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Physio-chemical Analysis of Gwarmi Clay Deposit, Wurno Local Government Area of Sokoto State, Nigeria

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----------**ABSTRACT**----------
The Chemical composition and physical properties of Gwarmi clay deposit in Wurno Local Government Area of Sokoto State, Nigeria has been investigated. The physical properties investigated were firing shrinkage, bulk density, apparent porosity, cold crushing strength, thermal shock resistance and refractoriness. The chemical analysis was performed using X-Ray Fluorescent (XRF) method, while the physical properties investigations were carried out following American Society for Testing and Materials (ASTM) stipulated standards. The result of the chemical composition shows that, the clay is rich in Oxides of Silica (SiO_2), Aluminum (Al_2O_3), and Iron (Fe_2O_3) with other oxides in trace amount. The Clay has a very low firing shrinkage of 1.5%, and higher water absorption of 14.95%. It has porosity of 19.0%, cold crushing strength of 304.55%kg/cm^{2}and thermal shock resistance of 20 cycles. The investigated properties proved that Gwarmi Clay deposit belongs to kaolinic fireclay deposit. The clay deposit can be used as refractory materials as well as in the development of load bearing structures such as improved cook stoves.

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I. INTRODUCTION

The term "Clay" applies to both materials having particle size of less than 2 micrometers and to the family of minerals that have similar compositions and structural characteristics (Olurunsogbon, 2007).

Clay is a complex inorganic mixture, whose composition varies widely depending on the geographical location. It is a natural substance occurring in great abundance in nature, being constantly formed on the earth's surface as a result of rock weathering (Aliyu,*et al., 2013*). Clay is composed of silica (SiO_2), Alumina (Al_2O_3) and water (H_2O) plus appreciable concentration of oxides of iron, alkali and alkaline earth, and contains groups of crystalline substances known as clay minerals such as quartz, feldspar and mica (Aliyu,*et al ., 2013*).

Clays have several properties; both chemical and physic- cal properties. These properties are responsible for the various areas of application of clays. Clays are used as ceramic and refractory materials, used in the formulation of drilling fluids, as binders in foundry moulds, as bind- ers for iron ore pellets in metallurgy, as catalyst in petro- leum refining and production of petrochemicals, soil remediation, bleaching of oils, clarification of wines etc (Paul, *et al ., 2012*).

There are vast deposits of clay spread across every region in Nigeria, though their properties differ from site to site on account of geological differences. The percentage of the minerals oxides (Fe_2O_3, MgO, CaOetc) in the clay ultimately determine the areas of applications of the clay such as in bricks, floor tiles, paper etc, while the presence of thealkali metals oxides (Na_2O, K_2O, CaO, etc) determine their suitability for making ceramic products (Aliyu,*et al ., 2013*).

Clay deposit may be formed in place as residual deposit in soil, but thick deposit are formed as the residual of secondary sedimentary deposition process after they have been eroded and transported from their original location of formation. Clay deposit is typically associated with very low energy depositional environments such as large lakes and marine deposits (Raw Material Research and Development Council, 2010).

Clay minerals are typically formed over long periods of time by the gradual chemical weathering of rocks, usually silicate-bearing, by low concentrations of carbonic acid and other diluted solvents. These solvents, usually acidic, migrate through the weathering rock after leaching through upper weathered layers. In addition to the weathering process, some clay minerals are formed by hydrothermal activity. Primary clays, also, known as kaolin, are located at the site of formation. Secondary clay deposits have been moved by erosion and water from their primary location (Raw Material Research and Development Council, 2010).

Clays are classified as (1) primary: (2) secondary or transported, i.e. sedimentary: and (3) residual, meaning that clay has been deprived of some of its original impurities by weathering or leaching. Many white or near-white plastic clays are residual, e.g. fireclays, from which most of the potassium, sodium, and iron compounds have been removed by leaching (Cardew, 2002).

All clays (whether swelling or non-swelling types) are hydrous aluminosilicates, and are therefore dominated by silica (SiO_2). Hence their SiO_2 content is not of great diagnostic help in their identification (Velde, 1992). Generally the elements Al, Mg, Fe, K, and to a lesser extent Na, and Ca, are used as indicators of clay types. Another important criterion is the basal spacing, i.e. the distance between the sheet layers of the crystals structures as determined by X-ray diffraction techniques. It is determined after heating to 200^oC to eliminate absorbed water and thus called the dried state. Swelled-spacing is determined using ethylene glycol vapour to expand the layers to a standard distance (Velde, 1992).

Olusola (1998) studied the refractory properties of Zungeru clay in Niger state and discovered that the moisture content, water of plasticity, and water of absorption were 7%, 40%, and 37.64% respectively. The values of fire shrinkage was 9.18%, apparent porosity was 22% while bulk density was 1.94 g/cm^3. He found that permeability was 80.4% while specific gravity was 2.82%. However, he found that specific density 3,33g/cm^3 recorded was higher than the international standard. The clay recorded a thermal shock resistance of 15 cycles while the refractoriness was >1200 ^oC making the clay good as lining materials for cookers.

The present study is aimed at determining the physiochemical properties of Gwarmi Clay deposits in Wurno local government of Sokoto State, Nigeria thereby determining its areas of applications.

II. MATERIALS AND METHODS

The clay sample was collected from Gwarmi clay deposit in wurno local government area of Sokoto state, Nigeria.The sample was collected at a depth of 0.5 m and analyzed. The specimen were dried, crushed, and then sieved.

2.1 Equipment used

The following equipments were used in the course of the investigation.
1. Weighing balances (Ohaus, model CS200, 0 – 200g), (0 – 100kg)
2. Mercury in bulb thermometer (0 – 100oC)
3. Plastic containers (50 -100 lt)
4. Glass funnel.
5. Beakers (150 - 500ml).
6. Measuring cylinders (50 -500ml).
7. Round bottom flasks (250, 500ml)
8. Ceramic crucibles (70ml, 250ml)
9. Desiccators
10. Electric ovens
11. Electric furnaces (1400^oC)
12. Ceramic and wooden mortars and pestles
13. Stainless steel spatula
14. Digital stop watch
15. Hydraulic press (50 tones) and X-ray fluorescence (XRF) machine

III. METHODOLOGY

The chemical composition of the clay was determined using X-ray fluorescence (XRF) method. This is a non-destructive analytical technique used to identify and determine the concentration of elements present in solid, powdered and liquid samples.

3.1 Determination of the Chemical Composition.

The chemical composition of the raw clay sample in wt % of (SiO_3, Al_2O_3, Fe_2O_3,etc) was examined using X-ray fluorescence/gravimetric methods, carried out at National Geoscience Research Laboratories Center, Kaduna, Nigeria.

3.2 Sample Preparation:

The sample was ground and sieved to -75 µm particle size. 4g of the sieved clay particles were intimately mixed with 1g of lithium tetra borate binder ($Li_2B_4O_7$) and pressed in a mould under a pressure of 10-

15 ton/m^2 into pellets. The pressed pellets were dried at 110 ^{0}C for 30 minutes in an oven to get rid of absorbed moisture and were finally stored in desiccators for analysis.

3.3 Determination of the physical properties

The physical properties were determined using American Society for Testing and Materials (ASTM, 2001) at the Raw Material's Laboratory of the "National Metallurgical Development Centre, (NMDC)" Jos, Plateau State, Nigeria. The physical properties determined include; linear shrinkage, water absorption, apparent porosity, bulk density, cold crushing strength, thermal shock resistance, and refractoriness.

3.3.1 Determination of Linear Shrinkage

To determine the shrinkage properties of the specimen, the specimen were pressed in a fabricated wood box of size 5x5x4cm. 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 verniercalliper. 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 linear shrinkage was calculated from equation (1).

Linear Shrinkage = $\frac{D_l - F_l}{D_l} \times 100$ … …………………………………………………(1)

Where, D_L = Dried Length, F_L = Fired Length

3.3.2 Determination of Apparent Porosity

A specimen of dimensions, 5cm x 5cm x 4cm size was tested. The specimen was dried over 12 hours at 110^{0}C in preparation for the test; it was taken directly from the oven for the test. The dry weight in air (D) of the specimen was measured. The specimen was then transferred into a 250ml beaker in empty vacuum desiccator. Water was then introduced into the beaker until the tested pieces were completely immersed. The specimen was allowed to soak in boiled water for 30 minutes being agitated from time to time to assist to release trapped air bubbles. The specimen was then transferred into empty vacuum desiccator to cool. The soaked weight (W) was recorded. The specimen was then weighed suspended in water using beaker place on balance. This gave suspended weight (S). The apparent porosity was determined according to ASTM Standards C 20/2007 using equation (2)

Apparent Porosity = $\frac{W - D}{W - S} \times 100$ …………………………………………………………… (2)

Where, W = Soaked weight

D = Dried weight

S = Suspended weight

3.3.3Determination of Bulk Density

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

Bulk Density = $\frac{DP_W}{W - S}$ *g/cm^3 ……………………………………………………………… (3)*

Where, D = Dried weight

W = Soaked weight

S = Suspended weight

P_w *= Density of water*

3.3.4Determination of Cold Crushing Strength Tests

A piecetest brick produced using the clay sample was prepared to standard cube size, of dimensions 50mm wide and 50mm high. It was then air dried for 24 hours and oven dried at a temperature of 110 ^{0}C for 12 hours and then fired in a furnace at a temperature of about 1200 ^{0}C for 6 hours.It was then cooled to room temperature. The specimen was then taken to the tensiometer where load was applied axially to the piece until crack was noticed. The load at which the specimen cracked was noted, which represents the load required for determining cold crushing strength of the test specimen. Cold crushing strength was then determined using equation (4)

$$CCS = \frac{\text{Maximum load (KN)}}{\text{Cross - sectional area (m2)}} = P/A \quad \ldots\ldots(4)$$

Where,

C.C.S = Cold Crushing Strength

P = Applied Load

A = Area of Load Applied

3.3.5Determination of Thermal shock Resistance

Specimen measuring 5cm x 5cm 4cm was used for this test.The prepared sample was inserted in a furnace which has been maintained at 900^{o}C. This temperature was maintained for 10 minutes. The specimen was removed with a pair of tongs from the furnace and then cooled for 10 minutes and observed for cracks. In the absence of cracks (or fracture),the specimen was put back into the furnace and reheated for a further period of 10 minutes and then cooled for another 10 minutes. The specimen was returned to the furnace for further 10 minutes. The process was continued until the test piece was cracked. The number of cycles of heating and cooling before cracking for the specimen was recorded as its thermal shock resistance.

3.3.6 Determination of Refractoriness (PCE)

The refractoriness or softening point was determined using the method of pyrometric cone equivalence (PCE). Test cones were prepared by mixing the clay sample aggregate with sufficient quantity of water to make the clay become plastic and molded by hand into a cone shape. The sample was dried and fired to a temperature of 900 ^{0}C in a muffler furnace. Pyrometric cones designed to deform at 1300 ^{0}C, 1400 ^{0}C, 1500 ^{0}C were placed round the sample and the temperature rose to above 1000 ^{0}C at 10 ^{0}C per minute. The heating was discontinued when the test cone bent over and leveled with the base of the disc. The pyrometric cone equivalent (P.C.E) of the sample was recorded to be t number of standard pyrometric cone corresponding to the time of softening of the test cone.

IV. RESULTS AND DISCUSSION

Table 1 shows the result of chemical composition in wt %, while the result of the physical properties is shown in Table 2.

Table 1: Chemical Composition of the Clay Sample (%)

Sample Location	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MnO	LOI	Al_2O_3/SiO_2
Gwarmi clay	60.20	18.00	6.77	5.89	0.62	0.02	0.92	0.3
*Refractory clay	46- 62	25 - 39	0.4– 2.7	-	0.2– 1.0	-	8-18	
*High melting clay	53-73	16-29	1-9	-	0.5-2.6	-	4-12	

*Nnuka and Agbo (2000)

Table 2: Physical Properties of test sample compared with standard clay

Sample Location	Linear shrinkage (cm)	Apparent Porosity (%)	Bulk Density (g/cm^3)	Cold crushing strength (kg/cm^2)	Thermal shock resistance (cycles)	Refractoriness (^{o}C)
Gwarmi clay	1.5	19.0	1.91	304.35	20	1500
*Fire clay	4-10	20-30	2.30	150	20-30	1500-1700

*Omowumi (2001)

V. DISCUSSION

The chemical composition of the clay sample tested showed that the alumina (Al_2O_3) content for the clay was 18.00%. The clay was found to qualify as highmelting clay but not as refractory clay. This is because the value of the alumina content lies withinthe recommended range for high melting clay as shown in Table 1.

The silica (SiO_2) content of the clay 62.20% meets the standard for refractory clay (46-62%).The iron iii oxide (Fe_2O_3) content of the clay sample is high and such level of oxide usually imparts a reddishcolour to clay when fired, so making it attractive as a ceramic raw material.In general the clay consist of three basic oxides of silica, alumina and iron with other oxides in trace amounts. The clay had alumina-silica ratio of 0.30. The alumina content agreed with Aderibigbe, (1989), who reported that in Nigeria, the major refractory clay deposits that is within the alumina-silicate raw materials are kaolin and fireclay deposits with alumina content of less than 45%. Similarly the clay sample contained some amount of lime (CaO) of less than 1%. In addition, the clay sample contained other different types of fluxing minerals, which were in trace amount. It is observed from Table 1, that the dominant components of the clay sample (SiO_2, Al_2O_3 and Fe_2O_3) clearly defined it as hydrated alumino-silicate type. The average silica + alumina + water (SiO_2 + Al_2O3 + Fe_2O_3) contents in the clay sample constitute about more than 84.97%.

Loss on ignition (L.O.I)

This is the combustion of volatile matter present in the clay. They are often required to be low. Asshown in Table 1, loss on ignition for the clay sample is much lower than 8% and 4% specified lower limit for refractory and high meltingclays respectively.

Linear Shrinkage

Results of the linear firing shrinkage are presented in Table 2. The result revealed that the linear shrinkage is 1.5%. RMRDC gave the linear shrinkage of Kankara deposit in Kastina State Nigeria to range from 9% to 12%; Chester, (1973), recommended linear shrinkage range of 7-10% for refractory clays. This value is lower than the recommended range of 4-10% for fireclay as reported by Omowumi (2001).This is more desirable as higher shrinkage values may result in warping and cracking of the clay product and may cause loss of heat in the furnace.

Apparent Porosity

The result of apparent porosity is shown in Table 2. The sample gave apparent porosity value of 19.00% which is within the acceptable range (10-30%) suggested for refractory clays by Chester (1973).

Cold crushing strength

The essence of undertaking this test is to determine the ability of bricks to withstand stresses in service. Table 2 shows the result of the cold crushing strength. Gwarmi clay gave a mean strength of 304.35 g/cm^3. However, Omowumi (2001), recommended 150 g/cm^3 as the minimum value for refractory clay materials. The high value obtained shows that Gwarmi clay can comfortably withstand impacts at low temperatures.

Thermal shock resistance

Result of the thermal shock resistance is displayed in Table 2. Result obtained gave the number of cycle to failure to be 20. This value fall within the 20-30 numbers of cycles recommended by Chester (1973).

VI. REFRACTORINESS

Table 2 shows the refractoriness of the sample. The result shows that refractoriness of the sample occurred at a temperature of 1500°C. The value is within the recommended range for fireclay refractories of 1500-1700OC as reported by Omowumi (2001). This shows that the clay can withstand the deformation temperature of about 1500 °C before fussing or bend under its own weight.

VII. CONCLUSIONS

Aninvestigation on the physio-chemical properties of Gwarmi clay was carried out and the following deductions can be inferred.

- The chemical analysis result shows that Gwarmi clay sample contain basically three basic oxides of Silica (SiO_2), Alumina (Al_2O_3) and Iron (Fe_2O_3) as the predominant substances with minor amounts of fluxing components (TiO_2, MnO and CaO).

- It was established that, the clay sample is siliceous in nature and can be classified as kaolinitic fireclay which can be used for the production of aluminosilicates refractory.

- The clay sample had a good cold crushing strength of 304.35 Kg/cm^2 which is higher than the standard of 150 Kg/cm^2.

- Gwarmi clay deposit can therefore, successfully be processed for use as refractory materials such as a ladle bricks, ramming mass, as well as in the development of load bearing structures such as improved cook stoves.

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