

Characterization of Selected Nigerian Kaolinites and Agricultural Waste Ashes as Materials for Sustainable Geopolymer Brick Manufacturing

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Abstract: There have been reports of an increasing rate of green house gas emissions from different sources resulting to adverse climatic changes all over the world. 2018 BBC Chatham House report stated that about 8% of the world's CO₂ emission come from cement production. Hence the need to look at alternative sources of cement production which would not compromise the expected strength and efficiency in building constructions as well as reduce CO₂ emission into the environment. This research presents an empirical study that investigates the Characterization of Selected Nigerian Kaolinites and agricultural waste ashes (Rice Husk Ashes) as materials for sustainable geopolymer brick manufacturing. A variety of parameters including solid-to-solid mix ratio, solid-to-liquid mix ratio, liquid-to-liquid mix ratio, presence of sand filler, curing duration, water absorptivity, and bulk density were examined to understand the extent or degree of geopolymerization as well as their influence on the mechanical properties of the clay-based geopolymers. From the results, it was observed that compressive strength of the geopolymer mixes increases with time progressively from 7days through 28days with Ikere clay sample of solid-to-liquid ratio 2.0 giving the highest compressive strength. The compressive strength test carried out on Rice Husk Ash-Clay geopolymers showed a tremendous increase in strength compared to the ones synthesised with clay samples alone. The geopolymer bricks also showed impressive strength with time increasing progressively from 7days through 28days. However, the geopolymer binders as well as the bricks produced with Ikere clay showed a greater compressive strength than that produced with Ikere clay samples. It was also observed that bricks made from RHA-CLAY geopolymer showed impressive strength compared to that made from ordinary Portland cement. It is obvious from these results that geopolymers could be a good alternative to contemporary method of cement production since it is more environmentally friendly.

Keywords: Geopolymers, Curing Duration, Water Absorptivity, Bulk Density, Compressive Strength, ASTM Standards

1. Introduction

Increasing emphasis on energy conservation and environmental protection has led to investigation of alternatives to customary building materials [1]. Among the goals of this research is reduction of greenhouse gas emissions arising from cement production through environmental friendly alternatives.

Currently, Portland cement is the leading material for industrial concrete demand worldwide, fulfilling a demand of over 1.5 billion tons annually; the production of Portland cement is energy-intensive and releases a significant volume of carbon dioxide (CO₂) to the atmosphere [1]. For each ton

of Portland cement manufactured, it is estimated that one ton of CO₂ would be released into the environment. The process involves very high temperatures (1400–1500°C), the destruction of quarries to extract raw materials, and the emission of greenhouse gases such as CO₂ and NO_x [2]. The costs associated with these energy requirements are substantial. Consequently, the need for further research on low energy alternative to cement production with decreased environmental impacts and enhanced economic benefits.

Geopolymerization is a complex multiphase process, comprising a series of dissolution-reorientation-solidification reactions [3-7]. First is the generation of reactive species or alkali activation, which is the dissolution of amorphous phases

(e.g., aluminosilicates) by alkali to produce small reactive silica and alumina; secondly is the reorientation, which involves transportation, orientation or condensation of precursor ions into oligomers; and thirdly, the actual setting reaction, which is the polycondensation process leading to the formation of amorphous to semi-crystalline aluminosilicate polymers [8]. However, these three steps can overlap with each other and occur almost at the same time, making it difficult to isolate and examine each of them separately.

Today, light weight construction materials are used to reduce the weight of building structures and improve thermal insulation efficiency of buildings [9]. Hence, lightweight geopolymer source materials and agricultural waste which would be used for this study include: Clay (kaolinite), and Rice Husk Ash (RHA). This study is focused on using cheap aluminosilicate materials to make geopolymers with excellent mechanical, chemical, and physical properties as well as long-term durability at room environment. Its main objectives include:

1. Using industrial by-products or low-cost materials to make geopolymers;
2. Examining the composition and mechanical properties of the resulting geopolymers;
3. Identifying and understanding the factors affecting the synthesis, composition, and mechanical properties of the synthesized geopolymers; and
4. Validate the potential applications of the end products in terms of their intrinsic properties.

The research seeks to examine the possibility of using Nigerian Aluminosilicate materials particularly clay sample from Ikare and Ikere and agricultural waste ashes (Rice Husk Ashes) as the base materials to making geopolymer bricks. Compressive strength, bulk density and water absorption tests are selected as the benchmark parameters. This is because comprehensive strength has an intrinsic importance in the structural design of concrete structures [10].

2. Experimental Procedure

2.1. Materials

Geopolymer source materials and agricultural waste used for this study include: Clay and Rice Husk Ash (RHA). The samples were sourced in various places within Ibadan and Lagos metropolis. Nigeria kaolin samples from Ondo (Ikare clay) and Ekiti (Ikere Clay) were used as sources of aluminosilicate oxides to synthesize geopolymer materials in this study. Three kilograms (kg) of each clay sample were sourced from Federal Institute of Industrial Research, Oshodi (FIIRO), Lagos State. Previous studies show that the Ikere clays are kaolinites rich with low amount of iron oxide minerals impurities [11]. The Ikere clay has a golden yellow colour while Ikare clay is light cream clay in raw form with some mica impurities. Sodium hydroxide and commercial sodium silicate obtained from FIRRO, Lagos State were used as activators; distilled water was used throughout the research to avoid the effect of unknown contaminants in mixing the geopolymer precursors. Rice Husks were sourced

from crop processing unit, International Institute of Tropical Agriculture (IITA), Ibadan. The cement used as control was Ordinary Portland cement bought from Bodija Market in Ibadan. The fine particles of sand used as aggregates were sieved using 212 μ m mesh size sieve and washed with distilled water before using it for geopolymer brick making. Figures 1 to 4 show the clay samples before and after calcination prior to geopolymer synthesis.



Figure 1. Non-Calcinated Ikere Clay Sample.



Figure 2. Calcinated Ikere Clay Sample.



Figure 3. Non-Calcinated Ikare Clay Sample.



Figure 4. Calcinated Ikare Clay Sample.

2.2. Sample Preparation

2.2.1. Preparation of Clay Samples

Sufficient amount of the clay sample were dried under the sun to reduce moisture content and cohesiveness. The sizes of the dried samples were reduced using a mortar and pestle to obtain particle aggregates which were then subjected to calcination in a muffle furnace set at 750°C for about 6hours. It was then screened to grains of 212µm particle size before use.

2.2.2. Preparation of Rice Husk Ash (RHA)

Sufficient amount of Rice husk samples were dried under the sun to reduce its moisture content. The dried samples were milled with milling machine to obtain particle aggregates. These particles were then ashed in a muffle furnace set at 450°C and sieved to grains of 212µm sizes.

2.2.3. Preparation of Geopolymer Activator and Geopolymer Synthesis

Sodium hydroxide solution (8M NaOH) were prepared and added to neutral sodium silicate activator in a given proportion to make it alkaline prior to the mixing of the geopolymer. The mixing was carried out at ambient temperature; different geopolymer mixes were synthesized with the calcined clay, Rice Husk Ash, and Sand which have already been prepared as stated above. These geopolymer Source materials (GSM) and Alkali Activator Solutions (AAS) were mixed together thoroughly in an electric mixer for about 15 to 20minutes to ensure sufficient reaction between the solid powder and Alkaline Activator Solution.

Furthermore, geopolymer specimens for mechanical characterization were subsequently prepared by pouring the geopolymer precursor into cylindrical molds with an inner diameter of 28mm and height of 37mm and allowed to dry in a laboratory ambient environment for about three days to set. The specimens were then demolded, followed by prolonged curing in plastic sealed bag for 7, 14, 21 and 28days to prevent excessive evaporation. To ensure reproducibility, two duplicate specimens were prepared for each geopolymer composition.

2.2.4. Preparation of Ordinary Portland Cement (OPC) Brick

Portland cement procured from Bodija market in Ibadan was used to make bricks to serve as control for the synthesized geopolymer bricks. OPC and sand mix ratio of 1:1, 1:2, and 1:3 were thoroughly mixed together in an electric mixer for about 15 to 20minutes to ensure sufficient reaction. Subsequently, OPC bricks were prepared by pouring the OPC-Sand Mixture into cylindrical molds with an inner diameter of 28mm and height of 37mm and allowed to dry in a laboratory ambient environment for about 24hours to set. The specimens were then demolded, followed by prolonged curing in a plastic sealed bag for 7, 14, 21 and 28days to prevent excessive evaporation. To ensure reproducibility, two duplicate specimens were prepared for each mix ratio and the compressive strengths determined.

3. Geopolymer-Mix Proportions

Different geopolymer mix-proportions were tested to ascertain the best geopolymer mix that will give the highest compressive strength. Sodium Hydroxide to Sodium Silicate ratio of 2.1 corresponding to 10.8g NaOH: 100g Na₂SiO₃ was used to activate the different geopolymer mixes. Geopolymer Source Materials to Alkali Activator ratio of 2.0, 1.4 and 1.3 were tested and the one that gave the highest compressive strength was used to make bricks; another set of geopolymers were also synthesized using clay and Rice Husk Ash with varying ratio.

From the previous work, Liquid to Liquid ratio that gave the highest strength is 2.1 (corresponding to 10.8g NaOH: 100g Na₂SiO₃ in the present work) [12]. Therefore, the following mix proportions were tried out to ascertain the geopolymer mix that would give the highest compressive strength.

3.1. Solid to Liquid Mix Ratio: 2.0

For every 200g of clay sample, 100g of the Alkali Activator Solution were added. The geopolymer mixtures were then mixed properly in an electric mixer for about 15 to 20minutes before molding in a photo tube. Entrapped air was excluded by vibrating the photo tube slightly on a solid surface.

3.2. Solid to Liquid Mix Ratio: 1.4

For every 140g of clay sample, 100g of the Alkali Activator Solution were added. The geopolymer mixtures were then mixed properly in an electric mixer for about 15 to 20minutes before molding in a photo tube. Entrapped air was excluded by hitting the photo tube slightly on a solid surface.

3.3. Solid to Liquid Mix Ratio: 1.3

For every 130g of clay sample, 100g of the Alkali Activator Solution were added. The geopolymer mixtures were then mixed properly in an electric mixer for about 15 to 20minutes before molding in a photo tube. Entrapped air was excluded by hitting the photo tube slightly on a solid surface.

4. Geopolymer Curing at Room Temperature

The molded samples were allowed to set at ambient temperature for 24hours. It was then demolded and cured at room temperature for 7, 14, 21 and 28days respectively in a plastic sealed bag to prevent excessive evaporation. The cured samples were examined every 7days to ascertain the level of improvement in their mechanical properties. After curing for 7days, the compressive strength tests conducted showed that the geopolymer solid to liquid mix ratio of 2.0 gave the highest compressive strength. This mix ratio was then used for subsequent geopolymer samples produced.

Figures 5 and 6 below are some of the samples of the synthesized geopolymers.



Figure 5. Geopolymer Samples synthesized from Ikere Kaolinite.



Figure 6. Geopolymer Samples synthesized from Ikare Kaolinite.



Figure 7. Compression test machine for measuring compressive strength and calcined kaolin geopolymers.

5. Mechanical Characterization of the Synthesized Geopolymers

5.1. Determination of Comprehensive Strength

The comprehensive strength of the geopolymer binders and bricks were calculated from the failure load which is the force (Newton) applied to break a test specimen divided by the cross sectional area (mm^2) resisting the load and reported in unit of (N/mm^2) [13]. This is important because the strength test can be used for quality control, acceptance of bricks, or for estimating the brick strength in a structure. The compressive strength of the geopolymers was measured by placing the sample in a compressive test machine plate and applying a force on the specimen with the testing apparatus until the specimen fails or crushes. Upon failure, the failure Load was recorded in Kilo Newton (KN). The compressive strength is then computed and recorded in N/mm^2 . Figure 7 is a compressive test machine used for the compressive test determination. Two geopolymers were measured for each geopolymeric specimen tested and the strength calculated using the expression:

$$\text{Compressive Strength } (\text{N}/\text{mm}^2) = \frac{\text{Load } (N)}{\text{Area } (\text{mm}^2)}$$

Where load is the applied force and the denominator is the cross-sectional area of the cylindrical specimen (Area of a Cylinder = $2\pi r^2 + 2\pi rh$).

5.2. Determination of Water Absorption Capacity

Water absorption capacity is the ability of a specimen to absorb and retain water. It is an important property that influences the durability of bricks. Geopolymers have hydrophilic character and micro-porous structure [14]. Water absorption capacity of the mature specimen was assessed by weighing the synthesized geopolymer brick in an analytical balance (W_d) and then immersing the sample completely in distilled water for 28 days at ambient temperature; the geopolymer sample was taken out, placed on a flat board and air dried for about 4 hours at room temperature. The weights (W_w) of the wet material were then measured in an analytical balance for each geopolymer test specimen and water absorption capacity calculated as follows:

$$\begin{aligned} \text{Percentage Water Absorption Capacity, } W\% \\ = \frac{(W_w - W_d)}{W_d} \times 100 \end{aligned}$$

Where W_w and W_d are Weight of wet and dry geopolymer respectively. ASTM specification for different types of cement bricks (C62, C216 and C902) and load bearing concrete masonry units were used to evaluate the quality of the synthesized geopolymer bricks [15]. The results for water absorption and comprehensive strength of the different geopolymers synthesized were compared with the ASTM specification standard. Figure 8 shows geopolymer samples immersed in water for water absorption capacity tests.

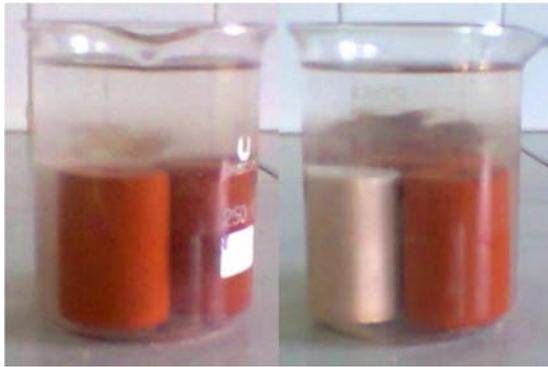


Figure 8. Water Absorption Capacity Tests specimen immersed in water for 28days.

5.3. Determination of Bulk Density

Density is defined as the average mass per unit volume; it is a measure of how much matter is squeezed into a given space. The more closely packed the molecules, the higher the density of the material. The mass of each geopolymer brick was measured in an analytical balance, the diameter and length of the specimen was determined using metric rule and the density was then calculated using the formula:

$$\text{Density} = \frac{\text{mass (g)}}{\text{Volume (cm}^3\text{)}}$$

5.4. Determination of Moisture Content of Rice Husk

Both the moisture and ash content of the unprocessed rice husk ash were determined by gravimetric method. For moisture content determination, about 10g of Rice Husk sample were weighed into a clean, dried evaporating dish and placed in an oven set at 105°C and allowed to dry for about 2hours. This sample was weighed periodically until a constant Weight was obtained. Constant weight refers to

repeated drying steps until two consecutive weights agree to within a specified precision, such as having two consecutive weights that does not differ significantly. The ash content was also determined in a like manner. The results were calculated as either the percent loss in weight or the percent moisture or water content as shown below:

Percentage Moisture Content

$$\frac{\text{Weight Loss or Weight of Water}}{\text{Weight of Sample}} \times 100$$

$$\frac{\text{(Weight before Drying - Weight after Drying)}}{\text{Weight before Drying}} \times 100$$

5.5. Determination of the Ash Content of the Rice Husk

The mineral particles and ash that remain after the ignition of Rice Husk sample were also calculated and reported. The mineral and ash are known as the residue. In any case, about 10g of the sample were accurately weighed into a clean, dry crucible and dried in a muffle furnace set at 450°C for about three hours until the sample was completely ashed as indicated by greyish to whitish colour which was observed. The changes can still be just simple evaporation of water and other components that would volatilize at the temperature used, although some procedures require that the sample be dried first. The calculation is also similar to loss on drying.

$$\text{Percentage Ash Content} = \frac{\text{Weight of Ash}}{\text{Weight of Sample}} \times 100$$

6. Results and Discussion

6.1. Compressive Strength Test

The results obtained for the compressive strength of the geopolymer bricks and binders are shown on the table below:

Table 1. Compressive Strength for Ikere and Ikare Kaolinite Comprising of Clay-to-Alkaline Activator Solution Ratios 1.3, 1.4, and 2.0

Curing Duration at Room Temp.	Compressive Strength±SD (N/mm ²) of Mix Ratio 1.3	Compressive Strength±SD (N/mm ²) of Mix Ratio 1.4	Compressive Strength±SD (N/mm ²) of Mix Ratio 2.0
IKERE GEOPOLYMER BINDERS			
7 Days	3.90±0.16	4.57±0.16	5.58±0.32
14 Days	5.13±0.31	5.35±0.32	6.47±0.32
21 Days	6.58±0.47	7.35±0.32	9.37±0.32
28 Days	7.81±0.32	10.6±0.16	13.2±0.31
IKARE GEOPOLYMER BINDERS			
7 Days	1.45±0.16	1.90±0.16	2.56±0.47
14 Days	1.67±0.16	2.34±0.16	3.01±0.16
21 Days	3.46±0.16	4.24±0.32	5.13±0.32
28 Days	4.57±0.16	5.35±0.32	8.25±0.32

From the results, it is obvious that compressive strength of the geopolymer mixes increases with time progressively from 7days through 28days. The strength is also dependent on the solid to liquid mix proportions as can be seen from the tables. The geopolymer mix of 2.0 gave the highest compressive strength for both clay samples. It is worthy to note however that the compressive strength depends on a number of factors such as temperature, time, geopolymer mix ratio, Alkaline Activator Solution among others. For instance, Ikere clay

cured at room temperature with varying ratio of geopolymer source material to alkaline activator solution gave different strengths as can be seen from the tables with mix ratio of 2.0 giving the highest compressive strength with time. Figures 9 – 12 show variations in compressive strength of the geopolymers with time for a given Solid to Liquid mix ratio as well as variations in compressive strength of the geopolymers with varying Geopolymer Source Materials to Alkaline Activator Solution (GSM-to-AAS) ratios.

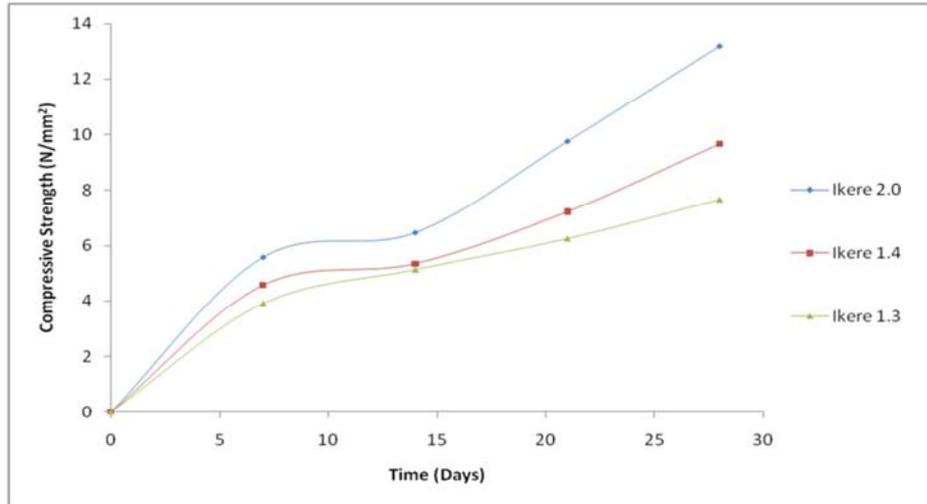


Figure 9. Variations in Compressive Strength of Ikere Geopolymer Binder with time for Clay-to-Alkaline Activator Solution Ratios 1.3, 1.4, and 2.0.

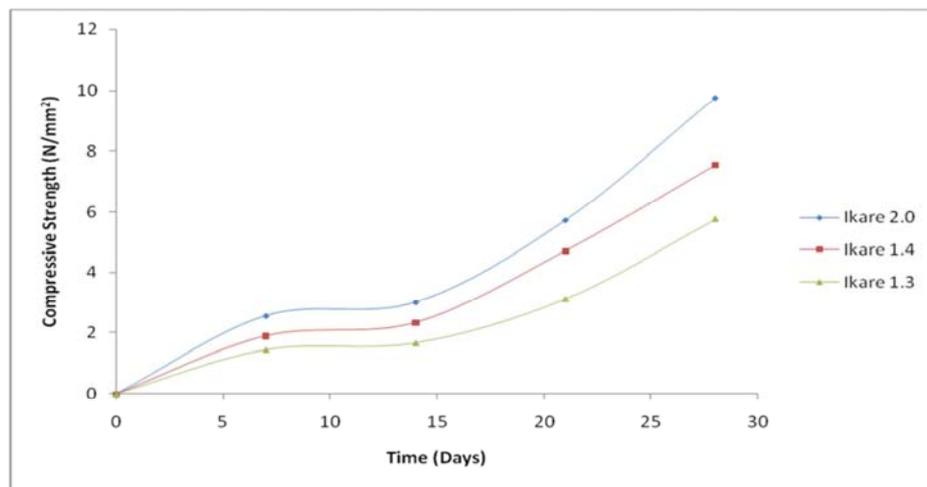


Figure 10. Variations in Compressive Strength of Ikere Geopolymer Binder with time for Clay-to-Alkaline Activator Solution Ratios 1.3, 1.4, and 2.0.

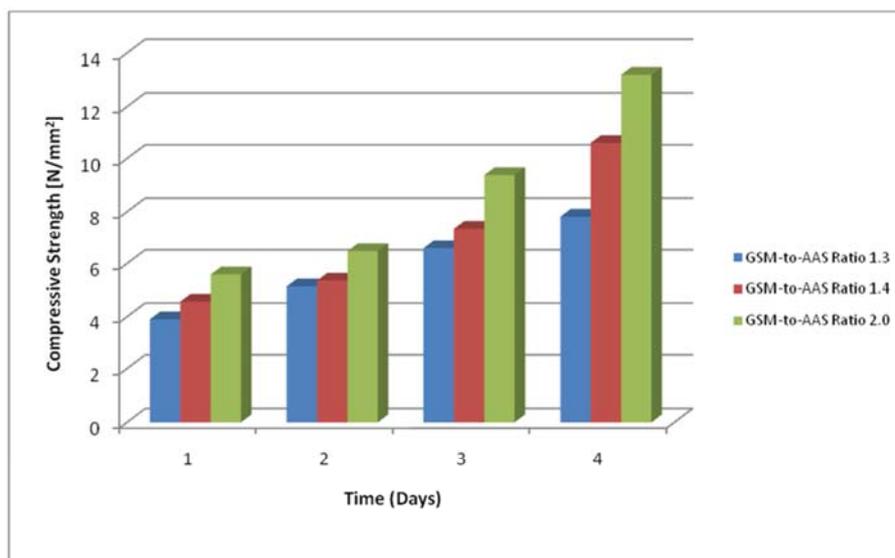


Figure 11. Compressive strength of Ikere Clay of Varying Geopolymer Source Materials to Alkaline Activator Solution Ratio against Time.

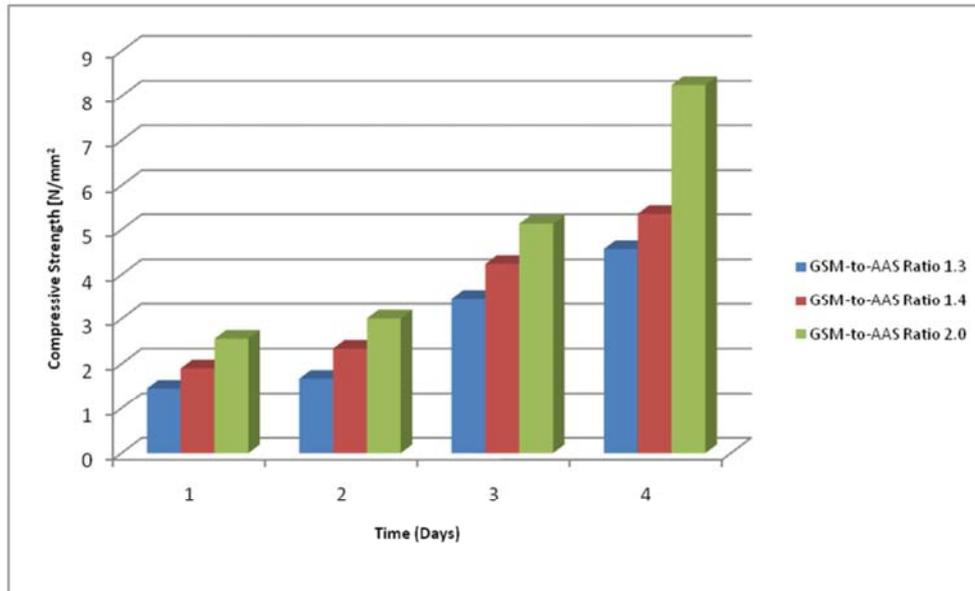


Figure 12. Compressive strength of Ikare Clay of Varying Geopolymer Source Materials-to-Alkaline Activator Solution Ratio against Time.

Subsequently, the geopolymer mix ratio that gave the highest compressive strength (i.e. mix ratio 2.0) was used to examine the effect of incorporating Rice Husk Ash on the compressive strength of geopolymer binders; also, this same mix ratio was used to make geopolymer bricks. The moisture

and Ash content of the Rice Husk Ash were found to be 7.69% and 20.03% respectively. The compressive strength results of the synthesised geopolymer samples are shown on the tables below:

Table 2. Compressive Strength of RHA-CLAY Geopolymer Binders Made From Ikere and Ikare Clay Samples Comprising of RHA-to-CLAY Ratios 1:1, 1:2, and 1:3.

Curing Duration at Room Temp.	Compressive Strength±SD (N/mm²) of Mix Ratio 1:1	Compressive Strength±SD (N/mm²) of Mix Ratio 1:2	Compressive Strength±SD (N/mm²) of Mix Ratio 1:3
IKERE GEOPOLYMER BINDERS			
7 Days	6.02±0.31	5.13±0.32	4.13±0.16
14 Days	7.58±0.32	6.91±0.31	5.69±0.47
21 Days	11.3±0.47	9.37±0.32	7.92±0.16
28 Days	15.4±0.31	13.6±0.31	9.70±0.16
IKARE GEOPOLYMER BINDERS			
7 Days	4.24±0.32	3.46±0.16	2.34±0.47
14 Days	5.91±0.16	4.35±0.47	3.35±0.32
21 Days	8.36±0.16	6.24±0.31	5.13±0.32
28 Days	12.9±0.32	9.48±0.16	7.69±0.16

Table 3. Compressive Strength of Geopolymer Bricks Made From Ikere and Ikare Clay Samples Comprising of Clay-to-Sand Ratios 1:1, 1:2, and 1:3.

Curing Duration at Room Temp.	Compressive Strength±SD (N/mm²) of Mix Ratio 1:1	Compressive Strength±SD (N/mm²) of Mix Ratio 1:2	Compressive Strength±SD (N/mm²) of Mix Ratio 1:3
IKERE GEOPOLYMER BRICKS			
7 Days	5.35±0.32	4.24±0.32	3.12±0.32
14 Days	6.13±0.16	5.35±0.32	4.24±0.32
21 Days	9.14±0.31	7.81±0.32	5.46±0.16
28 Days	13.1±0.16	9.37±0.32	6.91±0.32
IKARE GEOPOLYMER BRICKS			
7 Days	2.79±0.16	2.01±0.32	1.45±0.16
14 Days	3.46±0.16	2.90±0.32	2.34±0.16
21 Days	5.91±0.16	4.24±0.32	3.57±0.32
28 Days	9.81±0.32	7.25±0.16	7.25±0.16

Table 4. Compressive Strength of Ordinary Portland Cement Mixes at Room Temperature Comprising Ordinary Portland Cement-To-Sand Ratio 1:1, 1:2 and 1:3.

Curing Duration at Room Temp.	Compressive Strength±SD (N/mm ²) of Mix Ratio 1:1	Compressive Strength±SD (N/mm ²) of Mix Ratio 1:2	Compressive Strength±SD (N/mm ²) of Mix Ratio 1:3
7 Days	5.58±0.32	4.68±0.32	4.24±0.32
14 Days	6.36±0.47	5.69±0.16	4.35±0.47
21 Days	10.0±0.32	8.92±0.32	5.91±0.16
28 Days	13.6±0.32	11.4±0.32	7.92±0.16

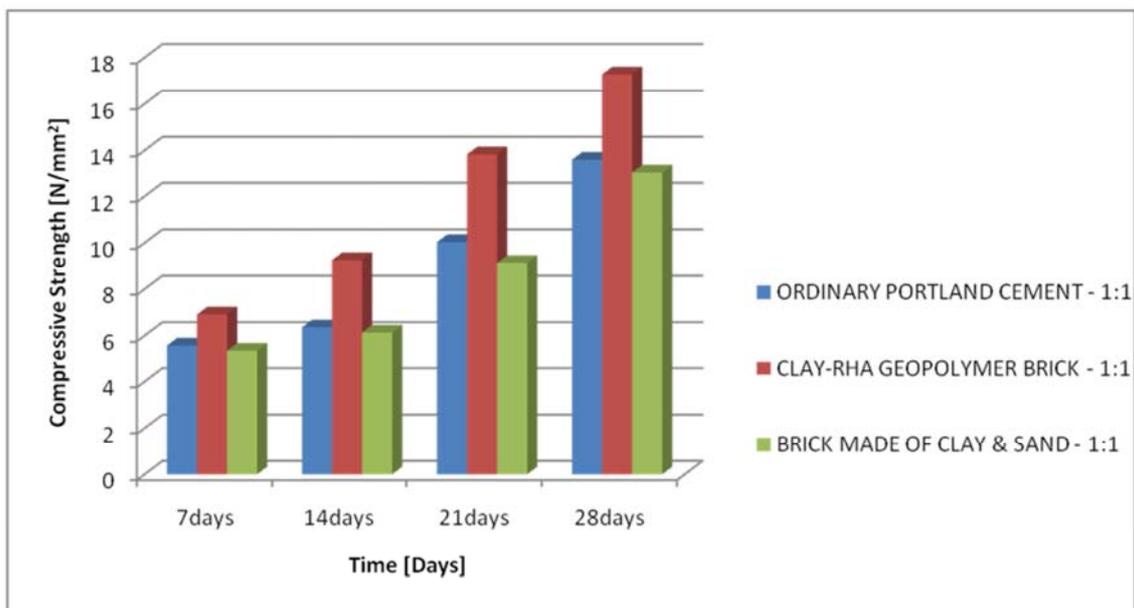
6.2. Influence of RHA on the Synthesised Geopolymer Binder

The compressive strength test carried out on Clay-Rice Husk Ash geopolymers shows a tremendous increase in strength of the synthesised geopolymer compared to the ones synthesised with clay samples alone. This can be seen in Table 2 above. For Ikere Clay-RHA geopolymer, the highest compressive strength obtained for Clay-to-RHA Ratio 1:1 was 15.4±0.31N/mm² against 13.2±0.31 N/mm² obtained with Ikere clay alone. Similarly, for Ikere Clay-RHA geopolymer of Clay-to-RHA Ratio 1:1, the highest compressive strength obtained was 12.9±0.32N/mm² against 8.25±0.32N/mm² obtained from Ikere clay alone.

Such improvements in strength can be explained by two possible reasons: (a) an increased amount of reactive silica from the RHA results in a higher density of the Si-O-Si bonds in the geopolymer, leading to a higher strength and stiffness; and (b) a

higher amount of RHA with a high specific surface area makes the end products more ductile [6]. Theoretically, the Si-O-Si bonds are stronger than those of Si-O-Al and Al-O-Al, implying that the strength of geopolymers should increase with the Si/Al ratio because the density of the Si-O-Si bonds increases with the Si/Al ratio [6]. The geopolymer bricks also showed impressive strength with time increasing progressively from 7days through 28days. However, the geopolymer binders as well as the bricks produced with Ikere clay showed a greater compressive strength than that produced with Ikere clay samples.

Ordinary Portland cement was used as a control in this experiment; from the results shown in Tables 2 to 4 above, bricks made from RHA-CLAY geopolymer showed impressive strength compared to that made from ordinary Portland cement. This shows the potential of these agricultural and industrial Wastes as materials for sustainable geopolymer brick manufacturing. Figure 13 below depicts this trend.

**Figure 13.** Compressive Strength of Ordinary Portland Cement, Ikere Clay-RHA Geopolymer Brick and Bricks Made of Ikere Clay and Sand Alone against Time.

6.3. Influence of Sand Filler on Ikere Clay-RHA Geopolymers

Ikere Clay-RHA geopolymer with mix- ratio of 50-50 (1:1) was selected to assess the influence of sand filler on the

mechanical properties of the synthesised geopolymer binders. The compressive test results obtained are shown in Table 5 below:

Table 5. Influence of Sand Filler on Ikere Clay-RHA Geopolymers Comprising 50:50:10, 50:50:25, and 50:50:50, of CLAY-RHA-SAND Mix Ratios Respectively.

Curing Duration at Room Temp.	Compressive Strength±SD (N/mm ²) of Mix Ratio 50:50:10	Compressive Strength±SD (N/mm ²) of Mix Ratio 50:50:25	Compressive Strength±SD (N/mm ²) of Mix Ratio 50:50:50
7 Days	6.91±0.31	5.80±0.32	4.35±0.47
14 Days	9.26±0.16	7.81±0.32	6.36±0.16
21 Days	13.8±0.32	11.8±0.32	8.59±0.16
28 Days	17.3±0.16	13.6±0.31	11.8±0.32

From the results obtained, it is obvious that the sand filler (at this given ratio) has detrimental influence on the compressive strength and stiffness of the binders. The compressive strength of the geopolymers increased with the addition of sand filler but decreased subsequently with increase in the amount of sand added. This is illustrated in the figure below.

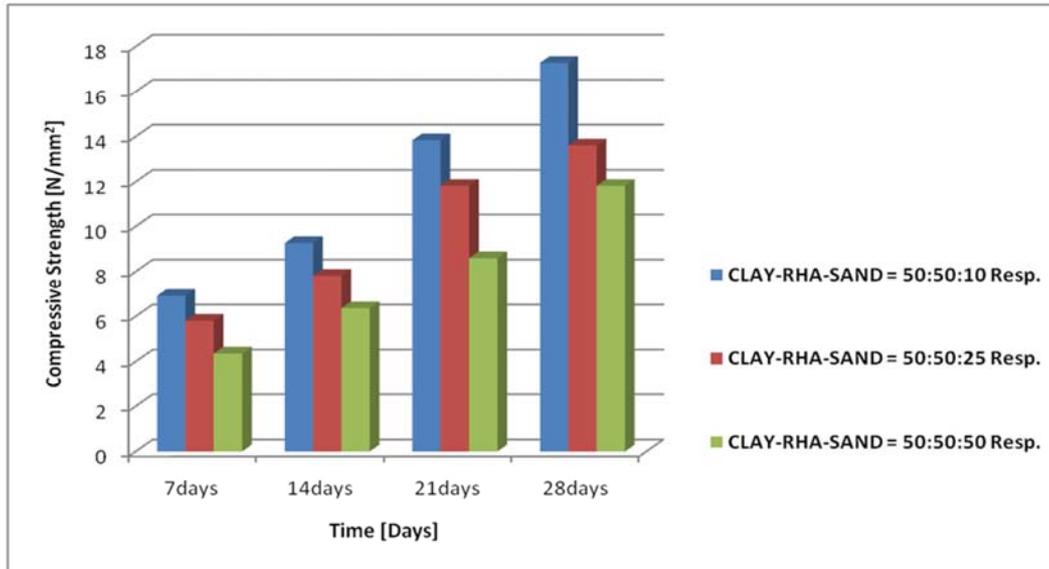


Figure 14. Compressive Strength of Clay-RHA-Sand Geopolymer Bricks Against Time.

Two possible reasons may account for this trend in the synthesized geopolymers: (1) the sand does not react with the geopolymer matrix; it is simply presents as inactive filler, and (2) the geopolymer already contains a high content of other constituents as inactive fillers, and adding more sand results in excessive concentration of inactive filler but insufficient geopolymer binder [16].

From these results, it is obvious that Ikere clay is a better clay for making geopolymer binder than Ikare clay; this could be probably due to variation in Al/Si content of the respective clay samples. Therefore Ikere clay is recommended for high compressive strength and environmentally friendly geopolymer synthesis when compared to its counterpart.

6.4. Comparison with American Society for Testing and Materials (ASTM) Specifications

Most of the time, ASTM specifications are used as

Table 6. ASTM specifications for different cement bricks and load-bearing concrete masonry units (ASTM C62, C216, C902, and C90).

Title of Specification	ASTM Designation	Weathering Conditions	Minimum Compressive Strength (MPa)	Minimum Water Absorption (%)
Building Brick	C62	SW	20.7	17
		MW	17.2	22
		NW	10.3	No Limit
Facing Brick	C216	SW	20.7	17

standards for certifying bricks suitable for a given construction purpose. In this study, ASTM specifications for different types of cement bricks (C62, C216, and C902) as well as load-bearing concrete masonry units (C90) were used to evaluate the quality of geopolymer materials. Table 6, summarizes the minimum compressive strengths and the maximum water absorptions for different types of cement bricks and load-bearing concrete masonry units.

Comparing the compressive strength of the synthesized geopolymer with the ASTM standard, geopolymers synthesized with Ikere clay satisfied ASTM specification standard C62, C90, and C216 under moderate or negligible weather condition. Therefore, this clay is suitable for making load bearing masonry, pedestrian and traffic paving brick except in severe weathering condition.

Title of Specification	ASTM Designation	Weathering Conditions	Minimum Compressive Strength (MPa)	Minimum Water Absorption (%)
Pedestrian and Traffic paving brick	C902	NW	17.2	22
		SW	55.2	-
		MW	20.7	-
		NW	20.7	-
Load-bearing Masonry	C90	-	13.1	17

SW indicates severe weathering condition, MW indicates moderate weathering condition and NW indicates negligible or no weathering condition (Mohsen and Mostafa, 2010) [17].

6.5. Water Absorption Test

Water absorption is an important property that influences the durability of bricks; the lower the water absorption, the higher the resistance to water infiltration and environmental damage [18]. Water absorption test were carried out on all the synthesized geopolymers by immersion in water at ambient temperature for 28days. For each geopolymer sample, two samples were immersed to ensure higher level of accuracy. The Bulk Density of the Geopolymers were also determined;

the mass of each geopolymer brick were measured in an analytical balance, the diameter and length of the specimen was determined using metric ruler and the density calculated using the formula:

$$\text{Density} = \frac{\text{mass (g)}}{\text{Volume (cm}^3\text{)}}$$

The results obtained are as shown in Tables 7 to 9 below:

Tables 7. Water Absorption and Bulk Densities of Ikere and Ikare Geopolymer Binders

Geopolymer Samples	Water Absorption Capacity (g)	Percentage Water Absorption Capacity (%)	Bulk Density (g/cm ³)
Ikere Clay 1.3	2.56	4.59	2.45
Ikere Clay 1.4	2.47	4.49	2.41
Ikere Clay 2.0	2.38	4.34	2.41
Ikare Clay 1.3	3.56	5.50	2.84
Ikare Clay 1.4	3.25	4.98	2.86
Ikare Clay 2.0	2.26	3.47	2.86

Tables 8. Water Absorption and Bulk Densities of Ikere and Ikare Geopolymer Binders Mixed with Rice Husk Ash (RHA).

Geopolymer Samples	Water Absorption Capacity (g)	Percentage Water Absorption Capacity (%)	Bulk Density (g/cm ³)
Ikere Clay + RHA 1:1	2.27	4.25	2.34
Ikere Clay + RHA 1:2	2.91	5.46	2.34
Ikere Clay + RHA 1:3	3.81	7.19	2.32
Ikare Clay + RHA 1:1	3.10	4.88	2.79
Ikare Clay + RHA 1:2	4.77	7.38	2.83
Ikare Clay + RHA 1:3	5.03	7.96	2.77

Tables 9. Water Absorption and Bulk Densities of Ikere and Ikare Geopolymer Bricks

Geopolymer Samples	Water Absorption Capacity (g)	Percentage Water Absorption Capacity (%)	Bulk Density (g/cm ³)
Ikere Clay + Sand 1:1	0.97	1.49	2.86
Ikere Clay + Sand 1:2	1.25	1.90	2.88
Ikere Clay + Sand 1:3	1.42	2.13	2.92
Ikare Clay + Sand 1:1	1.48	2.16	3.01
Ikare Clay + Sand 1:2	1.50	2.20	2.99
Ikare Clay + Sand 1:3	1.69	2.50	2.96

Tables 10. Water Absorption and Bulk Densities of Clay-Rice Husk Ash (RHA)-Sand Geopolymer Bricks and Bricks made of Ordinary Portland Cement (OPC).

Geopolymer Samples	Water Absorption Capacity (g)	Percentage Water Absorption Capacity (%)	Bulk Density (g/cm ³)
CLAY-RHA-SAND MIX: 50-50-10 (RESP.)	1.42	2.21	2.82
CLAY-RHA-SAND MIX: 50-50-25 (RESP.)	1.50	2.31	2.85
CLAY-RHA-SAND MIX: 50-50-50 (RESP.)	1.58	2.40	2.89
OPC 1:1	1.54	2.62	2.58
OPC 1:2	1.58	2.69	2.58
OPC 1:3	1.93	3.27	2.59

From these results, the water absorptivity of the geopolymer samples appears to be relatively low. The water absorption capacity of the various geopolymer samples increases with decrease in compressive strength of the geopolymers. It can be seen that all absorption values of the

geopolymer bricks were lower than the limit of ASTM standard specification given in Table 6 for different cement bricks and load-bearing concrete masonry units. When water passes through bricks, it invariably does so through separations or cracks between the brick units; hence, as the

bricks develop more strength, the geopolymer bonds and intermolecular spaces between the geopolymer units decreases and subsequently, it develops high resistance to moisture [18].

Bricks made from Ordinary Portland cement have about the same percentage of water absorption capacity to that made from clay geopolymers. It is also important to note that the water absorptivity was higher in Clay-RHA geopolymers compared to clay geopolymers alone indicating their improved porosity over the latter. Therefore, it can be concluded from the results that water absorptivity is directly proportional to porosity and inversely proportional to the compressive strength of the geopolymer samples.

7. Conclusion

Based on the results of the experiments compared to ASTM standard specifications, the following conclusions could be drawn:

- 1) The synthesized Clay-RHA geopolymers are a kind of composites consisting of pure geopolymer binders and other phases as fillers;
- 2) The synthesized geopolymer bricks have compressive strength comparable to bricks made from Ordinary Portland Cement.
- 3) The mechanical properties of the Clay-RHA geopolymers are highly complex and dependent upon an array of factors, such as alkalinity, raw material mix ratio, curing duration, Particle size of the geopolymer source materials, and uncertainties involving incomplete geopolymerization and side reactions;
- 4) The studied geopolymers have attractive compressive strengths of up to 17.3N/mm², which is comparable with most Portland cements, suggesting that the Clay-RHA geopolymers can be a potential cementitious construction material;
- 5) This potential technology, if proven successful, can generate significant environmental and economic impacts to the construction, manufacturing, and energy industries.
- 6) Sand filler has detrimental influence on the compressive strength of the geopolymers but decreased subsequently with increase in the amount of sand added.

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References

- [1] Sumajouw, D. J., and Rangan, B. V. (2004). Development of fly ash-based geopolymer concrete. *ACI Materials Journal*. 101 (6): 467-472.
- [2] Hardjito, D., Wallah, S., Sumajouw, D. &Rangan, B.(2004). Properties of Geopolymer concrete with fly ash as source material: effect of mixture composition, the 7th CANMET/ACI International Conference on Recent Advance in Concrete Technology, Las Vegas, USA.
- [3] Abdul-Hamid, A., Sulaiman, R. R., Osman, A., and Saari, N. (2007). Preliminary studies of the chemical composition of rice milling components stabilized by microwave heating. *J. Food Composition and Analysis*. 20: 627-637.
- [4] Davidovits, J., 1991. Geopolymers: Inorganic Polymeric New Materials. *Journal of Thermal Analysis*, 37: 1633-1656.
- [5] Buchwald, A., Hohmann, M., Kaps, C., Bettziehe, H. and Kuhmert, J. T. (2004). "Stabilized foam clay material with high performance thermal insulation properties", *CFI – Ceram Forum Int*. 81, E 39-42.
- [6] Duxson, P., Provis, J. L., Lukey, G. C., Mallicoat, S. W., Kriven, W. M. and van Deventer, J. S. J. (2005). "Understanding the relationship between geopolymer composition, microstructure and mechanical properties", *Colloids and Surfaces A: Physicochemical and Engineering Aspects* 269, 47-58.
- [7] Duxson, P., Fernandez-Jimenez, A., Provis, J. L., Lukey, G. C., Palom, A. and van Deventer, J. S. J. (2007). "Geopolymer technology: the current state of the art" *J. Mater Sci* 42, 2917-2933.
- [8] Palomo, A., Grutzeck, M. W., and Blanco, M. T. (1999). Alkali-activated fly ashes a cement for the future *Cement Concrete, Cement and Concrete Research*. 8, 1323-1329.
- [9] Van Jaarsveld, J. G., van Deventer, J. S. J., Lukey, G. C. (2002). "The effect of composition and temperature on the properties of fly ash- and kaolinite-based geopolymers", *Chemical Engineering Journal* 89, 63-73.
- [10] Pacheco-Torgal, F.; Castro-Gomes, J.; Jalali, S. (2008). Alkali-activated binders: A review. Part 2. About materials and binders manufacture. *Constr. Build. Mater.*, 22, 1315–1322.
- [11] Titiladunayo, I. F. and Fapetu, O. P. (2011) Selection of Clay for Furnace Lining in a Pyrolysis Process. *Journal of Emerging Trends in Engineering and Applied Sciences (JETEAS)*, 2, 938-945.
- [12] Ogundiran M. B. and Ikotun O. J. (2014). Investigating the Suitability of Nigerian CalcinedKaolins as Raw Materials for Geopolymer Binders, *Transactions of the Indian Ceramic Society*, 73, 138–142.
- [13] F. Pacheco-Torgal, J. Castro-Gomes, and S. Jalali, (2007). Cementitious building materials reinforced with vegetable fibres: A review. *Cement and Concrete Research*, 37, 933–941.
- [14] Fletscher, R. A., Mac Kenzie, K. J. D., Nicholson, C. L. and Shimada, S. (2005). "The composition range of aluminosilicategeopolymers". *J European Ceram. Soc*. 25, 1471-1477.

- [15] ASTM – American Society for Testing and Materials. (2005). *Annual Book of ASTM Standards*. C150–05. possibility of utilizing low kaolinitic clays in production of geopolymer bricks. *Ceramics – Silikaty* 54. 2: 160-168.
- [16] De Jong, B. & G. Brown, (1980). *GeochimCosmochim Acta*. 44 (3): 491.
- [17] Mohsen Q. and Mostafa N. Y. (2010). Investigating the
- [18] Lodeiro, G., Palomo, A. and Jiménez, F. (2007). “Alkali-aggregate reaction inactivated fly ash system”, *Cement Concrete Res.*, 37, 175-183.