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Comparative Study on the Addition of Cullet to Mowe and Ibamajo Clays for Ceramic Tiles Production

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Abstract

Waste broken glass known as cullet is one of the most common silicate wastes and has high volume in the urban wastes collection. Substantial amount of this wastes are being dumped in the city's garbage deposits. In the last few decades, there has been considerable research on the reuse of glass wastes as aggregate to produce glass-ceramic, mortars, cement and concrete. This work studies the effect of waste glass (cullet) addition in the production of ceramic wall tiles using Mowe and Ibamajo clays in Ogun State, Nigeria. Pulverized waste glass was prepared by sieving using a 1.8mm mesh size in order to get the required samples that will be used for pressing the ceramic tiles. Ceramic bodies (CB) were pressed (5.08cm x 5.08cm) and (2.5cm x 2.5cm) using a ceramic mass with 0, 10, 20 and 30 % of cullet powder, respectively. These were cured; oven dried and fired between the temperature ranges of 800-1200°C. The result of the linear shrinkage shows an increase in the linear shrinkage with increase in glass addition but this was still within the standard value. The average compressive strength value of Mowe and Ibamajo was in accordance with the standards also ($14.68N/mm^2-28.61N/mm^2$). The study provides a base for the conversion of wastes to wealth, specially, the recycling of broken glass with the results obtained.

Keywords: cullet, recycling, aggregates, ceramic bodies, wall tiles

1.0 INTRODUCTION

Broken glass is one of the municipal solid wastes which are hazardous to human beings. These types of wastes are threat to human safety and a burden on the environment due to its non-degradable nature and disorderly accumulation of wastes on sites. The increase in population growth lead to rise in the number of people using disposable and returnable materials in the cities and subsequently increasing the volume of residues produced (ABC- Associação Brasileira de Cerâmica,2002).

Ceramics products are alternative items in which glass waste can be added due to the wide use of pottery and ceramic wares in every home all over the world. The addition of glass wastes in different proportions may improve the performance of ceramic products and thereby contribute to the economic growth of a nation. Normally, ceramic tiles are produced by using clay, quartz, feldspar, etc., but these materials can be substituted with wastes materials, such as, waste glass powder. The waste glass powder can be used to replace the traditional fluxing agents, like, feldspar without changing the process.

Ceramic products are manufactured using high amount of fluxing agents, like, sodium and potassium feldspars, nefeline, talc and ceramic fritz (Gennaro *et al.* 2003). Ceramic tiles have various characteristics and the products can be used in homes, offices, etc., because of high mechanical resistance and surface hardness (Tucci *et al.*, 2004; Malleucci *et al.*, 2002).Soda-lime glass had no significant effect on semi-finished ceramic products but it influence remarkably on the firing behavior, increasing shrinkage and closed porosity, decreasing of open porosity and bulk density and lowering mechanical and tribological performances (Yurkov *et al.*, 2006)

The pioneer work that was carried out by Tyrell *et al.*, 1972 at the Tuscaloosa Metallurgy Research Laboratories has shown the benefit of adding waste glass powder in clay. The addition of glass reduces firing temperature and time resulting in a significant increase in production capacity of a plant (Tyrell, 1972). More than 95% of all manufactured glass is made from sodium oxide, calcium oxide, and silicon dioxide, commonly referred to as a soda-lime-silica composition. Soda lime glass is a vitreous silicate and these vitreous silicates are generated during the maturation of clay bodies and acts as fluxes during firing, thereby reducing clay body maturation temperatures. These were strong evidence that the addition of soda lime glass to clay body products could increase the efficiency of the process and therefore be a value-added application for recycled glass (Clean Water Center, 2009). Enhanced quality of ceramic products, economic savings of ceramic raw material as a result of glass addition, reduction in HF emissions and less glass waste disposal problems have been attributed to the use of the glass waste (Oliveira *et al*, 2004).

The aim of this work is to compare the effects of cullet addition on the ceramic properties of Ibamajo (IB) and Mowe (MO) clays from Ogun State for the production of wall tiles. The clays and glass used were characterized to determine their chemical compositions and physical properties.

2.0 Experimental Method 2.1 Material and Equipment

The materials used for this research work include: Clay materials and broken bottles (waste glass). Clay materials obtained from Ibamajo and Mowe in Ogun State and broken bottles were used in this research. Clay samples collected from Ibamajo, Mowe in Ogun State was at a depth of 1.5 meter using digger and shovel. The broken bottles used as additive was collected from different locations in Oshodi area of Lagos State (figure 2.1). These materials were tested in accordance with ASTM C-279 to ascertain their suitability for the production of ceramic wall tiles.



Figure 2.1: Broken glass collected

Waste bottles collected were cleaned by washing with H_20 and soap to remove contaminant in them and were air dried. The Hammer mill machine (Figure 2.2) was used to crush the air dried broken bottles before it was taken to the ball milling machine where it was ball milled for several hours (five hours) to fine powder (Figure 2.3). The broken bottles in powdered form were sieved using 0.8mm mesh size in order to get the required samples that will be used for pressing the ceramic tiles.

Hundred percent (100%) of the original materials collected were prepared before mixing it with grinded broken bottles in various volume fractions of 90:10 (B1, M1); 80:20 (B2, M2); and 70:30 (B3, M3) using the clay as the base.



Figure 2.2: Crushing of the broken bottles Figure 2.3: Ball milling the crushed bottles

Three specimen were produced for each sample of clay in order to study and analyze the effect of adding broken bottles to the clay samples under the following criterion; the percentage of milled broken bottles used to produce the specimens were 0% (B, M), 10% (B1, M1) 20% (B2, M2) and 30% (B3, M3) replacements in Ibamajo and Mowe clays respectively. In accordance with ASTM C-279, the clay samples and the milled glasses were measured at different ratios and mixed with 15wt% of H₂O properly. The samples were then tested for Fired Linear Shrinkage, Apparent Porosity, Compressive Strength, Thermal Shock Resistance (using muffle furnace), Water Absorption, Moisture Content, Refractoriness (using muffle furnace), and Loss on Ignition, Moisture Content and Bulk Density. The chemical composition was carried out using Atomic Absorption Spectrophotometer (AAS, PG990AFG). The result of the chemical analysis and physical analysis are shown in Table 3.2 and Table 3.3.

A 10MPa hydraulic press was used for pressing the tile specimen, cleaned, labelled and dried, subsequently, it was placed in an oven at 110°C for 24 h and fired (Figure 2.4). The oven-dried tiles were fired in a kiln to produce biscuit wares. The biscuit wares were fired at 800, 900, 1000, 1100 and 1200°C, respectively at a heating rate of 10°C/min up to 110°C, kept at this temperature for 30 min during the operation, and then heated up again to the firing temperature at 10°C/min and kept at this temperature for 2 h. After firing, the furnace was turned off and the ceramic wares were allowed to cool to ~ 60°C in the furnace before removal.



Figure 2.4: The pressed tiles with different composition of clay and milled Glass

2.1.1 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) and marked along a line to be able to determine the shrinkage degree from the original position after heat treatment. The distance between the two ends of the slabs was measured with Vernier Callipers. The samples were air dried for 24 hours and oven dried at 110° C for 24 hours. They were then fired at 800-1,100°C for 6 hours. The test pieces were cooled to 27° C and measurements of the degree of shrinkage were taken. The fired linear shrinkage was calculated using equation (2.1).

$$= \frac{(DL - Fl)}{DL} * 100$$
(2.1)

Fired shrinkage = DL ... Where, DL = Dried Length, FL = Fired Length

2.1.2 Apparent Porosity

Representative of test pieces of the ceramic mass were prepared and air-dried for 24 hours. The pieces were then oven dried at 110° C for 24hours and were fired at a temperature of 800-1,100°C; cooled and then transferred into desiccators and weighed to nearest 0.01g (Dried weight). The specimens were then transferred into 250ml beaker in empty vacuum desiccator. 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 in releasing 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 (2.2):

$$= \frac{(W-D)}{(W-S)} * 100$$
(2.2)

Apparent porosity = Where,

W = Soaked weight; D = Dried weight; S = Suspended weight

2.1.3 Bulk Density

Samples of each ceramic mass of standard sizes (5.08 cm x 5.08 cm) and (2.5 cm x 2.5 cm) were prepared. The specimens were air dried for 24hours and then oven dried at 110° C, cooled in desiccators and weighed to the accuracy of 0.0018 (Dried weight); after which the specimen was 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 was then suspended in water using beaker placed on a balance. The suspended weight(S) was taken and the bulk density was calculated using equation (2.3);

$$Bulk Density = \frac{D_{pw}}{(W-S)}g/cm^{3}$$
(2.3)

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

2.1.4 Compressive Strength

Test pieces of the ceramic mass were prepared to a standard size of sizes (5.08cm x 5.08cm) and (2.5cm x 2.5cm) on a flat surface. The test pieces were fired in a muffle resistance furnace at $800-1200^{\circ}$ C and the temperature maintained for 6 hours. The pieces were then cooled to 27° 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. Compressive Strength (CS) was calculated from equation (2.4);

Max.Load(KN)

 $C.A(m^2)$ CS =

Where, CA = cross-sectional area; CS = compressive strength. The Flexural strength was determined after firing the ceramic wares with control and automatic data recording, using EMIC instrumentation.

2.1.5 Thermal Shock Resistance

Test pieces of the ceramic mass measuring sizes (5.08cm x 5.08cm) and (2.5cm x 2.5cm) were prepared. The pieces were inserted in a muffle resistance furnace, which has been maintained at regular temperature of 900°C for 10 minutes. The pieces were removed with a pair of tongs from the furnace one after the other and then 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.

2.1.6 Refractoriness Value

The refractoriness of the ceramic mass was determined along with the standard Pyrometric Cone Equivalent (PCE) in which the test pieces were prepared and mounted on a refractory plaque along with some PCE Standard, that is, one whose melting point is slightly above and slightly below that expected of the test cone (ASTM Designation C-24). The plaque was then put inside the furnace and the temperature was raised at a rate of 100^oC per minute. The test continued until the tip of the test cone had bent over to level with the base

2.1.7 Moisture Content Analysis

The ceramic mass were air dried and weighed (W) and then placed in a furnace (Nabertherm, 1999 series) which was heated to a constant temperature of 110°C for 24hrs. The sample was taken out and 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 sand sample. The following expression (2.5) was used:

$$M.C = \frac{W - W_1}{W} \times 100$$

Where, M.C 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).

2.1.8 Water Absorption Test on the Biscuit Wares

The biscuit wares were weighed and soaked in a bowl of water for 2hours and then removed, wiped with dry foam and weighed. Water absorption of the biscuit wares carried out was calculated using (2.6).

$$\underline{B-A}$$

WA = A * 100.....(2.6)

Where, WA is water absorption, A is weight of biscuit ware before soaking. (g); B is weight of biscuit ware after soaking, (g).

2.1.9 Loss on Ignition

The Test sample was dried at 110°C and cooled. The sample in a clean and dried porcelain crucible was heated in a muffle furnace to a temperature of 900°C for 3hours. The loss on ignition (LOI) was calculated using equation (2.7).

$$LOI = \frac{m_2 - m_3}{m_2 - m_1} \times 100\%$$
 (2.7)

Where, m1 is the weight of the crucible (gm);m2 is the weight of clay and crucible (gm);m3 is the weight of the dried clay and crucible (gm).

2.1.10 Glazing

For glazing application, 25g of powder glaze was weighed and 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. Glazing was carried out by dipping the biscuit wares into a glaze paste by hand.

3.0 Results and Discussion

The chemical properties of the broken bottles and clays were determined. The results of the chemical analyses of the raw materials used are presented in Tables 3.1 and 3.2. From Table 3.2, the percentages of the silica (SiO₂) present in the clay samples were 54.0% and 56.0% for Ibamajo and Mowe, respectively. The chemical compositions of the clay samples closely followed those established for typical Kaolinite. Silica content above 46.5% indicates free silica (quartz) in the system which will enhance the ceramic properties (Singer et al., 1993). Table 3.1 depicts the chemical composition of the waste broken bottles, containing a relatively high percentage of SiO₂ (71.08%) in the material with 1.05% (Al₂O₃), 0.76% (MgO), 11.19% (CaO), 0.02% (ZnO), 14.52% (Na_2O) and 0.01% (K₂O). The presence of alkaline and alkaline earth oxides as shown above in the glass

composition will act as fluxing agents helping the sintering process of the ceramic products on the addition of the glass powder (Costa *et al.*, 2009).

The alumina (Al_2O_3) content of Ibamajo is lower than the recommended value of 26.70% as shown in Table 3.2. For good characteristics clay, the alumina should have a chemical composition above 26.70 % (Ryan, 1978). Low percentage of alumina lowers the coefficient of thermal expansion of ceramic wares and this may be as a result of the coupling reaction between Al^{3+} and K^+ in the system (El-Kheshen, 2003). The Fe₂O₃ content for Ibamajo and Mowe clays are 1% and 1.5% respectively, which fall within the standard value. The composition of Fe₂O₃ that is less than 2% indicates that the clay will usually burn white on firing while that above 2% will tend to change colour of the products to brownish or ruby-red depending on the percentages involved (Singer et al., 1993; Gupta, 2008).

The CaO and MgO content for the two clay samples were within the standard value except CaO in Mowe that falls below; Ibamajo; CaO= 0.3% and MgO = 0.5%, Mowe has CaO = 0.16% and MgO = 0.50% respectively. High percentage of CaO and MgO increases the shrinkage value of the materials but the value here would aid the bonding effect of the system as it was low (Gupta, 2008).

It was observed from the graph (Figure 3.1), that, loss on ignition (L.O.I.) increased with increase in temperature for the entire specimen (10%, 20% and 30%) of glass addition. At 1,000°C and above, the loss of mass was constant, indicating that the reactions occurred mostly >1000°C. Kaolinite in general, shows substantial loss of mass at temperatures below 600°C (Teixeira *et al.*, 2008). Increasing the amount of glass in the mixture reduces loss of mass as observed in the research. The values for L.O.I obtained for Ibamajo and Mowe clays are within the range recommended (Grimshaw, 1971).

The average fired linear shrinkage for the two clay samples are within the recommended values of 4-6% (Chester, 1973) (see Table 3.3). Higher shrinkage value may result in warping and cracking of the tiles during firing. At 800°C, for all the percentages of glass powder added to Ibamajo and Mowe clay, there was no change in fired linear shrinkage. 20-30% addition of glass powder to Mowe clay recorded constant fired linear shrinkage. At 900 and 1000°C, 10% glass powder in Ibamajo exhibited decreased fired linear shrinkage which may be attributed to continue reaction in the system. At 1,000°C and higher, there was an increase in fired linear shrinkage for samples with glass powder (10-30%) due to the presence of fluxing oxides, Figure 3.4-3.5.Between 950°C and 1,200°C, vitrification occurs due to the release of silicon oxide (SiO₂) which reacts with free oxides of alkaline earth metal and iron oxides forming glassy phase (Souza *et al.*, 1989). Some of the alkaline oxides are present in clays and some are released due to the breaking down of the structure of clay minerals on firing. Firing at 1,200°C, the fired linear shrinkage went below the recommended maximum limit of 6% and this agreed with Macedo *et al.*, 1996.

Apparent Porosity (A.P) of Ibamajo and Mowe clays has values of 16.11% which is below the recommended values. With the increase in firing temperature, the values for A.P tended to decrease and this may be attributed to the densification of the samples that occurred at higher temperature. The apparent porosity of the sample was approximately 20%, at 800 and 900°C. At 1200°C, a greater decrease in AP (AP < 15%) was observed for 30% glass powder in both Ibamajo and Mowe samples. Low percentage of apparent porosity enhances the entrapping of gases in the samples, which may be responsible for large number of pinholes on the glazed tile (B1, B3) and this phenomenon will adversely affects the life span of the tiles when in use (Gupta, 2008). The values may be enhanced by addition of fine grain additives, such as saw dust, baggage and rice husk. The average bulk density of Ibamajo and Mowe samples are shown in Table 3.3 and these values fall within the recommended values of the range of 1.7 - 2.1g/cm³ for ceramic tiles (ASTM C-279).

The compressive strength values obtained from Mowe samples were within the standard values of range between 22.9-59N/mm² (ASTM C-67) but Ibamajo samples have values below the recommended standard and this may be due to the high content of Na₂O and the lower silica content of 54% recorded. Thus, the Ibamajo tiles may have the lesser resistance to load, tension and shear stresses than Mowe tiles .Compressive strength was significantly changed at firing temperatures of 1100° C and 1200° C, due to the formation of liquid phase attributed to the glass powder. The incorporation of 20% and 30% glass powder showed better results compared to 10%. The strength characterizations of the samples show that the compressive strength of the blend sample varies between 14.68N/mm² – 28.61 N/mm² with respect to firing temperatures. The thermal shock resistances of the two clay samples were within the acceptable values of 26-30 cycles (De Bussy, 1972). This indicates that, there would be better performance of ceramic products when in use.

Flexural strength (FS) determined in a three-point flexure test was greater than 5 MPa, for the entire ceramic tiles. The minimum limit value for wall tile is 6.5 MPa (Souza *et al.*, 1989). Firing at 900°C showed a FS greater than the limit value for the production of wall tiles. At 1000°C, the graph shows that, the values were equal for the clays with and without glass powder (Figures 3.6 & 3.7). From 1100 to 1200°C, an increase in FS was recorded. At 1200°C, the Ibamajo tiles with glass powder show a FS greater than that of Mowe samples with the same percentages of glass powder (Figure 3.7). This value for FS is within the limit value for the production of pressed ceramic tiles of the BIII group (ASTM, C-74).

The moisture contents of the two clay samples were within the recommended range of 8-10% for moisture content of tile products (Allen, 1986). The moisture content of the clays collected from Mowe and Ibamajo were 10% and 8.6% respectively. This implies that Ibamajo will require more water for proper mixing of the material during processing.

The biscuit firing results show that, samples B2, M, M2 and M3 came out fine without any crack, while samples, B, M, and M2 cracked after firing. This may be due to stresses build-up by non-uniform drying of one side of the piece to the other, which will manifest during firing. After firing and subsequent cooling, glaze paste was applied to the samples. The samples were allowed to dry slowly at a high relative humidity (for several days) in order to prevent cracking, which the pieces were fired in a gas kiln. The glaze melted at a temperature of 1,025°C and the samples removed from the kiln after 12hours of cooling to avoid cracking.

Tile Sample B1 (which is 10% glass powder in Ibamajo clay) came out with fine shining surface and few pinholes. The pinholes may be as a result of un-hidden air trapped in the system. Tile B2 (20% glass powder in Ibamajo clay) came out fine after glazing and firing. The glaze was evenly distributed on the surface due to substantial adequate bonding of the individual material in the sample during firing. Tile B3 (30% glass powder in Ibamajo clay) which has water absorption of 12.4% presented a shining surface with some pinholes which may be due to the entrapment of gases in the ceramic body. The presence of pinholes on ceramic body could affect the quality and durability of the products. Sample M1 (10% glass powder in Mowe clay) came out with a dull surface which could be attributed to poor absorption of the glaze mixture during dipping. B3 as well have dull surfaces. On firing between 950°C and 1,025°C, vitrification occurs due to the release of silicon oxide (SiO₂) which reacts with free oxides of alkaline earth metals and iron oxides, forming glass (Souza et al., 1989). **Table 3.1: Chemical Analysis of Glass Sample (Soda-lime glass) compared with major**

Table 3.1: Chemical Analysis of 0	Flass Sample (Soda-lime glass) compared with maj
Components of Soda-lime Glass (Bref, 2009)

S/NO	PARAMETERS	LEVEL DETECTED (%)	LEVEL DETECTED (%){BREF,2009}
1.	Silicon oxide $(Si0_2)$	71.08	71-75
2.	Aluminum oxide (Al_20_3)	1.05	-
3.	Magnesium oxide (Mg0)	0.76	-
4.	Calcium oxide (Ca0)	11.19	10-15
5.	Zinc oxide (Zn0)	0.02	-
6.	Sodium dioxide (Na ₂ 0)	14.52	12-16
7.	Potassium oxide (K ₂ 0)	0.01	-

Table 3.2: Chemical Analysis of Clay Samples

Sample Location	Chemical Analysis, (%)								
	Sio ₂	Al_20_3	Fe ₂ 0 ₃	Ca0	MgO	K ₂ O	Na ₂ O	Tio ₂	L.O.I
Ibamajo	54.0	22.80	1.0	0.30	0.20	0.30	1.0	0.86	16.73
Mowe	56.0	26.93	1.5	0.16	0.50	1.10	0.65	1.09	10.21
ASTM Standard	57.0	26.70	1.60	0.20	0.70	2.0	0.10	1.10	12.15
Table 2.2. Dhuring Duementing of the Class Semuclas									

Table 3.3: Physical Properties of the Clay Samples

Properties	Ibamajo	Mowe	Standard
Fired Linear Shrinkage (%)	4.00	6.00	4-6
Apparent Porosity (%)	16.11	16.11	30max
Bulk Density (g/cm ²)	1.76	1.72	2.3
Compressive Strength (N/MM ²)	14.68	28.61	22.9-59
Thermal Shock Resistance (No of Cycles)	30	30	20-30
Moisture Content (%)	8.60	10.00	8-12.00
Water Absorption (%)	2.60	2.67	2.6-2.7
Refractoriness (°C)	1500	1460	1400-1500















4.0 Conclusion

In view of the above results and discussion, the following can be reached:

- 1. Tiles B2, M, M2 and M3 samples came out fine without any crack on the ceramic bodies, while Tiles B, M, and M2 cracked after firing. This may be attributed to stresses build-up by non-uniform drying of one side of the piece to the other.
- 2. Addition of 10-30% of broken glass reduces the softening points of Ibamajo and Mowe clays in the range of 50-250°C and this amount of reduction are significant for energy savings. There was an improvement in the physical-thermal properties of the ceramic wares produced as recorded in Table 3.3.
- 3. The strength characterizations show that the compressive strength of the samples vary from 14.68 N/mm²-28.61 N/mm². The results are in tandem with the established technical characteristics standard recommended for ceramic wares production (Grimshaw, 1971).
- 4. The primary raw material usage is reduced by the addition of glass powder. It was established that, during firing, glass powder accelerate the densification process in the system and the results of tests show that the addition of broken glass powder in clay for the production of ceramic wares (tiles) has economic value.

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