

*Full Length Research Paper*

# The potential of binary blended geopolymer binder containing Ijero-Ekiti calcined kaolin clay and ground waste window glass

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The aim of this study was to investigate alkaline reactivity potentials of calcined clay and ground waste window glass with NaOH/Na<sub>2</sub>SiO<sub>3</sub> solution to form geopolymer, an inorganic binder. A calcined clay (CC) sourced from Ijero-Ekiti, Nigeria, was replaced by various proportions (0, 25, 50 and 75%) of ground waste window glass (GWWG) and subjected to alkaline activation by NaOH/Na<sub>2</sub>SiO<sub>3</sub> solution. X-ray fluorescence (XRF) and Fourier Transform Infrared (FTIR) spectroscopy were applied to characterise the materials and the resulting geopolymers. The XRF results revealed the main oxides of Ijero-Ekiti clay as SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> and for GWWG as SiO<sub>2</sub> and Na<sub>2</sub>O. The FTIR results confirm the clay as kaolin clay which is suitable for geopolymer synthesis. The synthesised geopolymer binders were cured at room temperature for 7, 14, 21 and 28 days. Their compressive strengths, dry density and water absorption were measured. Geopolymers with ground waste window glass (GWWG) indicated higher strengths at both early and late curing times. The final 28-day compressive strength values of GWWG-CC-based geopolymers observed was in the range of 17.3±0.6 to 23.1±0.7 MPa compared with 11.6±0.4 to 14.5±1.4 MPa for 100% CC-geopolymers. Calcined clay replacement up to 75% glass yielded the highest strength. Addition of window waste glass enhanced both early and late strength gain of the geopolymers and improved physical properties. Therefore, GWWG-CC-geopolymers could serve as potential binders in making green construction and building materials.

**Key words:** Ijero-Ekiti kaolin clay, waste window glass, recycling, clay-waste-glass-geopolymer, compressive strength development.

## INTRODUCTION

Conventional bricks used in building are produced either from natural clay which are cured by firing in high-temperature kiln or by cementing method using ordinary Portland cement (OPC) as the binder (cement-clay brick) as documented by Hwang and Huynh (2015). Producing

bricks from these materials and methods have associated impacts of high energy consumption and release of CO<sub>2</sub> into the environment. Additionally, clay is a known non-renewable natural resource. Continuous mining of it may lead to environmental degradation. Finding an alternative

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binder with partial replacement of clay with waste materials will lead to reduction in clay mining and offer environmental benefits in terms of waste recycling.

Climate change and waste management are parts of environmental problems threatening sustainable development. Cement production industry has been of tremendous benefit to development in the areas of construction and building. However, it remains the leading source of CO<sub>2</sub> emission, a major greenhouse gas which causes global warming. Production of one ton of Portland cement has been reported to release approximately one ton of CO<sub>2</sub> to the atmosphere from combustion of carbon fuel in the kiln (Davidovits, 1994). The CO<sub>2</sub> is produced from calcination of limestone and silica (0.55 ton) during cement manufacturing as shown in the reaction:  $5\text{CaCO}_3 + 2\text{SiO}_2 \rightarrow (3\text{CaO}, \text{SiO}_2)(2\text{CaO}, \text{SiO}_2) + 5\text{CO}_2$  and consumption of fossil fuels in the kiln (0.40 ton). Following this, there is a need for an alternative binder to replace or complement cement usage in building and construction which will result in reduction of CO<sub>2</sub> emission.

Development of inorganic polymer, termed as geopolymer, has been researched as low CO<sub>2</sub> binder which requires much less energy to be produced. According to Davidovits (1994), geopolymer technology has potential to reduce CO<sub>2</sub> emission caused through the cement industries by 80 to 90%. Production of 1 kg OPC was documented to consume about 1.5 kWh energy with the emission of about 1.0 kg CO<sub>2</sub> and each fired-clay-brick consumed approximately 2.0 kWh energy and emission of about 0.41 kg CO<sub>2</sub> into the environment (Hwang and Huynh, 2015).

Geopolymers are three dimensional aluminosilicate materials which function as binders. They are produced by the reaction of concentrated alkali hydroxide and silicate solutions with solid materials that are rich in alumina and silica. Natural solid materials that have been used include thermally treated kaolin clay, volcanic ash, by-products (fly ash, blast furnace slag, etc.) and metallurgical wastes (Ogundiran and Kumar, 2015; Tchakoute et al., 2013; Buchwald et al., 2009; Andini et al., 2008; Nugteren et al., 2009; Rickard et al., 2011). Source materials, synthesis conditions and the intended uses determine the characteristics of geopolymers. For most applications, the desired properties of geopolymers include fast or slow setting, high early compressive and tensile strengths, high ultimate strength, low shrinkage and permeability, high acid and fire resistance (Davidovits, 1994).

Solid waste management is a major challenge globally. Recycling of solid wastes is the most preferred option to open disposal or landfilling due to contribution to environmental sustainability. This has resulted in a renewed interest to recycle waste glass in concrete in place of disposal on land or landfilling (Shao et al., 2000; Shayan and Xu, 2006). Glass is amorphous and has high silica content, which are the primary requirements for a

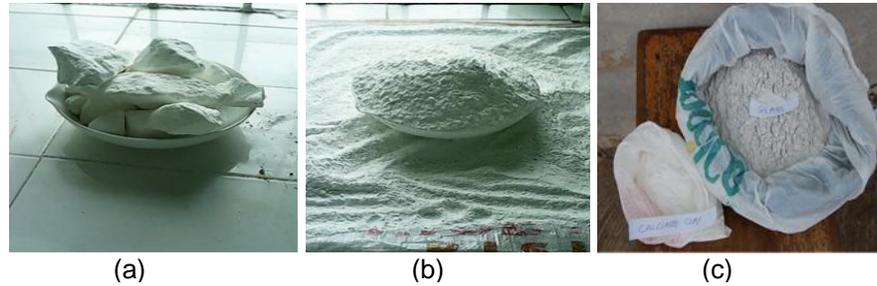
pozzolanic material. A particle size of 75 µm or less has been reported to be favourable for pozzolanic reaction (Shao et al., 2000). Investigations have been carried out to use ground or powdered waste glass to partially replace cement in concrete production (Shayan and Xu, 2006; Park et al., 2004; Shao et al., 2000). Few studies have also shown the geopolymerisation potentials of waste glass with other materials such as commercial metakaolin, fly ash and blast furnace slag (Redden and Neithalath, 2014; Lin et al., 2012). Reports are limited on the production of geopolymer from kaolin clay and waste glass. Use of alkaline activated calcined clay and waste window glass in the production of binder for building materials will protect the environment. This will be possible through recycling of solid waste in place of disposal, reduction in mining of non-renewable clay in building material, CO<sub>2</sub> emission and energy required in cement production.

Waste window glass is abundant, found mainly at demolition sites and as residues at construction sites. In developing countries majority of demolition glass has not found recycling method but rather abandoned at the demolition sites or dump at waste disposal sites thereby causing pollution of the environment. Glass is amorphous with high silica content which may produce alkaline aluminosilicate reactivity. These characteristics present the need for recycling waste window glass into useful products. The use of ground waste window glass (GWWG) in combination with Ijero-Ekiti calcined clay may result in geopolymer binder of desirable properties which can be utilised in building materials in place of fired-clay-brick or cement-clay bricks.

Thermal processing of Nigerian kaolin clay has been reported to result in geopolymer binders (Ogundiran and Kumar, 2015; Ogundiran and Ikotun, 2014). Kaolin is available as "waste" after mining of other minerals and can also be mined at low cost. However, continual mining is not sustainable. Therefore, this study was designed to investigate the effects of partial replacement of Ijero-Ekiti calcined clay with ground waste glass to produce geopolymer binders. Effects of GWWG on the compressive strength, density and water absorption capacity of Ijero-Ekiti calcined clay geopolymers were evaluated.

## MATERIALS AND METHODS

A Nigerian kaolin tagged as Ijero-Ekiti kaolin clay was sourced from natural deposits in Ijero local government, Ekiti state. The clay was whitish in nature. Large amount of the clay sample was dried under sun to reduce its moisture content. The size of the clay sample was reduced using rolling rod. The crushed clay was oven-dried at 105°C for 6 h and later sieved to 212 µm using Endicott Ltd, London, stainless steel sieve. The clay sample was heat treated in a laboratory muffle furnace set at 700°C for about 6 h (Ogundiran and Kumar, 2015). The raw, crushed and calcined clay samples are shown in Figure 1. The waste window glass was obtained from demolition sites. The broken pieces of window glass were



**Figure 1.** Material samples. (a) Untreated raw clay; (b) Treated uncalcined clay; (c) Calcined clay and ground window glass.

**Table 1.** Mix proportion used on a weight basis to produce a geopolymer cylinder

% ratio of CC: GWWG	CC (g)	GWWG (g)	Activator (g)
100:0	23.0	-	19.8
75: 25	17.3	5.70	19.8
50: 50	11.5	11.5	19.8
25: 75	5.70	11.3	19.8

**Table 2.** Particle size distribution of raw and calcined clay.

Particle size	< 212 $\mu\text{m}$	< 150 $\mu\text{m}$	< 106 $\mu\text{m}$	< 63 $\mu\text{m}$
Raw clay	3.54	46.6	38.4	9.64
Calcined clay	3.40	7.91	58.8	28.8

thoroughly washed with water to remove particles and debris and also with distilled water and finally air-dried. The dried samples were crushed, ground and sieved to 63  $\mu\text{m}$  particle size in accordance with particle size of 75  $\mu\text{m}$  or less to initiate pozzolanic reaction (Shao et al., 2000).

### Material characterization

The chemical composition of calcined clay and ground window glass was determined using X-Ray Fluorescence Spectrometer (PAnalytical XRF machine PW2400). Structural characteristics of the samples were measured by Fourier transformed infrared spectroscopic method. The infrared spectra were recorded on a Perkin-Elmer Fourier Transform Infrared (FTIR) Spectrum BX spectrometer set to scan frequencies across a range of 4000 to 400  $\text{cm}^{-1}$ .

### Particle size analysis

Particle size distribution of the raw clay and calcined clay was determined using normal dry sieve technique (Darweesh, 2001). This was done using a Mechanical Sieve Shaker. 100 g each of the 212  $\mu\text{m}$  raw and calcined clay was weighed accurately on an analytical weighing balance. Stack of sieves set comprised of 212, 150, 106 and 63  $\mu\text{m}$ . The sieve with larger openings was placed above the sieve with smaller openings. A bottom pan was placed under the 63  $\mu\text{m}$  sieve. The raw clay was placed into the stack of

sieves from the top and the stack was covered. The stack of sieves was run through a sieve shaker for few minutes. The amount of raw clay retained on each sieve and the bottom pan was weighed. The results of the sieve analysis are presented in Table 2. Calcination improved reduction in the particle size of Ijero-Ekiti clay.

### Synthesis and characterisation of geopolymer

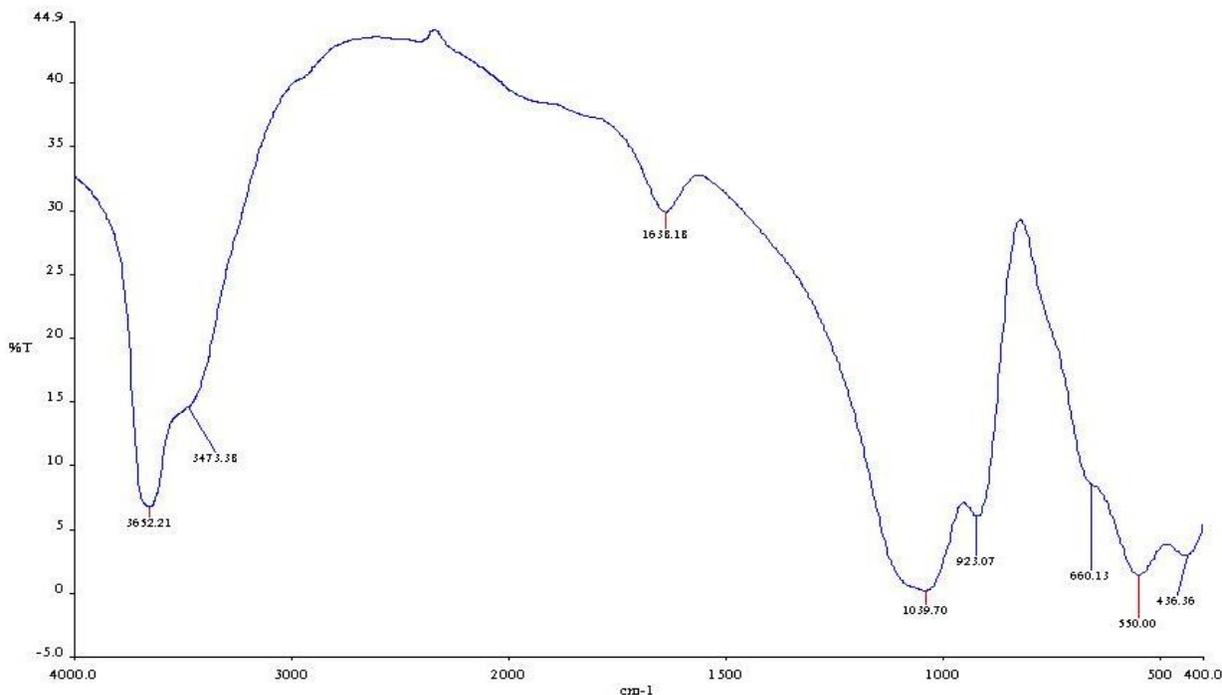
Mixture of sodium silicate ( $\text{Na}_2\text{SiO}_3$ ) and sodium hydroxide (NaOH) solutions both of industrial grade was used as activator. Sodium silicate solution has chemical composition of 30.1%  $\text{SiO}_2$ , 9.4%  $\text{Na}_2\text{O}$  and 60.5 %  $\text{H}_2\text{O}$ . The NaOH was in the form of white flakes. Eight molar (8 M) NaOH solution was prepared by dissolving the appropriate quantity in water. The alkaline activator was prepared about 24 h before use with mixing ratio of (1:1) sodium silicate/NaOH solution.

The percentage ratio of calcined clay: ground waste window glass of 100:0, 75:25, 50:50 and 25:75 were prepared. The mix proportions are presented in Table 1.

The solid starting materials were dry mixed in a mixer for a few but consistent minutes to homogenise the samples. Predetermined amount of the activator was added and mixed again for a few minutes until a consistent or thixotropic paste was obtained. The pastes were cast into cylinders of 29 mm diameter to a height of 30 mm and vibrated on a sieve shaker for 5 min for compaction and reduction of entrapped air. The moulds were covered and allowed to set at room temperature for 3 days. The geopolymer samples were removed after 3 days and kept in sealed plastic bags to cure

**Table 3.** Chemical composition of calcined Ijero-Ekiti clay and waste window glass (Wt. %).

Variables	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	Mn <sub>2</sub> O <sub>3</sub>	SrO	ZnO	LOI
Calcined clay	52.9	45.5	0.07	-	0.12	-	-	0.19	-	0.02	0.01	-	0.01	1.46
Window glass	72.1	5.28	0.30	0.34	0.08	0.01	11.7	0.85	0.10	0.00	0.01	0.05	-	-

**Figure 2.** FTIR spectrum of raw Ijero-Ekiti kaolin clay.

at ambient temperature. The compressive strength at 7, 14, 21 and 28 days curing was measured. For each curing time, test was conducted on minimum of three geopolymer samples using compression test machine (ELE International, ADR Touch, 2000). The result was recorded as mean  $\pm$  SD compressive strength for the closest two. Water absorption after 7 days and dry density of the geopolymers after 28 days were measured as reported previously (Ogundiran and Ikotun, 2014).

## RESULTS AND DISCUSSION

### Material characterization

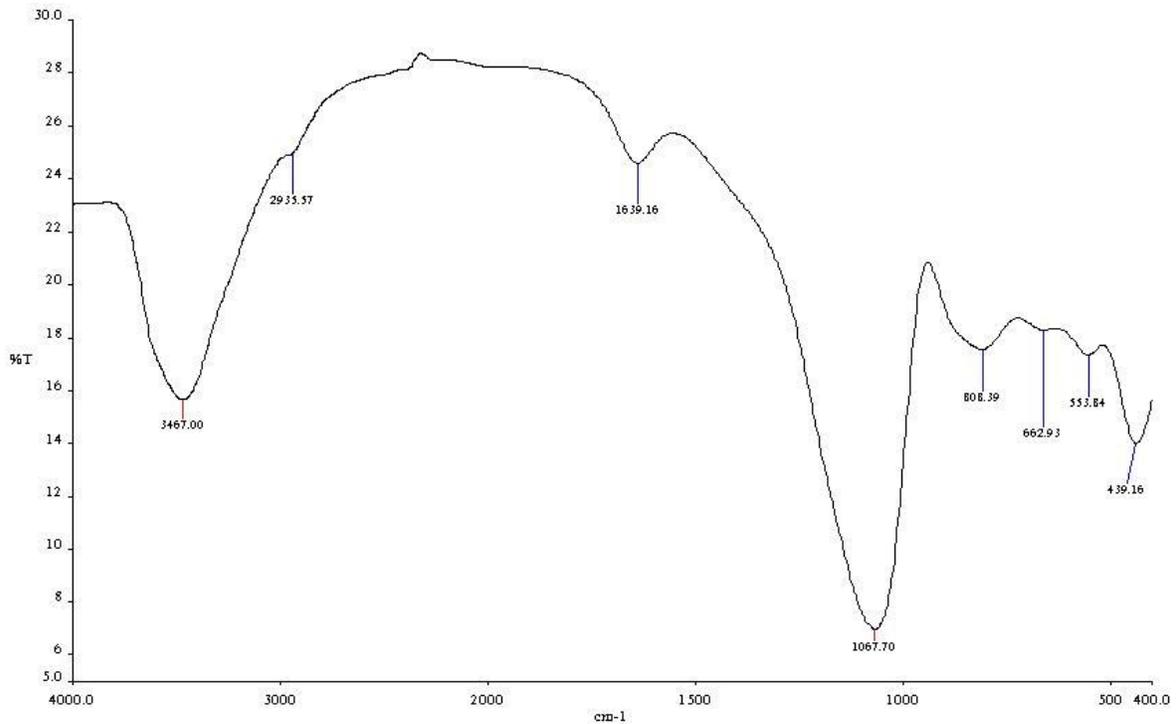
#### X-ray fluorescence analysis

The chemical composition of the calcined clay and ground waste window glass (GWWG) are presented in Table 3. The main components of this clay are silica and alumina. However, the alumina (45.5%) content of Ijero-Ekiti kaolin clay is much higher than that of Ikere kaolin clay (27.4%) reported previously (Ogundiran and Kumar, 2015). This may have impact on the alkaline reactivity of

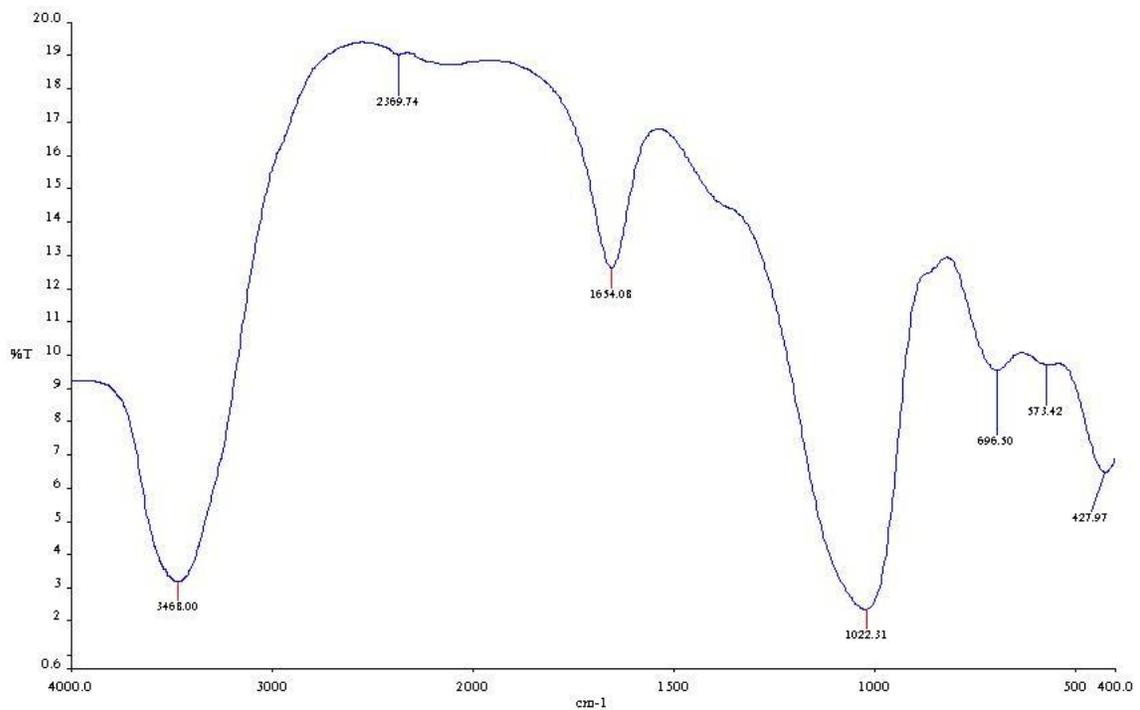
this clay and also compensate for the low alumina content of the glass. The glass has high silica content (72.1%) followed by Na<sub>2</sub>O (11.7%) which presents this window glass as Soda-lime glass. Other minerals are present in minimal amount. The most familiar type of glass is soda-lime glass, which is composed of about 75% silicon dioxide (SiO<sub>2</sub>), sodium oxide (Na<sub>2</sub>O) from sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>), lime (CaO), and several minor additives.

#### Fourier Transform Infrared (FTIR)

The FTIR spectra of Ijero-Ekiti raw and calcined clay and calcined clay-geopolymer are shown in Figures 2 to 4. In the IR spectrum of raw clay, strong bands at 3652 and 3473 cm<sup>-1</sup> of OH stretching vibrations were observed which are due to hydroxyl groups attached to Al octahedron sheet (Ogundiran and kumar, 2015). In addition, band associated with OH bending vibrations of Al-OH was detected at about 923 cm<sup>-1</sup> while band associated with Si-O-Si stretching vibrations were



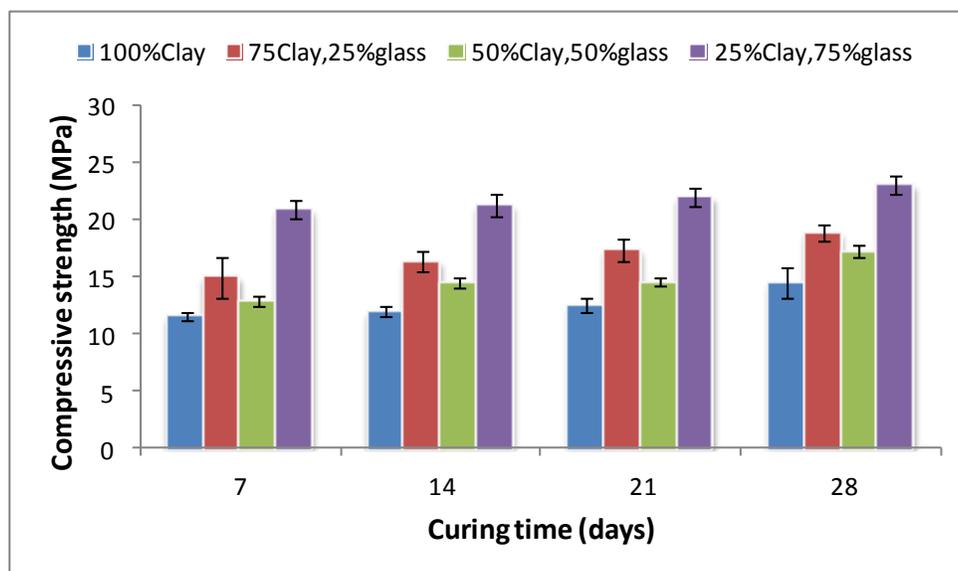
**Figure 3.** FTIR spectrum of Ijero-Ekiti calcined clay.



**Figure 4.** FTIR spectrum of Ijero-Ekiti calcined clay geopolymer.

observed at 1039 cm<sup>-1</sup>. All these bands establish the presence of kaolinite in Ijero-Ekiti clay. The Si–O

stretching vibrations were observed at, 660, 550 and 436 cm<sup>-1</sup> indicative of the presence of quartz. The band at



**Figure 5.** Compressive strength development of calcined clay-waste window glass geopolymers.

1638 was associated with stretching vibrations of OH of adsorbed water.

In the FTIR studies of the calcined clay, the  $3652\text{cm}^{-1}$  observed in the raw kaolin clay which indicated the presence of OH stretching of inner surface hydroxyl groups of kaolinite disappeared in the calcined clay (metakaolin clay) implying that the raw kaolin clay lost some amount of water during the calcination process at  $700^\circ\text{C}$ . The H-O-H stretching vibration observed at  $3467\text{cm}^{-1}$  and H-O-H bending vibration at  $1639\text{cm}^{-1}$  have been attributed to water absorbed from the atmosphere by the calcined clay. The Si-O stretching and bending vibrations of metakaolinite were observed at  $1068$  and  $439\text{cm}^{-1}$ , respectively. The band at  $808\text{cm}^{-1}$  was due to 4-coordinated Si-O-Al stretching vibration.

The FTIR spectrum of Ijero-Ekiti calcined clay geopolymer is shown in Figure 4. Similar to calcined clay, characteristic bands at  $3468$  and  $1654\text{cm}^{-1}$  corresponding to adsorbed atmospheric water were observed in the FTIR spectrum calcined clay geopolymer. In geopolymer, Si-O stretching vibration was observed at lower wave number ( $1022\text{cm}^{-1}$ ) than that of the calcined clay ( $1068\text{cm}^{-1}$ ). This indicates that the condensation of Si-O tetrahedron in the geopolymer is lower than that in the metakaolin. The band at about  $700\text{cm}^{-1}$  has been linked to disruption of the Al environment (Barbosa et al., 2000).

### Compressive strength of geopolymers

The compressive strength values of Ijero-Ekiti calcined clay and Ijero-Ekiti calcined clay-GWWG-geopolymers

are shown in Figure 5. The compressive strength of each geopolymer mix was the mean $\pm$ SD of two geopolymers with closest results. The standard deviation of all the tests except in two cases was less than 10%. The results demonstrate that the strength of all the geopolymers increased with curing time, suggesting continuous chemical reactions by the geopolymers and this gave rise to stronger strength gain. All geopolymers which contained GWWG exhibited higher compressive strength than 100% calcined clay-geopolymer at all curing ages. The higher the quantity of glass, the higher the compressive strength except for 50% GWWG replacement. Geopolymer with 75% glass replacement exhibited the highest compressive strength at all ages followed by the geopolymer with 25% glass. Clay-glass-geopolymer displayed better early and later strength gain. At one week curing, 75% glass addition increased the compressive strength by 44.5% at 7 days. The compressive strength value of 100% clay-geopolymer was about 37.2% lower than the strength of 75% glass-geopolymer at 28 days. The difference in the results corroborates the silica content of the glass as an active component in the mixture which participated in the geopolymerisation reaction. The results obtained from XRF analysis show high content of  $\text{SiO}_2$  in the composition of ground window glass. Addition of GWWG possibly improved more participation of Si-O-Si bond in geopolymer network of clay-GWWG-geopolymers. The Si-O-Si bond is stronger than Al-O-Al bonds (Duxson et al., 2005). The Al-O-Al bonds were probably more in the clay-geopolymer and this accounted for lower strength. Fletcher et al. (2005) prepared geopolymers from mixtures of dehydroxylated kaolinite and also found that

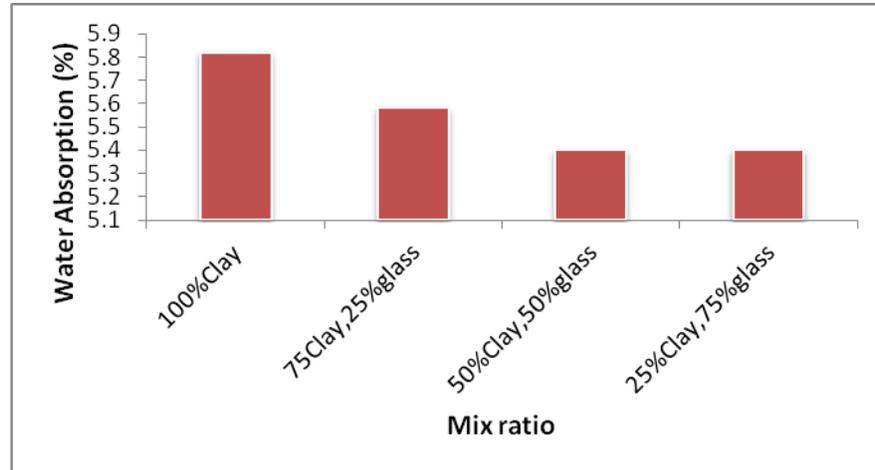


Figure 6. Water absorption of geopolymers at 7 days.

high-alumina compositions exhibited low compressive strength. As revealed by XRF results (Table 1), Ijero-Ekiti clay is exceptionally rich in alumina content (45.5%). The difference in the strength may also be attributed to the high content of  $\text{Na}_2\text{O}$  (11.7%) in the waste glass. It was reported that alkalis in concrete could act as catalyst in forming calcium silicate hydrate at early stage and promoting early strength development (Shao et al., 2000). For glass containing concrete, Shao et al. (2000) reported increase in early strength and no decrease in late strength in the presence of 16%  $\text{Na}_2\text{O}$  from the soda lime glass. Similarly, since Ijero-Ekiti clay is exceptionally rich in alumina content, addition of glass which is high in  $\text{Na}_2\text{O}$  might have resulted in the formation of sodium aluminosilicate (N-A-S-H) gel which is stronger and stable (Redden and Neithalath, 2014). The results confirm the possibility of using mixture of waste soda lime glass and calcined Ijero-Ekiti clay as geopolymer binder.

### Water absorption of geopolymer

The results of water absorption test at 7 days for the geopolymers are shown in Figure 6. As the GWWG increased, water absorption decreased. Geopolymer samples consisting of 100% calcined clay showed the highest percentage of water absorption capacity while geopolymers with higher content of GWWG had the least. The decrease could be attributed to silica content of glass participating in the formation of geopolymer network, which in turn reduced the amount of pores in the network. All the geopolymer samples had water absorption below 6% which is lower than the acceptable values set by ASTM C62, C216, C902 and C90 (Mohsen and Mostafa, 2010). This demonstrates that replacing calcined Ijero-Ekiti clay with GWWG made the geopolymers to be less porous and improved their ability to limit water

penetration into the geopolymer structure. Water absorption is an important parameter which determines geopolymer durability. Pores in the geopolymer network may give way to penetration of water into the geopolymer. The ability of water to infiltrate into a geopolymer structure will affect its durability. A durable geopolymer should possess high water resistance capacity.

### Dry density of geopolymers

The results of dry density of the geopolymers at 28 days are shown in Figure 7. The effect of GWWG addition on the density of the geopolymers was appreciable. Increase in the amount of GWWG led to decrease in density. The GWWG-calcined clay-geopolymers can be applied as binder in the production of lightweight construction and building materials. On the other hand, if desired to be applied as binder in the production of heavyweight construction and building materials, the bulk density can be improved by addition of aggregates such sand and coarse aggregates.

### Conclusion

The FTIR spectra of raw Ijero-Ekiti, calcined Ijero-Ekiti and geopolymer products showed changes in chemical bonding, confirming Ijero-ekiti clay as suitable for geopolymer synthesis. Addition of ground window glass to calcined clay improved both the early and late mechanical strength gain of the geopolymers and decreased their water absorption capacity. The geopolymer contained up to 75% waste window glass had compressive strength of 23.1 MPa at 28 days compared with calcined-clay geopolymer counterpart

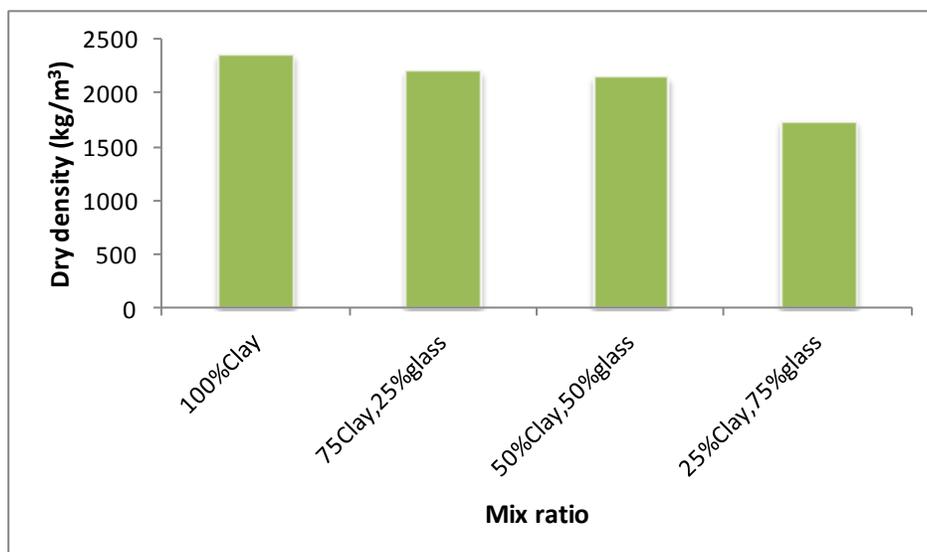


Figure 7. Dry density of geopolymers at 28 days.

which had compressive strength of 14.5 MPa. Clay-glass geopolymer displayed better early and later strength gain. However, the dry density was decreased with glass addition, which on modification by addition of aggregates can be improved where necessary. Geopolymer, synthesised using mixture of calcined clay and waste window glass, has high compressive strength, increase early and late strength gain, low water absorption capacity. Therefore, it could serve as potential binder in making green construction and building materials. Application of waste window glass in geopolymer synthesis will be economical, sustainable and also serves as effective way of recycling waste glass which requires environment friendly management. It will enhance less use of non-renewable clay in making building material.

### Conflict of Interest

The authors have not declared any conflict of interest.

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