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Original Research Article

Improving the Properties of Indigenous Wall Tiles using Recycled Waste Glass Bottles as Reinforcement/Glaze Material

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ABSTRACT

This research was conducted to improve the properties of indigenous wall tiles. Results obtained revealed that at 800 ^oC, the reinforced sample showed average linear shrinkage of 11%, increasing with cullet content (%) but showing reducing effect at elevated temperature of 1075 ^oC. Beyond 1075 ^oC, the samples with higher composition of cullet were found to have melted and fused together with high vitrification except sample of 50 % of clay with 10% of glass surviving. Results further revealed that the flexural strength of the tiles produced increased with the increase in glass reinforcement up to 20 % composition beyond which deterioration resulted owing to loss of surface water and dehydroxylation of clay causing break in homogenous interaction and propagation of cracks. It recorded its highest flexural strength of 47.01 kgF/cm^2 at reinforcement (glass bottle powder composition) of 20 % and least at 10 % composition where it had flexural strength of 37.4701 kgF/cm^2. The porosity and water absorption of the tiles decreased with the increase in the reinforcement (waste glass bottle powder composition). The porosity reduced from 16 to 6% at waste bottle composition between 10 to 25%, respectively: while the water absorption characteristics showing similar trend decreased from 8.2% to 2.0% at waste bottle composition between 10 to 25%, respectively. Glaze composition of 70% glass and 30 % clay depicted good and desirable glossy effect.

1. INTRODUCTION

Tiles are flat thin slab of fired clay, used to cover roofs, floors and walls. The use of Tiles has become a very prominent feature in the building industry. However, it must be noted that in these modern times, there are tiles of varying materials. There are wooden tiles, linoleum tiles, plastic tile and marble tiles. All these are products of scientific research. As pointed out by Ali (2004), the functional value of tiles is in its use in building construction. This is not only for their aesthetic values but load carrying and liquid impervious abilities. He observes that most modern houses are now embellished with tiles of various types, sizes and colours.

The use of tiles could be traced back in history to about 875 BC, when the Assyrians used glazed tiles to create wall relief decorations in their palaces. These reliefs tell stories to commemorate the military victories and hunting expeditions of their kings who ruled some twenty-eight hundred years ago in what is now Iraq (Lewis and Lewis, 2009).

Within Igbo cultural homestead, pot sheds have been found to be used as floor tiles within domestic homes. Pieces of broken pottery were used to pave the floor and entrance path to the living houses. In some homesteads, even oil palm kernel shells have been found to be used on floors as tiles (Mannan *et al.,* 2006; Olaniyi *et al*., 2019).

Basically, tiles are made of clay and fired to high temperatures to transform them into very hard-rock-like materials that are impervious to water (Idowu, 2014; Abuh *et al.,* 2019). Technically, firing converts the clay tile to ceramics: a state in which the clay product cannot disintegrate in water or any other liquid. The clay body could be a composition of earthen ware clay with the addition of kaolin and feldspar, to enhance its physical properties of texture, reduced shrinkage rate, green and firing strength. Several modifications of in tiles characteristics can be achieved depending on the intended application. In the presence other finely ground materials, vitrified wall tiles which is glass-*like* due to high temperature are *most* suitable for wet areas *like* bathroom, kitchen etc.

Waste glass bottle poses serious nuisance to the environment constituting waste management burden to man owing to its inert and non- biodegradable nature (Abuh *et al*., 2019). This makes its disposal very difficult as it can remain active in a dumpsite for a thousand years. Ceramics have been found to be alternatives for utilizing these wastes (Abuh *et al.*, 2019). Waste glass bottle crushed into different particle sizes often referred as cullet (ranging from coarse aggregates to very fine powder depending on the intended use) is commonly used as additive in tile reforming due to its low softening temperature (Furlani *et al.*, 2010). It has been shown that these new ceramics (cullet reinforced) can be formed with higher density, higher hardness, lower drying shrinkage, less water absorption and other good physical qualities (Loryuenyong *et al*., 2009; Sahar *et al.,* 2011)

Ali (2004) observed that "about 80% of tiles used in Nigeria today are imported from abroad, since the few tile producing industries cannot satisfy the huge demand for the product". This observation is very serious and poses a challenge to Nigerian contemporary potters or ceramists who no doubt are in a better position to assist in salvaging this aspect of Nigerian economy. Nigeria is blessed with abundant natural supply of clay mineral which should not be allowed to be waste but must be harnessed for indigenous tile production and establishment of cottage industries. Ali (2004) argued that the country blessed with such useful raw material as clay cannot afford to import tiles from Italy, Spain and China at very exorbitant cost. This new thrust towards local industrial scheme, will replace the age-old traditional pottery practice that is on the decline in most local communities. However, our local tiles production has not been able to compete favourably and even replace quality foreign products owing to issues of standardization and functional property improvement (Ali, 2004). This has resulted in preference being given to imported product in spite the attendant cost and economic sabotage (Idowu, 2014; Ibeh, 2019). To curb this ugly trend therefore, calls for devoted efforts on property improvement on our indigenous tiles to conform to standards and compete if not totally replace the imported counterparts.

Secondly, our environment is littered with broken bottles that pose very serious health hazard. These broken bottles could be recycled and utilized as reinforcement or glaze to enhance the qualities of our indigenous wall tiles thereby underscoring the waste to wealth concept. Hence, the aim of this study was to improve the qualities of indigenous wall tiles through the use of recycled waste bottle powder as reinforcement and glaze material.

2. MATERIALS AND METHODS

2.1. Materials

The basic raw material for this study is the raw clay which was dug from a site at Eha-Alumona in Nsukka Local Government Area of Enugu State, Nigeria. The other constituents are kaolin which contains silica and feldspar which also provides silica and alumina, waste broken coloured bottles that were crushed, ground and recomposed to form suitable reinforcement and glaze for the tiles, plaster mould and the metal die or cutter used in the production of the wall tiles from the clay slab. A kiln located in the foundry workshop of Institute of Management and Technology (IMT), Enugu, Nigeria was used for firing of the wall tiles to bring them to good finish. Kaolin was purchased from the retail outlet at Kenyatta chemical market Enugu, Nigeria, feldspar was obtained from the quarry along Enugu-Abakalliki road in Ebonyi State, Nigeria, while waste bottles were gathered from dump sites including those of brewery companies at 9th mile, Enugu, Nigeria. Veneer caliper, tape, gauges, digital weighing balance (0.1 g accuracy), stop watch etc. were among the apparatus used.

2.2. Design and Production of Mould

A designed metal cutter with definite pattern was used to produce the mould for the tile production. This metal cutter was constructed with ½" metal flat bar with a suitable welded onto the upper edge of the cutter. This cutter was used for cutting the tile shape from rolled out slab of the clay body. Before cutting out the shapes, the slab of clay body was allowed to harden to the leather hard stage which ensures that drying shrinkage will be reduced.

2.3. Sample Preparations

2.3.1. Clay sample

The raw clay was soaked in a large plastic bin containing sufficient water and allowed for about four days to properly disintegrate and become loose. After four days, the soaked clay was thoroughly stirred with a suitable pole to form a liquid slurry. This slurry was passed through a 30-mesh sieve into another large plastic bin. The sieving process helped to eliminate small clay lumps, stones, and all unwanted particles or impurities. The sieved clay was allowed to stand for about four days while the clay particles sediment and the clear water at the top was decanted using a siphoning tube. The de-watering process continued until a very thick mass of clay is left at the base of the bin. The thick mass of clay was poured onto a very clean concrete floor so that the excess water was absorbed leaving lumps of plastic clay. The lumps of clay were cut to pieces and set under the sun to dry. They were further broken to pieces, pulverised in ceramic containers and fired in a kiln to about 1105 ^{o}C to eliminate all the physical combined water. After cooling, they were crushed and ground in a mortar and graded by sieving with an 80-mesh sieve. This provided the earthenware clay in powder form to enable definite predetermined measures to be taken when compounding the clay body. Three components (clay, kaolin and felspar) (Plate 1) were mixed in different proportion of 3:1:1 of clay, kaolin and feldspar, to form the clay body for the production of the tiles. The ratio was to

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produce a workable clay body with adequate plasticity and low shrinkage rate. About 5 – 10 % water by weight was added to form a semi-dry composition suitable for dry pressing method of production. Grog from ground fired clay was also added to assist in drying and firing strength.


(a) Clay


(b) Kaolin

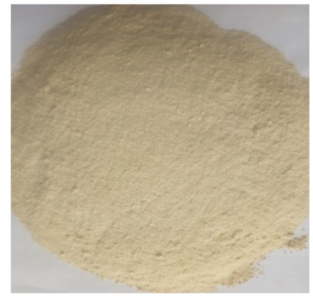
(c) Feldspar

Plate 1: Three components of clay body

2.3.2 Waste glass bottle (cullet)

Basically, the waste coloured bottles were of the malt and larger beer products. Malt bottles provided the brown colour while beer bottles provide the green colour. The broken bottles were carefully washed and allowed to dry. Using a metal pestle and wooden mortar, the broken bottles were crushed and ground to powder of different particle sizes (Plates 2 and 3). The powder was passed through a hundred mesh sieve to eliminate some larger particles. These larger particles were poured back into the mortar for further grinding. In this way, the coloured broken bottles were crushed and ground to powder differently. Further sieving was carried out with an 80 mesh sieve to secure a finer grade of bottle powder.


Plate 2: Coarse and fine particles of glass bottle powder (Green)


Plate 3: Coarse and fine particles of glass bottle powder (Brown)

2.3.3. Reinforced tile sample preparation

Samples of 60% clay 20% kaolin and 20% feldspar compositions (as control sample) and a clay body at 50% earthen ware plus 50% kaolin were used for the preparation of two set of tiles. The flat tiles of 15.6 cm and 9.4 cm dimension were produced by cutting out the piece from a sheet clay slab rolled out on a very wide board for each composition. This dimension includes 20% allowance for drying and firing shrinkages. After trimming and fettling, they were set out to dry. Other pieces that served as raw trials were also prepared from the same clay samples. After drying the shrinkage property of tiles were determined. Another set of samples were prepared using glass powder as reinforcement in clay body. Clay body of earthen ware clay and glass powder were produced in four different proportion at 50% clay + 10% glass powder, 40% clay + 20% glass, 25% clay + 25% glass powder while kaolin and feldspar were kept at their constant composition. These clay bodies were rolled out as sheet on a flat board from which stripes of clay tiles measuring 12 cm x 4 cm x 1 cm were cut and labeled according to the compositions. The dimensions adopted for the sample were informed by the test equipment and cutter design allowing for shrinkages. Bricks measuring 12 cm x 5 cm x 4 cm were also produced from clay mixed with 20% Grog and fire clay mixed with 20% grog (for purpose comparison). The cut tiles (Plate 4) were exposed to room temperature to dry. At this stage, each piece was fettled. Fettling is the trimming and smoothing of a leather hard clay product especially the sharp edges left by the moulds. This was done with a fine-bladed knife.

Plate 4: Cut samples

2.3.4. Cullet preparation as glazing material

Glass and clay were sieved with an 80 mesh sieve to obtain very fine articles size powder. The glass powder and the clay were mixed in their different proportion of 90% glass powder + 10% clay, 80% glass powder + 20% clay, 70% glass powder + 30% clay and a control mixed of 100% glass. The last mixture was 100% glass which was very difficult to apply because of its weight. All these mixtures were applied onto sample pieces of bisque ware and subjected to temperature of 1075 ^{o}C. Melted glass flows freely like a liquid because of the large quantity of flux and low quantity of alumina. The fluxing agents in the glass are sodium oxide, potassium oxide and calcium oxide. These constitute one of the major constituents of feldspar. While the other constituents are silica the glass former and alumina increases the viscosity or the thickness or hardness of the glass. Therefore, to increase the viscosity of the powdered glass, the alumina portion will be increased. The major source of alumina in this circumstance is kaolin which essentially is made up of silica and alumina. To this end, a percentage of kaolin was introduced into the powdered glass and tested for fitness on the tile. The recomposed glass mixed with water with a little addition of gum Arabic to enable it stick on to the tile at the raw stage. The water serves as the vehicle that transfers the glaze particles unto the ceramic ware. As the ware in its porosity absorbs the water, the glaze particle remains in position. A test was carried out by applying a thin layer of the liquid recomposed glass onto the surface of sample tiles. The tiles were placed in a slant in a test kiln and fired to 1075 ^{o}C. With the melted glass coat remaining in position is an indication that the re-composition of the glass to glaze is successful. However, the method of applying the glaze on the ceramic tiles was by dipping. This method ensures an even application of the glaze on the ceramic surface. As control, a tile sample was glazed with regular glazing material to be able to be compared with sample glazed with cullet.

2.3.5. Drying of samples

All the produced samples were exposed to room temperature to dry. At the end of drying, the amount by which the flat tiles have shrunken were determined. Thereafter, biscuit firing to temperature of 800 ^{o}C and glost firing to temperature of 1075 ^{o}C for the wall tiles that were produced in the kiln

2.4. Evaluation Procedures

The produced samples were subjected to physical and mechanical tests. They were evaluated for shrinkage, mechanical strength (flexural/modulus of rupture), apparent porosity and water absorption capacity as described by (Abuh *et al*., 2019)

2.4.1. Physical examination

The physical tests were carried out as described by Abuh e*t al.* (2014) before firing and after firing at the experimental temperatures. Samples were subjected to close observation for surface morphology to ascertain the effect of increase in cullet on physical appearance, the tendency of a uniformly distributed grains, contact surface, porosity and water absorption.

2.4.2. Determination of shrinkage

The percentage shrinkage was determined using Equation 1 following the procedures presented by Idowu (2014) and Abuh *et al.* (2019).

$$\text{Shrinkage, } S = \frac{P_L - D_L}{P_L} \times 100 \quad (1)$$

Where P_L is plastic length and D_L is the dry length. If the shrinkage is to be determined after firing to biscuit temperature of 800 ^{o}C, it is calculated thus

$$S = \frac{P_L - F_L}{P_L} x \ \frac{100}{1} \quad (2)$$

Where F_L is the fired length

2.4.3. Determination of flexural strength

Flexural strength (modulus of rupture) which is the amount of force (F) the tile can take without breaking or permanently deforming was calculated using Equation 3 (Idowu, 2014; Pena *et al.,* 2016; Ibeh, 2019; Abuh *et al.,* 2019).

$$\sigma = 3FL/2wd^2 \tag{3}$$

Where σ = flexural strength, (kgF/cm^2), L = length of the sample (cm), w = width of the sample (cm) and d = depth of the sample (cm)

2.4.4. Determination of water absorption

The procedure for determining water absorption consists of weighing the samples after they are completely dried (dry mass), and then immersing them in boiling water for 2 hr. The heating system is then switched off and the ceramic pieces remain immersed in water to cool naturally for 4 hr (water immersion line) for saturation. After this procedure, the excess moisture on the sample surfaces is removed with damp cloth and their mass is again measured (wet mass). The water absorption is calculated with the Equation (4).

$$WA = \frac{M2-M1}{M2} \times 100 \tag{4}$$

Where WA is the water absorption (%) M_1 is the dry mass (g) and M_2 is the wet mass (g).

2.4.5. Determination of apparent porosity

The vacuum method evacuates the air from a chamber with the tiles inside and then immerses the tiles in water following the procedure presented by Idowu (2014). Once again, the tiles are weighed before and water immersion to determine the apparent porosity (Alves and Boschi, 2015).

2.4.6. Micro-structural examination

Microstructural examination was conducted on the samples to ascertain the distribution/ dispersion of waste bottle powder reinforcement on the clay matrix. Samples of the produced tiles of different reinforcement and composition were treated and subjected to micro-examination. The specimens were mounted on the stage of the metallurgical microscope (AP2000MTI with Daheng digital camera of maximum magnification of X100) and adjusted to give a proper view of the structure and then it was captured as it appeared on the monitor of the attached system

3. RESULTS AND DISCUSSION

Table 1 presents the percentage shrinkage of different clay samples. Results revealed that at 800 ^oC, the earthen ware glass reinforced sample showed 11% shrinkage. The higher linear shrinkage was consistent with findings of Abuh *et al.* (2019) due to replacement of kaolin in the clay with glass powder which flow at high temperatures creating holes. It is also attributed to the high vitrification, densification and glassification of the clay particles intermingling with large quantity of iron oxide which is a major constituent of earthen ware clay (Abuh *et al.,* 2018). The iron oxide acts as a fluxing material and reduces the melting point of common red clay. At the end of firing at 1075 ^oC, the samples were found to have melted and fused

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together. Only the mixture of 50% of clay plus 10% of glass survived with very high vitrification. There was evidence of over firing attributable to the large quantity of iron oxide in the earthen ware clay (Abuh *et al.,* 2018).

Table 1: Percentage shrinkage of different clay samples at temperature of 800 °C

S/N	Sample	Plastic length	Dry/fired length	Shrinkage (%)
1	Clay brick	10	9.5	5
2	Fire clay brick	10	9.5	5
3	Tile samples	15.6	14.6	6.41
4	Glass Reinforced clay	10	8.9	11

Table 2 presents the mechanical properties of different tile samples. The flexural strength of the samples was found to increase with the increase in glass reinforcement. The samples recorded highest flexural strength of 47.01 kgF/cm^2 at glass bottle powder composition of 20% and least at 10% composition where it had flexural strength of 37.4701 kgF/cm^2. It was observed that beyond 20% glass powder composition, the strength decreased with loss of plasticity due to loss of surface water and dehydroxylation of clay causing break in homogenous interaction as the clay particles are forced apart weakening the strength. On the other hand, the porosity and water absorption of the tiles decreased with the increase in the reinforcement (waste glass bottle powder composition). The porosity reduced from 16 to 6 % at waste bottle composition between 10 to 25%, respectively. The water absorption characteristics decreased from 8.2 to 2.0% at waste bottle composition between 10 to 25%, respectively. Though these values were above the standard 0.5 % water absorption rate but there was significant improvement with introduction of cullet. The increase in strength with increase in glass/cullet at elevated temperature may be due to interaction of the oxides producing mullite and free silica in the form of cristobalite as suggested by Abuh et al. (2018). The silica content is increased with increase in cullet which is melted at high temperatures by interacting oxides to produce soda lime glass which confers hardness when cooled to ambient conditions. Increasing the glass powder tends to increase the amount of free silica present which melts with increase in temperature to form a uniform dense structure that greatly confers strength on the body.

Table 2: Mechanical properties of different samples at temperature >800 ≤ 1075 °C

Samples	Waste bottle powder composition (%)	Flexural strength (kgF/cm ^{2})	Apparent porosity (%)	Water absorption (%)
Control sample	0	31.20	42	28
A	10	34.47	16	8.6
B	15	39.95	13.4	
C	20	47.01	10	2.8
D	25	45.72	6	2

The values as shown in Table 2 show a decrease in porosity with increase in cullet. Porosity alone would have encouraged higher utilization of cullet as values tend to zero but cracks develop and propagate at increase beyond 20% cullet. However, with the recommendation of previous work, these cracks can be avoided at temperatures below 1000 °C (Abuh et al., 2019). It was further observed that water absorption decreased with increase in cullet. The absorption rate is tending to zero because of its highest vitrification and loss of porosity. The value obtained at 20% cullet at 1075 °C being 2.8 % was around the recommended value of 2.6-2.7% (Pena *et al.*, 2016; Chester, 1973).

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Table 3 shows the glazing composition and observed effect at 1075 ^{o}C on the samples. Upon glazing and after firing, the samples were allowed to cool. On examination, the 100 % glass melted and showed sign of running effect. It could not stick effectively on the vertical wall of the test sample. The second and third samples 90 % glass + 10 % clay, 80 % glass + 20 % clay, also melted and showed signs of running down the vertical walls of the list samples. The fourth sample of 70 % glass and 30% clay showed signs of higher viscosity. There was no sign of running effect. The flat tiles when glazed with the 70 % glass and 30 % clay mixture and subjected to a firing temperature of 1075 ^{o}C depicted evidence of improvement with glossy effect. The tiles produced from only the earthen ware body, showed signs of over firing and warpage. The tiles produced with the 50% earthen ware and 50% kaolin, showed signs of being normal. They did not show signs of over firing and this could be attributed to the presence of kaolin which is more refractory than earthen ware clay. Its refractoriness according to Idowu (2014) enabled the clay body to withstand the greater high temperature at 1075 ^{o}C. The tiles were able to withstand warpage. Therefore, 70% glass + 30% clay mixture, proved very adequate for the tile glazing. Tile melt was very effective and fitted the body without crazing and shivering.

Table 3: Glazing composition and observed effect at 1075 ^{o}C

S/N	Glass/clay mixture	Observed effect
1	100% glass on test piece	Melted completely with running effect
2	90% glass and 10% clay mixture	Melted completely with running effect
3	80% glass 20% clay mixture	Melted completely with slight running effect
4	70% glass and 30% clay mixture	Melted completely with higher viscosity

4. CONCLUSION

Tiles produced from the composition of clay body and variation of 10 - 20% glass reinforcement, exhibited good quality with improvement in flexural strength, overall reduction in percentage shrinkage, reduction in water adsorption and apparent porosity with high densification due to cullet dispersion in the tile matrix. Improvement in strength with increase in cullet makes the tiles very suitable for flooring. However, beyond 20% glass powder composition, the strength decreased (from 47.01 kgF/cm^2 to 45.72 kgF/cm^2) with loss of plasticity and this unusual behaviour was due to loss of surface water and dehydroxylation of clay resulting in break in homogenous interaction as the clay particles were forced apart weakening the strength. It is therefore, recommended that tile reinforcement should be kept below 20% cullet compositions. Water absorption capacity significantly reduced (from 8.2% to 2.0%) with glass powder reinforcement of tiles at elevated temperature of 1075 ^{o}C owing to high vitrification leading to desirable loss of porosity thereby making it suitable for wall application to prevent wetting. Finally, though clay and glaze have the same constituents of silica (SiO_2) which is the glass former, composition of glaze with the 70 % glass and 30 % clay when applied on tiles and subjected to a firing temperature of 1075 ^{o}C depicted evidence of improvement on surface effect, aesthetics and water impermeability characteristics of wall tiles.

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6. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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