EVALUATION OF MICROSTRUCTURE AND COMPRESSIVE STRENGTH PROPERTIES OF BINARY BLEND ALKALINE ACTIVATED MORTAR

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Abstract
Alkaline Activated materials have been proven recently to be the way of converting Agricultural, thermal and industrial waste into construction repair materials. This paper evaluates the microstructure and compressive properties of Cassava Peel Ash (CPA) incorporated Metakaolin (MK) based Alkaline Activated Mortar (AAM). Sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH) solution were used as an alkaline activator and NaOH in varying concentrations of 6, 9 and 12 M. The mass ratios of sodium silicate to sodium hydroxide (NS: NH) and the binder to fine aggregate (B: A) were fixed to 2.5 and 0.4 respectively. The specimens were subjected to a curing temperature of 60ºC for a duration of 24 hours. The compressive strength of the synthesized AAMs was determined at 3, 7 and 28 days. The use of Scanning Electronic Microscope (SEM) was used to study the microstructure of the mortar. It is demonstrated that 50% of MK replaced CPA of 12m of NaoH achieved the highest compressive strength as much as 47.6 N/mm^2 at 28 days curing. The strength and microstructural characteristics of the synthesized AAMs was significantly improved. The study showed that the use of Cassava Peel Ash and Metakaolin in the production of sustainable green geopolymer mortar is a suitable alternative repair material that is feasible both technically and environmentally.

Keywords: Alkaline Activated Binder; Metakaolin; Cassava peel ash; Compressive strength.

Introduction
The world population is increasing by the day and for that reason, a better life is demanded and protecting the environment is the world's biggest challenge (Global warming) (Mittal & Mittal, 2013). By 2020, Portland cement (PC), the most common produced component in concrete, is expected to reach 4.4 billion metric tons (Mehta & Ashish, 2020). It's no secret that the manufacture of Portland cement has negative environmental repercussions, which may be ascribed to the release of around 0.8 tons of carbon dioxide (CO_2) per ton of cement produced, as well as quarrying and the use of non-renewable natural resources (Das & Neithalath, 2018). Huseien *et al.* (2018), put it succinctly that by the year 2020, an increase in CO_2 and other greenhouse gas emissions to100% would impact the global warming indices and climate change unless the emission is inhibited. Adding to the existing problem of CO_2 emission is the waste management problem. According to Sadh *et al.* (2018), the availability of agricultural and industrial waste when left unused causes environmental degradation and this has led to the clamour for cementless mortar. Researchers have been working tirelessly to produce revolutionary construction materials to achieve alternative binders in order to reduce emissions and the difficulties that come with them (Rashad, 2013). Alkali activated materials was introduced as new sustainable construction materials as an alternative to ordinary Portland cement (Huseien *et al.,* 2018; Pourakbar & Huat, 2017). According to Pacheco-Torgal. (2015), The total replacement of cement was made possible by Davidovits in 1979 through the

introduction of geopolymer sometimes referred to as AAM and AAMs commonly use alumino-silicate based nature available and waste materials (industrial and agricultural waste (i.e. pozzolana) and some of the source materials used to produce AAM is; Metakaolin, fly ash, palm oil fuel ash, rice husk ash and slag. Cassava peel constitutes between 20-35% of the weight of tuber, especially in the case of hand peeling. Based on a 20% estimate, about 6.8 million tonnes of cassava peel is generated annually and 12 million tonnes is expected to be produced in the year 2020 (Raheem *et al.,* 2020). Studies have it that CPA meets the requirement for a pozzolana, which is in line with ASTM C618 (2012) requirement of 70% minimum for pozzolanas (Ofuyatan *et al*., 2018; Ogunbode & Akanmu, 2012). Despite much research, the binary blend source material of Metakaolin-cassava peel ash has not been evaluated. As such, the use of Alkali Activated Materials such as MK and CPA as a total replacement of cement is to address issues of waste management, global warming and other environmental related issues. The objective of this study is to evaluate the performance characteristics of cassava peel ash and metakaolin blended alkali-activated mortar with a view to developing an enhanced AAM from an agricultural by-product and geologically based solid. The use of Alkali Activated materials such as MK and CPA as a total replacement of cement is to address issues of waste management objectives to reduce, reuse and recircle (3R). This is targeted towards arresting the concerns of global warming and other environmental related issues ravaging the earth.

Material Characterization

The two precursor materials used in this study are metakaolin and cassava peel ash, the liquid used are NaOH and Na_2SiO_3, water, superplasticizer and fine aggregate.

Metakaolin (MK)

Kaolin powder was acquired from Alkalari, Bauchi State, Nigeria. It was calcined at 750°C for 4 hours. MK has a distinctive off-white colour close to that of the parent kaolin (Plate 1). The appearance of kaolin has changed from pure white to floral whitish after the dehydrocyclization process. The specific gravity of MK is 2.2, the chemical compositions of MK were determined using X-ray Fluorescence Spectroscopy (XRF). The XRF results revealed that the major constituents of MK are silicon oxide (SiO_2) and alumina oxide (Al_2O_3). Other components include ferric oxide (Fe_2O_3), calcium oxide, magnesium oxide, potassium oxide, etc. The typical chemical composition of MK is depicted in Table 1.

Cassava Peel Ash (CPA)

Cassava peel is a waste material generated from cassava plant. In the present study, Cassava peel was collected from Doko village in Lavun LGA of Niger state and was used as raw materials (Binder). CPA possesses both cementitious and pozzolanic properties. CPA was gotten from dried Cassava peel calcinated at 500°C for 2hours and it is Dark Ash in colour (Plate 1). The specific gravity of CPA is 2.3 Table 1 summarizes the chemical composition of CPA.

Plate I: Powered sample of MK and CPA

Table 1: Chemical compositions of CPA and MK (mass%)

Materials	SiO2	Al2O3	CaO	SO_3	Fe2O3	Na2O	K2O	TiO2	MnO	MgO	LOI
MK	72.39	20.35	0.01	-	1.12	0.34	3.12	0.90	0.02	0.12	2.35
CPA	80.83	0.77	4.24	0.83	1.55	0.06	5.50	-	0.05	-	

Figure 1 illustrates the X-Ray Diffraction (XRD) patterns of CPA and MK. The XRD pattern of CPA revealed a pronounced broad hump with diffraction peaks at 2α values in the range of 27-56°. Few sharp crystalline diffraction peaks indicates its dominant amorphous phase and crystalline phases of 111,220,330. The XRD pattern of MK demonstrates an outstanding crystalline phase material with obvious detectable quantities of kaolinites and silica. Plate II displays the SEM images of CPA and MK used in this study. The surface morphology of CPA clearly revealed gelatinous appearance with irregular globular shaped particles as shown in Plate II (a), whereas MK manifested irregular pellet-like and angular particles arranged disorderly (Plate II b).

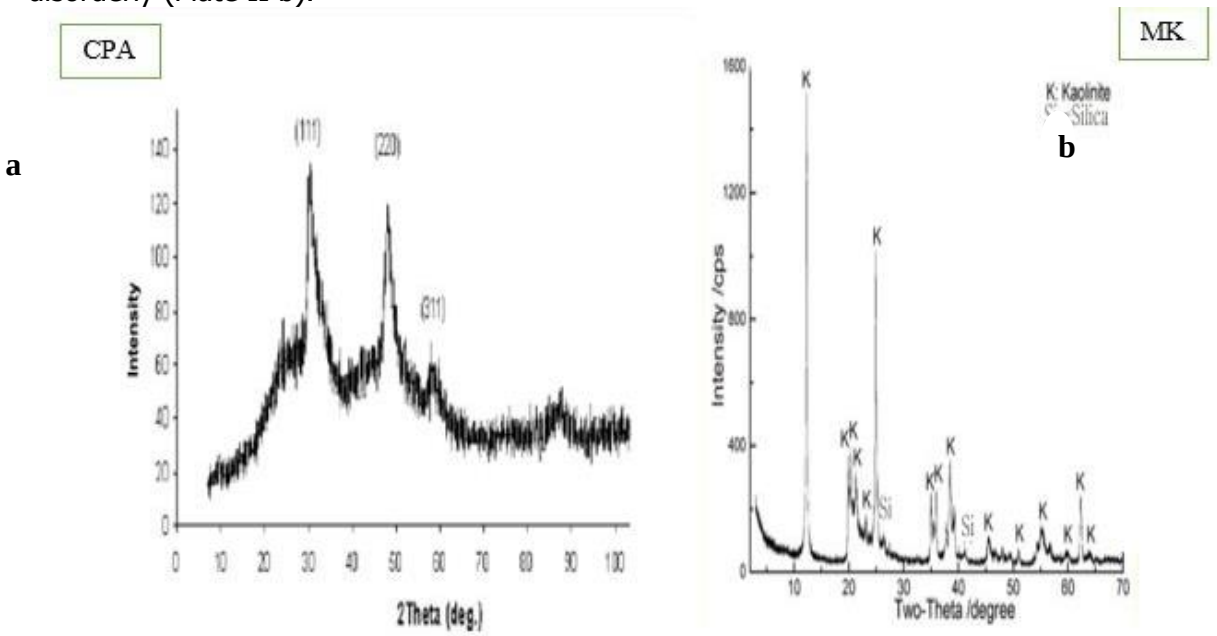

Figure 1: XRD of (a) CPA and (b) Mk

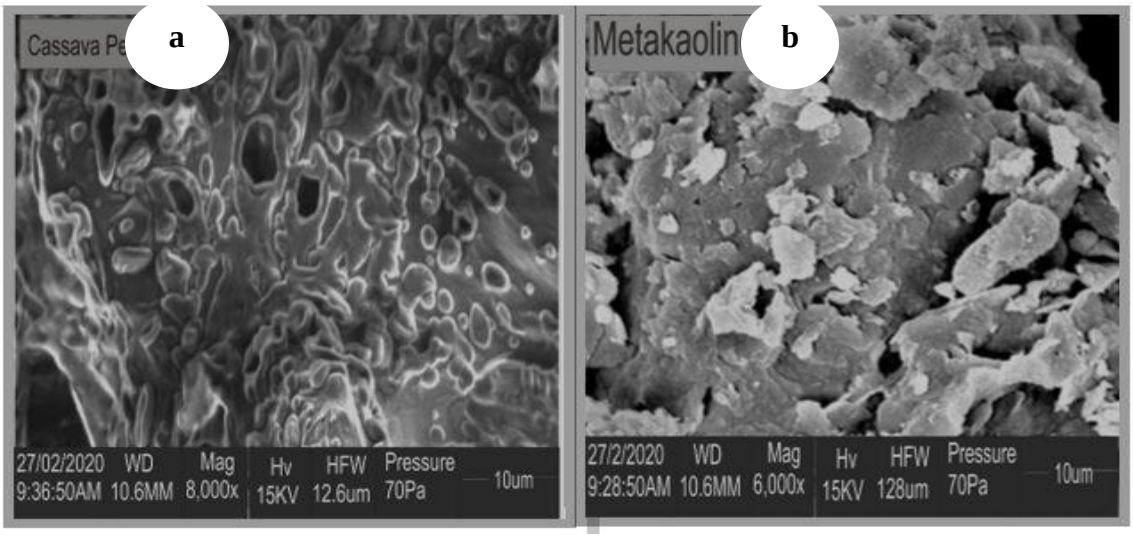

Plate II: SEM of (a) CPA and (b) MK.

Alkali solution

A mixture of sodium silicate (NS) and sodium hydroxide (NH, purity 98%) alkaline solution used in the present study (Plate III). These were used to activate the alumina and silica in MK and CPA. The NS solution was composed of SiO2 (27.0 mass%), Na2O (14.0 mass%) and H2O (51.0 mass%). These chemicals were purchased from Niger State. A different amount of pellet was dissolved in water to prepare NH solution of various molar concentrations (6, 9 and 12M). The solution was left for 24h to be cool, then it was added to a NS solution to prepare the final alkaline solution. The ratio of sodium silicate to sodium hydroxide (NS: NH) was 1:2.5 fixed for all mixtures of alkaline solution. Table 2 illustrate the details of the Alkaline solution composition

Plate III: Images of NaOH and Na_2SIO_3

Table 2: Composition of Alkaline Solution

Alkaline Solution mass%	NaOH solution (NH)			Na2SiO3 solution (NS)			NS:NH
	Molarity M	Na2O mass%	H2O mass%	SiO2 mass%	Na2O mass%	H2O mass%	
S1	6	18.6	81.4	27.0	14.0	59.0	2.5
S2	9	27.9	72.1	27.0	14.0	59.0	2.5
S3	12	37.2	62.8	27.0	14.0	59.0	2.5

Fine aggregate

Siliceous River Sand was used to prepare all mortar specimens. Finess modulus of the aggregate and specific gravity were discerned to be 2.6 and 2.4 respectively.

Method of Mix

The AAM mortar was prepared using water to solid ratio (w/s) 0.27, 0.26 and 0.25. The water content is the total water in activator and additional water whilst the solid is the CPA and MK and solid part of the activator. The ratio of binder to sand was 1: 2.5. Present AAMs were prepared by mixing MK with CPA over a period of 4 min at dry condition to achieve a homogenous mixture of fine aggregates as illustrated in Plate IV. Then, the acquired mixture was activated by adding the alkaline solution to obtain a thorough mixed mortar cast into 50 mm cube mould. The casting was performed in three layers, where each layer was compacted. The samples were left for 24 h after casting before subjected to oven curing at 100°C for 24 hours. In Addition, OPC mortar with 0.5 w/c ratio and sand-cement ratio (S/C) of 2.5 was also cast for comparison. They were tested for 3, 7 and 28 days to evaluate the compressive strength (according to ASTM C109, 2020) and other mechanical properties. Table 3 depicts the achieved three different phases of mixtures.

Table 3: Mix proportion of AAM

Phase	Binder mass%		Alkaline solution type	S: B mass%	B: A mass%	H2O: binder mass%
	CPA	MK	NaOH molarity			
1	100	0	NHNS		0.50	1.2.5
0.4		6,9 &12				
2	50	50	NHNS		0.50	1.2.5
0.4		6,9 &12				
3	0	100	NHNS		0.50	1.2.5
0.4		6,9 & 12				

Plate IV: Images of AAM paste for MK-MK/CPA-CPA

Casting and curing

The fresh AAM was cast in a 50 mm x 50 mm x 50 mm cube mould and remould after a rest period of 24 hours of casting. The specimens were further cured in an oven at a temperature of 80 °C for a 24-hours duration. After removing the specimen from the oven, the specimens were maintained at the ambient condition of 26ºC until test day. Eighty-one specimens of AAM were prepared for testing to determine the mechanical strength at 28days.

Testing

The compressive strength test was performed on AAM according to BS EN 196-1 (2016) by using mechanical testing. The specimens were loaded with constant load until failure occurs. The compressive strength value was determined by the average of 3 specimens testing for each different type of mixture. The compressive strength for paste was being tested for 3, 7 and 28 days.

Results and Discussion

The effect of molar concentration on strength

Strength of AA mortar cured at 100°C for 24 hours at the age of 3, 7 and 28 days for 6, 9 and 12 molar concentrations were presented in Figures 2, 3 and 4 for 100% CPA, mix MK & CPA and 100% MK. For 6M concentration of CPA, blended MK & CPA and MK, the strengths were 11.6, 12.9 and 18.3, 27.6, 31.6 and 36.7 and 11.6, 15.9 and 28.3 MPa respectively. For 9M concentration of CPA, blended MK & CPA and MK, the strengths were 8.8, 10.6, and 15.8, 29.9, 38.8 and 46.0 and 18.6, 21.5 and 27.3 MPa respectively while for 12M concentration of CPA, blended MK & CPA and MK the strengths were 12.7, 14.2, and 20.6, 33.2, 44.4 and 47.6 and 37.3, 40.6 and 46.2 MPa respectively. There was generally a linear increase in strength from day 3 curing today 28. 100% CPA Specimens of 9molar concentrations exhibited lower compressive strength compared to Mixed and 100% MK. 12 molar concentration of Mixed CPA & MK gives higher strength of 47.6 MPa at 28 days of curing.

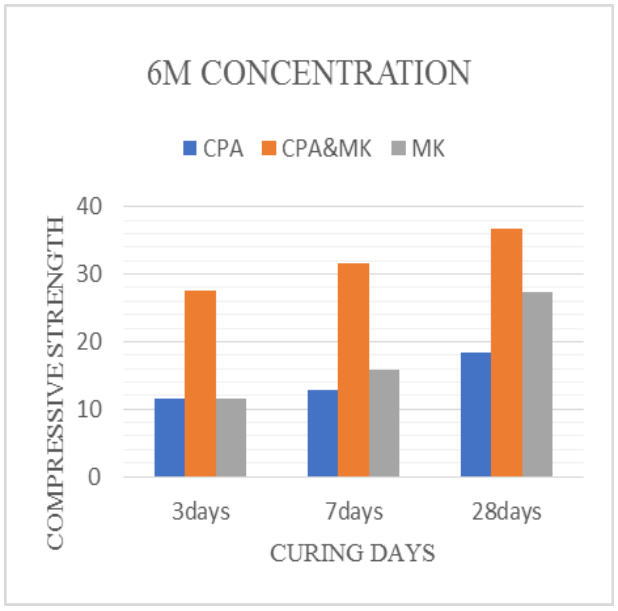

Figure 2: Compressive strength of 6M

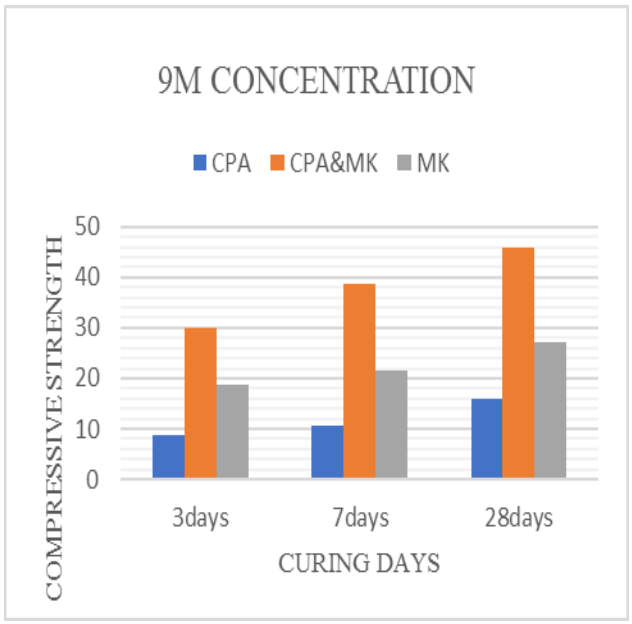

Figure 3: Compressive strength of 9M

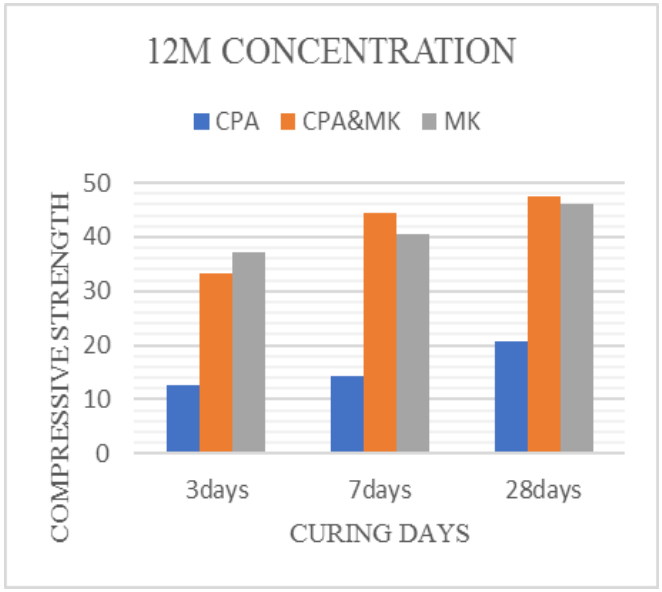

Figure 4: Compressive strength of 12M

Microstructure Analysis

The microstructure of CPA, MK and CPA/MK alkaline activated mortar paste was observed using Scanning Electron Microscope (SEM) at 28days. Plate's V (a), (b) and (c) shows SEM images of samples of 9M of CPA, MK and MK/CPA alkali-activated mortar crushed at 28 days. In Plate V (a) CPA, the orange circle colour revealed a less dense network with large pores which is as a result of entrapped air bubbles due to lack of adequate compaction and the yellow circle colour showed some threadlike particles which is an indication that some particles did not react. This might be responsible for lower strength. Plate V (b) MK appeared relatively homogeneous with the orange colour circle indicating a partially dense surface with smaller pores and the yellow

colour circle showing few unreacted particles. Plate V (c) CPA/MK showed a dense and compacted surface that appears non-porous. It, therefore, revealed a strong bond formed between them.

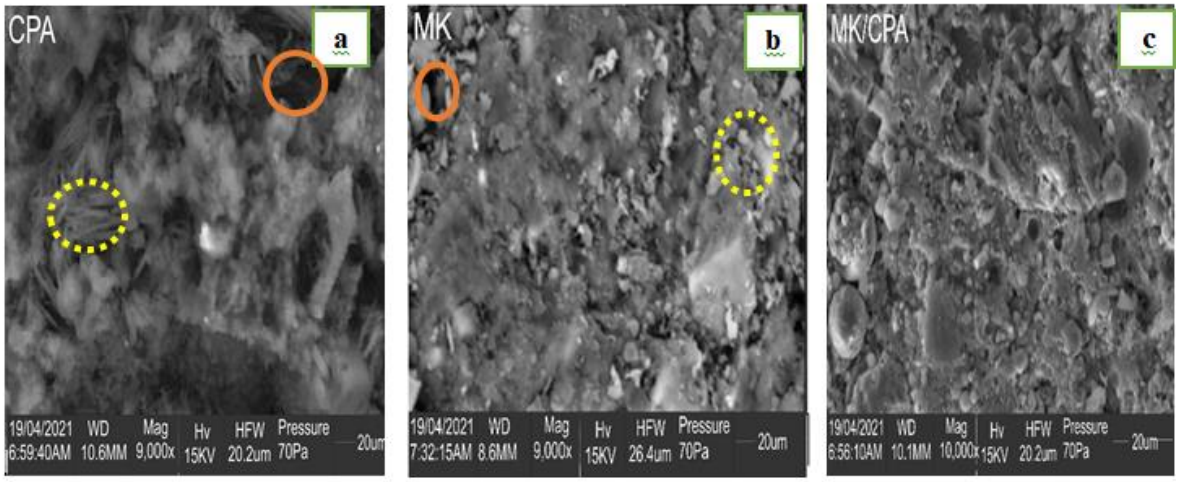

PLATE V: SEM images of (a) CPA, (b) MK and (c) MK/CPA for 9M at 28days

Conclusions

In this paper, evaluation of the compressive properties of binary blend alkaline activated mortar, cured under elevated temperature and mainly based on alumino-silicate precursors from agricultural waste (CPA) and Metakaolin was discussed. The main outcomes of this study are listed below:

(a) The Blended CPA/MK (50%/50%) based AAM sample with 12 molar concentration of NaoH produced the highest compressive strength.

(b) The alkali activation of MK and CPA with sodium silicate and sodium hydroxide solutions produced materials with equitable high compressive strength, when prepared under NaoH molarity of 9 and above.

(c) All AAM samples, cured for 28 days, had the highest compressive strength when compared with others cured at 3 and 7 days.

(d) The microstructure of the CPA based geopolymers, blended MK/CPA based AAM and MK based AAM was observed by SEM, and the results showed that:

i. The blended CPA/MK (50%/50%) samples had higher homogeneity and the lowest number of pores in comparison to the other samples (CPA (100%/0%) & MK (100%/0%) with the least unreacted CPA and MK from the alkaline activator.

ii. This sample also produced the highest compressive strength of 47N/mm^2 after a period of 28 days. This suggests that the dissolution of the aluminosilicate in the geopolymerization process in the blended CPA/MK sample produced the highest compressive strength. While, the pores in the AAM matrices had led to the lower compressive strength of the other mix type.

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