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Influence of Sulphuric Acid on the Compressive Strength of Ternary Blended Geopolymer Mortar

[1*]Ogunbode E. B., [2]Musa S., [3]John A., [1]Dauda A. A., [2]Musa B. & [1]Garba Y. Y.
[1]Department of Building, Federal University of Technology Minna
[2]Department of Quantity Surveying, Niger State Polytechnic, Zungeru
[3]Department of Civil Engineering, Niger State Polytechnic, Zungeru
[*]**Corresponding author**: ezekiel@futminna.edu.ng

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The deteriorating effect of acid media on cement based constructions has become a worrying problem all over the world. These media generally occur as solutions in various branches of the industry, acid rains and mists, and acid ground-waters. A very popular form of acid attack on concrete that is usually referred to as biogenic sulphuric acid attack also occurs in both industrial and urban sewer systems. The emergence of new cementitious materials, like geopolymer cements, during the past decades necessitates detailed experimental work and research activities to investigate their durability in aggressive acid environments. The study therefore explored the development of alkali-activated CPA-SHA-MK ternary blended geopolymer mortar (GPM) using sodium silicate (Na_2Si_3) and sodium hydroxide (NAOH) solutions with 9M constant concentration as alkaline activators under both the aggressive and ambient-temperature curing media. The mass ratio of sodium silicate to sodium hydroxide (NS: NH) and as well as the binder to fine aggregate were fixed at 2.5 and 0.8 respectively. The durability of the ternary blended geopolymer mortar was examined through acid resistance test using 50 mm cubes after 28, 56 and 90 days of curing. The results revealed that the setting time prolonged as the replacement levels of RHA-MK increased at a decrease in the replacement levels of CPA. The results also showed that both the PCM and GPM samples studied suffered mass and strength losses in the acid solution and the loss increases at an increase in the hydration periods. The strength losses were observed to be higher in PCM mix (12.19 N/mm^2 at 90 days) as compared to the GPMs (6.67 N/mm^2 at 90 days) while the mix incorporated 50% CPA, 33% MK and 17% RHA (C50M33R17) was observed to be better compared to other mixes in durability behaviour. The study therefore recommends C50M33R17 mix proportion for good durability performance.

Keywords: Cassava Peel Ash (CPA), geopolymer, Metakaolin (MK), Rice Husk Ash (RHA), sulphuric acid

INTRODUCTION

Over the years, Ordinary Portland Cement (OPC) has been widely employed as mortar binder, concrete binder and various building substances worldwide. It is known that, large scale manufacturing of OPC causes serious pollution in the environment in terms of considerable amount of greenhouse gases emission (Duxson *et al.*, 2007; Rashad *et al.*, 2013). The OPC production alone is accountable for nearly 6 to 7% of total CO_2 emissions as estimated by International Energy Agency (IEA) (Palomo *et al.*, 2011). Among all the greenhouse gases, approximately 65% of the global warming is ascribed to the CO_2 emission. The emitted greenhouse gases such as CO_2, SO_3 and NOx from

the cement manufacturing factories can cause acid rain and damage the soil fertility (Zhang *et al.*, 2011). Generally, the industrial consumption of raw materials is around 1.5 tonnes per each tonne of OPC production (Rashad, 2013a). To surmount such problems, efforts have been dedicated to developing novel construction materials to achieve alternate binders such as geopolymer (Rashad, 2013b).

Geopolymers are the alumino-silicate polymers which consist of three dimensional amorphous structures formed due to the geopolymerization of alumino-silicate monomers in alkaline solution (Rowles & O'connor, 2003). In the past, intensive studies have been carried out on calcined clays (metakaolin) or industrial wastes such as FA, palm oil fuel ash and slag (Chang, 2003; Kong *et al*., 2007; Temuujin *et al*., 2010). A reaction pathway was proposed involving the polycondensation of orthosialiate ions (hypothetical monomer) (Provis *et al*., 2005). The mechanism of geopolymerization process (Dimas *et al*., 2009) is based on three steps: (i) dissolution in alkaline solution, (ii) reorganization and diffusion of dissolved ions with the formation of small coagulated structures and (iii) polycondensation of soluble species to form hydrated products. Compared to OPC, alkali activated mortars are well-known for their excellent properties such as high compressive strength (Nmadu *et al*., 2021; Burciaga-Díaz *et al.*, 2013; Zhang *et al*., 2010), low shrinkage (Chi *et al*., 2012; Zhang *et al*., 2010), acid resistance (Palomo *et al*., 1999), fire resistance, devoid of toxic fumes emission (Duxson *et al*., 2007), low thermal conductivity (Zhang *et al.,* 2010), excellent heavy metal immobilization, high temperature stability (Yao *et al*., 2009), low manufacturing energy consumption for construction purposes and several engineering applications (Zhang *et al*., 2010).

Mortars and concretes made with Portland cement deteriorate in aggressive environment of sewers, mining, mineral processing, acid rain or acid ground-water (Harrison, 1987; Bakharev *et al*., 2003; Allahverdi & Skvara, 2005; Bakharev, 2005). Most of the commercial repair materials owing to their low durability and sustainability perform poorly under aggressive chemical and adverse weather environmental conditions. Geopolymer prepared from the waste materials with high content of aluminium-silicate and alkaline activator solution has emerged as a leading repair material (Huseien *et al.*, 2017). However, new binders are prerequisite for enhanced acid resistance performance, better sustainability and environmental affability.

Sulphuric acid, generated by sulphur/sulphide-oxidising bacteria has been identified as the corroding acid in sewer systems (De Belie *et al.*, 2004; Lavigne *et al.*, 2016; Huber *et al.,* 2016). Unfortunately, the relative inaccessibility of sewer networks poses considerable challenges for maintenance and repair (Scrivener & De Belie, 2013). Sulphuric acid can also be present in groundwater or produced from the oxidation of sulphur bearing compounds in backfill, such as pyrite, causing degradation to concrete substructures (Bassuoni & Nehdi, 2007). The dissolution of hydrogen sulphide can also form sulphuric acid with a low pH on the concrete walls of geothermal wells (Pyatina & Sugama, 2016). Therefore, sulphuric acid is a major cause of degradation of concrete structures.

EMPIRICAL STUDIES

The majority of studies in the literature on the acid resistance of GP materials report favourable performance. Rostami and Brendley (2003) studied the sulphuric acid resistance of fly ash concrete and PC concrete with the addition of silica fume. After 90 days exposure to 20% sulphuric acid, the fly ash concrete had a mass loss in the region of 4% compared to 25% for the PC based concrete. Similarly, Thokchom *et al*. (2009a) reported that fly ash GP mortars had much better performance in terms of mass loss when exposed to 10% sulphuric acid than their heavily corroded PC counterparts. An increase in mass loss was also reported when the alkali dosage (% of Na_2O) was increased. However, in a later publication it was stated that increasing the alkali dosage results in a higher residual compressive strength after sulphuric acid attack (Thokchom *et al*., 2009b). Bakharev (2005) studied the resistance of fly ash GP pastes and PC pastes to 5% sulphuric acid. A superior performance was observed for the fly ash GP paste which was attributed to much lower calcium content. Lee and Lee (2016) studied the resistance of fly ash and slag GP mortars to 10% sulphuric acid. They reported higher resistance of blends with lower slag content due to the nature of the binding gel produced. On the other hand, Lloyd *et al.* (2012) reported that increasing the slag content increases the resistance of GP pastes exposed to sulphuric acid with pH controlled at 1.0. Allahverdi and Skvara (2006) studied the mechanism of sulphuric acid attack on fly ash and slag GP pastes containing 50% of each. They reported an ion exchange between the samples and attacking acid followed by shrinkage cracks and the formation of gypsum.

The present study developed an environmentally friendly geopolymer mortar with broad arrays of applications in the construction industry and exhibiting durability characteristics by introducing three pozzolanic materials in enhancing the acid resistance characteristics of the geopolymer mortar.

MATERIALS AND METHODS

Materials

The materials used included cement (CEM 1), Rice Husk Ash (RHA), Metakaolin, Cassava Peel Ash (CPA), fine aggregate, alkaline solutions (Sodium Silicate (Na_2SiO_3) and Sodium Hydroxide (NaOH)), water and superplasticizer (Master Rheobuild 1100).

Portland cement type CEM II/A-LL, 42.5 N from Dangote Cement Company conforming to BS EN 1971-1 (201) and NIS 444-1 (2003) was used as the main binder (PC). The cement was obtained from local cement merchant in Minna and effort was made to ensure that the supply was obtained from the most recent stock and kept in dry position.

The RHA used in this study work was collected from a local grain mill in Garatu village along Minna-Bida Road, Bosso LGA, in Niger State. The collected RHA was then burnt in open air with a locally fabricated incinerator. The resulting RHA was dried and sieved to eliminate larger materials and to lessen the carbon content. A local milling device was used to ground the resulting burnt RHA particles to a size smaller than 150 µm. Finally, the ground ash was sieved with a 75 µm sieve size and particles passing through were used as the RHA for the experiment. The RHA powder obtained was white in colour, which was an indication of complete burning of all carbon and impurities within the husk.

River sand with maximum size of 4.75 mm was used in this study. Clean potable water as specified by BS EN 1008 (2015) available within the

concrete laboratory of Department of Building, School of Environmental Technology, Federal University of Technology, Minna was used for mixing of the mortar and other laboratory use in accomplishing the study

Methods

The methods adopted in conducting the material analysis (physical and chemical), fresh properties and durability properties are presented in this section. The acid resistance test of the ternary-blend alkali activated mortars involved the evaluation of acid resistance of the specimens tested.

Material characterisation

The material characterizations involved the examination of both the chemical and physical properties of the constituent materials. The physical analysis carried out on the sand, RHA, CPA, MK were particle size distribution analysis (PSD), specific gravity and moisture content. The chemical analysis conducted on RHA, CPA and MK samples was X-ray Fluorescent (XRF) analysis for determination of oxide composition in accordance to ASTM C618 (2015) using XRF analyser. Also, setting time and flowability of the mortar mix were also evaluated.

Mix proportioning and specimen production

The study investigated the varying composition of solid binder as independent variable on the durability properties of ternary blend GPM. A trial mix was conducted prior to the casting of experimental specimens. The control variable of the study were the specimens having Portland cement only (PCM) and the other mixes were those with variations in binder composite, a total of five mortar samples were cast with increasing CPA content of 10 %, 30 %, 50 %, 70 % and 90 %. While the remaining contents was two-third of MK and one-third of RHA as shown in Table 1.

Table 1: Mix proportion of Alkali Activator Mortar for optimum CPA content

Mix ID	CPA (%)	MK (%)	SHA (%)	W/B Ratio	Fine Agg.	Na_2SiO_3	NaOH
C90M07S03	90	7	3	0.35	2.5	0.25	0.1
C70M20S10	70	20	10	0.35	2.5	0.25	0.1
C50M33S17	50	33	17	0.35	2.5	0.25	0.1
C30M47S23	30	47	23	0.35	2.5	0.25	0.1
C10M60S30	10	60	30	0.35	2.5	0.25	0.1

Specimen Testing and Data Collection

The test specimens were tested for the acid resistance test. The choice of acid solution and its concentrations was based on practical utilization of concrete as a construction material in sewage pipes, mining, and food processing industries.

Acid resistance test

The resistance of the geopolymer mortar to acid attack was studied by immersion of cube specimens (50 x 50

mm) in 5% solutions of sulphuric acid with pH of 0.8. The testing medium was replaced monthly with fresh solutions. The compressive strength of specimen (50 x 50 mm) was measured at 28, 56 and 90 days of exposure. The assessment of the geopolymer specimens in acidic environment was carried out based on the performance from weight loss and strength loss. These were all done in consonance with previous studies on testing the effect of acidic environment on geopolymer containing pozzolanic materials (Aiken *et al.,* 2018; Valencia-Saavedra *et al.,* 2020).

RESULTS AND DISCUSSION

Materials Characterization

Physical properties of the constituent materials

The results in Table 2 revealed that the fine aggregate conformed to the medium sand classification according to Shetty (2009) with uniformity coefficient (C_u) of 2.39, coefficient of curvature (C_c) of 0.94, specific gravity value of 2.82 and fineness modulus (FM) of 2.65. These are indications that the fine aggregate was appropriate for the production of geopolymer mortar (GPMs). The sand was used at saturated surface dry conditions and its grading was measured according to ASTM C33/C33M (2019)

Table 2: Summary of physical properties of the constituent materials

Item	Sand	RHA	CPA	MK	PC
D_{10}	360				
D_{30}	540				
D_{60}	860				
C_u	2.39				
C_c	0.94				
FM	2.65				
SG	2.82	2.8	1.8	2.59	3.15

XRF characterization of binders of RHA, CPA and MK

The chemical composition and loss on ignition of CEM II (Portland cement) as received and RHA, CPA and MK determined by XRF are shown in Table 3. The outcome of the test revealed that the RHA, CPA and MK contained majorly SiO_2. Furthermore, it can be seen that the SiO_2 present in them as revealed by the result are RHA=95%, CPA=80.83% and MK=72.39% with silica-sesquioxide (S-S) ratio (SR) of RHA=166.67, CPA=34.84 and MK=3.37 with aluminium-sesquioxide ratio (AR) of RHA=3.75, CPA=0.50 and MK=18.17 respectively which according to ASTM C618 (2015) is affirmed to be a very strong reactive Class F pozzolan with the sum of silica (RHA=95%, CPA=80.83% and MK=72.39%) higher than the specified 70%, alumina (RHA=0.45%, CPA=0.77% and MK=20.35%) and ferric oxides (RHA=0.12%, CPA=1.55% and MK=1.12%). Also, the CEM II contained 60% CaO which was an indication that the PC was majorly calcium oxide which is in conformance with the oxide composition reported in literature (Shetty, 2009) for CEM II.

Table 3: Oxide Composition of Binder Constituents (RHA, CPA and MK)

Oxides	RHA (%)	CPA (%)	MK (%)	CEM II (%)
SiO_2	95.0	80.83	72.39	25.64
Al_2O_3	0.45	0.77	20.35	5.24
Fe_2O_3	0.12	1.55	1.12	7.15
CaO	0.84	4.24	0.01	60.35
MgO	0.45	-	0.12	0.41
SO_3	0.10	0.83	-	0.11
K_2O	1.50	5.50	3.12	0.05
Na_2O	0.03	0.06	0.34	0.31
M_2O_5	0.05	-	-	0.04
P_2O_5	0.72	-	-	0.03
LOI	0.74	2.10	2.35	0.67
$SiO_2 + Al_2O_3 + Fe_2O_3$	95.57	83.15	93.86	12.68
SR	166.67	34.84	3.37	2.07
AR	3.75	0.50	18.17	0.73
Total	100	100	100	100

Fresh Properties Test

Before the production of the specimens, fresh GPM mixtures were examined for workability using flow table test as described in BS EN 1015-3:1999 as a measure of workability of the GPMs mixtures presented in Table 4. Master Rheobuild plasticizer was used as a high range water reducer and was administered at constant concentration of 1.0% by weight of binder (bwob). Flow table test was used to observe the spreading of the fresh geopolymer mortar by repeatedly tapping for 25 times on a level surface. Throughout the experiment, the rapid setting of ternary-based GPM was noticed as shown in the Table 4. The fresh mortar began to set within five to twenty minutes (5-20 mins) right after thoroughly mixing process for the C90M07R03 mix followed by C70M20R10, C50M33R17, C30M47R23 and C10M60R30 with the initial and final setting times of twenty and sixty minutes (20-60 mins), twenty-five and sixty-five minutes (25-65 mins), thirty and hundred minutes (30-100 mins), forty-five and one-twenty minutes (45-120 mins) respectively. However, the GPM had a very flash setting with initial setting time of one and half hours (1 hr. 30 mins) when compared with the reference mortar (PCM) having final setting time of three hours (3 hrs.). The results revealed that at a decrease in the CPA content, the setting time increases. This finding is in agreement with the study of Shamsolketabi (2020). Furthermore, the result also showed a descending trend in the spreading width as observed with a decrease in the replacement levels of CPA. Higher CPA content exhibited higher workability and faster settings of the GPM. Hence, it was discovered that the RHA incorporation contributed to the reduction in the spreading width as a result of its hygroscopic nature and thereby accelerate the geopolymerization of the specimens (Gao *et al*., 2016). Lower spreading width achieved by increasing the replacement level of MK-RHA attributed to the content of CaO and thus accelerate the geopolymerization as its rapid reaction with alkali activator (Khan *et al*., 2016).

Table 4: Fresh Properties of ternary blended GPMs

Variables	Spreading Width (mm)	Setting time (min)	
		Initial	Final
PCM	220	90	180
C90M7R3	210	5	20
C70M20R10	200	20	60
C50M33R17	190	25	65
C30M47R23	150	30	100
C10M60R30	140	45	120

Influence of Sulphuric Acid Attack on the Compressive Strength of GPMs

The mass loss and the compressive strength were monitored in order to understand the primary degradation mechanism.

Mass loss

The mass change in mortar samples during the period of immersion in H_2SO_4 solutions is shown in Figure 1. The rate of mass loss in both the PCM and GPMs sample exposed to H_2SO_4 solutions was increasing from one cycle to the next during the hydration periods but the loss was highly pronounced in PCM with 31.25%, 33.30% and 34.10% at 28, 56 and 90 days respectively compared to the GPMs. The results also revealed that the sample C90M7R3 reached maximum mass loss of 29.50%, 32.55% and 32.95% at 28, 56 and 90 days respectively when exposed to H_2SO_4 solutions with 5% by weight of curing water concentrations. The same trend was followed in other GPM mixes with an increase in the mass loss as the hydration periods increases and with the GPM samples C70M20R10 having the values of 24.20%, 23.99% and 26.45% at 28, 56 and 90 days respectively followed by the GPM mix C50M30R20 having the lowest mass loss of 16.50%, 18.25% and 19.22% at 28, 56 and 90 days respectively. The GPM mix C30M47R23 had the values of 20.25%, 25.05% and 26.59% while 29.50%, 30.69% and 33.45% were for C10M60R30 GPM mix. This is similar to the outcome of Aiken *et al.*, 2018 study on effect of sulphuric acid on geopolymer material.

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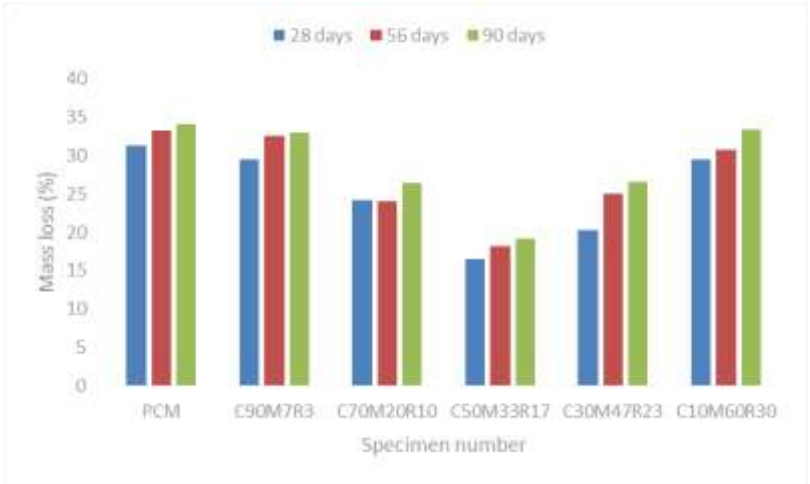

Figure 1: Mass loss of ternary blended GPMs subjected to H_2SO_4 attack

In contrast, the PCM mass loss was above the mass loss in all the GPM mixes. As evidenced by the colour change of the sample's surface, the difference was most probably related to the decalcification of hep and formation of a layer sulphate salts, gypsum and ettringite (Peyvandi *et al*., 2015), on the surface of the sample. Most likely, these salts also formed in the pores of the outermost surface layer of the samples. At this stage, low degradation was observed and the mass loss was the result of high degree of hep decalcification and most importantly, the result of progressive degradation of the surface layer caused by pressure exerted by expansive crystals of the salt formed inside the pore structure. The results corroborated the findings of Aiken *et al.* (2018) and Valencia-Saavedra *et al.* (2020).

Strength loss

Results of GPMs compressive strength cured in both ambient and sulphuric acid (H_2SO_4) media at various hydration periods (28, 56 and 90 days) are shown in Figure 2. At the end of 90 days curing age, the strength of mortar specimens immersed in H_2SO_4 solution is compared with those cured in normal ambient temperature. The difference between the GPMs compressive strength cured in ambient temperature and those immersed in H_2SO_4 solution is referred to as the strength loss. From the results in Figure 2, it was clear that the whole specimens suffered strength losses and the loss of some mixes were not drastically detrimental. The strength loss was observed to be more in reference mortar (PCM) when compared to GPMs (C90M7R3, C70M20R10, C50M33R17, C30M47R23 and C10M60R30). The C50M33R17 mix exhibited the lowest loss in strength with the highest residual strength of 94%, 90% and 85% at 28, 56 and 90 days respectively followed by C30M47R23 having residual strengths of 84%, 82% and 81%, C70M20R10 with residual strengths of 82%, 81% and 81% and as well as 80%, 77% and 74% for the GPM mix C10M60R30 and 70%, 61% and 58% for the GPM mix C90M7R3 at 28, 56 and 90 days respectively. The strength losses values of 8.40 N/mm^2, 11.31 N/mm^2 and 12.19 N/mm^2 were observed for PCM mix at 28, 56 and 90 days respectively. Whereas, the strength loss values of 8.51 N/mm^2, 9.30 N/mm^2 and 10.16 N/mm^2 were

recorded in C90M7R3, 5.25 N/mm^2, 6.19 N/mm^2 and 7.16 N/mm^2 for C70M20R10, 4.94 N/mm^2, 6.16 N/mm^2 and 7.40 N/mm^2 for C30M47R23 , 6.05 N/mm^2, 7.08 N/mm^2 and 9.10 N/mm^2 for C10M60R30 and as well as the GPM mix C50M33R17 having the lowest strength loss values of 2.67 N/mm^2, 4.20 N/mm^2 and 6.67 N/mm^2 with the highest residual strength as said earlier at 28, 56 and 90 days respectively. However, the result also revealed that irrespective of the mix, the loss in strength for both the PCM and GPMs kept increasing at an increase in the hydration periods while the loss is more pronounced in PCM in comparison with the GPMs. The superior behaviour of GPMs is corroborated by the findings of Aiken *et al.* (2018) that reported lower strength loss of GPMs while that of PCM was higher.

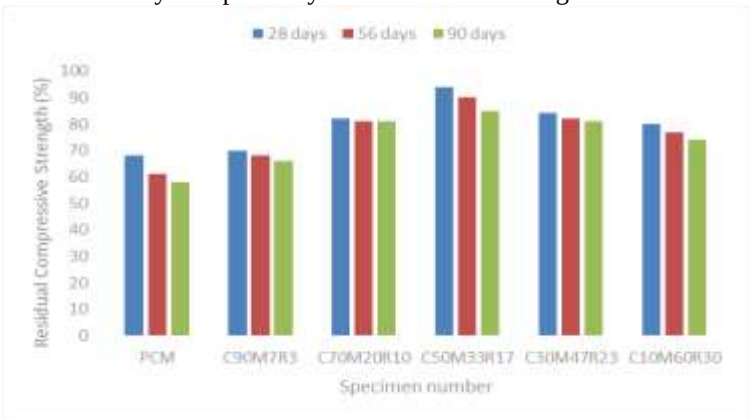

Figure 2: Strength losses as caused by H_2SO_4 attack on ternary blended GPMs

CONCLUSION

The entirely GPM mixes in H_2SO_4 environment had a better strength compared with the reference mortar (PCM). Sulphuric acid as a curing medium resulted in loss in the mass and compressive strength values of both the PCM and GPMs but the loss was more pronounced in PCM. Acid immersion studies indicated that geopolymer mortars have shown better acid-resistant properties. Ternary blended of CPA, MK and RHA with the composition of C50M33R17 should be adopted as the SCMs for 9M concentrations of alkali activators for better durability performance.

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