

## Application of Nkwo-Alaike Clay for the Production of Ceramic Wares Using Cullet as Sintering AID

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**ABSTRACT :** Nkwo-Alaike clay which was collected from Imo State, South-East Nigeria, was characterized and cullet was added as sintering aid (flux) in order to decrease the firing temperature thereby saving cost of fuel utilization. The cullet collected were milled into powder and batches containing mixture of 10%, 20%, 30% cullet powder with clay were prepared and moulded under 30MPa in order to evaluate the effect of cullet powder on sintering of the ceramic products. The samples produced were gradually fired to 900-1050°C with soaking time of 1hour at 5°C/min. Physical and thermal properties of the samples were investigated, from the results, the composition containing 20% cullet which was fired at 1050°C before its maturity had the best mechanical strength (13.80–28.61MPa) and no pinholes or cracks were recorded. The overall result show that, the firing temperature was reduced from 1150°C to 1050°C, making it possible to use the Nkwo-Alaike clay with cullet addition for the production of wall or floor tiles at 20% addition.

**Keywords:** cullet, flux, sintering, soaking time, strength, tiles.

### I. INTRODUCTION

Most solid residues generated by both public and private activities are sometimes being disposed in landfills. The availability of glass wastes from the recovery of landfills is expected to increase with the activities of glass producers [1], these needed to be converted for economic purpose, as in ceramic production.

Ceramic products are widely used in homes, as wall and floor tiles, water closet (WC), Jugs, plates, etc. Clay is mostly used as the raw material for their production. They are manufactured using high amount of fluxing agents like, sodium and potassium feldspars, nefeline, talc and ceramics fritz [2]. Ceramic tiles have various characteristics and it can be used in many different places because of its high mechanical resistance and surface hardness [3].

The main constituent of clay is silica (SiO<sub>2</sub>) and alumina (Al<sub>2</sub>O<sub>3</sub>) with the addition of other materials to attain the desire product standard. In this work, waste glass (cullet) powder which contains about 71% silica (SiO<sub>2</sub>) was added to clay in various proportions for the production of ceramic tiles in order to determine its effects on the ceramic properties. The production of tiling materials are one of the priorities of the traditional ceramic industry [2]. As a result, several studies related to the substitution of conventional raw materials in tile-making (i.e. clays, sands and feldspars) by other natural resources or industrial wastes were carried out during the last decade [1,3,4]. Promising results have been obtained using glass cullet waste [3, 5,11] cathode ray tube (CRT) glass [1, 6], different industrial residues [4] and various volcanic rocks [2]. In the work carried out by [1,3,6], the optimal amount of the replacement, that is, cullet or CRT were between 5–10 wt.%.

The crystalline proportion of the final ceramic products may be increased by using glass-ceramic frits [7] or compositions with high crystallization trends [8]. In this case, due to re-crystallization processes during the heat treatment, as the amount of residual amorphous phase decreases while increasing the crystalline proportion will have a positive effect on the mechanical properties of ceramic products [9]. The major focus of these studies was to use Nkwo-Alaike clay base with cullet powder as alternative flux.

### II. MATERIALS AND METHOD

The materials used for this study include: clay materials and broken bottles (cullet). The clay materials used was collected from Nkwo-Alaike deposit in Imo State; the depth is 2.0m down the threshold and 7m interval from three different points using digger and shovel. The broken bottles used as additive was collected from the immediate environment.

The waste broken bottles collected were washed with Omo-Detergent and water to remove contaminants in them after which they were air dried. Hammer crusher machine was used to crush the air dried bottles before it was disc milled for 5 hours (five hours) to fine powders. The powders were sieved with a 0.8mm mesh size in order to get the finer samples that will be added to the clay samples before pressing.

These materials were prepared and tested in accordance with [10] to determine their suitability for the production of ceramic wares. In carrying out the test, the following were determined; fired linear shrinkage (F.LS), compressive strength (CS), water absorption (WA), bulk density (BD), apparent porosity (AP), thermal shock resistance, refractoriness, moisture content and loss on ignition (LOI), and as well as the chemical analysis. The detail analyses on the experiments are enumerated below.

The cullet collected were milled into powder and sieved. Hundred percent (100%) of the original materials (Nkwo-Alaike clay) collected was prepared and formed into tiles as a control sample. The clay as a base material was mixed with cullet in various weight fractions of 90:10; 80:20; and 70:30.

A mould pattern or die for the ceramic tile was constructed specially using mild steel plate of the dimensions: length 14cm, width 12cm, depth 5cm, and thickness 1cm of two pieces each. The plates were machined to give the squareness and depth required. After which it was mounted on ceramic tile press to begin the production of the tile samples.

The clay was mixed with the cullet (ceramic mass) in batches as indicated above. The clay and the cullet with 6-8% water content (200ml of water) were homogenized in a Bluebird-Model 12SS clay mixer and the green sample with initial dimension of (5.08cm x 5.08cm) and (2.5cm x 2.5cm) were obtained by pressing; using hydraulic ceramic tile press of 10-50MPa capacity at 30MPa. Four specimens were pressed from each sample mixed in order to study and analyze the effect of cullet addition on the clay, especially, the firing temperature. Table 2.1 shows the mix ratios in percent by weight of the clay sample and cullet used in the research.

**Table 2.1: Percent by weight of the clay and cullet**

S/N	Specimens	Mix Ratio		
		Clay (%)	Cullet (%)	Water (ml)
1	N	100	0	200
2	N <sub>1</sub>	90	10	200
3	N <sub>2</sub>	80	20	200
4	N <sub>3</sub>	70	30	200

The tiles produced were air dried for 14 days after which they were oven dried at a temperature of 200<sup>0</sup>C for 3 days in order to remove the moisture content. The oven-dried tiles were fired in a gas kiln for a period of 8hrs at gradual of 800<sup>0</sup>C, 900<sup>0</sup>C, 1,000<sup>0</sup>C and 1,100<sup>0</sup>C respectively and as well soaked at 800<sup>0</sup>C for 2hours to produce biscuit wares. The Biscuit wares were allowed to cool for 24hours before removing them from the kiln.

### 2.1 Fired Linear Shrinkage

Test pieces of the ceramic mass were made into standard slabs of 5.08cm x 5.08cm and 2.5cm x 2.5cm. The pieces were marked along a line to be able to determine the shrinkage degree from the original position after the application of heat. The distance between the two ends of the slabs was measured with Vernier Calipers after which, the samples were air dried for 24 hours and oven dried at 110<sup>0</sup>C for 24 hours. They were then fired at 1,100<sup>0</sup>C for 6 hours. The samples were cooled to room temperature and measurements of the degree of shrinkage were taken. The fired linear shrinkage was calculated using equation (2.1).

$$\text{Fired shrinkage} = \frac{(DL - FL)}{DL} * 100 \quad (2.1)$$

where, DL = Dried Length; FL = Fired Length.

### 2.2 Compressive Strength

Test pieces were prepared to a standard size of 76.2mm cube on a flat surface. They were dried and fired in a Nabertherm (1999) model muffle resistance furnace at 1,100<sup>0</sup>C and the temperature maintained for 6 hours. The pieces were then cooled to 27<sup>0</sup>C and then placed on a compressive tester (Testometric M-500-25KN) and load applied axially by turning the land wheel at a uniform rate till failure occurs. The manometer readings were recorded and comprehensive strength (CS) was calculated using equation (2.2);

$$C.S = \frac{Max .Load (KN )}{C .A (m^2)} \quad (2.2)$$

where, C A = cross-sectional area; CS = compressive strength.

### 2.3 Water Absorption Test on the Biscuit Wares

The biscuit wares were trimmed and dressed, then each of them were weighed and then soaked in a bowl of water for 2hours and then removed, wiped with dry foam and weighed in accordance with [10].The water absorption was calculated using formula (2.3).

$$WA = \frac{B - A}{A} * 100 \quad (2.3)$$

where, A= is weight of biscuit ware before soaking (g); B= is weight of biscuit ware after soaking, (g).

### 2.4 Bulk Density

Samples of the clay materials measuring 60mm x 60mm x 15mm were prepared. The specimens were air dried for 24hours and then oven dried at 110<sup>0</sup>C, cooled in desiccators and weighed to the accuracy of 0.0018 (Dried weight); after which the specimens were transferred to a beaker and heated for 30 minutes to help get rid of the trapped air. The specimens were cooled and soaked weight (W) taken. The specimen each was then suspended in water using beaker placed on a balance. The suspended weight(s) each piece was taken and the bulk density was calculated using equation (2.4).

$$\text{Bulk Density} = \frac{D_{pw}}{(W - S)} g / cm^3 \quad (2.4)$$

where, D= Dried weight; W= Soaked weight; S=Density of water.

### 2.5 Apparent Porosity

Representative of test pieces of the ceramic mass were prepared, samples of cylindrical shape of 1.65 cm diameter were moulded in dry pressing under 30MP and air-dried for 24 hours. The pieces were then oven dried at 110<sup>0</sup>C for 24hours and then fired at a temperature of 1,100<sup>0</sup>C, cooled and transferred into desiccators and weighed to nearest 0.01g. The specimens were then transferred into 250ml beaker in empty vacuum desiccators. Water was then introduced into the beaker until the test pieces were completely immersed and allowed to soak in boiled water for 30 minutes, agitated from time to time to assist to release trapped air bubbles. The soaked weight (W) was recorded. The specimens were then weighed suspended in water using beaker placed on balance. This gave suspended weight(S); the apparent porosity was calculated using equation (2.5):

$$\text{Apparent porosity} = \frac{(W - D)}{(W - S)} * 100 \quad (2.5)$$

where, W = Soaked weight; D = Dried weight; S = Suspended weight.

### 2.6 Thermal Shock Resistance

Test pieces measuring 50mm x 75mm were prepared. The pieces were inserted in a Nabertherm (1999) model muffle resistance furnace, after drying, which has been maintained at regular temperature of 900<sup>0</sup>C for 10 minutes. There were then removed with a pair of tongs from the furnace one after the other and cooled for 10 minutes. The process continued until test pieces were readily pulled apart in the hands. The numbers of heating and cooling in cycles for each piece were recorded as its thermal shock resistance.

### 2.7 Refractoriness Value of the Ceramic Mass

The refractoriness of the ceramic mass was determined along with the known Standard (Pyrometric Cone Equivalent, PCE) in which the test pieces were prepared and mounted on a refractory plaque along with some PCE, that is, one whose melting point is slightly above and slightly below that expected of the test cones (ASTM. Designation C24). The plaque was then put inside the furnace and the temperature was raised at a rate of 100<sup>0</sup>C per minute. The test continued until the tip of the test cone had bent over level with the base to evaluate its refractoriness value.

### 2.8 Moisture Content Analysis

The ceramic mass were air dried and weighed ( $W$ ) and then placed in a Nabertherm (1999) model furnace which was heated to a constant temperature of 110°C for 24hrs. The sample was taken out cooled in desiccators and re-weighed ( $W_1$ ). The loss in weight gives the amount of moisture content (MC) which can be expressed as percentage of initial clay sample. The following expression as is in equation (2.6) was used for the calculation:

$$M.C = \frac{W - W_1}{W} \times 100 \quad (2.6)$$

where, MC= is the moisture content (%);  $W$ = is the weight of the sample before drying (g);  $W_1$ =is the weight of the sample after drying (g).

### 2.9 Chemical Analysis

Chemical analyses of the materials were carried out using Atomic Absorption Spectrophotometer (AAS, PG 990 AFG) in order to determine the clay chemical and cullet compositions. Loss on Ignition (LOI) was also carried out on the samples and the results are shown in Tables 3.1-3.2.

### 2.10 Biscuit Firing

The pressed tiles were fired in a gas kiln for a period of 8hours at gradual temperatures of 800, 900, 1,000 and 1,100°C. It was soaked at 800°C for 2hours to obtain biscuit wares. The Biscuit wares were allowed to cool for 24hours before removing them from the kiln; Table 3.4 shows the result of the biscuit wares.

### 2.11 Glazing

The glazing was carried out using 25g of powder glaze which was milled for 1hour in the ball milling machine after which, 400litres of water was added and mixed to form a homogenous mixture before sieving the mixture with 0.8mm sieve. The biscuit wares were dipped in glaze paste at interval of 10 seconds beginning with the biscuit that has the highest water absorption capacity. The samples were fired in a gas kiln and the glaze mass melted at a temperature of 1,025°C. The samples were removed from the kiln after 12hours of cooling to avoid cracking. The aim of glazing was to make the tile resistant to dirt, stain and water and as well as giving an esthetic look.

## III. DISCUSSION OF RESULTS

In this work, Nkwo-Alaike (NA) clay obtained from Imo State and broken bottles (cullet) were characterized. The results of the chemical analyses of the raw materials used are presented in Tables 3.1-3.2. Table 3.1 depicts the chemical composition of the waste glass, containing a relatively large percentage of  $\text{SiO}_2$  (71.08%) in the material with 1.05% ( $\text{Al}_2\text{O}_3$ ), 0.76% ( $\text{MgO}$ ), 11.19% ( $\text{CaO}$ ), 0.02% ( $\text{ZnO}$ ), 14.52% ( $\text{Na}_2\text{O}$ ) and 0.01% ( $\text{K}_2\text{O}$ ). The presence of alkaline and alkaline earth oxides in the broken bottles (cullet) composition will act as fluxing agents helping the sintering process of the ceramic products on the addition of the cullet[11].

The percentage composition of silica ( $\text{SiO}_2$ ) present in Nkwo-Alaike clay is 56.7% (see Table 3.2). Silica content above 46.5% indicates free silica (quartz) in the system which will enhance the ceramic properties [11, 12], therefore, silica content in NA will improve ceramic properties of the sample investigated.

The alumina ( $\text{Al}_2\text{O}_3$ ) content of NA fell below the recommended value of 26.70% as shown in Table 3.2. For good characteristics clay, the alumina should have a chemical composition of 26.70 % or above [12]. Low percentage of alumina reduces the coefficient of thermal expansion of ceramic products as a result of the reaction between alumina and potassium ion [13].The  $\text{Fe}_2\text{O}_3$  (1.76%) for the NA clay is slightly higher than the recommended value of 1.60%. Composition of  $\text{Fe}_2\text{O}_3$  less than 2% in clay will lead to whitish color after firing, while that above 2% will tend to change the colour to brownish or ruby-red as a result of oxidation [14].The percentage of  $\text{CaO}$  and  $\text{MgO}$  in the clay sample is within the standard value; 0.3% and 0.2%.respectively. High percentage of  $\text{CaO}$  and  $\text{MgO}$  may increase the shrinkage value of the materials when fired.

As observed from the graph (Figure 3.1), L.O.I. increased with increase in temperature for the entire specimen (10%, 20% and 30%) of cullet. At 1,000°C and above, which is the sintering temperature; the loss of mass was constant, indicating that the reaction (dehydration, burning-off of the impurities, etc.) is almost completed. Increasing the amount of cullet in the mixture reduces loss of mass as observed in the research. The values for L.O.I obtained for different batches of the mixtures were within the range as recommended [15].The moisture content of the clay sample is 9.5% which is within the recommended range of 8-10% an indication that a moderate addition of water will be required.

The chart showing the comparative analysis of the physical properties of the clay sample is displayed in Figure 3.5. The average fired linear shrinkage for the samples are within the recommended values of 4–6 % [16, 17] (see Table 3.3). Higher shrinkage value may result in warping or cracking of the ceramic products during firing. 20-30% addition of cullet in NA clay recorded no visible change in the fired linear shrinkage of the mix below 1,000°C. At 1,000°C and above, there was an increase in fired linear shrinkage for samples with 10-30% cullet which may be due to the presence of fluxing oxides (Figure 3.3). Firing at 1,200°C, the fired linear shrinkage went above the recommended maximum limit of 6% which led to cracking of the ceramic products [18].

The apparent porosity (A.P) for NA clay is 27.77% which falls within the standard values of 10–30 % which is the recommended value [15]. With the increase in firing temperature, the value for A.P tended to decrease and this may be attributed to the densification of the samples that occurred at a greater stage of the firing. The apparent porosity of the mix (N1, N2, and N3) was approximately 30% at 800°C and 900°C. At 1,000°C and above, there was a continuous fall in A.P for different cullet percentages added. At 1,200°C, a greater decrease in A.P (A.P < 15%) was recorded at 30% cullet addition due to the burning off of some impurities. On the other hand, low percentage of apparent porosity enhances the entrapping of gases in the ceramic products which may be responsible for large number of pinholes on the glazed tiles (N3) and this phenomenon will adversely affects the life span of the tiles when in use [19].

The average bulk density of the clay is shown in Table 3.3 and this value fall within the recommended values of the range of 1.7–2.1g/cm<sup>3</sup> for ceramic tiles [15]. This shows that the clay can be used for the purpose it was intended for. However, the ceramic mass containing 10-30% cullet present a decrease in bulk density with increasing firing temperature, which may be due to the presence of some impurities, such as, TiO<sub>2</sub>, CaO and K<sub>2</sub>O.

The compressive strength of NA clay was 27.60MPa and it was within the standard values of 22.9-59MPa [15]. Thus, the NA clay when processed into tiles will have strong resistance to load. The strength characterizations of the blended samples show that, the compressive strength varies from 13.8–27.60MPa with respect to firing temperature (Figure 3.4). For the samples fired at 900°C, there was an initial increase in strength with the addition of the cullet. Firing to 1,050°C witnessed gradual increase in strength for 10%-30% cullet addition.

The thermal shock resistances of the samples were within the acceptable values of 26-30 cycles [20]. This result is a pointer to a better performance of the ceramic products when used for the purpose they were intended for.

As shown in Figure 3.2, a reasonable decrease of water absorption was observed for all samples as the percentage of addition of cullet increases. This decrease was found to be more pronounced on firing at 1,025°C and 1,100°C. This may be due to cohesion between the particles of the mix.

Biscuit firing of mix N1, N2, and N3 came out without crack on the ceramic bodies, while samples N had cracks and pinholes. The cracks may be due to the original nature of virgin material. Blending with cullet showed lower pinholes compared with N. It can be observed from Figure 3.2 that, the blended samples have low water absorption compared with original material. The water absorption experienced a significant reduction over a short range of temperature. It means that the addition of cullet to NA clay will drastically reduce the firing temperature and consequently the time of firing.

**Table 3.1: Chemical Analysis of the Broken Bottles (Cullet) as compared with Breef (2009)**

S/No.	Parameters	Level detected (%)	Level detected(%) {BREF,2009}
1.	Siliconoxide (SiO <sub>2</sub> )	71.08	71-75
2.	Aluminum oxide (Al <sub>2</sub> O <sub>3</sub> )	1.05	-
3.	Magnesium oxide (MgO)	0.76	-
4.	Calcium oxide (CaO)	11.19	10-15
5.	Zinc oxide (ZnO)	0.02	-
6.	Sodium dioxide (Na <sub>2</sub> O)	14.52	12-16
7.	Potassium oxide (K <sub>2</sub> O)	0.01	-

**Table 3.2: Chemical Analysis of Nkwo-Alaike Clay**

S/No.	Parameter	Chemical Analysis (%)	
		Nkwo-Alaike	*Standard
1	SiO <sub>2</sub>	56.7	40-60
2	Al <sub>2</sub> O <sub>3</sub>	26.55	25-45
3	Fe <sub>2</sub> O <sub>3</sub>	1.76	1-5
4	MgO	0.30	0.2-1
5	CaO	0.20	0.2-1
6	Na <sub>2</sub> O	0.85	<2.0
7	K <sub>2</sub> O	0.08	<2.0
8	TiO <sub>2</sub>	1.68	<2.0
9	LOI	9.76	5-14

\*Grimshaw R.W (1971); Chesti J.H (1973)

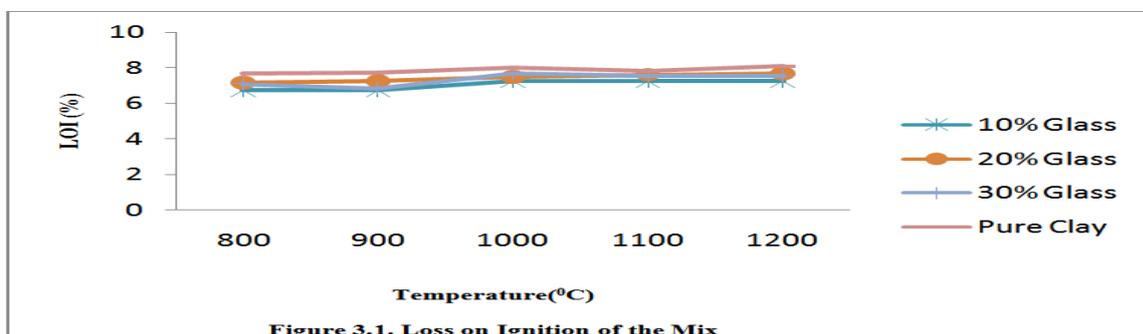
**Table3.3: Physical and Thermal Properties of Nkwo-Alaike Clay**

Properties	Nkwo-Alaike	*Standard
Fired Linear Shrinkage (%)	4.0	4-6
Apparent Porosity (%)	27.77	10-30
Bulk Density (g/cm <sup>3</sup> )	1.72	1.7 – 2.1
Compressive Strength (MPa)	27.60	22.9-59
Thermal Shock Resistance (No of Cycles)	26	20-30
Moisture Content (%)	9.50	8-12.00
Water Absorption (%)	2.64	2.6-2.7

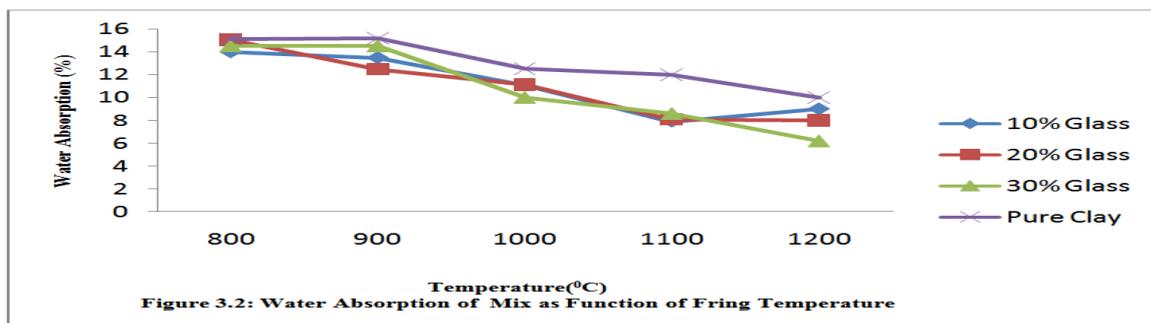
\*Grimshaw R.W (1971); Chesti J.H (1973)

**Table 3.4: Biscuit Firing Result**

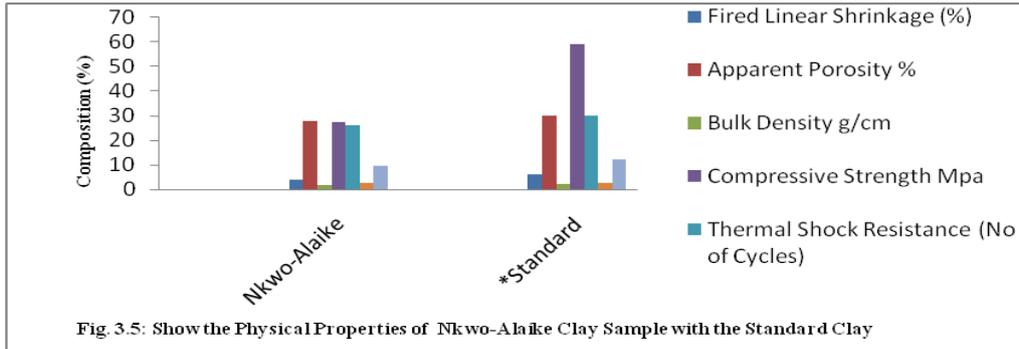
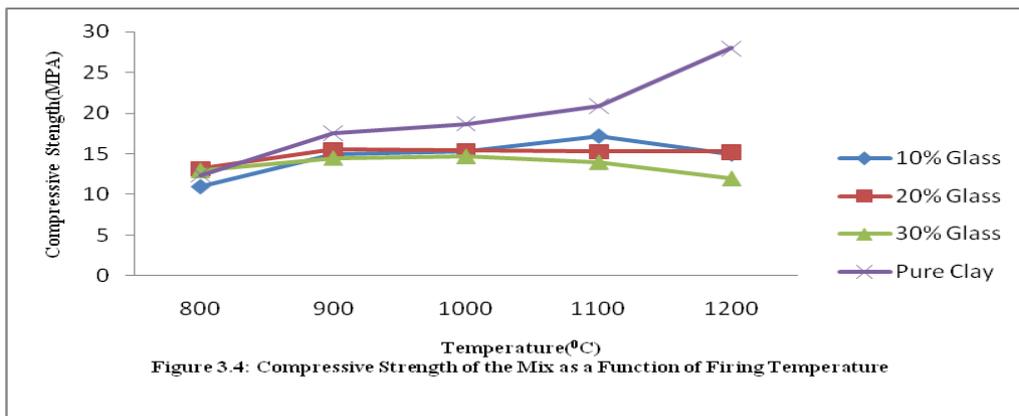
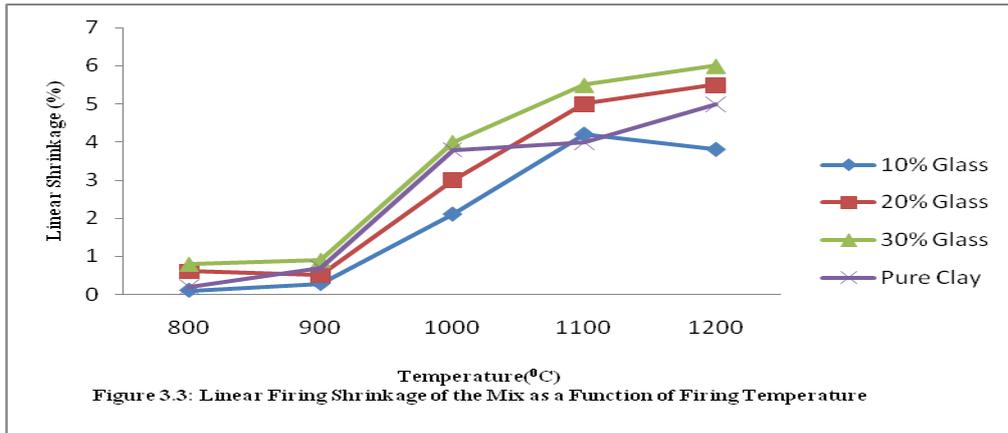
S/N	Test Specimen (Clay : Cullet)	Observations	
		Biscuit Firing	Glaze Firing
1	N (100:0)	Has cracks	-
2	N1(90:10)	Fine	Shiny surface
3	N2(80:20)	Fine	Few pinholes
4	N3(70:30)	Fine	Few pinholes



**Figure 3.1. Loss on Ignition of the Mix**



**Figure 3.2: Water Absorption of Mix as Function of Fring Temperature**



\*Grimshaw R.W (1971); Chesti J.H (1973)

#### IV. CONCLUSION

From the results obtained from all the experiment carried out, it shows that, it is possible to utilize broken bottles (cullet) as alternative raw material (reducing the primary raw material usage) to lower the sintering temperature during the production of ceramic wares. With the addition of cullet which served as fluxing agent during the firing process, the firing shrinkage was curtailed as well. The additions of cullet lower the water absorption of the tiles as observed.

It was established that during firing, cullet accelerate the densification process in the system and this was clearly shown in the results. The addition of cullet to clay for the production of ceramic tiles has economic value, that is, reduction of the firing temperature and time leading to energy savings. There was also an improvement in the physical properties of the ceramic tiles produced. The compressive strength of the samples produced range from 13.68–28.61 MPa and the results obtained generally confirmed that, blending of Nkwo-Alaike with cullet for the production of ceramic tiles will yield good ceramic products.

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