Firing temperature algorithm on the physiochemical properties of *Ishiagu* clay deposit for refractory application

Chidinma Christiana EZEOFOR*, Kovo Godfrey AKPOMIE, Ebuka Leonard ONYEYILIM

Department of Pure and Industrial Chemistry, Faculty of Physical Sciences, University of Nigeria, Nsukka, PMB 410001, Enugu State, Nigeria.
E-mail(*) chidinma.ezeofor@unn.edu.ng

*Corresponding Author, phone: +2348184636365

Abstract

This study investigated the chemical and physical characteristics of clay samples from Ebonyi State in Nigeria with a view to determining them for refractory application. The chemical analysis of the clay showed silica (48.3%) and alumina (21.7%) as the main constituents while other metal oxides such as Fe_2O_3 (6.8%), Na_2O (2.9%), K_2O (2.7%), MgO (1.2%), CaO (0.9%) and MnO (0.3%) are present in minute quantities. As the firing temperature increased from 900 to 1200^{o}C, the analysis varied from 2.98 to 10.56 for linear shrinkage, 34.01 to 27.02% for apparent porosity, 2.52 to 2.37g/cm^3 apparent density and 1.66 to 1.73g/cm^3 bulk. The clay showed low plasticity with a modulus of plasticity of 1.21, making moisture of 24.84 and had a refractoriness of up to 1200^{o}C. From the result of this study, it is shown that Ishiagu clay has good refractory and industrial potentials and can be used in the manufacture of refractory bricks, tiles, high melting clay, ceramics and colour vase but requires the addition of other materials to achieve the desired properties.

Keywords

Ishiagu clay; Chemical and physical characteristics; Ceramics, Industrial; refractory potential

Introduction

Clay is a plastic material containing fine-grained minerals that solidifies by losing its water constituents during firing or drying [1, 2]. Chemically, they are hydrous aluminium silicates that contains small amount of impurities such as potassium, sodium, calcium, magnesium or iron [3]. Refractoriness is an essential property in modern industry as a result of their capacity to resist high temperature, ability to react with the environment without melting, inert, possession of reversible thermal expansion and resistance to thermal shocks [4]. Refractory are non metallic materials comprising of silicon and aluminum oxides with the ability to resist both physical and chemical stress, possess high melting temperature and retaining their structural properties at such temperatures (>1100^{o}C) [5, 6].

The characterization and chemical composition of clay deposit in any region determines its use and applicability in the area and gives a general information on the usage either in paints, textiles, metallurgy, ceramics, rubber, pharmaceuticals etc. [7]. Researchers have carried out studies on characterization and mechanical properties of clay obtained from different parts of Nigeria in order to supply basic information on their uses [8]. For instance, some clays such as Ezzodo, Bende, Dabagi, and Odukpani clay have been characterized and found useful for industrial, insulating, ceramic and refractory applications [3, 8-10].

Consequently, the local demands for refractory products are very high with respect to the population yet most of the products are imported. In Nigeria, technological development has led to the development of Iron and steel industries together with other industries that use refractories such as Ajeokuta steel complex in Nigeria which on completion will need about 3600 tones of refractory bricks worth over N22 million [11]. Similarly electric cooker, oven and furnace manufacturing industries use refractories as their main gathering materials. These refractories are currently being imported and cost the manufacturers about N3.95 million. In addition, the steel rolling mills in Nigeria will consume about N4.5 million worth of refractories when they are brought to use [11]. This indeed is discouraging, irrespective of the huge availability of clay raw materials in Nigeria as well as intellectual researchers for production of refractory products of high quality and standards.

In light of this situation, there has been an upsurge of interest in the area of developing good refractories from large deposit of clay spread across every region in Nigeria in the last two decades. This includes some local clay deposit from Benue state and Ikete Ekiti which have been investigated with good refractory and mechanical property result to meet local need [12, 13]. Yaim et al. also reported that clays from Chanchanga, Bida Suleya and Zungeni all in Niger state have better refractory and physical properties compared with imported ones [14]. In response to their development, the industrial potential of more clay deposit in Nigeria needs to be investigated [14].

Owing to the tremendous demand of refractories in most glass, metal industries and so on, researchers at various place in Nigeria have been motivated to analyze different clay deposits from different parts of the country in other to ascertain their suitability in refractory application. Therefore, this study aimed to characterize Ishiagu clay deposit from Ebonyi State in other to substantiate its utilization in refractory applications.

Materials and methods

Sample collection and pretreatment algorithm

Ishiagu clay was obtained from Ishiagu in Ivo local government area in Ebonyi state, Nigeria by random sampling at different points. The clay was taken at a depth of 1.55m and mixed properly to obtain a uniform and homogenous sample. The cone and quartered method was used to obtain a representative sample as described by Abubakar et al. [9]. The method involves pouring the sample to take a conical shape, dividing it into quarters, discard the two quarters sitting opposite each other while the other two are combined to constitute a reduced sample, this is continued until an appropriate sample size remains. 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 0.425mm 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^{o}C for 3hrs, pulverized and passed through a mesh sieve of size 0.18mm. 1.6kg of the clay was weighed and mixed with appropriate amount of water to make it plastic for the moulding process.

Physicochemical characterization algorithm

Molding of the test pieces

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

Making moisture determination

This was determined by weighing the cylindrical test pieces immediately after molding and recording as the wet weight W_o. The test pieces air dried for 24 hrs and then dried in an oven at 105^oC 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 (%) = $(W_o-W_i)/W_o*100$

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 venier 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 = Ho/Hi

Determination of modulus of rupture

Five long rectangular pieces were made and air dried for 7 days after which they were oven dried at 105^oC until a constant weight was obtained. Four of the pieces were fired to their respective temperatures of 800, 900, 1000, 1100^oC 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) = $3PL/2BH^2$

Shrinkage determination

Immediately after molding of the rectangular test pieces, a vernier caliper was used to insert a 5m mark on each of them; this was recorded as the original length *Lo* (cm). The test pieces were then air dried for 7days and then dried in an oven at 105°C until a constant weight was obtained. The shrinkage from the 5cm mark was then determined and recorded as the dried length, *Ld* (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 5cm mark were then determined and recorded as the fired length, *Lf* (cm). The shrinkage was then calculated:

Dry Shrinkage (%) = 100*(Lo-Ld)/Lo

Linear Shrinkage (%) = 100*(Ld-Lf)/Ld

Fired Linear Shrinkage (%) = 100*(Lo-Lf)/Lo

Determination of water of absorption

The fired test pieces obtained after firing were then weighed and the weight recorded as dry weight, *M1* (g). Thereafter, the test pieces were soaked in water for one hour, then removed, cleaned and weighed immediately and recorded as soaked weight, *M2* (g). The water of adsorption was then calculated:

Water of Absorption (%) = 100*(M2-M1)/M1

Porosity and density determination

After the procedure described above was completed. The suspended weight of the test pieces were then determined by the use of a lever balance and recorded as *M3* (g). The apparent porosity, apparent density and bulk density were then calculated:

Apparent Porosity (%) = 100*(M2-M1)/(M2-M3)

Apparent Density = M1/(M1-M3)

Bulk Density = M1/(M2-M3)

Chemical analysis algorithm

0.2g of the clay was weighed into a beaker and 10ml of *aqua regia* (HCl + HNO_3 in the ratio 3:1 respectively) was added and digested in a hot plate in a fume cupboard. 10ml of Hydrofluoric acid was also added to aid the digestion process. After digestion, 30ml of de-ionized water was added and the mixture filtered through a filter paper into a 250ml 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 (Buck scientific model 210-VGP). The concentration of metal oxide in the clay was expressed in mg/L.

Results and discussion

The result for the characterization of Ishiagu clay deposit is shown in Table 1.

Table 1. Physicochemical properties of Ishiagu clay

Parameters	**Value**
Al_2O_3 (%)	21.7
SiO_2 (%)	48.3
Fe_2O_3 (%)	6.8
CaO (%)	0.9
K_2O (%)	2.7
Na_2O (%)	2.9
MgO (%)	1.2
MnO (%)	0.3
LOI (%)	14.9
Colour before firing	Brown
Colour after firing	Reddish brown
Refractoriness (^oC)	1200
Modulus of Plasticity at 900^oC	1.21
Making moisture (%) at 900^oC	24.84
Wet-Dry Shrinkage (%)	2.5
Dry-Fired Shrinkage (%)	10.56
Total Shrinkage (%)	12.8
Apparent Porosity (%)	27.02
Apparent density (%)	2.37
Bulk Density (%)	1.73
Water Absorption (%)	15.62
Modulus of Rupture (Kg/cm^3)	16.50
Green modulus of rupture (Kg/cm^3) at 900^oC	2.32

The major compositions of Ishiagu clay were silica and alumina while the other metal oxides are present in negligible proportions. This makes the clay fall under the class of Alumino-Silicate refractories. The silica content of Ishiagu clay was found to satisfy the clay requirement for paper (45.0-45.8%) and paint (45.3-47.9%) [15]. However, the silica level was found to be lower than that required for glass (80-90%), refractory bricks (>51.7%) and high melting clay (53-73%) [16]. The alumina composition of Ishiagu satisfies the requirement for high melting clay (16-29%) and glass (12-17%) but fell short of the standard required for production of ceramics (>26.5%), Refractory bricks (25-44%), paper (33.5-36.1%) and paint (37.9-38.4%) [15, 16]. Alumina content of clay is a strong indicator for its refractoriness. In other words, the higher the amount of alumina, the higher is the refractoriness of the clay [17]. Since the percentage of alumina present in Ishiagu clay was low it is likely to have moderate refractory properties. The Fe_2O_3 content of Ishiagu clay was found to be high and above the standard required for ceramics (0.5-1.2%), refractory bricks (0.5-2.4%), glass (2-3%) and paper (0.3-0.6%) but below the requirement for paper production (13.4-13.7%) [17]. Also, the clay can still be incorporated in the production of high melting clay which needs about 1-9% of Fe_2O_3 [15]. Moreover, such high level of Iron oxide usually give a reddish colour to the clay body when fired increasing its suitability in the production of ceramic products such as flower vase that appreciates such coloration [17].

The reddish brown colour of Ishiagu clay after firing can be attributed to the presence of Fe_2O_3 as shown in Table 1. This limits the use of this clay in making white ware products [18]. The high Iron oxide content also affects the high temperature characteristics of the clay eg fired strength thereby making it act as flux in high refractory materials (17, 19).

The presence of alkali oxides (CaO, K_2O and Na_2O) in reasonable amounts as shown in Table 1 indicates the tendency of Ishiagu clay to have low or moderate refractoriness. This is as a result of the use of these alkali oxides as mild fluxes which reduce the vitrification temperature and refractoriness of the clay during firing [20] by combining with the oxides of silica and alumina to form eutectics. Fig. 1 shows the quantity in which the metal oxides are present in Ishiagu clay.

The loss on Ignition (LOI) (14.9%) of Ishiagu clay met the standard requirement for the manufacture of ceramics (>8.18%), refractory bricks (8-18%), high melting clays (5-14%), 12.-15% recommended for Kaolinitic clays [15, 16]. This 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 [21]. A low LOI is desired in production of low porous ceramic products as higher LOI gives rise to higher porosity in the manufactured products because of loss of LOI components during firing. It is observed from Table 1 that Ishiagu clay has moderate refractoriness and did not show any sign of failure at 1200°C but at higher temperature (>1300°C), signs of failure was observed. This suggests that Ishiagu clay did not meet the standard requirement of 1580-1750°C for refractory materials [22]. This could be attributed to low alumina and presence alkali metal oxide fluxes in Ishiagu clay, which reduces its refractoriness. Similar refractoriness was reported by Etukudoh et al. [8] in the characterization of Ezzodo for its industrial potentials as well as Adiabo clay as reported by Abubakar et al. [22].

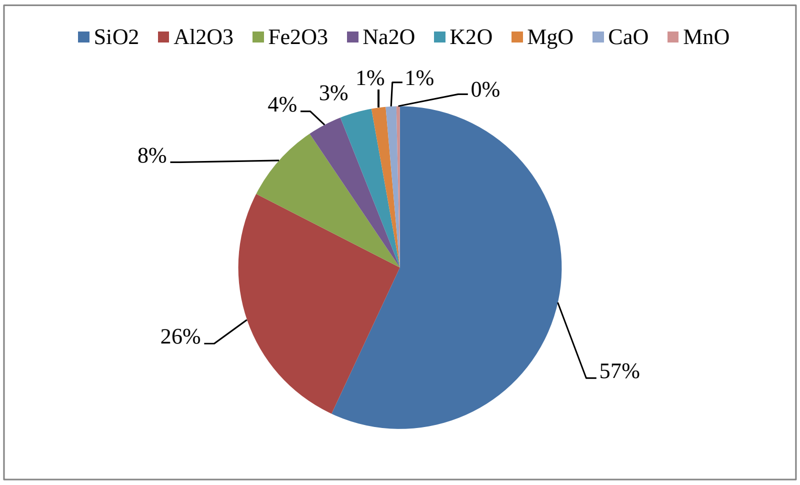

Figure 1. *Distribution of the quantity of metal oxides present in Ishiagu clay*

The physical properties of Ishiagu clay determined after firing at temperatures of 900 to 1200°C are shown in Fig 2-8.

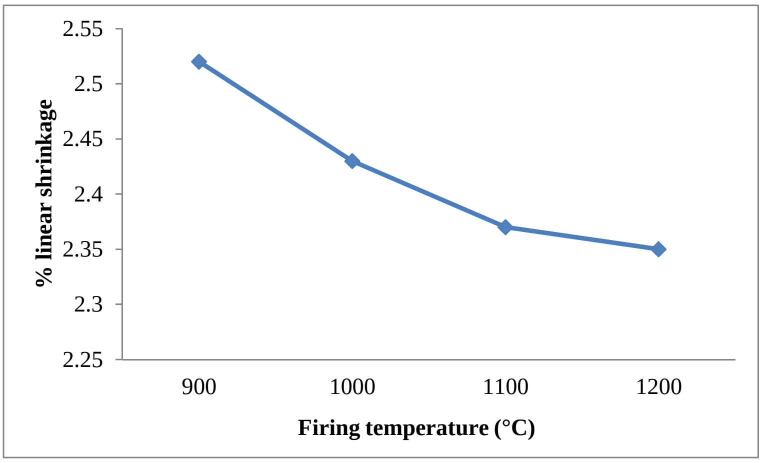

Figure 2*. Effect of firing temperature on the linear shrinkage of Ishiagu clay*

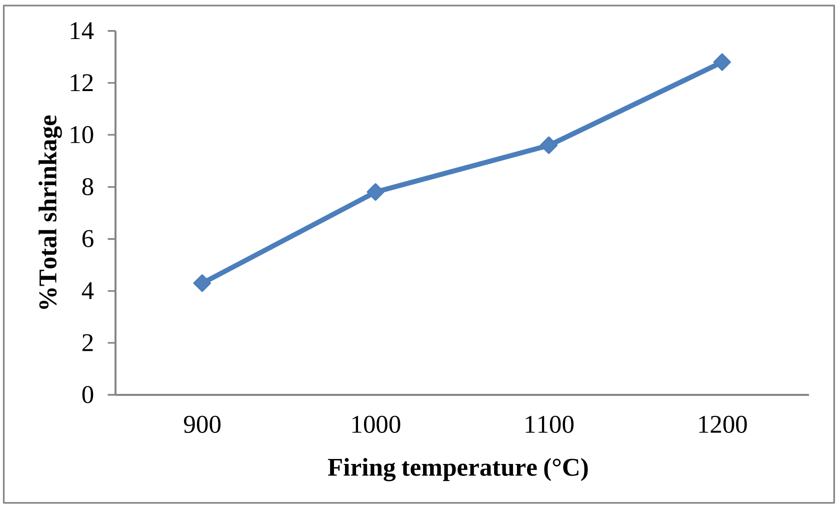

Figure 3. *Effect of firing temperature on the total shrinkage of Ishiagu clay*

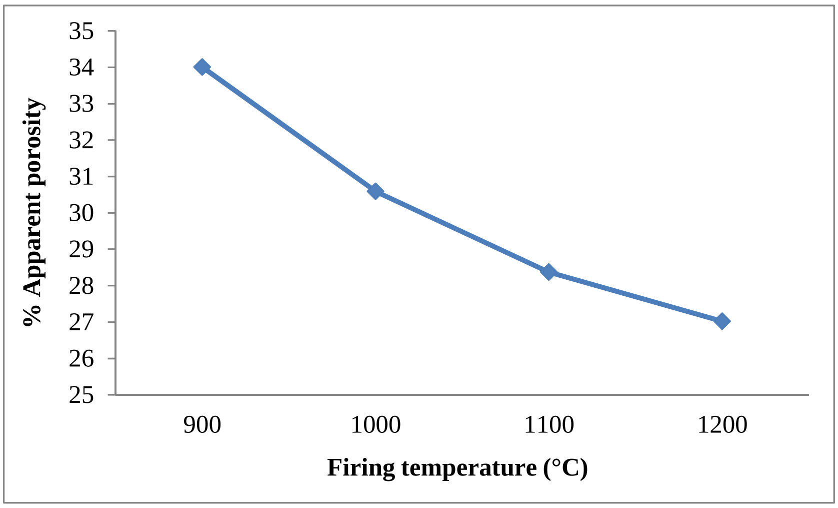

Figure 4*. Effect of firing temperature on the apparent porosity of Ishiagu clay*

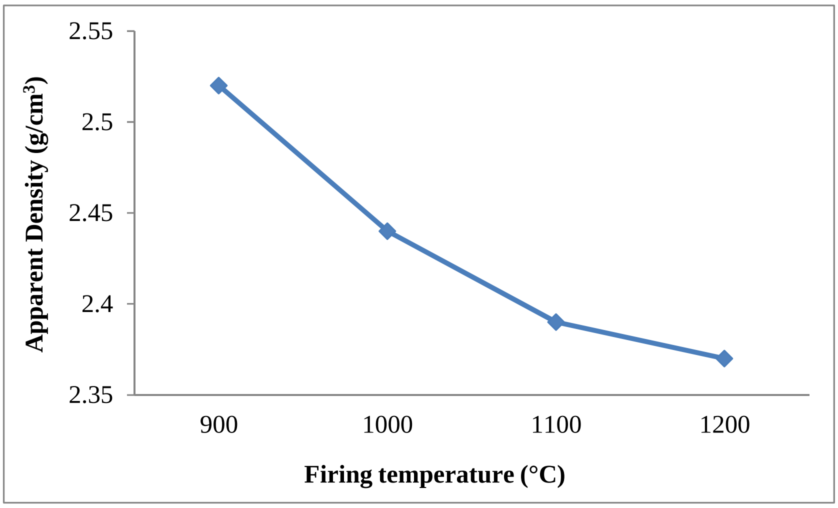

Figure 5. *Effect of firing temperature on the apparent density of Ishiagu clay*

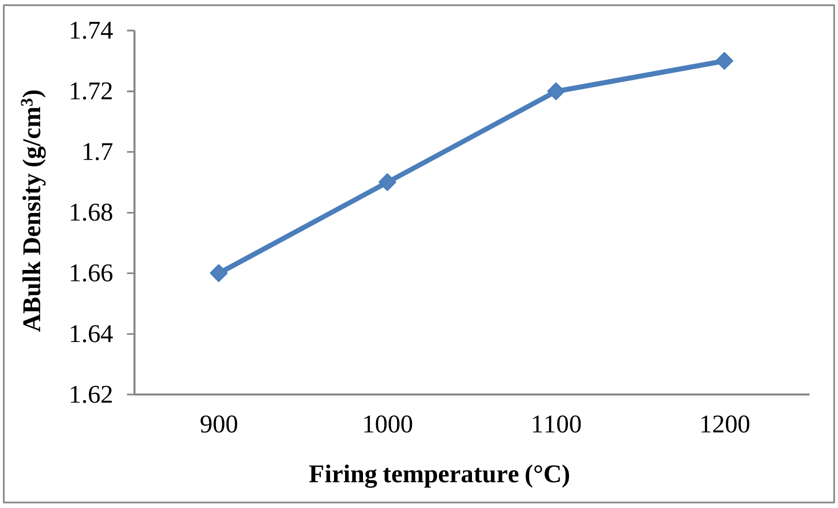

Figure 6. *Effect of firing temperature on the bulk density of Ishiagu clay*

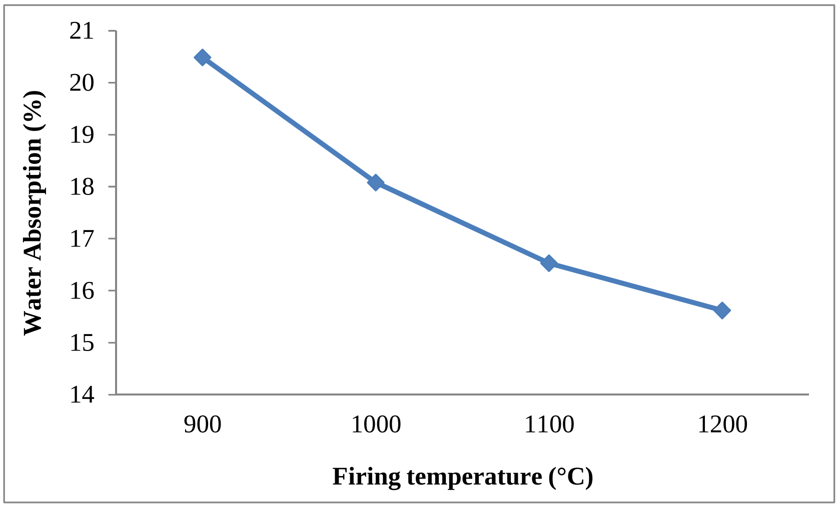

Figure 7. *Effect of firing temperature on the water absorption of Ishiagu clay*

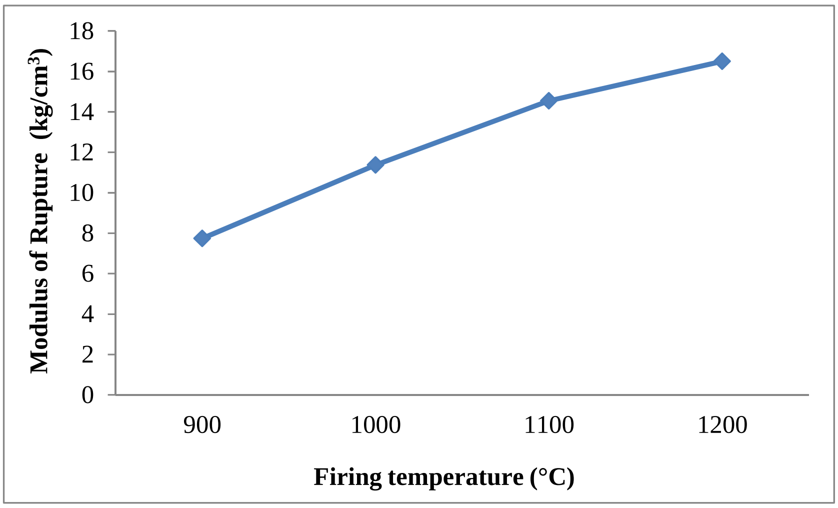

Figure 8. *Effect of firing temperature on the modulus of rupture of Ishiagu clay*

Shrinkage of a clay particle is one of the important factors to be considered. This is because low shrinkage gives rise to good insulating characteristics [23] creating a desired finished product. Figure 2 shows the result of dry-fired (linear) shrinkage after firing at different temperatures. It is observed that an increase in the linear shrinkage of the clay from 2.98 to 10.56% with increasing temperature from 900 to 1200°C was obtained. The firing shrinkage in a furnace is related to the total mineralogy of the clay body in which almost every mineral constituent of the original clay stay apart. As the firing temperature increases the individual grains in the compacted clay body may shrink within themselves but overall shrinkage of the clay takes place when the reaction between adjacent grains occurs [24]. The value obtained at the highest temperature was high. This could be because of fineness of the particles; finely-grained particles shrink more than those of coarse grain [25]. Additionally the mineral composition and presence of mica and quartz, which are main components of rocks underlying the area, can also affect the volume shrinkage.

The linear shrinkage of Ishiagu clay was within the range of 4-10% required for fireclays [8]. Moreover the values were also within the standard of 7-10% for aluminosilicates and kaolinites [26]. This suggests that Ishiagu clay is not only an aluminosilicate clay but also may be from a kaolinite origin. Figure 3 also shows an increase in the total shrinkage of ishiagu clay from 4.3 to 12.8% with increase in firing temperature. The total shrinkage and wet-dry shrinkage is not considered as an important factor when describing the quality of clay because of the variation in their value with the making moisture during firing [22]. As shown in Table 1, the making moisture of 24.84% was obtained for Ishiagu clay that is high and accounts for wet-dry and total shrinkage values recorded because of removal of water during drying.

Porosity is used for measuring the degree of vitrification and directly indicates volume shrinkage. The apparent porosity of Ishiagu clay with increase in firing temperature is shown in Figure 4. A decrease in the porosity from 34.01 to 27.02% with increase in firing temperature (from 900 to 1200°C) was observed. This decrease is because of increase in shrinkage with increasing temperature as shown earlier, which give rise to the sticking together and decrease in porosity of the clay particles. This is because as the samples shrink with firing temperature, the number of pores is reduced. The values of apparent porosity was within the recommended value for the manufacture of fire bricks (20-80%) [27] as well as 20-30% and >23.7% for production of fire clays and siliceous fire clays respectively [8]. The clay can be optimized in the production of insulating properties by the addition of some carbonaceous materials to help improve its porosity and insulating property since the higher the pores in between the clay particles, the higher its ability to trap heat.

The result of the bulk density of Ishiagu clay is shown in Figure 6. This property is important in the transportation or handling of a refractory material. It is affected by different factors such as particle size, treatment during manufacturing and nature of materials in the clay samples. The bulk density value of the sample showed an increase from 1.67 to 1.73g/cm^3 with increase in firing temperature (Figure 6). This shows that the clay is more closely packed and dense as the shrinkage increases thereby increasing the strength of the clay as a whole. The apparent density follows an opposite trend giving rise to a decrease from 2.52 to 2.37g/cm^3 as the firing temperature increases (Figure 5). This is anticipated as apparent density always follows an opposite trend to the bulk density of fired clay bodies. The bulk density of Ishiagu clay obtained at 1100°C and 1200°C of 1.72 and 1.73g/cm^3 respectively met the internationally accepted standard of 1.7-2.1g/cm^3 required for building and fire clays [28]. Also the apparent density was within the standard range of 2.3 to 3.5g/cm^3 for building and fireclays [29].

Figure 7 showed the effect of firing temperature on the water absorption of Ishiagu clay. Similar trend as the apparent porosity was obtained in which a decrease in water absorption of the clay body with increase in firing temperature was observed. Water absorption decreased from 20.49 to 15.62 as the firing temperature increases from 900 to 1200^{o}C. This is because the water absorption depends on the presence of pores. That means the higher the apparent porosity of the fired body the higher the water absorption and vice versa. Since pores are responsible for absorption of water, the decrease in water absorption can be ascribed to a decrease in porosity of the clay as temperature increases.

Fig 8 showed an increase in the strength or modulus of rupture (MOR) of Ishiagu clay with increase in firing temperatures. The value of MOR increased from 7.74 to 16.50Kg/cm^3 as firing temperature increased from (900 to 1200^{o}C). This increase can be attributed to the compactness and rigidity of clay body as temperature increases giving rise to shrinkage of the particles. At a high temperature above 1000^{o}C, the amount of liquid formed increases and on cooling, this liquid solidified mainly to form glass which acts as a cement to bind the mass together thereby giving rise to great strength on the body [30]. The MOR obtained at all temperatures fell within the wide range of 1.4 to 105KgF/cm^2 required for manufacture of different products [19].

The modulus of plasticity of Ishiagu clay in this study was found to be 1.21, this is lower than that of 1.35 reported for Ezzodo clay [8] and 1.33 reported for Adiabo clay [22]. This low value of Ishiagu clay indicates poor plasticity which is undesirable because the clay will not have good workability and cannot be easily moulded into shape. The low plasticity of Ishiagu clay may be due to the high silica content and moderate alumina content recorded. Similar results have been reported by researchers [31].

Conclusions

Careful observation of the result showed that Ishiagu clay contains aluminium oxide (Al_2O_3) and silica (SiO_2) as main constituents making them suitable as aluminosilicates refractory materials. The clay also showed moderate refractory properties (1200^{o}C) which is below the standard requirement for refractory clay (1500^{o}C) but can be enhanced using other insulating materials. Analysis of the result shows that the physical properties of the clay fall within the specification except its low plasticity which can be as a result of high content of silica and moderate content of alumina. The clay was found to be suitable for the production of ceramics, high melting clay, tiles, refractory bricks and colored vaze but not completely. This means that other materials can be used as enhancers to the clay to obtain the desired properties. For Ishiagu clay to be used as furnace lining material, the percentage composition of impurities such as Fe_2O_3 (6.8%) has to be reduced to minute amount by magnetic sieve. The composition of alumina (21.7%) needs to be increased to specification by mixing it with other refractory clay with higher percentage composition of aluminum oxide.

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