Physiochemical Properties of Ajona-Obukpa Clay Mineral for Refractory and Industrial Application

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ABSTRACT

This study evaluates the characterization of Ajona- Obukpa clay obtained from Enugu State, Nigeria for its industrial applicability. The chemical analysis of the clay showed silica (46.8%) and alumina (21.1%) as the major constituents while other metal oxides such as Fe_2O_3 (6.2%), CaO (3.1%), K_2O (2.5%), and Na_2O (2.8%) are present in appreciable amounts. The physical analysis showed a variation in the linear shrinkage (2.38 to 4.86%), total shrinkage (5.50 to 8.00%), apparent porosity (30.49 to 21.05%), and apparent density (2.44 to 2.43 g/cm^3), bulk density (1.69 to 1.79 g/cm^3), water absorption (17.99 to 11.76%) and modulus of rupture (10.75 to 19.06 kgF/cm^2) with increase in firing temperature from 900 to 1200°C. The clay showed moderate plasticity with a modulus of plasticity of 1.35 and had a refractoriness of up to 1200 °C. The result of this study showed that Ajona-Obukpa clay has good industrial potentials and can be utilized in the manufacture of ceramics, high melting clays, refractory, bricks, tiles and color vase but requires additives to help obtain the desired properties. It can therefore be utilized to help reduce the bulk of clay minerals imported from foreign nation into Nigeria.

(Keywords: Ajona-Obukpa clay, refractory, industrial use, geochemistry).

INTRODUCTION

Clay is an unconsolidated rock matter, with very fine grain, which is plastic when wet, and undergoes ceramic change to become hard and stony when heated. The ceramic industries are the major users of clay. These industries consume approximately 70% of all clays marketed in crude or beneficiated form and those marketed only as finished products [1].

Natural clay minerals are well known to mankind as far back as the days of civilization. Owing to their low cost, abundance in most continents of the world, high sorption potential for ion exchange, clay materials are indispensable candidates as adsorbents [2]. Clay is an important material for engineering works. It is a traditional material commonly used in construction industries, such as in the manufacturing of clay bricks, pipes, ceramic tiles, and constructional devices. Nigeria is highly blessed with this resource and has a reasonable supply naturally [3]

Clay is mainly composed of silica, alumina, and water plus appreciable concentration of oxides of iron, alkali and alkaline earth, and contains groups of crystalline substances known as clay minerals such as quartz and feldspar [2]. The presence of the minor oxide impurities occurring in variable amounts in clays tends to impart some properties to the clay, which are of technical value. Characterization of the clay available in any region helps in its applicable and general usage either in ceramic, drilling mud, refractories, plastics, paints, textiles and adhesives, paper foundry, pharmaceuticals, rubbers [4]. However, appropriate use of clay depends on its properties, composition, shrinkage, plasticity, feasibility, among others.

Over the years, many researchers have reported the characterization and properties of clays found in different parts of Nigeria in order to provide useful information on their applicability [5-7]. Consequently, such clays as Mbaukwu clay, Isiagu clay, Ezzodo clay, Ukpor, Uzalla clay, Kankara, Adiabo, and Dabagi among others have been characterized and found useful applications for industrial purposes and ceramics [1,8,9].

Moreover, the local demands for refractory products are very high because of its numerous applications in ceramics and steel industries yet most of the products are imported. The exorbitant cost of importation of refractories has led to unfavorable trade balances for manufactures and even government which is responsible for the hike in the prices of the finished products (e.g., electric cooker, oven, glass wares, tiles, ceramics etc.). In addition, the steel rolling mills in Nigeria will consume about ₦4.5 million worth of refractories when they are brought to use [10]. This is ridiculous compared to the abundance of both raw materials and human capacity for research and production of ceramics products of international standard and quality. Another major problem is the fact that most of the imported ceramic wares are made with colors and glazes that are not consumable and very dangerous to the human body [11].

In southeastern Nigeria there is an abundance of clay deposits, which have not been characterized and may be useful in the manufacture of ceramics and most industrial products. Ajona-Obukpa clay is one of such, which is present in abundant amounts in Nsukka local government area of Enugu State, Nigeria. Literature search revealed that there is dearth of information on its properties or characterization for its industrial applications. This study therefore investigates the characterization and potentials of Ajona-Obukpa clay for its industrial uses to help reduce the high rate of importation of clay products in Nigeria. The chemical and physical properties of the clay was determined in order to substantiate its application in the manufacture of useful industrial products.

MATERIALS AND METHODS

Sample Collection and Pretreatment

Ajona-Obukpa clay sample was obtained by random sampling at different points in Obukpa, Nsukka local government area of Enugu state, Nigeria. The samples were taken at a depth of 1.55 m and mixed properly to obtain a homogenous mixture. The cone and quartered method was employed to obtain a representative sample as described [12]. Thereafter, the collected clay was dispersed in excess water in a pre-treated plastic container and stirred vigorously to ensure proper dissolution. The dissolved clay was then filtered through a 0.425 mm mesh sieve to get rid of unwanted particles and plant materials. The filtrate was allowed to settle, after which excess water was decanted off. The clay was then sundried and oven dried at 100°C for 3 hours, pulverized, and passed through a mesh sieve of size 0.18 mm. 1.6 kg of the clay was weighed and mixed with appropriate amount of water to make it plastic for the molding process.

Molding of the Test Pieces

The clay was then molded into three types of shapes using metallic molds and the application of lubricants to the surface of the molds to prevent the test pieces from sticking to the surface. The first shape is cylindrical with a width of 3.5 cm and height 3 cm, the second is a rectangular piece with length 8 cm, width 4 cm and height 1.5 cm, while the third has a long rectangular shape with length 9.5 cm, width 2 cm and height 1.5 cm.

Determination of Making Moisture

This was determined by weighing the cylindrical test pieces immediately after molding and recorded as the wet weight, W_o. The test pieces air-dried for 24 hours and then dried in an oven at 105 °C until a constant weight was recorded. After drying the test pieces were weighed and the dried weight recorded as W_i. The making moisture was then calculated.

$$Making\ Moisture\ (\%) = \frac{[W_o - W_i]100}{W_o}$$

Determination of Relative Plasticity

The relative plasticity was determined using the cylindrical test pieces. The original height, H_o of the test pieces were obtained by the use of the Vernier caliper by taking the average of three sides. Afterwards, a manual plastometer machine was used to deform the test pieces. The deformation height, H_i was recorded taking the average of three sides. The relative plasticity was then calculated [8].

$$Relative\ Plasticity = \frac{H_o}{H_i}$$

Determination of Modulus of Rupture

Five long rectangular test pieces were made and air dried for 7 days after which they were oven dried at 105°C until a constant weight was obtained. Four of the pieces were fired to their respective temperatures of 800, 900, 1000, and 1100°C in a laboratory kiln (Fulham Pottery).

The electrical transversal strength machine was used to determine the breaking load, P (Kg). A Vernier caliper was used to determine the distance between support L (cm) of the transversal machine. The height, H (cm) and the width, B (cm) of the broken pieces were determined and the average value obtained from the two broken parts was recorded. The modulus of rupture was then calculated:

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

Shrinkage Determination

Immediately after molding of the rectangular test pieces, a Vernier caliper was used to insert a 5 m mark on each of them, this was recorded as the original length *Lo* (cm). The test pieces were then air dried for 7 days and then dried in an oven at 105°C until a constant weight was obtained. The shrinkage from the 5 cm mark was then determined and recorded as the dried length, L_d (cm). Afterwards, four of the dried samples were fired to their respective temperatures of 800, 900, 1000, and 1100 °C each temperature corresponding to a particular test piece. The shrinkage of the test pieces from the 5 cm mark were then determined and recorded as the fired length, L_f (cm). The shrinkage was then calculated:

$$Dry\ Shrinkage\ (\%) = \frac{100[L_o - L_d]}{L_o}$$

$$Linear\ Shrinkage\ (\%) = \frac{100[L_d - L_f]}{L_d}$$

$$Fired\ Linear\ Shrinkage\ (\%) = \frac{100[L_o - L_f]}{L_o}$$

Determination of Water Absorption

The fired test pieces obtained after firing were then weighed and the weight recorded as dry weight, M_1 (g). Thereafter, the test pieces were soaked in water for one hour, then removed, cleaned and weighed immediately and recorded as soaked weight, M_2 (g). The water of adsorption was then calculated:

$$Water\ of\ Absorption\ (\%) = \frac{100[M_2 - M_1}{M_1}$$

Porosity and Density Determination

After the procedure described above was completed. The suspended weight of the test pieces was then determined by the use of a lever balance and recorded as M_3 (g). The apparent porosity, apparent density and bulk density were then calculated:

$$Apparent\ Porosity\ (\%) = \frac{100[M_2 - M_1]}{M_2 - M_3}$$

$$Apparent\ Density = \frac{M_2}{[M_1 - M_3]}$$

$$Bulk\ Density = \frac{M_1}{[M_2 - M_3]}$$

Loss on Ignition (LOI)

The weight of an empty porcelain crucible was determined and recorded as W_1, 2 g of the dried pulverized clay was added and the weight of the crucible + clay was determined, W_2. The sample was then ignited in the laboratory kiln at 1200 °C. After, the cooling of the sample the weight of the crucible + sample after ignition was determined, W_3. The loss on Ignition was then calculated:

$$Loss\ on\ Ignition\ (LOI) = \frac{100[W_2 - W_3]}{[W_2 - W_1]}$$

For LOI:

W_1 = 9.42 g, W_2 = 11.42 g, W_3 = 11.068 g

Chemical Analysis

0.2 g of the clay was weighed into a beaker and 10 mL of aqua regia ($HCl + HNO_3$ in the ratio 3:1, respectively) was added and digested in a hot plate in a fume cupboard. 10 mL of Hydrofluoric acid was also added to aid the digestion process. After digestion 30 mL of de-ionized water was added and the mixture filtered through a filter paper into a 250 mL volumetric flask and made up to the meniscus mark with de-ionized water. The sample was then analyzed for the elemental composition by the use of the Atomic Absorption spectrophotometer (AAS) (Buck scientific model 210 VGP). The concentration of metal oxide in the clay was expressed in mg/L. The percentage composition of the elements in the clay was calculated from the equation:

% Composition = 100CV/M

Where C (mg/L) is the elemental composition obtained from the AAS, V (L) is the volume of the volumetric flask in which the digested solution was diluted and M (mg) is the mass of sample digested.

RESULTS AND DISCUSSION

Chemical Analysis

The result for the chemical analysis of Ajona-Obukpa clay is shown in Table 1. It is observed that silica (SiO_2) and alumina (Al_2O_3) form the major composition of the clay while other metal oxides are present in smaller amounts. The silica content of Ajona-Obukpa clay was found to be low and fall short of the requirement for the manufacture of ceramics (>60.5%), refractory bricks (>51.7%), high melting clays (53-73%) and glass [13]. However, the silica level was found to higher than that for paper (45.0-45.8%) and paint (45.3-47.9%) [14].

The alumina composition of Ajona-Obukpa clay fell short of the requirement for the manufacture of ceramics (>26.5%), Refractory bricks (25-44%), paper (33.5-36.1%), paint (37.9-38.4%) but met the standard required for high melting clays (16-29%) and glass (12-17%) [13,14]. The percentage composition of alumina in clay is a strong indicator for its refractoriness and the higher the amount of alumina the more refractory the clay [15]. The low percentage of alumina indicates that Ajona-Obukpa clay is likely to have low or moderate refractory properties.

The Fe_2O_3 content of Ajona- Obukpa clay was quite high and above the standard required for ceramics (0.5-1.2%), refractory bricks (0.52.4%), glass (2-3%) and paper (0.3-0.6%) but below that required for paper production (13.4-13.7%) [15]. However, the clay can still be used in the manufacture of high melting clays which requires only 1-9% of Fe_2O_3 as reported by Grimshow [14]. Besides, such high levels of iron oxide usually give a reddish color to the clay body when fired making it suitable for some ceramic products such as flower vase which requires such coloration [15]. The presence of Fe_2O_3 was responsible for the reddish-brown coloration obtained after firing as shown in Table 1. The coloration makes the clay unsuitable for white ware products [1]. In addition, the iron content in the clay may be desirable, acting as fluxes in infusible products and highly refractory materials [12]. This is because the high iron content in the clay greatly affects the high temperature characteristics of the clay [15].

Table 1: Chemical Composition of Ajona-Obukpa Clay.

Metal Oxides	Conc (mg/L)	% Composition
SiO_2	374.4	46.8
Al_2O_3	168.8	21.1
Fe_2O_3	49.6	6.2
Na_2O	22.4	2.8
K_2O	20	2.5
MgO	12.8	1.6
CaO	24.8	3.1
MnO	3.2	0.4
LO1 at 1200^0C	-	15.4

The presence of alkali oxides (CaO, K_2O and Na_2O) in reasonable amounts in Ajona-Obukpa clay as shown in Table 1 indicates the good fluxing ability during firing at low temperatures to form glasses of complex composition towards giving a vitreous structure to the ceramic product [17]. The presence of these oxides in clay acts as mild fluxes, they combine with the oxides of silica and alumina on firing to for eutectics and so reduce the vitrificaion temperature and refractoriness of the clay [18]. This further suggests that Ajona-Obukpa clay is likely to have low or moderate refractory properties as stated earlier.

As observed in Table 1, the Loss on Ignition (LOI) of the clay is 15.4% which accounts for the water vapor from dehydroxylation reactions in the clay minerals, carbonate decomposition into CO_2 and oxides as well as burning out of organic matter or other impurities present in the clay [18].

The LOI of Ajona-Obukpa clay met the standard for the manufacture of ceramics (>8.18%), refractory bricks (8-18%) and high melting clays (5-14%) [20]. A high LOI usually implies higher porosity in the manufactured products due to the removal of LOI components during firing while a low LOI is sometimes desired in the manufacture of low porous ceramic products.

As shown in Table 1, Ajona-Obukpa clay had moderate refractory properties and did not show any sign of failure at 1200⁰C. However, at higher temperatures of 1300⁰C signs of failure were observed. The implication is that, although the sintering level was high, it is not enough to fall within the internationally accepted range of 1580-1750⁰C for refractory materials [1], this result is corroborated by the low alumina and the presence of alkali metal oxide fluxes in Ajona- Obukpa clay which reduces its refractoriness. Similar refractoriness was reported by Abuh *et al.* [1] in the characterization of adiabo clay for its industrial potentials.

Linear Shrinkage

Some of the physical properties of Ajona- Obukpa clay determined after firing at temperatures of 900 to 1200 ˚C are shown in Figures 1-7. The shrinkage of a clay body after firing is a very important factor to be considered. High shrinkage values may result in warping and cracking of the clay and this may result in loss of heat and create an undesired finished product. The result for the linear shrinkage of Ajona-Obukpa clay after firing at different temperatures is shown in Figure 1.

It is observed that an increase in the linear shrinkage of the clay from 2.38 to 4.86% with increase in the firing temperature from 900 to 1200⁰C was recorded. This minimal increase may be attributed to the removal of certain components in the clay body with increase in temperature resulting in the sintering and subsequently vitrification of the clay body. This implies that as the temperature is increased the clay tends to compress and may result in decrease in porosity.

The linear shrinkage of the clay at firing temperatures of 1100⁰C (4.03%) and 1200⁰C (4.86%) was within the range of 4-10% required for fireclays [12]. However, the values are lower than the standard of 7-10% for aluminosilicates and kaolinites [21]. This indicates that Ajona-Obukpa clay is not from a kaolinite origin as deduced from the low percentage of alumina present. The low shrinkage values suggest a high content of non-fluxing impurities [1].

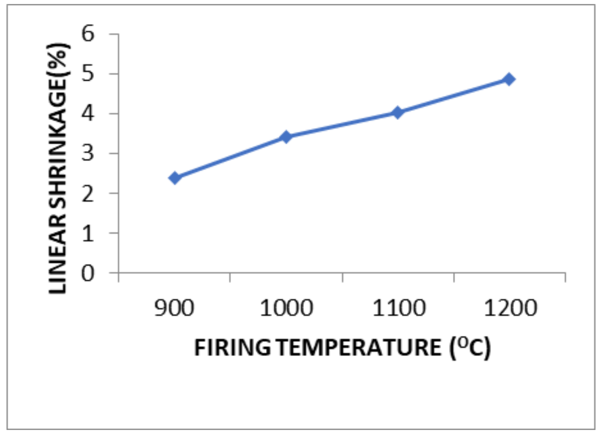

Figure 1: Effect of Firing Temperature on the Linear Shrinkage of Ajona-Obukpa Clay.

Total Shrinkage

Also, Figure 2 showed an increase in the total shrinkage of Ajona-Obukpa clay from 5.50 to 8.00% with increase in the firing temperature. However, the total shrinkage and the linear shrinkage (Table 1) are of little importance since their values change with the making moisture during firing [1].

As seen in Table 1, the making moisture of 18.73% was obtained for Ajona- Obukpa clay which is quite high and accounts for the linear and total-shrinkage recorded due to removal of such water during drying.

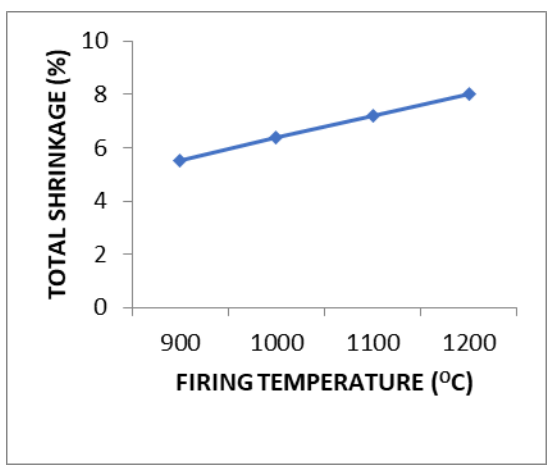

Figure 2: Effect of Firing Temperature on the Total Shrinkage of Ajona-Obukpa Clay.

Apparent Porosity

The apparent porosity of the clay with firing temperature is shown in Figure 3. A decrease in the porosity from 30.49 to 21.05% with increase in firing temperature was observed. This decrease is due to the increase in shrinkage with increasing temperature as stated earlier, which resulted in the coming together and closure of the pores of the clay body.

The values of the apparent porosity were within the range of 20-80% required for the manufacture of fire bricks [22] but higher than the standard range of 20-30% and (>23.7%) for production of fireclay and siliceous fireclays respectively [4]. However, the apparent porosity of 21.05% obtained at 1200⁰C for Ajona- Obukpa clay was within the range.

The clay could still be utilized in the production of insulating materials by the addition of some carbonaceous materials to help improve its porosity and insulating properties. Also, the slightly high porosity of the clay can be reduced drastically by the addition of glaze in the final product when a porous surface is not required in the manufacture.

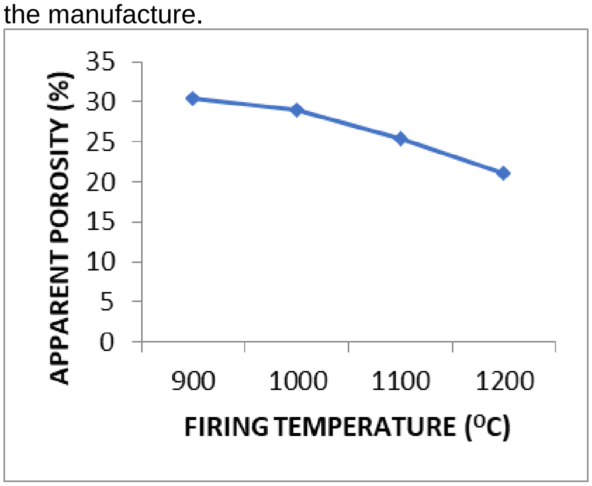

Figure 3: Effect of Firing Temperature on the Apparent Porosity of Ajona-Obukpa Clay.

Bulk Density

The bulk density of the clay showed an increase from 1.70 to 1.79 g/cm^3 with increase in firing temperature (Figure 4). This implies that the clay becomes more compact and dense as the shrinkage increases and thus is expected to have a progressive increase in strength of the clay body.

Apparent Density

A reverse trend was obtained in Figure 5 for the apparent density in which a decrease from 2.44 to 2.27 g/cm^3 was observed with increase in firing temperature. This is expected as the apparent density always shows an opposite trend to the bulk density of fired clay bodies. The apparent density is seldom discussed in most publications in details as the bulk density, as the latter is related to the weight of the body to an extent. The bulk density of Ajona-Obukpa clay obtained at 1100⁰C and 1200⁰C of 1.75 g/cm^3 and 1.79 g/cm^{3,} respectively was within the internationally accepted standard of 1.7-2.1 g/cm^3 required for building and fireclays [23]. The same also applies to the apparent density which was within the standard range of 2.3-3.5 g/cm^3 as reported by Ryan [24].

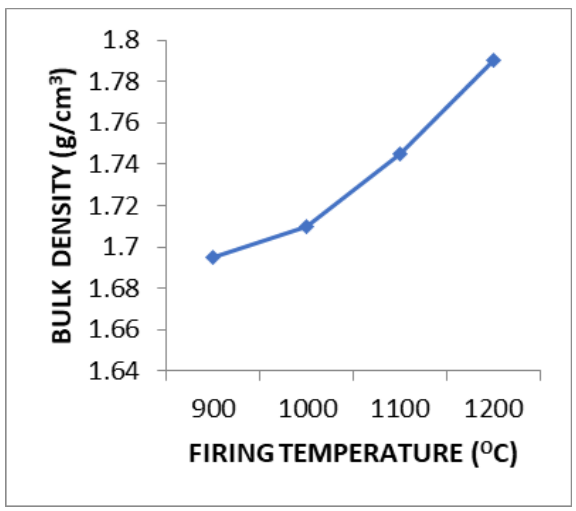

Figure 4: Effect of Firing Temperature on the Bulk Density of Ajona-Obukpa Clay.

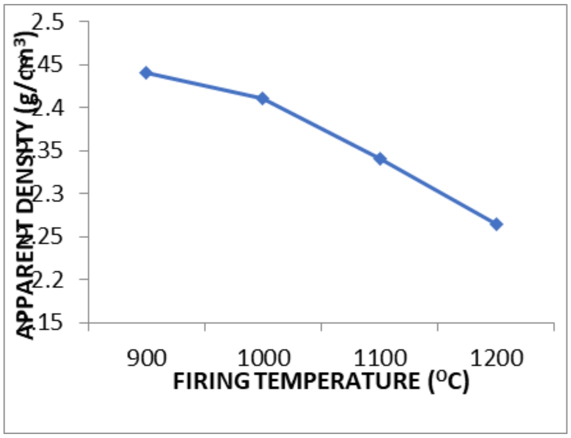

Figure 5: Effect of Firing Temperature on the Apparent Density of Ajona-Obukpa Clay.

Water Absorption

The result of the water absorption of Ajona-Obukpa clay at different firing temperatures is shown in Figure 6. From the result, a decrease in the water absorption with increase in firing temperature of the clay was recorded. In fact, with increase in the firing temperature of the clay from 900 to 1200⁰C the water absorption showed a decrease from 17.99 to 11.76%. This decrease is attributed to an increase in shrinkage and decrease in porosity of the clay body with increase in firing temperature.
The presence or addition of glaze to the clay body also helps reduce the water absorption when required due to the reduction in porosity as stated earlier.

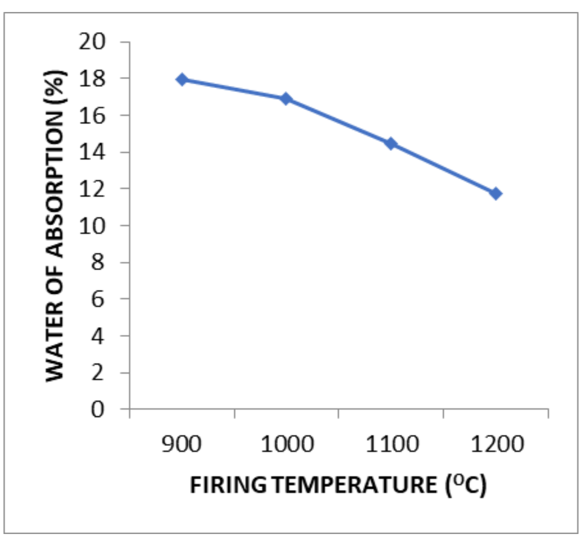

Figure 6: Effect of Firing Temperature on the Water Absorption of Ajona-Obukpa Clay.

Modulus of Rupture

Figure 7 showed an increase in the strength or modulus of rupture (MOR) of Ajona-Obukpa clay with increase in firing temperature. An increase in the MOR from 10.79 to 19.06 kg/cm^2 with increase in the firing temperature from 900 to 1200⁰C was recorded.

This increase is due to the fact that the clay body becomes more compact and rigid as temperature increases as the body shrinks together. The increase can also be attributed to bond formation in the glassy phase of the body [25]. The alkali metal oxide fluxes in the clay body combine to form some considerably low temperature melting compounds, which increases the strength of the body on cooling. The MOR obtained at all temperatures was within the wide range of 1.4 to 105 kg/cm^2 generally required for the manufacture of a wide variety of product [25].

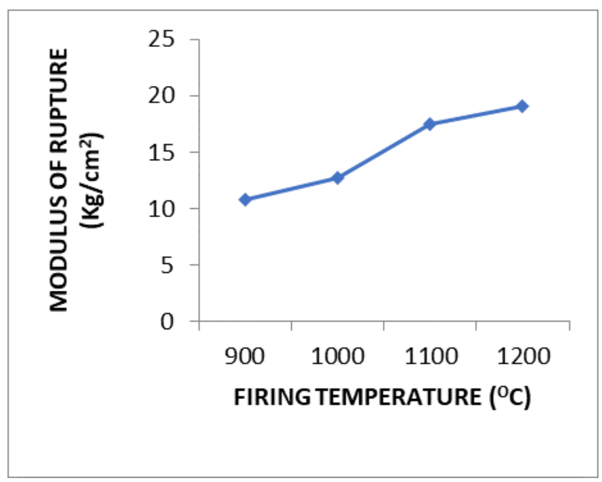

Figure 7: Effect of Firing Temperature on the Modulus of Rupture of Ajona-Obukpa Clay.

Modulus of Plasticity

The modulus of plasticity of Ajona-Obukpa clay as shown in Table 1 was found to be 1.47, this is higher than that of 1.33 reported for adiabo clay which was said to have moderate plasticity [1]. The higher values reported for Ajona-Obukpa clay indicated better or higher plasticity which is desirable as the clay will tend to have good workability and can easily be molded into shape.

The plasticity recorded makes the clay suitable for many industrial products but the high Fe_2O_3 content and other oxide impurities limits its use in white wares and refractory. However, the clay can be utilized as an additive to improve the plasticity of short clays. The moderate plasticity of Ajona-Obukpa clay may be due to the low alumina content and the high silica content recorded. Similar results have been reported by other researchers [1,25,26].

CONCLUSION

Summarily, the result showed that Obukpa contains aluminum oxide Al_2O_3 and silica SiO_2 as main constituents making them suitable as aluminosilicates refractory materials. The refractoriness of the clay was moderate at 1200^0C though below the standard requirement for refractory clay (1500^0C). The physical analysis of the clay falls within specification especially the higher plasticity of 1.47 as compared to 1.21 and 1.31 reported for Isiagu clay and Ezzodo clay, respectively. The high value makes Ajona-Obukpa clay possess good workability and can be easily molded into various shapes. The clay was found to be useful in the for the production of paper, paint, high melting clays, glass, and ceramics.

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