

Re-use of Waste Glass Bottles in enhancing properties of Metakaolin-based Geopolymers: Synthesis and Characterization

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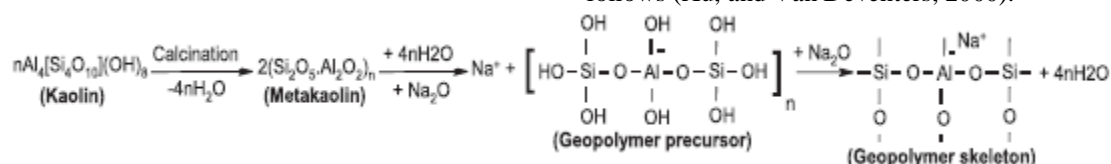
ABSTRACT: Geopolymer technology has emerged and is gaining international attention as it does not only contributes towards recycling, and the minimization of carbon dioxide emissions, but it also offers an economically feasible alternative to Ordinary Portland Cement (OPC) or high strength cement (HSC) due to its enhanced mechanical and chemical properties. This work is aimed at looking for an alternative to OPC due to the amount of carbon dioxide it produces as well as recycling of glass bottles. In this study, wasted glass bottles of particle size 63 μm up to 50 wt. % were added into 212 μm sieved Kaolin clay already calcined at 700 $^{\circ}\text{C}$ for 6 hours. The mixture for each ratio was dissolved in alkaline activator made up of mixture of 8 M NaOH and Na_2SiO_3 in the ratio of 1:1 in an electric mixer. This was moulded in a cylindrical container, demoulded on the third day into sealed nylons and cured at room temperature for 7, 14, 21 and 28 days. The compressive strength, water absorption capacity and density, were thereafter investigated. Prior to the geopolymer synthesis, the raw materials were characterized using FTIR and XRF to know the presence of essential functional groups and chemical composition respectively which qualified it as geopolymer binders. The compressive strength as high as $18.8 \pm 3.85 \text{ N/mm}^2$ (28 curing days), percentage water absorption as low as $1.64 \pm 0.19 \%$ and bulk density of 1783.05 ± 10.3 were achieved for geopolymers containing 15 % glass content. Hence, it is feasible to use Coca cola waste glass bottles up to 50 % wt. inclusion for manufacture of geopolymers.

Key words: Alkaline activator; Carbondioxide; Geopolymer; Kaolin; Waste glass

I. INTRODUCTION

Nowadays, there is great concern regarding the global warming caused by CO_2 from cement production and increasing amount of the industrial wastes such as plastics and glass. Portland cement used in most building and civil constructions are increasing every year [0.4~1.0 tons of CO_2 is produced to make 1 ton of cement and it is as much serious as 7 % of total CO_2 amount produced from anthropogenic activities (UNEP, 2002). Therefore many researchers have focused on reducing CO_2 production and substitute materials for cement with the consideration of ecological industry in the future (Zhao et al., 2007; Komnitas, and Zaharaki, 2007).

Recently, there has been a great development in a new type of high performance inorganic cementitious material; geopolymeric cement, around the world in addressing these challenges which is not only as benefits to the environment but also to the economy. Geopolymer represents a kind of inorganic polymer with SiO_4 and AlO_4 tetrahedra being the structural units (Davidovits, 1989). In general, geopolymers, are alternative aluminosilicate-based cementitious materials (Davidovits, 2008). Basically, metakaolin is a common industrial clay mineral which can be produced through the calcinations of kaolin. Metakaolin is characterized by pozzolanic properties and is considered as the preferred raw-material in geopolymer synthesis as follows (Xu, and Van Deventers, 2000).



Nowadays, geopolymer studies are receiving note-worthily increasing attention because they may be used as a viable economical alternative to organic

polymers and inorganic cements in diverse applications, such as military, aircraft (Giancaspro et al. 2006), high-tech ceramics (Goretta et. al. 2006),

thermal insulating foams (Buchwald et al. 2004), fire-proof building materials (Valeria et al. 2003), protective coatings, refractory adhesives (Bell et al. 2005) and hybrid inorganic-organic composites (Zhang et al., 2004; Li et al., 2005). This interest is due to their exceptionally high thermal and chemical stability, excellent mechanical strength, adhesive behavior and long-term durability. In addition, early researchers have demonstrated that geopolymers are cheap to produce and can be made from a great number of minerals and industrial by-products, including pozzolana (Allahverdi et al. 2008; Verdolotti et al. 2008), natural aluminosilicate minerals (Xu, and Deventers, 2000). Moreover, they are environmentally friendly materials from the point of view of reducing greenhouse effects caused by CO₂ emission from the manufacturing of Portland cement (Khale and Chaudhary, 2007; Zhang, et al., 2010). It is believed that these advantages will extend beyond this in the future. Re-use of waste glass in geopolymer synthesis can reduce energy consumption and CO₂ emission associated with the industrial production of sodium silicate solutions (Brykov and Korneev, 2008) and can prevent the improper deposition of such kind of waste in landfills.

Hence, the importance attached to recycling and valorization of all manners of industrial waste and by products, to enhance environmental protection (Yao et al., 2009; Onipede and Bolaji, 2004).

II. MATERIALS AND METHODS

2.1. Sample Collection

The Kaolin was sourced from the site where it is being mined in Ijero Local Government area, Ekiti State, whereas the Coca cola glass bottles were sourced from waste bottle dealers at Mokola Roundabout, Ibadan North-West Local Government area, Oyo State, Nigeria.

2.2. Sample Preparation

The collected clay sample was scraped to remove impurities, it was then chopped and sun-dried to reduce moisture content and cohesiveness. The dried material was then crushed using rolling rod to obtain particle aggregates which was subjected to calcinations in a muffle furnace set at 700 °C for 6 hours (Ogundiran and Ikotun, 2014). Thereafter, it was screened to grain size less than 212 µm particle size.

The collected glass bottles were washed copiously with tap water, rinsed with distilled water and air-dried. The dried bottles were carefully crushed and ground with milling machine then

screened to grain size less than 63 µm particle size at Geology Department, University of Ibadan, Nigeria.

2.3. Raw Material Characterization

Samples characterization was performed by particle size, chemical and spectroscopic observations.

2.3.1. Particle Size Analysis

The sieve analysis determines the grain size distribution curve of soil samples by passing them through a stack of sieves of decreasing mesh opening sizes and by measuring the weight retained on each sieve.

The grain size distribution and surface area of 212 µm particle size raw clay and calcined clay was determined by normal sieve technique (<212, <150, <106, <63 µm).

Procedure

100 g each of the sieved raw clay and sieved calcined clay (<212 µm) were weighed using weighing balance. This was poured on Sieve compartment arranged in the order; 150 µm, 106 µm, 63 µm and source pan set on a motorized sieve shaker. The shaker was switched on and the samples shaken for 10 mins. Various portions that settled on these sieves were collected in a pre-weighed containers and reweighed. The weight of various portions were deducted and calculated as percentage mass per sample (Ogundiran and Ikotun, 2014).

2.3.2. Fourier Transformed Infrared (FTIR) of Raw and Calcined Clay and Ground Glass Bottles

212 µm raw and calcined clay and 63 µm glass were dried in an oven at 105 °C for 2 hours. 1 mg of each sample were mixed with 200 mg of KBr in an agate mortar, ground for few minutes, then, pressed into pellets in a die set with hand press. A thin and transparent KBr pellets (windows) was obtained. The pellets in a collar was mounted on the sample holder of FTIR spectrometer, scanned and FTIR absorption spectra obtained and recorded in the 4000 – 400 cm⁻¹ range with resolution of 4 cm⁻¹ on Perkin-Elmer Fourier Transform Infrared Spectrum BX Spectrometer.

2.3.3. X-Ray Fluorescence (XRF) Analysis of Raw Clay, Calcined Clay and Glass Bottles

The clay and glass bottle samples were dried in an oven set at 105 °C for 4 hours and placed in a plastic sealed containers and thereafter analyzed with XRF (P Analytical XRF machine PW2400 model) for its chemical composition (SiO₂, Al₂O₃, CaO, MgO, Fe₂O₃, Na₂O, K₂O, TiO₂, MnO, P₂O₅ and LOI)

2.4. Preparation of Geopolymer Activator

The alkaline solution used, 8 M sodium hydroxide (NaOH), was prepared by dissolving 160 g sodium hydroxide pellets (98 % purity) in 500 mL distilled water. The commercial sodium silicate used was clear, colourless and slightly viscous. The chemical composition of the silicate was Na₂O-12 %, SiO₂-30 % and H₂O-58 %. The NaOH and sodium silicate were mixed in the ratio of 1:1 and allowed to stand overnight before use (Ogundiran and Ikotun, 2014).

2.5. Procedure for Geopolymer Synthesis

The percentage ratio of calcined clay to ground glass bottles (source materials) was selected as 100:0, 85:15, 70:30 and 50:50. The components of each of the mixtures were accurately weighed. The solid starting materials for each mixing ratios was mixed in a mixer for 5 minutes to homogenize the samples. The activator (mixture of 8 M NaOH and sodium silicate) was then added to the solid mixture in a mixer and mixed again for 5 minutes until thixotropic paste was obtained. The paste obtained was cast using cylindrical containers, 29 mm inner diameter to 29 mm height and vibrated for few minutes for compaction and reduction of entrapped air. The moulds were covered and allowed to set at room temperature and atmosphere pressure for 3 days. The molded samples were then demolded followed by prolonged curing in plastic sealed bag for 7, 14, 21 and 28 days to prevent excessive evaporation (Ogundiran and Ikotun, 2014).

2.6. Mechanical Characterization of the Synthesized Geopolymer

2.6.1. Determination of Compressive Strength

The compressive stress in obtained experimentally by means of a compressive test. Compressive strength is calculated by dividing the maximum load by the original cross-sectional area of a specimen in a compressive test. In other word, it is calculated from the failure load which is the force (Newton) applied to break a test specimen divided by the cross sectional area (mm²) resisting the load using the strength the following expression.

$$\text{Compressive strength (N/mm}^2\text{)} = \frac{\text{load (N)}}{\text{area (mm}^2\text{)}}$$

Where load is the applied force and the denominator is the cross sectional area of the cylinder (Area of a cylinder = $\pi d^2/4$).

Before each test, the specimen were sandpapered to reduce the samples to exactly 29 mm height where necessary. The samples were then analysed using ELE compression machine (ADR

Touch 2000) at a space rate of 0.5000 kNs. Compressive strength of all the geopolymers made from metakaolin and the mixture of metakaolin and ground glass bottles cured for different days were determined separately by crushing three test samples, calculating compressive strength for each and recording the closest two values. Also the mean and standard deviation for each batch was calculated and recorded (Yao et al., 2009; Ogundiran and Ikotun, 2014).

2.6.2. Determination of Water Absorption Capacity

Water absorption capacity of the specimen was evaluated by weighing the synthesized geopolymer brick on analytical balance (W_d) and then immersing the sample completely in distilled water for 7 days at ambient temperature. The geopolymer samples were then taken out, placed on a flat board and air dried for about 4 hours at room temperature. The weight of the wet material (W_w) was measured in an analytical balance for each geopolymer test specimen and water absorption capacity calculated as follows (Yao et al., 2009; Ogundiran and Ikotun, 2014).

$$\text{Percentage water absorption capacity, } w \% = \frac{(W_w - W_d)}{W_d} \times 100$$

Where W_w and W_d are weight of wet and dry geopolymer respectively.

The percentage water absorption all the geopolymers made from metakaolin and mixture of metakaolin and ground glass bottles cured for 28 days were determined separately and the closest two values were recorded. Also the mean and standard deviation for each batch were calculated and recorded.

The American Society for Testing and Materials (ASTM) specifications for different cement bricks and load bearing concrete masonry unit was used to evaluate the quality of the synthesized geopolymer based on its compressive strength and water absorption to ascertain the quality of the synthesized geopolymers.

2.6.3. Determination of Dry Density

The mass of each geopolymeric specimen was measured on analytical balance. Also the diameter and the height of the specimen was determined using metric ruler and the density calculated as follows (Yao et al., 2009; Ogundiran and Ikotun, 2014).

$$\text{Density} = \frac{\text{mass (kg)}}{\text{volume (m}^3\text{)}}$$

Where volume of cylinder is $\pi r^2 h$

III. RESULTS AND DISCUSSION

3.1. Raw Material Characterization

3.1.1. Particle Size Analysis

Table 1 and 2 show the particle size distribution of raw and calcined clay used respectively. The predominant particle size of both clays was in the range of 63-150 μm .

Table 1: Particle Size distribution of raw clay

Sieve aperture size (μm)	Mass of material retained, Ri (g)	Percentage of material retained Ri/Mi x 100	Cumulative % Passing 100-(Ri/Mi x 100)%
212	0.00	0.00	100
150	3.54	3.60	96.4
106	46.6	47.5	48.9
63	38.4	39.1	9.82
0.00	9.64	9.82	0.00

(Source pan)

Total dry mass (Mi) = 98.2

Table 2: Particle size distribution of calcined clay

Sieve aperture size (μm)	Mass of material retained, Ri (g)	Percentage of material retained Ri/Mi x 100	Cumulative % Passing 100-(Ri/Mi x 100)%
212	0.00	0.00	100
150	3.40	3.44	96.6
106	7.91	8.00	88.6
63	58.8	59.5	29.1
0.00 (Source pan)	28.8	29.1	0.00

Total dry mass (Mi)= 98.9

3.1.2. FTIR Analysis

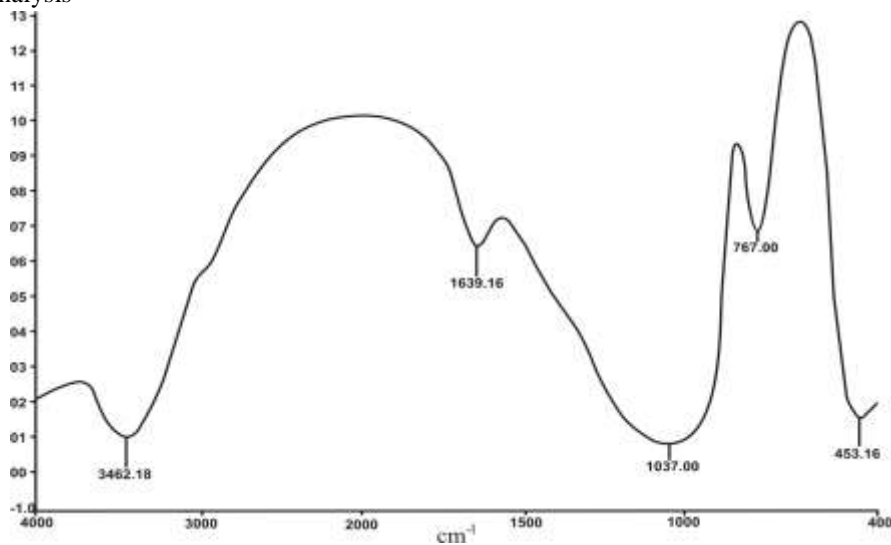


Figure 2: FTIR Spectrum of Coca Cola glass Bottle

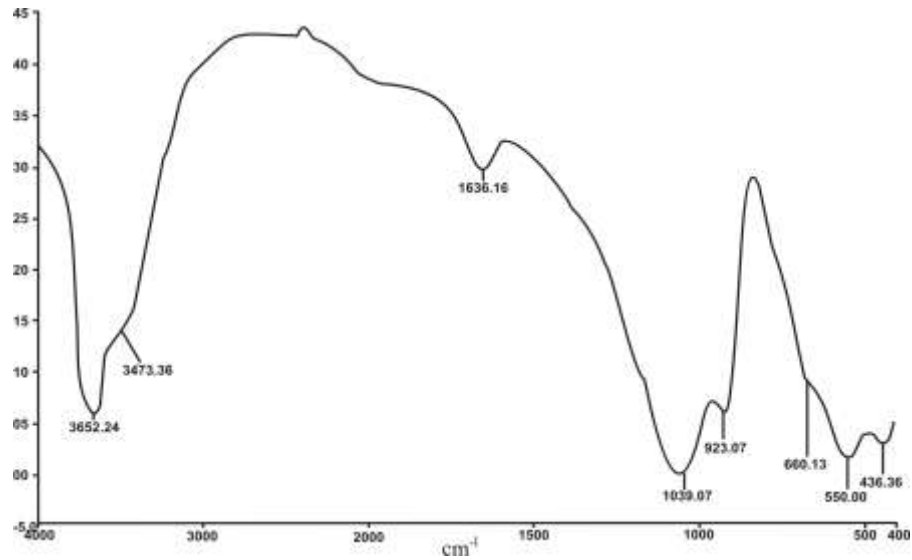


Figure 3: FTIR Spectrum of Raw Ijero Clay

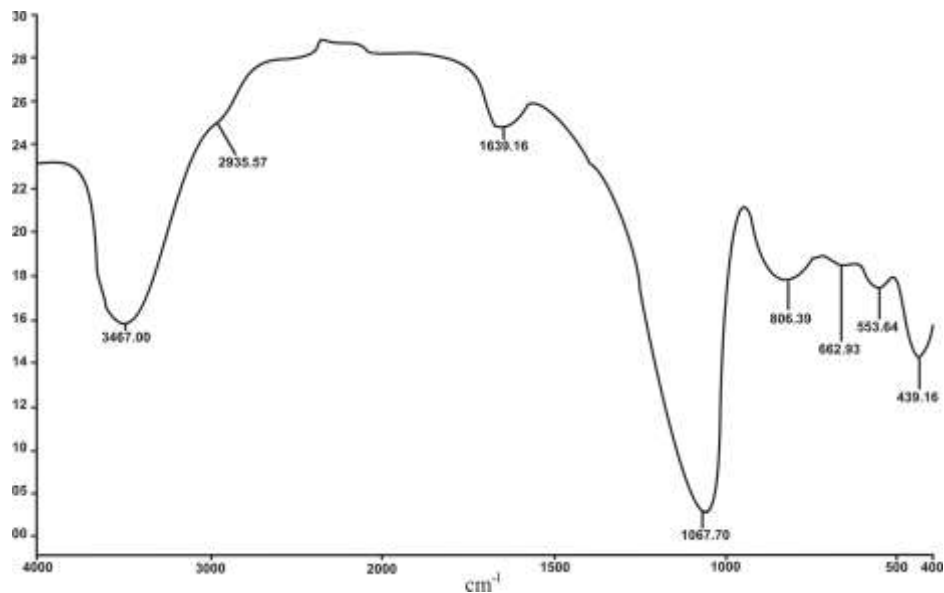


Figure 4: FTIR spectrum of calcined Ijero clay

Table 3, 4 and 5 show various bands and their corresponding vibrational modes for glass, raw clay and calcined clay respectively. The FTIR spectra for glass bottle, raw clay and calcined clay are shown

in Figure 2, 3, and 4 respectively in the range of wave numbers 4000 - 400 cm^{-1} .

Table 3: Assignment of FTIR Bands on the Spectrum for Waste Glass (Torres et al., 2009)

Wave number (cm^{-1})	Assigned to
3462.18	reticular ν -OH
1639.16	σ -H-O-H
1037.00	ν_3 (Si-O) asymmetric stretching
767.00	ν Si-O (in the SiO_4 tetrahedron)
453.16	ν_4 (O-Si-O) binding vibration

Intense band at 900-1100 cm^{-1} associated with SiO (ν_3 (Si-O)) asymmetric stretching vibrations

in the SiO_4 tetrahedral group present in the silicates, and another at 450 - 470 cm^{-1} due to the ν (O-Si-O)

bending vibrations in the SiO₄ groups, likewise in the silicates.

Table 4: Assignment of FTIR bands on the spectrum for raw clay (Torres-Corrasco et al., 2014)

Wave number (cm ⁻¹)	Assigned to
3652.24	vibration modes of adsorbed water
3473.36	Deformation modes of OH ⁻
1636.16	-Si-O-Si asymmetric stretching vibration
1039.70	-Si-O-Si bending vibrations
923.07	Vibrational modes of Si-O-Si units
660.13	-Si-O-Si stretching vibrational modes
550.00	Al-O asymmetric stretching vibration of AlO ₄
436.36	-Si-O-Si bending vibrations ν ₄ Si-O out-of-plane bending vibrations

Table 5: Assignment of FTIR bands on the spectrum for calcined clay (Dimas et al., 2009; Panagiotopoulou, et. al., 2007)

Wave number (cm ⁻¹)	Assigned to
3467.00	Deformation modes of OH ⁻
2935.57	-Si-O-Si- Vibrations
1639.16	-Si-O-Si- Vibrations
1067.70	-Si-O-Si- asymmetric stretching vibrations
806.39	Vibrational modes of -Si-O-Si units or Al-O of AlO ₄
662.93	Vibrational modes of -Si-O-Si units or Al-O of AlO ₄
553.64	-Si-O-Si- bending
439.16	-Si-O-Si- bending

In figure 3 and 4, it was clearly observed that water removal took place in the later which shows the effectiveness of calcinations. The peak at 3652 cm⁻¹ was attributed to physically bond water on silanol group which can be easily removed from clay at high temperature (preferably 700 °C). At higher temperature, the dehydroxylation by condensation of surface hydroxyl groups from the internal hydrogen bonded silanol groups began which complete at about 700°C. The peak at 3467.00 of the calcined clay is associated with the isolated non interacting surface silanol group (Dimas et al., 2009; Panagiotopoulou, et. al., 2007).

3.1.3. X-RAY Fluorescence (XRF)

The chemical composition of glass, raw and calcined clay (Table 6) was determined by the XRF method. It revealed that the percentage composition of the sum of essential oxides; SiO₂ and Al₂O₃, in both clays is greater than 70%. Raw materials which meet the field SiO₂ + Al₂O₃>70% and SiO₂O₃ might be suitable to form geopolymeric binders (Buchwald et al., 2005). The Ijero clay having attained more than 70% as the sum of chemical compositions of silica and alumina and that of silica being greater than alumina, it is therefore qualified to be used as geopolymer binders.

Table 6: Chemical Composition of Raw Clay, Calcined Clay and Glass Bottle
% by weight

Composition	Raw clay	Calcined clay	Glass Bottle
SiO ₂	45.2	52.9	44.1
Al ₂ O ₃	39.0	45.5	0.91
Fe ₂ O ₃	0.07	0.07	0.33
CaO	0.00	0.00	6.49
MgO	0.09	0.12	0.28
Na ₂ O	0.00	0.00	7.31
K ₂ O	0.19	0.19	0.30
SO ₃	0.01	0.00	0.12
TiO ₂	0.00	0.00	0.07
P ₂ O ₅	0.02	0.12	0.01

Mn ₂ O	0.02	0.01	0.01
Cr ₂ O ₃	0.00	0.00	0.07
SrO	0.00	0.00	0.03
ZnO	0.01	0.01	0.00
LOI	1.54	1.46	31.5

3.2. Mechanical Characterization of the Synthesized Geopolymer

3.2.1. Compressive Test

The compressive strengths of metakaolin-glass mixtures activated with the NaOH/Na₂SiO₃ solutions are presented in Table 7. The compressive strength for all the mixed ratios generally increased with the curing days. According to the test results, the best compressive strength value of 18.8±3.85 N/mm² was obtained from the clay-to-glass ratio 85:15 at 28 days. The 85:15 clay-to-glass ratio showed high compressive strength compared to other ratios for all curing days among geopolymers containing the waste

glass. The compressive strength decreased as the amount of waste glass in the mixture increased. The low compressive strength value of the geopolymer with high glass content could be attributed to the fact that glass cannot be activated by the alkaline solution under the work condition in this research. The mechanical strength of the mixed ratios conformed to one or more specifications set by ASTM (Table 9). The value were acceptable and suitable for some load-bearing applications such as building bricks, pedestrian and traffic paving bricks and load bearing masonry.

Table 7: Compressive Strength of Ijero Metakaolin-Glass Bottle Geopolymer Comprising different Clay-to-glass ratios

Curing duration	Compressive strength (N/mm ²) for different Mixing Ratios			
	100:00	85:15	70:30	50:50
7 Days	11.9±0.32	12.8±0.32	12.7±0.85	11.9±0.11
14 Days	12.0±0.64	14.8±1.93	13.1±2.68	12.6±0.21
21 Days	12.2±2.03	17.0±0.21	14.2±0.11	14.5±0.75
28 Days	16.7±0.43	18.8±3.85	15.8±0.11	15.4±1.82

3.2.2. Water Absorption and Bulk Density

Water absorption is normally an important factor to determine the degree of densification of geopolymer. Evidently, the results in Table 8 and Figure 8 showed that the percentage water absorption of the geopolymer increase with increased glass content. The lowest value of percentage water absorption, 1.38±0.00%, was obtained for mix 100:0

and the highest value was 2.12±0.13% for mix 50:50. An increase in water absorption for geopolymers containing waste glass could be attributed to the increasing amount of opened pores, induced by a number of glass particles oozing out onto the geopolymer surface. However, the percentage water absorption generally is low and meets the acceptable standard set by ASTM (Table 9).

Table 8: Water absorption and bulk densities of Ijero metakaolin-glass bottle geopolymer comprising different clay-to-glass ratios

Geopolymer Samples Clay + glass	Water absorption capacity (g)	Percentage water absorption capacity (%)	Bulk density (kg/m ³)
100:00	0.48±0.01	1.38±0.00	1782.30±9.19
85:15	0.59±0.06	1.64±0.19	1783.05±10.29
70:30	0.59±0.01	1.75±0.02	1717.82±5.72
50:50	0.83±0.03	2.12±0.13	1779.42±12.94

Table 9: ASTM Specification for Different Cement Bricks and Load-Bearing Concrete

Title of Specification	ASTM Designation	Weathering condition	Minimum compressive Strength (N/mm ²)	Maximum water (%)
Building brick	C62	SW	20.7	17

		MW	17.2	22
		NW	10.3	No Limit
Facing brick	C216	SW	20.7	17
		NW	17.2	22
Pedestrian and Traffic paving	C902	SW	55.2	-
Brick		MW	20.7	-
		NW	20.7	-
Load bearing masonry	C90	-	13.0	17

SW indicates severe weathering condition, MW indicates moderate weathering condition and NW indicates negligible or no weathering condition (ASTM, 2005).

IV. CONCLUSIONS

This work has successfully accomplished the development of geopolymers for industrial applications using inorganic materials as the constituent. The research characterized the geopolymer starting materials (Clay mineral and waste Coca cola bottles) and the resulting geopolymers for potential application in the building industry which minimizes the degradation of the environment, safe for use, promotes use of renewable resources and energy saving.

Based on the results of this study, it is feasible to use waste glass bottles from bottling companies as a mixture for manufacture of geopolymers. Waste glass bottle can be mixed with kaolinite in different proportions. The results showed that with proper curing, waste glass bottle inclusion up to 50 wt. % did not cause detrimental effects to the properties of geopolymer. However, the compressive strength of the geopolymers decreased while the water absorption of geopolymers was significantly increased as the waste glass content increased. It is concluded that glass bottle composition of geopolymer when mixed with clay up to 50 % wt. has good enough compressive strength, low percentage water absorption and acceptable density for potential structural applications.

Potential Conflicts of Interest

The authors declare no known conflict of interest.

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