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Strength Properties of Metakaolin-Fly Ash based Geopolymer Concrete

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Abstract:
Under this study, the combine compressive strength of concrete mortar containing a locally prepared pozzolana of Metakaolin and an adhesive agent to partially replaced Ordinary Portland Cement (OPC) was checked. The material was found to have many properties of the OPC and can pertly replace the cement for some certain required strength. Several specimens were prepared in the proposed research work to study the behavior of fly ash and metakaolin-based geopolymer concrete. Activating agents include sodium hydroxide and sodium silicate. Ordinary Portland cement will thus take the place of native industrial by-products (such as fly ash) and metakaolin. The specimens' mechanical and durability properties were investigated. Microimaging analysis techniques was also used to evaluate the proposed concrete's microstructural properties. Furthermore, a critical review of literature on the behavior of geopolymer concrete as a construction material was first established. Other experimental procedures such as the determine the optimum mix composition of fly ash and metakaolin and the determine the optimum percentages of sodium hydroxide and sodium silicate was also conducted.
Key Word: *Compressive Strength, Metakaoline, Geopolymer concrete, Fly Ash, Green House Gasses.*

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I. Introduction

Because of its importance and advantages in the construction industry, concrete is one of the most widely used construction materials on the planet. However, cement production (a necessary component of concrete) consumes a significant amount of energy. As a result, significant amounts of carbon dioxide (CO2) and other greenhouse gases (GHGs) are released into the atmosphere. It was discovered that one ton of cement produces one ton of CO2 and other greenhouse gases. These gases result of the environmental issues associated with cement production, the construction and building industry needed to develop in a more sustainable material to replace cement.

As a result, cement production emitted 1.5 billion tons of CO2 globally, accounting for around 5% of total GHG emissions per year. If the current downward trend persists, the figure will reach 6% by 2015. (Ken et al., 2015). The proposed study examines the feasibility of fully replacing OPC with a mix of fly ash and metakaolin-based geopolymer. Activating agents include sodium hydroxide and sodium silicate. Since geopolymer concrete is a relatively new research field in Nigeria, there hasn't been much research done on its properties yet.

Davidovits coined the word "geopolymer" in 1978 to define a class of mineral binders with a chemical composition similar to zeolites but an amorphous microstructure. He also proposed the term "poly(sialate)" for the chemical classification of silico-aluminate-based geopolymers. Ordinary Portland/pozzolanic cements form calcium-silicate-hydrates (CSHs), which are responsible for the strength and other properties of cement-based materials; however, geopolymers gain structural strength by polycondensing alumina and silica precursors with a high alkali content (Wallah and Rangan, 2006).

Similarly, in this research, we characterize the samples using scanning electron microscopy (SEM), X-ray fluorescence (XRF), and X-ray diffraction (XRD). The study also evaluates the developed concrete's economic feasibility in comparison to ordinary Portland cement concrete. Concrete developed by the construction industry will be identified. Ordinary Portland cement (OPC) is made by heating various raw materials in a kiln at about 1450 degrees Fahrenheit. After thermal power plants and steel, Portland cement manufacturing is the most energy-intensive operation, according to Today's Concrete Technology (2010). Per ton, it consumes 4GJ of energy. Furthermore, 1 ton of Portland cement necessitates approximately 2.8 tonnes of raw material, which necessitates the use of fossil fuel for quarrying, packaging, transportation, and unloading of the material to the facility (Criado et al., 2005 and Guo et al., 2010). Furthermore, it was discovered that 1 ton of cement produces approximately 1 ton of CO_2 and other GHGs. (Celik et al., 2014; Chong et al., 2013; Pourkhorshidi et al., 2010 Turanli et al

Furthermore, we examine the properties of the new concrete using locally available materials to test the toughness and microstructural properties of geopolymer concrete in this report. The properties of fly ash and metakaolin-based geopolymer concrete combined with sodium hydroxide and sodium silicate as triggering agents are also investigated. The findings were compared to OPC concrete to see whether fly ash and metakaolin-based geopolymer could be used as an OPC.

Geopolymer, which described a family of mineral binders with a chemical composition like zeolites but with an amorphous microstructure, was developed by Davidovits in 1978. He also suggested using the term 'poly (sialate)' for the chemical description of geopolymers based on silico-aluminate. It is known that ordinary Portland/pozzolanic cement formed calcium-silicate- hydrates (CSHs), which are responsible for the strength and other properties of cement-based materials. More so, geopolymers used a high alkali content and polycondensation of alumina and silica precursors to achieve structural strength (Wallah and Rangan, 2006). Geopolymers are alternative cementitious materials made by combining silica and alumina-rich pozzolanic materials. Fly ash (FA) and ground granulated blast-furnace slags are examples of these materials (GGBS). Other resources include: include potassium hydroxide (KOH) and soluble silicates (in most cases), such as sodium silicate, in which dissolved Al_2O_3 and SiO_2 species polymerize to shape a three-dimensional amorphous aluminosilicate network with strength comparable to or greater than OPC concrete (Ken et al., 2015). As a result, geopolymers are also known as alkali-activated aluminon silicate binders.

The ability of geopolymer binders to replace conventional OPC binders was bolstered by the fact that there is a plethora of industrial by-products produced in various industries that have been discovered to be suitable for use as geopolymer source materials, all of which are posing disposal challenges. Fly ash (FA), for example, is a by-product of the coal-fired power plant industry that accounts for 75–80 percent of global annual ash output (Joseph and Mathew, 2012).

As a result of the ongoing challenge of minimizing OPC use in the construction industry, as well as the challenges of disposing of industrial by-products in different industries, geopolymer binder has a strong chance of replacing OPC binder in the construction industry (Ken et al., 2015).

Kenne et al. (2015) investigated the effects of kaolin calcination rate on metakaolin-based geopolymer properties. They calcined Kaolin samples of the same mass at 700°C for 30 minutes at different calcination rates (1, 2.5, 5, 10, 15, and 20°C/min) to obtain metakaolin, which was then used to make geopolymers. Thermal analysis, chemical analysis, XRD, FTIR, particle size distribution, basic surface area, bulk density, setting time, and compressive strength were used to characterize and test the kaolin, metakaolins, and geopolymers. Metakaolins, except at 1C/min, contained residual kaolinite, whose quantity increased with the rate of kaolin calcination and affected the characteristics of geopolymers, according to FTIR and XRD studies. As the rate of kaolin calcination increased, the setting time increased (226 min (rate of 1C/min) – 773 min (rate of 20C/min)), while the compressive intensity decreased (49.4 MPa (rate of 1C/min) –20.8 MPa (rate of 20C/min). They came to the conclusion that low-temperature calcination of kaolin is needed for the production of geopolymers with high compressive strength and short setting times.

II. Material and Methods

Materials

The materials used in this research work were the Dangote brand of Ordinary Portland Cement, river sand (fine aggregate), metakaolin, coarse aggregates, and water.

Cement

The cement used in this study was the Dangote brand of Ordinary Portland Cement. The cement was subjected to various standardized tests to check its quality and ensure its conformity with NIS:444-1 (2003) and BSEN197-1 (2000).

Metakaolin (MK)

The kaolin used to produce the MK was obtained from Kankara L.G.A. of Katsina state. The sample of the metakaolin is heated to burn at about 700°c using a kerosene vaporization burner kiln. A pyrometer monitored the temperature until a required level was attained. After that, the ash could cool and then be allowed to pass through a 212 microns sieve. The MK conformed to ASTM C 618 -12a:1978, Type N pozzolanas specification

Aggregate
Locally available river sand was used as fine aggregate. Before using it, sieve analysis following BS 812-103.1(1995) was done to determine its grading.

Water
According to BSEN1008 (2002), water for concrete should be of portable quality (pH -6.8 to 8.0).

Methods

Chemical Composition of Dangote brand of Ordinary Portland cement
The Dangote brand of Ordinary Portland Cement was subjected to a chemical composition test following BSEN196-2 (1995). The chemical composition of the Dangote brand of OPC was determined using X-Ray Fluorescence (XRF). The XRF is a method for qualifying the elements in a material sample.

Physical Properties of Dangote brand of Ordinary Portland Cement
The physical properties of the Dangote brand of Ordinary Portland Cement, which are specific gravity, fineness, standard consistency, and setting time and soundness tests, were determined according to BSEN197-1 (2000).

Grading and Specific gravity of Fine Aggregate
The summary of particle size distribution result is given in Table 3.5, while the detailed result is given in Appendix A-3. Also, the specific gravity of the fine aggregate was determined following BS 812-2(1995).

Consistency of Cement-MK paste
Consistency tests of the Cement and Cement-MK pastes were performed following BSEN196-3 (1995). Concrete pastes made from MK was used to partially replace cement in various percentages. The percentages are 0, 3, 5, 8, and 10% by weight. The control specimen has a percentage of 0 percent. Cement and RHA were thoroughly mixed in dry powdered form in correct proportions for the other percentages. They then thoroughly combined the ingredients with the appropriate amount of water before performing the consistency test. AS0, AS3, AS5, AS8, and AS10 were assigned to each Cement/MK mixture percentage.

For example, AS0 represents 0%, AS2.5 represents 2.5 percent, AS5 represents 5%, AS7.5 represents 7.5 percent, and AS10 represents 10%.

The Vicat apparatus is used to determine normal consistency through a trial-and-error method. Several Cement/MK pastes were made with various amounts of mixing water until the plunger could penetrate 5-7mm. The amount of water required for normal consistency is expressed as a percentage of the dry binder's weight.

Setting Time of Cement-MK paste
BSEN196-3 was used to determine the initial and final setting times of Cement-MK pastes (1995).

Mortar Prismatic Specimen Preparation
Mortar is typically made up of a binder and sand mixture. As specified in BSEN196-1, the mortar used here is one-part binder material to three parts sand (1:3). (1995). The various cement and MK mixture percentages were prepared in a dry powdered form. This was used as a binder, and it was mixed in a ratio of one-part binder to three parts sand, with a water/cement ratio of 0.5, according to BSEN196-1 (1995). M0 represents one part of the cement-containing binder and 0 percent MK (control) mixed with three parts sand in the mortars. M2.5, on the other hand, is a part of the binder that contains cement that has been partially replaced with 2.5 percent MK mixed with three sand. M5 refers to a portion of the binder that contains cement that has been partially replaced with 5% MK mixed with three sand. M8 is a one-part binder containing cement that has been partially replaced with 8% MK and three parts sand. Furthermore, M10 is a one-part binder that contains cement that has been partially replaced with 10% MK and three sand. The labels M0, M2.5, M5, and so on are merely for identification. The quantities of materials used for each mixture are shown in Table 1.

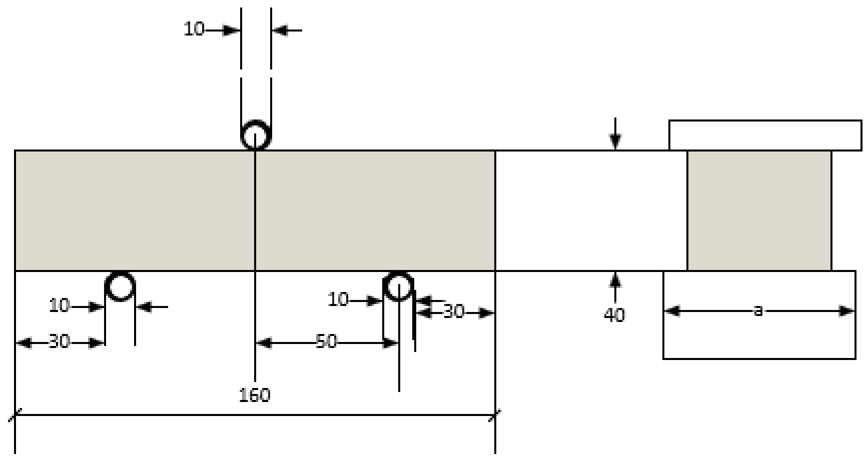
Table 1; Quantities of Materials Used for Mortar (quantity to produce three specimens)

S. No	Description	Cement (g)	MK (g)	Fine Aggregate (g)	Water (g)
1	M0	450	0	1350	225
2	M2.5	436.5	13.5	1350	225
3	M5	427.5	22.5	1350	225
4	M7.5	414	36	1350	225
5	M10	405	45	1350	225

Cement-MK Mortar Drying Shrinkage Test
Before the flexural test, the shrinkage test was performed on the prismatic mortar specimens by measuring their length to determine any shrinkage after thoroughly drying them according to BSEN196-1 (1995).

Cement-MK Mortar Flexural Strength Test

Sixty (60) prismatic test specimens with a cross-section of 40mm x 40mm and a length of 160mm were made and tested for flexural strength in accordance with BSEN196-1 (1995) recommendations. These specimens were cast, demolded after twenty-four (24) hours, and then cured in water according to standard procedure until tested at 2,400°F. The center point loading method was used to determine the flexural strength as specified in BSEN196-1 (1995). The loading arrangement is as shown in Figure 1.



a) Longitudinal Section b) Cross Section

Figure 1: Loading arrangement for the determination of mortar flexural strength

Compressive Strength Test on Cement-MK Mortar

After a flexural strength test, the prism halves were put through a compression test. The compressive area was determined by centering the prism halves laterally to the hard steel auxiliary platens (because the prism halves have an irregular form). The platens must be 40mmx40mm in size and at least 10mm thick, according to BSEN196-1 (1995). The relative altitude of the upper and lower platens was fixed during loading. The result of the passed through the specimen's center. As shown in Figure 3.2, the load was gradually increased at a rate of 2400 200 N/s throughout the load application until fracture.

The halves of the prisms were labeled CS0, CS2.5, CS5, CS7.5, and CS10, respectively. The labels CS0, CS2.5, and CS5 were only used for identification and to represent the different percentages of Cement replacement with MK.

Using the auxiliary steel plates, the specimens were tested as 40mm equivalent cubes. Each cube was then subjected to a compressive strength test.

Scanning Electron Microscope (SEM)

SEM (Scanning Electron Microscope) is a type of microscope that uses electrons (SEM). A scanning electron microscope (SEM) was used to examine the Cement-sand incorporating MK on a microscopic level (phenom proX). The SEM mortar samples were made with the mixture described in the following sections. The sample was placed on a double adhesive sticker and sprayed for 5 seconds in a sputter coater machine. The sample now has a conductive property. The sample stub was then placed on a charge reduction sample holder before being charged into the machine column and the SEM doors were closed.

The machine was allowed to settle for a few seconds before setting the parameters. 15kv was used to image the sample, which was set at 1000x magnification and focused using a rotary knob. In Navcam mode, a clear and proper image was created, which was then transferred to electron imaging mode. The image was then transferred to the Phenom suite software, which performed fiber metric/pore measurements and generated a report. It was saved in a folder named after the sample that was used. The same method was used to determine the pore size and fiber content of the sample.

III. Results And Discussions

Dangote brand of Ordinary Portland Cement

The result of chemical composition analysis of Dangote brand of Ordinary Portland cement (OPC) using XRF is compared with BS EN197-1(2000) requirements as shown in Table 4.1 below.

Table 2: Comparison of Chemical Composition of Dangote brand of Ordinary Portland Cement with BSEN197-1(2000) Specification

Elemental Oxide	Dangote brand OPC Composition (%)	BS EN197-1(2000) Requirement
SiO_2	28.57	Max. 35.5%
Al_2O_3	2.8	Max. 6.3%
CaO	79.12	Limit not specified
Fe_2O_3	4.28	Max. 6.5%
K_2O	0.24	Less than 0.6
MnO	0.01	Limit not specified
SO_3	1.6	Max. 3.5%
TiO_2	0.16	Limit not specified
V_2O_5	0.06	Limit not specified
Cr_2O_3	0.038	Limit not specified
LOI	4.05	Max. 5%

From the above table, the oxide composition values were observed to be within an acceptable limit. Thus, the Dangote brand of OPC can be said to be of sound quality in terms of chemical composition and has satisfied the standard requirement.
Also, the results obtained from the physical properties of the cement were compared with
BS EN197-1(2000) requirement as shown in Table 4.2 below.

Table 3: Physical Properties of Dangote brand of Ordinary Portland Cement in Comparison with BS EN197-1(2000) Specification

S. No	Parameters Tested	BS EN197-1(2000) requirement	Test Result
1	Specific Gravity	3.15	3.12
2	Standard Consistency	26%-33%	30.33%
3	Setting Time (minutes) 1. Initial Setting Time 2. Final Setting Time	 ≥ 45mins < 10hours	 93mins 146mins
4	Soundness	≤ 10mm	0.5mm

From Tables 2 and 3, it could be observed that the cement has satisfied the requirements, and thus it is a standard OPC.

Analysis of Chemical Composition of MK

The rationale behind conducting XRF analysis was to determine the MK's oxide composition and ascertain the percentage composition of the amorphously of the silica therein. The result of XRF is shown in Table 4.3 below.

Table 4; Chemical properties of MK

OXIDE	MK (%)
AL_2O_3	39.80
Fe_2O_3	2.64
SiO_2	54.00
CaO	1.57
MgO	Trace
Na_2O	0.79
K_2O	0.12
LOI	0.018

The ash's SiO2 content was 54.00 percent, according to the table above, and supplementary cementing materials keep the amorphous SiO2 to a minimum. The results also revealed the different oxide compositions of other important oxides, which could be useful in the hydration of cement paste and concrete. According to ASTM

C618, MKA calcined at 700oC for 3 hours contains more than 50% of silica, alumina (39.89 percent), and ferric oxide (2.64 percent) (1993). It was discovered that the alkali content (Na2O) was 0.79 percent. This value is much lower than the maximum alkali requirement for pozzolana, which is 1.5 percent. For pozzolana to be used with reactive aggregate, it must have a high alkali content (Siddique, 2008). In this case, the pozzolana will not be suitable for use in construction projects involving reactive aggregates. The obtained Loss on Ignition (LOI) was 0.018 percent. This is far less than the maximum of 10% required by ASTM C618 for pozzolanas (1993). Higher LOI means more unburned carbon, which lowers the ash's pozzolanic activity. Although unburned carbon is not pozzolanic, its presence in the mixture may serve as a filler.

Table 4: Chemical Composition of Kaolin and Cement.

Oxide	Content %	
	Cement	Kaolin
Al_2O_3	5.2	41.0
SiO_2	21.1	52.8
TiO_2	0.2	0.088
K_2O	0.6	-
CaO	66	0.957
Na_2O	0.4	-
MgO	3.1	0.011
MnO	0.02	-
Fe_2O_3	3.21	0.099
Loss on Ignition	8.0	09

Table 5: Specific Gravity of Metakaolin and Portland cement

Material	Mass of materials (g)	Volume of materials (ml)	% difference
Metakaolin	138	150	2.48
Portland cement	189	150	3.12

Table 6: Slump Test

Types of concrete	Slump (mm)	% Difference
OPC concrete	31	26
Binary Concrete	23	

Table 7: Average Compressive Strength

Concrete samples	Age(days)			
	7	14	28	90
OPC concrete	12.8	37.4	41.1	41.4
Binary concrete	25.5	35.3	46.1	47

Table 8: Average Tensile Strength

Concrete samples	Age(days)			
	7	14	28	90
OPC concrete	1.35	2.07	3.01	3.84
Binary concrete	1.35	2.07	3.84	5.39

Meta-Kaolin (MK): Specific gravity

The result of specific gravity of MK was determined and found to be 2.48; this value is less than 3.12 for Portland cement, as presented in Table 4. Therefore, the lower specific gravity of MK compared to cement will result in a higher volume of water of the same weight to form a standard paste.

Meta-Kaolin (MK): Fineness

The MK is assumed to have satisfied the fineness requirements that qualify it to be placed on the same scale with OPC since it was sieve through a sieve with a smaller aperture. Hence, it is more refined than cement.

Fine aggregate

The result of fine aggregates particle size distribution was presented in Table 9. At the same time, its specific gravity was determined and found to be 2.54.

Table 9: Particle Size Distribution of the Fine Aggregate

BS Sieve Size (mm)	Cumulative Total Passing (%)
2.0	100
1.6	88.5
1.0	64.75
0.6	34.25
0.15	12.25
0.075	0.75
Pan	0.0

The fine aggregates properties were evaluated and found to be well-graded and fall within the grading zone 2 of BS 882(1992). The fine aggregates was therefore found suitable for concrete making.

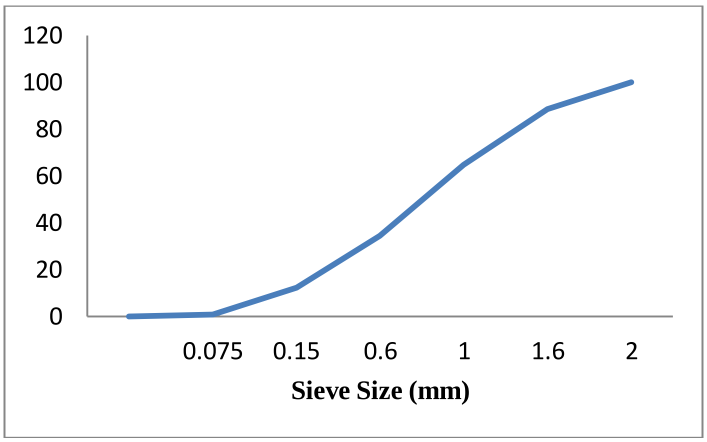


Figure 2: Particle Size Distribution curve (Fine Aggregates)

Meta-Kaolin (MK) on the Consistency

The paste consistency results were presented in Table 10. Each value of the result is an average value of three tests.

Table 10: Average Consistency of Cement-MK paste

S. No	Paste No.	Consistency (%)
1	AS0	30.33
2	AS2.5	32.82
3	AS5	34.38
4	AS7.5	37.50
5	AS10	39.63

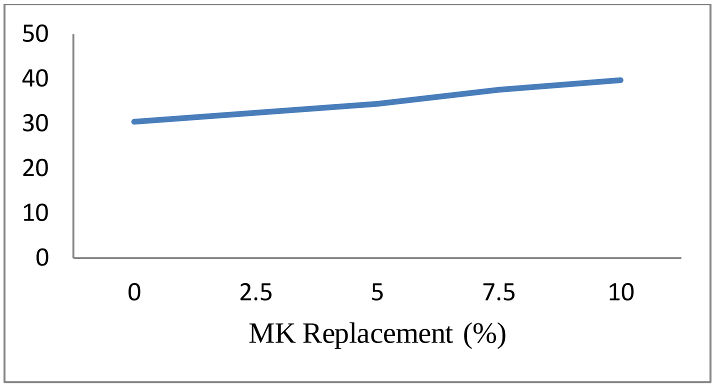


Figure 2: Relationship of consistency to various percent replacement of MK

It can be seen that as the MK replacement increases, the consistency increases; the graph shows a linear relationship. The MK added to the cement raises the carbon content, which raises the amount of water needed to achieve reasonable workability. Based on the amount of water required to achieve a flow equal to within 5% of the control mixture in the strength activity index test mortar, ASTM C 618 (1993) requires a water consistency not to exceed 115 percent of the control mixture.

While BS3892-1 (1997) stipulated a water requirement of 30% Pulverized Fuel Ash (PFA) and 70% Ordinary Portland Cement, intimately mixed water should not exceed 95% of the Portland cement alone. AS2.5 (2.5 percent replacement) outperformed the control paste AS0 (0 percent replacement) by 7.59 percent, and AS5 (5 percent replacement) outperformed the control by 11.78 percent based on these criteria. By 19.12 percent, AS7.5 (7.5 percent replacement) outperformed the control. Finally, AS10 (10% replacement) outperformed the control by 23.47 percent. All of the RHA mixes with OPC met the BS3892-1(1997) and ASTM-C618 consistency requirements (1993).

Samples with high porosity and LOI of MK was discovered that the normal consistency of cement-MK pastes required more water than cement paste alone. Cement paste had a standard consistency of 30.33 percent, while cement-MK pastes had a consistency of 32.82 to 39.63 percent, depending on the MK content. The higher the MK content in the paste, the more water was needed to keep the paste's normal consistency, as previously reported by (Jaturapitakkul and Roongreung, 2003).

It could be observed that the consistency of the control mixture was approximately 30.33%; however, water required for standard consistency linearly increased with an increase in MK content. This is because ashes are hygroscopic, and the specific surface area of MK is much higher than cement; therefore, it needs more water; this finding agrees with the work of (Ganesan et al., 2008).

Effect of MK on the Setting Time

The results of setting times of Cement- MK paste was presented in Table 4.6. Each value of the result is an average value of three tests. The detailed result is given in Appendix A-5.

Table 11: Average Initial and Final Setting Time of Cement- MK paste.

S. No	Paste No.	Initial Setting Time (min)	Final Setting Time (min)
1	AS0	93	146
2	AS2.5	97	154
3	AS5	119	190
4	AS7.5	102	170
5	AS10	107	220

The effect of MK on initial and final setting times when partially replaced with 0, 2.5, 5, 7.5 and 10% MK is shown in Figure 3 (Appendix).

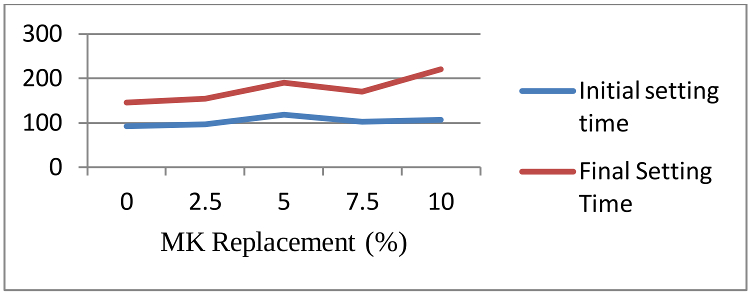


Figure 3: Relationship of setting times to various percent replacement of MK.

The initial and final setting times increased with an increase in MK content, as shown in Figure 4.3. The low rate of hydration in the paste containing MK could explain the increase in set times with an increase in replacement level. Because a high rate of hydration is associated with a high cement content, it is obvious that replacing cement with MK will result in a low heat of hydration and, as a result, a slow rate of setting.

Cement pastes had initial and final setting times of 93 and 146 minutes, respectively. As a result, the setting time of cement-MK pastes was longer than that of cement-based pastes. The initial and final setting times are compared with the BS EN197-1(2000) recommendations presented in Tables 4.7 and 4.8

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Table 12: Initial Setting Time of Cement-MK Compared with BS EN197-1(2000) Requirements

S/No.	Paste No.	Initial Setting Time (min)	BS EN197-1(2000) Requirement (min)
1	AS0 (0 percent replacement)	93	≥45min
2	AS2.5 (2.5 percent replacement)	97	
3	AS5 (5 percent replacement)	119	
4	AS7.5 (7.5 percent replacement)	102	
5	AS10 (10 percent replacement)	107	

Table 13: Final Setting Time of Cement- MK Compared with BS EN197-1(2000) Requirements

S/No.	Paste No.	Final Setting Time (min)	BS EN197-1(2000) Requirement (min)
1	AS0 (0 percent replacement)	146	<10hrs (600min)
2	AS2.5 (2.5 percent replacement)	154	
3	AS5 (5 percent replacement)	190	
4	AS7.5 (7.5 percent replacement)	170	
5	AS10 (10 percent replacement)	220	

All the setting times are within the recommended range based on BS EN197-1(2000). Setting time retardation is advantageous during hot weather, allowing more time to place and finish the concrete. However, during cold weather, pronounced setting time retardation can significantly delay finishing operations and causes disruption in a concrete setting.

Effect of MK on the Soundness

The soundness of Cement- MK paste results were presented in Table 4.9 below. Each value of the result is an average of three tests. The detailed result is given in Appendix
A-6.

Table 14: Average Soundness of Cement- MK paste.

S. No	Paste No.	Soundness (mm)
1	AS0	0.5
2	AS2.5	1.0
3	AS5	1.0
4	AS7.5	1.0
5	AS10	1.0

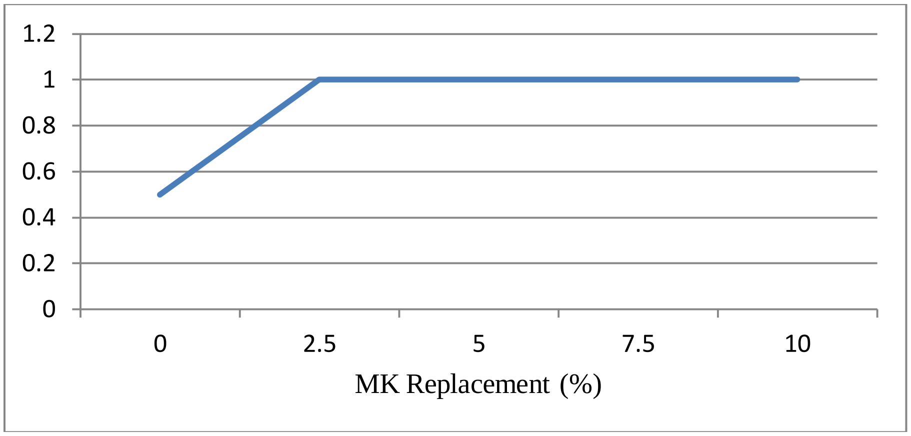

Figure 4: Relationship of soundness to the various percentage of replacement of MK.

From Figure 4 above, it could be deduced that soundness generally increased with an increase in cement replacement with MK from 0% and remained constant for the other replacements levels. Furthermore, it was observed that the ash has no adverse effect on the soundness.

Furthermore, the soundness result was compared with BS EN197-1(2000) recommendations presented in Table 4.10.

Table 15: Soundness of Cement- MK paste Compared with BS EN197-1(2000) Requirements

S. No.	Paste No.	Soundness (mm)	BS EN197-1(2000) Requirements (mm)
1	AS0 (0 percent replacement)	0.5	≤10mm
2	AS2.5 (2.5 percent replacement)	1.0	
3	AS5 (5 percent replacement)	1.0	
4	AS7.5 (7.5 percent replacement)	1.0	
5	AS10 (10 percent replacement)	1.0	

Soundness is the ability of paste to maintain volume after setting. The increase in the RHA may be due to hard-burned CaO with water which results in the formation of calcium hydroxide.

All the various paste mixes (AS0 to AS10) satisfies the code requirements, as they are within the recommended range of less than 10mm for cement, based on BS EN197-1(2000).

Effect of MK **on Mortar Drying Shrinkage**

The effect of MK replacement on mortar shrinkage conducted at 2,7,14, and 28 days are shown in Figure 4.5. From the figure, the shrinkage increased with an increase in curing time. Also, the shrinkage increased apparently with an increase in MK replacement.

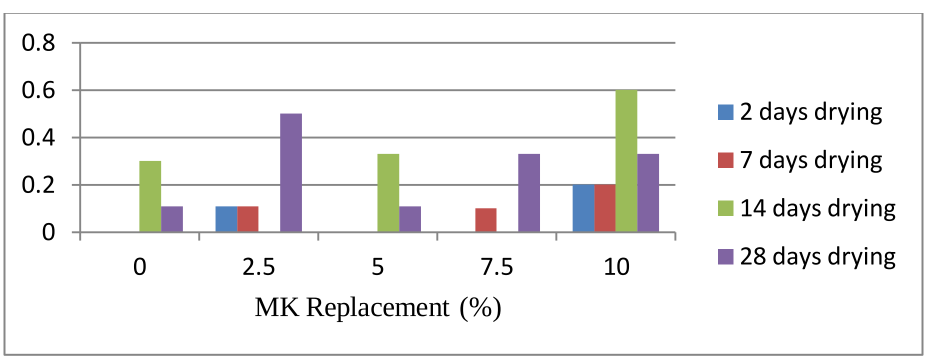

Figure 5: Relationship of drying shrinkage to various percentage replacement of MK.

From Figure 4, the drying shrinkage increased as the curing age increases for all the percentage replacements. Although it could be seen that the 2 days drying shrinkage was less than the 7 days drying shrinkage, the same trend was also observed at 14 and 28 days.

The 28 days drying shrinkage as presented in Table 3.14 and its corresponding percentage reduction from the original specimen (40x40x160) mm are compared with the recommendations of ASTM-C618 (1993) and presented in Table 4.15.

Table 16: Comparison of 28 days Drying Shrinkage with ASTM-C618 (1993) Recommendations.

S. No.	Sample No.	Drying Shrinkage (mm)	Percentage Reduction (%)	ASTM-C618 (1993) Recommendations (%)
1	DS0 (0 percent replacement)	0.11	0.07	<0.03
2	DS2.5 (2.5 percent replacement)	0.50	0.31	
3	DS5 (5 percent replacement)	0.11	0.07	
4	DS7.5 (7.5 percent replacement)	0.33	0.21	
5	DS10 (10 percent replacement)	0.33	0.21	

The ASTM-C618 (1993) standard specifies a maximum drying shrinkage of 0.03 percent of the original length after 28 days. As a result, it was clear that none of the samples met the requirement. DS5 (5 percent replacement) of drying shrinkage 0.07 percent of the original length, on the other hand, has the same value as the control sample (DS0). DS2.5, DS7.5, and DS10, on the other hand, shrink to 0.31, 0.21, and 0.21 percent of their original length after drying. As a result, all of the samples have values greater than or equal to the code recommendation of less than 0.03 percent of the original length. The reduction in MK particle size, which increased pozzolanic activity and contributed to the pore refinement of the MK concrete matrix, may be to blame for the increase in drying shrinkage. This is also in line with the findings of (Mehta and Monteiro, 2006), who concluded that pore refinement additives in concrete result in higher shrinkage and creep.

Effect of MK on Mortar Flexural Strength

The flexural test results on (40x40x160) mm rectangular prisms were presented in the figure below. Each value of the result is an average of three tests.

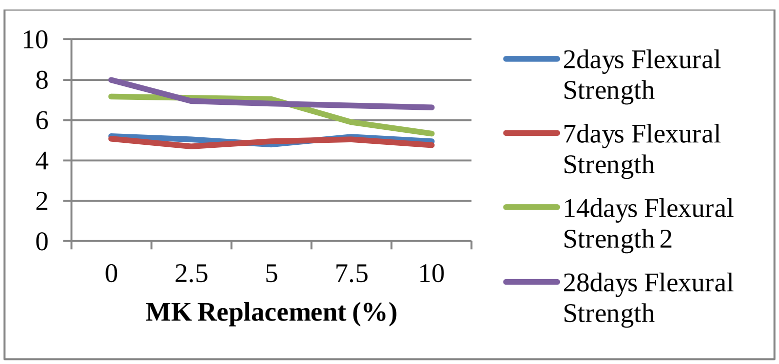

Figure 6: Relationship of Cement- MK mortar flexural strength to various percentage replacement of MK.

The flexural strength of Cement-MK mortar increased with curing time and decreased with an increase in the percent of MK content, as shown in Figure 4.6. Figure 4.6 also shows that the MK is gradually participating in the hydration process, as the rate of change in flexural strength due to varying percentage replacements of MK is gradually decreasing with age, as indicated by the gradual reduction of the slope for the 2, 7, 14, and 28 days.

Figure 4.7 depicts the development of flexural strength over two, seven, fourteen, and twenty-eight days of curing. It can be seen that the graph has the typical parabolic shape, indicating that concrete strength developed at a high rate at first and then declined. At 28 days, the strength also tended to converge. This adds to the evidence that MK behaves like a pozzolana that works with mortar, as previously stated (Nuruddeen and Ejeh, 2012).

Effect of MK on Mortar Compressive Strength

The results of Cement- MK mortar compressive strength was presented here in Tables 4.20 to 4.23. Each value of the result is an average of three tests.

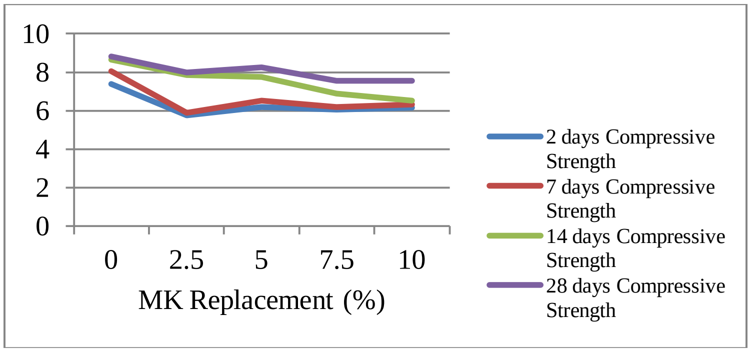

Figure 7: Relationship of Cement- MK mortar compressive strength to various percentage replacements.

As shown in Figure 4.8, the compressive strength of Cement-MK mortar increased with curing time and decreased with cement replacement with MK. When compared to the control, the value of compressive strength at 5% replacement was higher for all curing ages; thus, the 5% replacement level is a critical percentage value. For 2,7,14, and 28 days, the rate of decrease in compressive strength with an increase in the percentage of MK differs. The lower the percentage reduction in compressive strength, the longer the curing time.

The compressive strength of Cement- MK mortar was generally lower than the compressive strength of Ordinary Portland Cement mortar in all cases. The percentage of MK replacement in the mix increased, resulting in a decrease in compressive strength. For all curing ages, however, a 5% replacement of cement with MK yielded the highest strength. Following BS 5628-1, all other percentage replacements achieved the targeted compressive strength of 6N/mm2 for 28 days (1991).

Table 17 shows the percentage reduction in compressive strength of the Cement- MK mortar when compared to the control mortar.

Table 17: Reduction in Compressive Strength

MK content (%) /Age (Days)	**Reduction in Compressive Strength (%)**			
	2	7	14	28
0.0	0.0	0.0	0.0	0.0
2.5	22.3	26.7	9.5	9.5
5.0	16.5	19.0	10.3	6.4
7.5	18.1	22.9	20.5	14.2
10.0	16.9	21.4	24.7	14.2

It could be observed from above table, the percentage reduction increased with increases in the percentage of MK. A minimum reduction was attained at 5% replacement for almost all the curing ages. The 28 days compressive strength result presented in Table 18 is compared with the requirement of BS 5628-1(1991).

Table 18**:** Comparison of 28 days Mortar Compressive Strength with BS 5628-1(1991) Requirements.

0	Sample No.	Compressive Strength (N/mm^2)	BS 5628-1(1991) Requirement (N/mm^2)
1	CS0 (0 percent replacement)	8.81	>6.00
2	CS3 (2.5 percent replacement)	7.97	
3	CS5 (5 percent replacement)	8.25	
4	CS7.5 (7.5 percent replacement)	7.56	
5	CS10 (10 percent replacement)	7.56	

It could be seen that all the samples have satisfied the requirements of BS 5628-1(1991), where $6N/mm^2$ is stipulated as the minimum compressive strength for mortar. Furthermore, it could be observed from the graph that there is a rapid rate of increase at the initial stage, which is followed by a gradually diminishing rate with time, forming the characteristic parabolic curve.

The early strength gain could be attributed to the pozzolanic reaction of the available silica from the MK and the amount of C-H available from the hydration process and micro filler effect. This early strength gain caused by MK is in agreement with the work of (Middenhorf et al., 2005; Naji et al., 2010).

Effect of MK **on Mortar Microstructure (SEM)**

Scanning Electron Microscopy (SEM) is becoming a versatile tool for studying microstructure, interfacial transition zone, and hydration progress. The SEM technique studied selected specimens to have a closer look at the microstructure and evaluate hydration product and morphology.

Figures 8 (a, b and c) are SEM Plates showing the structural images of our samples (1000x Magnification). Table 19 shows the average pore size ratio of mortar M0, M5, and M10, respectively. The cement was partially replaced with 0, 5, and 10% MK subjected to 28 days of curing.

From Figures 8 (a-c), it could be observed that SEM of cement mortar replaced with 0% MK at 28days has a dense structure, with CSH occupying most of it. The other SEM of the mortar in Figure 8 b and c show a denser structure with more significant and more numerous CH.

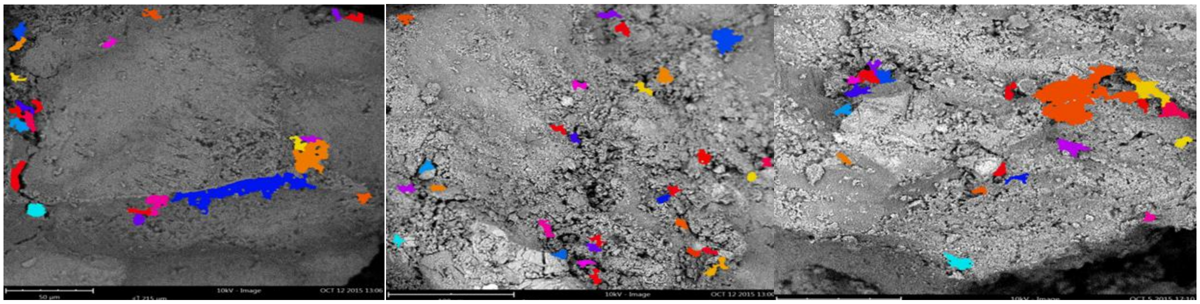

Figure 8 (a, b & c): Pore images of the specimens at (0%, 5%, and 10%) replacement respectively.

Table 19: 28 days Cement- MK Mortar Average Pore Size Ratio

S. No.	Sample	Average Pore Size Ratio (%)
1	M0 (0 Percent replacement)	22.82
2	M5 (5 Percent replacement)	5.19
3	M10 (10 Percent replacement)	19.74

It could be seen from Figure 8b that is at 5% MK replacement, the average pore size ratio was less than that of the control specimen (0% replacement). Similarly, the average pore size ratio increased from 5.19% at 5% to 19.74% at 10% MK replacement. These values are less than that of the control, which was 22.82%. This further supports the optimum percent replacement of OPC with MK of 5%.

It could also be observed that the porosity decreased with an increase in the MK content. This could be attributed to the higher silica content in the MK, which agrees with the work of (Naji et al., 2010), who found out that a decrease in the total porosity is attributed to the change occurring in the pore size distribution because MK could react with the calcium hydroxide to form C-S-H.

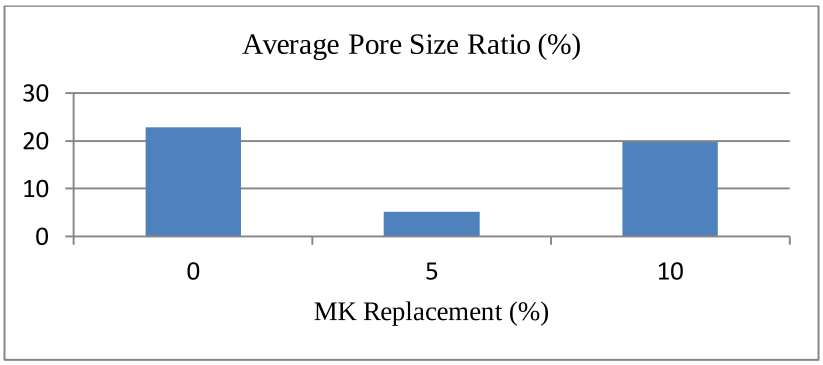

Figure 9: Relationship of average pore size ratio to various percentage replacement of MK.

IV. Conclusions

Based on the experimental results and discussions, the following conclusions could be drawn:

- Chemical analysis of rice husk ash (MK) using XRF indicates the presence of significant oxides such as SiO_2 (91.5%), CaO (2.84%), Fe_2O_3 (1.96%); minor oxides such as MnO (0.247%), TiO_2 (0.11%), V_2O_5 (0.004%), etc. and Loss on Ignition was found to be 9.18%.
- The chemical analysis using XRF and physical properties of the Dangote brand of OPC indicates that it is sound and is Ordinary Portland cement.
- The consistency, soundness, initial and final setting times of cement increased with MK content.
- The compressive and flexural strengths of MK-Mortar decreased with an increase in MK content while drying shrinkage of MK-Mortar increased apparently with an increase in MK content.
- Scanning Electron Microscopic (SEM) image of 0% MK -Mortar at 28days has a dense structure while for 5 and 10 % replacement of MK -Mortar showed a denser structure with more significant and more numerous CH; also, the average pore size ratio decreases with increases in the MK content, but 5% replacement level has the minimum.

V. Recommendations

- Based on the result of this work's findings, an optimal 5% MK replacement of the Dangote brand of OPC is recommended for use as partial replacement of cement. In addition, the replacement will aid in waste disposal and utilization of Metakaolin.
- It is also recommended that the Cement-MK mortar be used as a general-purpose mortar.

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