter was filled with $2000cm^3$ of water in a bel jar put in place. The orifice was opened and then taken for $2000cm^3$ of water to displace equal volume of air through the specimen taken. The pressure difference was measured using manometer.

$$p = \frac{vxh}{pxAxt} or \frac{vh}{pAt}$$

Where p = permeability meter
V = volume or air passed through the specimen (cm^2)
h = height of specimen
A = cross-sectional area of the specimen
p = pressure head under which the air has passed
t = time of flow in seconds

$$or\ p = \frac{30072}{pxt} or \frac{30072}{pt}$$

Thermal shock resistance: The test was carried out using 50mm x 50mm specimens. They were inserted into a muffle furnace and heated to 900^0C and held for 15 minutes. They were removed quickly from the furnace, and placed on fire bricks and allowed to cool for fifteen minutes, after the 15 minutes cooling, they were then returned to the furnace and the process repeated for 30 cycles. They were seen at the end of the 30 cycle not to have deformed.

Refractoriness: Pyrometric cone equivalent test was used to determine refractoriness specimen with 50mm pyramid height and 15mm rectangular base were used. They were put inside a refractory plague with two Seeger cone of 12 and 13 and oven dried at 110^0C. The temperature equivalents for the two Seeger cones were 1340^0C and 1348^0C respectively. They were put in a furnace and temperature raised at the rate of 100^0C per minute until the two Seeger cones bent over level with the base. Upon observation, it was noted that the specimen had not deformed or let alone melting. The firing was continued until temperature of 1400^0C was attained, yet the specimen remained undeformed.

The refractoriness was estimated using Shuen'sformular.

$$refractoriness, K(^0C) = \frac{360 + Al_2O_3 - RO}{0.228}$$

Where, Al_2O_3 = % alumina in the clay
RO = sum of all other oxides besides silica
360, 0.228 = constants.

Loss on ignition: 50g of the clay sample was oven dried 110^0C and cooled in a desicator. The dried sample was put inside the crucible and the weight of the crucible and the sample were recorded (m^2). The crucible with its contents were cooled in a desicator and then re-weighed (m^3)

3.0 Results and Discussion

The results of the research are presented in Table 3.1 – 3.4

Table 3.1 shows the chemical composition of the clay deposit from its chemical composition, the clay fall within the range of Aluminium-silicate type of clay because of its high value of Alumina (Al_2O_3) and Silica (SiO_2) which are highest among other chemical composition of the clay sample.

The results of the granulomeric particle size analysis and other physical properties tests are presented in Tables 3.2-3.4. From the table 3.2, it was shown that the clay has fine particle size of 5.0 – 2.0 of about 28% and fairly coarse particle of about 35% respectively. This indicated that the clay sample is siliceous with fairly large particles sized. The result of the other physical properties tests of the clay sample are shown in Table 3.3 and 3.4. The Table 3.3 and 3.4 shows that the shrinkage, modulus of rupture, cold rushing strength fell within the international standard Organization specifications. The porosity measures the ease with which liquid gas slip through the refractory materials[8]. Porosity affects the strength of refractory material as well as its insulating compatibilities. The average apparent porosity of the clay sample was found to be 20.73 as shown in Table 3.3but it was out of the range as quoted by [9] to be 20%-30% for fire clay or with most refractory bricks. The high apparent porosity value will enhance the penetration of molten metal, molten slag, and this will corrode the refractory bricks faster if applied in melting furnaces. The average value of the fired linear shrinkage of Umulumgbe clay sample is 7.2% as shown in Table 3.3 which is within the stipulated range of 7-10% for fireclay refractories as given by [9]. This firing shrinkage normally gives an indication of the firing efficiency. The bulk density of the sample was found to be 1.84g/cm^3 which falls within the range quoted for high alumina refractories of 2.29 kg/cm^3 – 2.89 kg/cm^3 as quoted by [1].

At higher temperature, the low melting point constituents of the clay body tend to melt, oxidize and fuse to the highly refractory constituents, thereby closing the pores and increase the formation of a denser body and strengths due to strong formation [10].

Table 3.1: Chemical Analysis of Umulumgbe clay deposit

Compound	SiO_2	Al_2O_3	CaO	TiO_2	Fe_2O_3	CO_3O_4	CuO	As_2O_3	ZrO_2	M_0O_3
Conc Unit	51.0 %	26.0 %	0.261 %	1.79 %	4.269 %	0.034 %	0.021 %	0.011 %	0.275 %	0.025 %

Compound	Pr_2O_3	Eu_2O_3	HfO_2	Re_2O_7	PbO	CdO	BaO	CaO_2
Conc Unit	0.02%	0.10%	0.031%	0.058%	0.040%	1.81%	0.04%	0.14%

Table 3.2: Granulomeric particle size analysis

Size (mm)	Quantity (%)
5.0 – 2.0	28
2.0 – 0.5	16
0.5 – 0.1	26
< 0.1	35

Table 3.3: Physical properties of Umulumgbe clay deposit

Parameter/Temperature	900^{0}C	1000^{0}C	1100^{0}C	1200^{0}C
Wet-Dry Shrinkage (%)	4.40	5.30	5.50	6.20
Dry-Fired Shrinkage (%)	1.67	1.85	2.14	2.14
Total Shrinkage (%)	6.0	6.10	6.60	7.20
Apparent Porosity (%)	22.74	21.08	19.52	18.38

Apparent Density (g/cm^3)	2.29	2.27	2.22	2.16
Bulk Density (g/cm^3)	1.77	1.79	1.82	1.84
Water Absorption (%)	12.83	11.78	10.92	10.81
Modulus of Rupture (kg/cm^3)	24.07	24.43	26.81	28.45
Green Modulus of Rupture (kg/cm^3)	12.73			
Modulus of Plasticity	1.47			

Table 3.4 : Other physical properties

Property	Quality
Plasticity ratio	1.3.1
Thermal shock resistance	26
Green strength (kgf/cm^2)	22.40
Moisture content (%)	26.59
Estimated Refractoriness ^{0}C	1,654

4.0Conclusion

This research work characterizedUmulumgbe clay deposit for its usefulness as refractory materials. This study has revealed that Umulumgbe clay sample has alumina (Al_2O_3) and silica (SiO_2) as its predominant oxides. From the physical propertiestests carried out, it was showed that some properties such as linear shrinkage, bulk density, thermal shock resistance as shown in Table 3.4 were compared favourably with the features of the fireclay refractories as reported by some researchers. This will ease off the importation of refractory bricks materials and improve the local content of our ceramic product.

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