Influence of Sulphate Attack on Compressive Strength of Millet Husk Ash as an Alternative to Silica Fume in Internally Cured High Performance Concrete

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Due to the difficulties in maintaining failing concrete structures built with normal concrete (NC), high-performance concrete (HPC), which is extremely dense and has a low water-binder content, has been developed. However, it has been observed that inadequate internal curing in HPC can lead to autogenous shrinkage and micro-cracking. Previous research has indicated that internal curing (IC) agents, such as pre-saturated lightweight aggregate (LWA) and superabsorbent polymers (SAP), are frequently used to address this issue. It is also noteworthy that the production of HPC necessitates the use of additional supplementary cementitious materials (SCM), particularly silica fume, which is not easily accessible in Nigeria or other Sub-Saharan African countries. This article presents the report of an effort to replace silica fume in HPC with millet husk ash (MHA) as a SCM material. The HPC specimens produced was internally cured with pre-soaked pumice as LWA and SAP respectively, with the view to establish the effectiveness of MHA for production of HPC. The results of 28, 56 and 90 days for concrete specimen immersed in ordinary water and in 5% of $MgSO_4$ for C55/67 HPC mixtures for 100 mm concrete cubes having 7.5% silica fume content is compared with MHA based HPC of varied MHA contents of 2.5%, 5%, 7.5%, 10% and 15%).The results reveal 2.5%MHA performed best when compare with the control (i.e 7.5%SF) in resisting 5% $MgSO_4$ attack with a strength loss factor of 1.89, 1.81 and 1.31% at 28, 56 and 90 days of age and was recommended for use as SCM and IC-agent in HPC.
Keywords: Millet husk ash (MHA); Silica fume (SF); Superabsorbent polymers (SAP); Pre-soaked pumice; and High-performance concrete (HPC)

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Introduction

Cement, water, and aggregates are combined to create concrete, a composite material (Neville, 2012; Mudashiru *et al*., 2021). The foundation of the material is cement which when mixed with water forms a paste that binds aggregates together to form a hard material called concrete. The strength of concrete is commonly considered as the most valuable property because it usually gives an overall picture of the quality of concrete and it is one of the most vital element in structural design which is specified for compliance purpose (Olawuyi *et al*., 2020; Mudashiru *et al.*, 2021). Many materials are being added to the concrete mix to improve its properties both in fresh and hardened states.

High-performance concrete (HPC), an alternative to normal concrete (NC), was developed in response to issues with the deterioration and early failure of concrete structures constructed using NC (Mudashiru *et al*., 2021). Due to its high strength, low water-to-binder ratio (W/B) and high modulus of elasticity, HPC is mostly utilized in the construction industry these days to create tall buildings, construction of tunnels and bridges (Aïtcin, 2004; Orosz, 2017). Thinner structural components can be built with HPC, creating a structure with more aesthetic appeal (Nduka *et al*., 2020). Buildings with more usable spaces will require less steel to be constructed, and the pressure on the overall structure will be lessened when structural members are built using HPC. Therefore, architects and designers can easily attain greater architectural flexibility, buildable structural shapes and forms, and nearly free reinforcing bars, which results in cheaper labour (Wang *et al*., 2015).

When water is added, and densification occurs in the concrete, the quantity of cement and the added supplementary cementitious materials (SCMs) in HPC mix typically results in a rise in temperature. According to Savva *et al*. (2018), on the direct relationship between ambient temperature and cementitious materials, the hydrated gel's porosity is increased because the ambient temperature typically affects the cementitious materials' grains This phenomenon causes a fast reaction that obstructs the uniform distribution of hydration products. Because of the combined effects of hydration and pozzolanic reaction, the integration of SCMs induces autogenous shrinkage, chemical shrinkage, and self-desiccation, which has increased the water demand in the concrete (Wu *et al*., 2017). Using an internal curing method is one of the most efficient and effective ways to address these issues in concrete production. Due to HPC's low permeability, external curing is insufficient to allow water to permeate the concrete, making the IC method one of the most effective and efficient ways to reduce the risk associated with autogenous shrinkage (Olawuyi *et al*., 2017, Mudashiru *et al*. 2021). Hence, the incorporation of

IC agents which has the ability to absorb water and release it in to the system when the need arises.
Studies on the application of several IC agents in the manufacturing of HPC have been conducted; the most widely employed IC agents in the literature are LWA and superabsorbent polymer (SAP) (Mudashiru *et al*., 2021). In order to determine the efficacy of Nigerian supplementary cementitious materials (MHA) and internal curing agent (pre-soaked pumice) in HPC production, this article will concentrate on the influence of sulphate attack on the compressive strength of millet husk ash (MHA) as an alternative to silica fume (SF) in internally cured high performance concrete.

Materials and Methods

Materials

Binders including Portland cement (PC) and SCMs such as SF and MHA, fine and coarse aggregates, superplasticizer, pumice, SAP, magnesium sulphate, and water were the materials used in this study. The Portland cement used in this investigation was Dangote brand 3X (Grade 42.5N), whose characteristics meet BS EN 197-1: 2016 standards. It was purchased in Gidan-Kwano, Minna, Niger State, from a cement store. The MHA that was utilized as SCM was obtained from the burning of the husk in the Concrete Laboratory of the Department of Building, Federal University of Technology, Minna, Niger State, using a locally built incinerator. The burning was done outdoors for roughly twenty-four hours at a temperature of no more than 700 degrees Celsius. After that, the material was allowed to cool before being harvested and ground using a grinding machine. According to ASTM C430-2014, the milled MHA was sieved with a 75µm mesh before being placed in an airtight polythene bag for storage. The SF used as the second SCM for this study was purchased from a Construction Chemical company in Lagos.
Natural sand retained on 300 µm sieve, which is the prerequisite for HPC production, was utilized as the fine aggregate in this study (Shetty, 2004; Neville, 2012; Nduka *et al*., 2020; Olawuyi *et al*., 2020). The sieve analysis was used to analyse the physical properties of the sand, such as its specific gravity (SG), fineness modulus (FM), coefficient of uniformity (Cu), coefficient of curvature (Cc), and dust content. In accordance with typical HPC mixes found in the literature, crushed granite stone that passed through a 13.50 mm sieve size and retained on at least a 9.50 mm sieve size was used as coarse aggregate (Aitcin, 2004; Beushausen and Dehn, 2009; Neville, 2012; Olawuyi and Boshoff, 2018; Nduka *et al*., 2020; Olawuyi *et al*., 2021). To reduce the mixture's water requirement, the coarse aggregate was cleaned to get rid of dust particles.
According to Olawuyi and Boshoff (2018), a Superabsorbent polymer (FLOSET 27CS) with a grain size of ≤ 600 µm, manufactured in France by SNF Floerger, was added at 0.2% by weight of binder (bwob). This was done by taking into account that the tea-bag test yielded a SAP absorption capacity of 12 g/grams, which complies with the requirements of the SAP specification for the production of HPC (Olawuyi *et al*., 2021). The thermoset polymer called SAP is precisely the covalently cross-linked polymers of acrylic acid and acrylamide that are produced by polymerization in bulk solutions and neutralized by alkali hydroxide. Pre-soaked pumice, which was utilized in this investigation, was a porous igneous rock that was crushed to a maximum size of 12.5 mm after being created by explosive volcanic eruptions. Before being used, the pumice was drained after being submerged in water for 24hrs (Olawuyi *et al*., 2020).
According to the specifications of BS EN 1008 (2002), potable water sourced from Federal University of Technology, Minna, Niger State was used for the mixing at 0.3 w/b in this investigation **(**Ogunbayo *et al*., 2018**).** The chemical admixture (superplasticizer) utilized in the typical HPC combinations was sky 504 Masterglenium polymer-based polycarboxylic ether (PCE) superplasticizer from Armorsil Manufacturing Incorporation. It was applied at a concentration of 1.5% by weight of binder as reported by Olawuyi *et al*. (2021).

Methods

Properties of constituent materials
X-ray fluorescence (XRF) was used to analyse the chemical compositions of binders (MHA, SF, and OPC) at the National Geoscience Research Laboratory in Kaduna State and the Laboratory of the Ewekoro Factory of Lafarge Plc. Following the calcination, grinding, and sieving processes, about 100g of these binders were placed in sealed polythene bags and dispatched to determine the oxide compositions in compliance with BS EN 197-1: 2016. Wet sieving was used to ascertain the aggregates samples' particle size distribution. The aggregates and binders' specific gravities were carried out.

Production of HPC specimens
The mix design approach for material proportioning for HPC production was selected based on the work of Aitcin (2004) which was cited by Nduka *et al*. (2020) and Olawuyi *et al*. (2021). The mean goal strength of C55/67 at 28 days was chosen. The following table displays the HPC material proportioning: 0.2% SAP by weight of the binder and 5% pre-soaked pumice by weight of coarse aggregate (bwoca).

Table 1: Materials mix proportioning of the HPC mixtures

Mix proportion	Materials (Kg/m^3)								
	CEM II	**SF**	**MHA**	**F/A**	**C/A**	**PP**	**SAP**	**SP**	**Water**
M0a	499.5	40.5		700	997.5		1.08	8.1	156
M0b	499.5	40.5		700	997.5	52.5		8.1	156
M1	526.5		13.5	700	997.5	52.5		8.1	156
M2	513		27	700	997.5	52.5		8.1	156
M3	499.5		40.5	700	997.5	52.5		8.1	156
M4	486		54	700	997.5	52.5		8.1	156
M5	459		81	700	997.5	52.5		8.1	156

NB: CEM= Portland cement, SF= silica fume, MHA= millet husk ash, F/A= fine aggregate, C/A= coarse aggregate, PP= pre-soaked pumice, SAP= superabsorbent polymer, and, SP= superplasticzer
M0=92.5%PC+7.5%SF; **M1**=97.5%PC+2.5%MHA; **M2=**95%PC+5%MHA; **M3**=92.5%PC+7.5%MHA; **M4**=90%PC+10%MHA; **M5**=85%PC+15%MHA
a=0.2% SAP, **b=**5% pre-soaked pumice

For the SAP internally cured HPCs, the SAP contents (0.2% bwob) were utilised, along with an additional 12.5g/g of water for SAP absorption; for the Pumice internally cured HPCs, the coarse aggregate of the pre-soaked saturated pumice was measured and used at 5% by weight. Prior to compressive strength testing, the cast 100 mm cube HPCs were demoulded and cured for 28, 56 and 90 days by submerging them completely in water and 5% $MgSO_4$ solution (Olawuyi *et al*., 2021).

Results and Discussion

Physical and chemical properties

The results of the XRF analysis of the powder binders (MHA, SF, and PC) are shown in Table 2 below. The total of the major oxides (SiO_2 + Al_2O_3 + Fe_2O_3) yields 87.20%, which is above the 70% minimum requirement as stated in the ASTMC 618 (2012) standard, indicating that the MHA, is a class N pozzolan. According to ASTM C618 (2012) it is an extremely strong and reactive class F Pozzolan, as shown by the SF main percentage of SiO_2 (96.20%). The total ferric oxides (SiO_2+Al_2O_3+Fe_2O_3), alumina, and silica for the SF (96.91%) exceed the 70% threshold given for the Class of Pozzolan in ASTM C618 (2012). On the other hand, calcium oxide

Table 2: Oxide Composition of Binder Constituents

Oxides	MHA (%)	SF (%)	CEM II (%)
SiO_2	71.05	96.20	25.64
Al_2O_3	14.66	0.45	5.24
Fe_2O_3	1.49	0.26	7.15
CaO	1.55	0.05	64.35
MgO	0.73	0.03	0.41
SO_3	0.67	0.10	0.11
K_2O	5.21	0.02	0.05
Na_2O	1.16	0.02	0.31
M_2O_5	2.06	0.50	0.04
P_2O_5	1.19	0.4	0.03
LOI	2.10	1.02	0.00
SiO_2 + Al_2O_3 + Fe_2O_3	87.20	96.91	38.03

(CaO) makes up the majority of the PC. This conforms to oxides composition for CEM II Portland cement found in literature (Neville, 2012; Mehta & Monteiro, 2014).

The physical characteristics of the aggregate utilized in the investigation are displayed in Figure 1 and Table 3. The fine aggregate's uniformity coefficient (Cu) of 2.39, coefficient of curvature (Cc) of 0.94, and fineness modulus (FM) of 2.88 indicate that it complies with Shetty's (2004) medium sand categorization. The study's coarse aggregates are in the class of uniformly graded stone, with a coefficient of uniformity (Cu) of 1.32 and a coefficient of curvature (Cc) of 0.92. Table 3 shows that coarse and fine aggregates pre-soaked pumice and crushed granite are both suitable for producing HPCs.

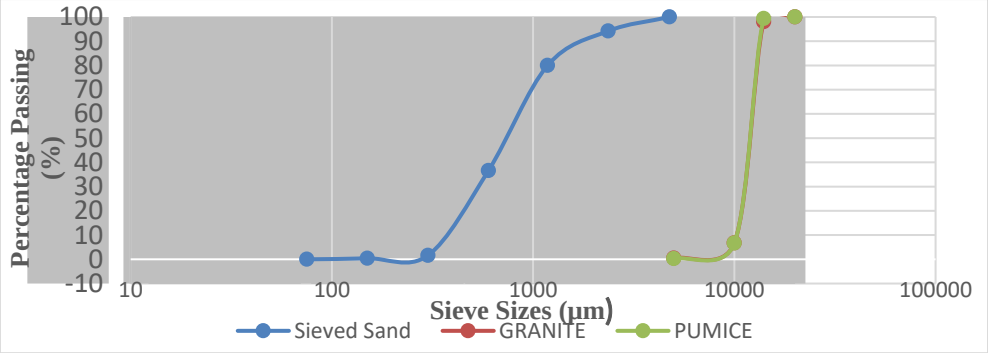

Figure 1: sieve analysis/particle size distribution of aggregates

Table 3: Summary of sieve analysis of aggregates

Item	Sand	Granite	Pumice
D_{10}	360	10000	10000
D_{30}	540	11000	11000
D_{60}	860	13000	13000
C_u	2.39	1.3	1.3
C_c	0.94	0.93	0.93
FM	2.87		

Table 4 present the specific gravity of the constituent materials (PC, MHA, SF and aggregates). The results gave the values as 3.14, 2.63, 2.24, 2.85, 2.85 and 1.77 for PC, MHA, SF, fine aggregate, Granite and pumice respectively. The results reveal that the values are inconformity with the previous report in literature (Neville, 2012).

Table 4: Specific Gravity of PC, MHA, CCW and aggregates

Materials	PC	MHA	SF	F/Agg.	Granite	Pumice
Specific gravity	3.14	2.63	2.24	2.85	2.85	1.77

Influence of sulphate attack on the compressive strength of the HPCs

Physical/visual assessment, mass loss, residual compressive strength and strength loss factor was used to determine the influence of sulphate attack on the various HPC mix.

Physical / visual assessment

The physical/visual observation of the various HPCs specimen cured in 5% magnesium sulphate ($MgSO_4$) solution at different curing age is presented in Table 6. At 28, 56 and 90 days, a whitish $MgSO_4$ crystal and frost precipitation was observed on the surfaces of the concrete cubes for all the HPC mix. A slight deterioration at the corners and cracks on the surface of the entire specimen was noticeable. The samples containing SAP has fine crack, the pre-soaked pumice incorporated HPC on the other hand was observed to have a very fine and tiny cracks at 28 and 56 days. At 90 days, reduction in cracks formation was observed. The reduction in the crack at 90 days was as a result of internal water reservoir which was provided by the IC-agents which aided further hydration process and as a result of formation of additional C-S-H resulting from the reaction of the binary binders (Olawuyi *et al*., 2021). None of the HPC specimen was distorted. Formation of white deposit especially in all the specimens sample may amount to physical deterioration in a longer period other than the period used for this study (Olawuyi *et al*., 2021).

Table 6: Physical characteristics of internally cured HPCs exposed to sulphate solution

Mix ID	Surface texture	Size	Colour	Edge	Shape
Physical characteristics of HPC at 28 days					
M0a	Slightly deteriorated	No change	Whitish deposit	Fine cracks	Perfect cube
M0b	Slightly deteriorated	No change	Whitish deposit	Very fine cracks	Perfect cube
M1	Slightly deteriorated	No change	Whitish deposit	Very fine cracks	Perfect cube
M2	Slightly deteriorated	No change	Whitish deposit	Very fine cracks	Perfect cube
M3	Slightly deteriorated	No change	Whitish deposit	Very fine cracks	Perfect cube
M4	Slightly deteriorated	No change	Whitish deposit	Very fine cracks	Perfect cube
M5	Slightly deteriorated	No change	Whitish deposit	Very fine cracks	Perfect cube
Physical characteristics of HPC at 56 days					
M0a	Smooth	No change	Whitish deposit	Perfect	Perfect cube
M0b	Smooth	No change	Whitish deposit	Very fine cracks	Perfect cube
M1	Smooth	No change	Whitish deposit	Very fine cracks	Perfect cube
M2	Smooth	No change	Whitish deposit	Very fine cracks	Perfect cube
M3	Smooth	No change	Whitish deposit	Very fine cracks	Perfect cube
M4	Smooth	No change	Whitish deposit	Very fine cracks	Perfect cube
M5	Smooth	No change	Whitish deposit	Very fine cracks	Perfect cube
Physical characteristics of HPC at 90 days					
M0a	Smooth	No change	Whitish deposit	Perfect	Perfect cube
M0b	Smooth	No change	Whitish deposit	Perfect	Perfect cube
M1	Smooth	No change	Whitish deposit	Perfect	Perfect cube
M2	Smooth	No change	Whitish deposit	Perfect	Perfect cube
M3	Smooth	No change	Whitish deposit	Perfect	Perfect cube
M4	Smooth	No change	Whitish deposit	Perfect	Perfect cube
M5	Smooth	No change	Whitish deposit	Perfect	Perfect cube

M0=92.5%PC+7.5%SF: **M1**=97.5%PC+2.5%MHA: **M2**=95%PC+5%MHA: **M3**=92.5%PC+7.5%MHA; **M4**=90%PC+10%MHA: **M5**=85%PC+15%MHA: **a**=0.2% SAP 5%: **b**= pre-soaked pumice

Mass loss

Figures 2, 3 and 4 provide results of the mass loss of various HPCs mix cured in $MgSO_4$ at 28, 56 and 90 days of age. From figure 2, the mass loss values obtained for the controls (M0a and M0b) at 28 days in $MgSO_4$ solution are: 1.89% and 1.49% respectively while the values for M1, M2, M3, M4 and M5 are; 1.51%, 1.56%, 2.16%, 2.32% and 2.84% respectively.

From Figure 3, the values for mass loss for the controls (M0a and M0b) at 56 days of age are; 1.53%, and 1.24% while for M1, M2, M3, M4 and M5 their mass loss values are; 1.38%, 1.43%, 1.89%, 1.97% and 2.34%. From figure 4, at 90 days of age, the values for mass loss for the controls (M0a and M0b) are; 1.19% and 1.02% while for M1, M2, M3, M4 and M5 are; 1.18%, 1.24%, 1.54%, 1.72% and 1.97.

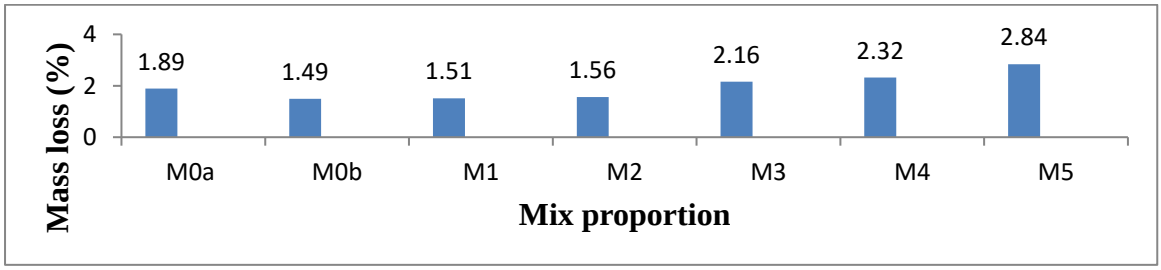

Figure 2: Mass loss of internally cured HPCs subjected to $MgSO_4$ at 28 days

M0=92.5%PC+7.5%SF: **M1**=97.5%PC+2.5%MHA: **M2**=95%PC+5%MHA: **M3**=92.5%PC+7.5%MHA; **M4**=90%PC+10%MHA: **M5**=85%PC+15%MHA: **a**=0.2% SAP: **b**=5% pre-soaked pumice

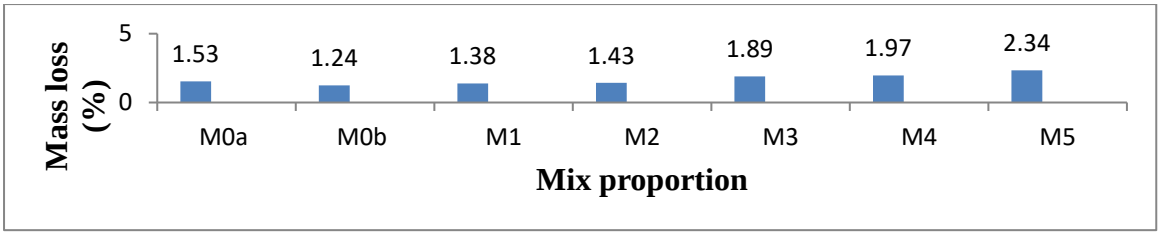

Figure 3: Mass loss of internally cured HPCs subjected to $MgSO_4$ at 56 days
M0=92.5%PC+7.5%SF: **M1**=97.5%PC+2.5%MHA: **M2**=95%PC+5%MHA: **M3**=92.5%PC+7.5%MHA; **M4**=90%PC+10%MHA: **M5**=85%PC+15%MHA: **a**=0.2% SAP: **b**=5% pre-soaked pumice

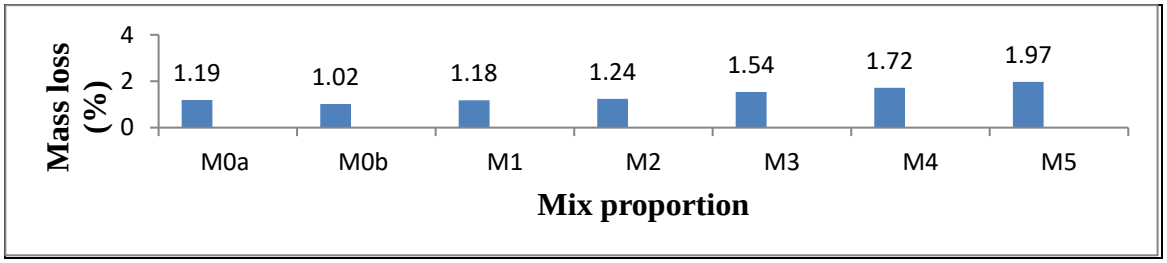

Figure 4: Mass loss of internally cured HPCs subjected to $MgSO_4$ at 90 days
M0=92.5%PC+7.5%SF; **M1**=97.5%PC+2.5%MHA; **M2**=95%PC+5%MHA; **M3**=92.5%PC+7.5%MHA; **M4**=90%PC+10%MHA; **M5**=85%PC+15%MHA
a=0.2% SAP; **b**=5% pre-soaked pumice

Generally, from the figures, it shows that all the HPCs specimens show similar behaviour of mass losses of the specimen. The phenomenon behind this was as a result of non-absorption of the sulphate solution at 28, 56 and 90 days immersion in $MgSO_4$ (Mudashiru *et al*., 2021). From the figures, it was observed that at 28, 56 and 90 days of age, SF based HPC internally cured with SAP (M0a) suffer more mass loss of 1.89%, 1.53% and 1.19% when compared with SF based HPC internally cured with pre-soaked pumice (M0b) with mass loss value of 1.49%, 1.24% and 1.02%. From the figures, it was observed that SF based HPC internally cured with pre-soaked pumice (M0b) with mass loss value of 1.49%, 1.24% and 1.02% suffer less mass loss when compared with MHA based HPC internally cured with pre-soaked pumice (M1, M2, M3, M4 and M5) with M5 having the highest mass loss of 2.84%, 2.34% and 1.97% at 28, 56 and 90 days of age. also, from the result it was observed that and increases in proportion of MHA lead to an increase in mass loss for all the curing ages with 15% of MHA (M5) having the highest value of mass loss of 2.84%, 2.34% and 1.97% while 2.5% MHA (M1) has the lowest value of mass loss of 1.49%, 1.24% and 1.02% at 28, 56 and 90days.

From the figures, it was also observed that the mass losses of the HPCs decrease at a decrease rate as the curing age increases. The reason behind this may be attributed to additional hydration of C-S-H (Olawuyi *et al*., 2021)

Strength loss factor (SLF)

The effect of $MgSO_4$ on the compressive strength of the various HPCs was expressed in terms of strength loss factor (SLF) expressed in percentage, and these are illustrated in Figures 5, 6 and 7. From Figure 5, the strength loss factors for the controls (M0a and M0b) cured in $MgSO_4$ at 28 days is: 1.75% and 1.72% while for M1, M2, M3, M4 and M5 the values are; 1.89%, 2.45%, 2.89%, 2.96% and 3.12%. At 56 days of immersion in sulphate solution, the SLF values from figure 6 for the controls (M0a and M0b) are: 1.73% and 1.69% while for M1, M2, M3, M4 and M5 the SLF are; 1.81%, 2.36%, 2.81%, 2.88% and 3.03%. In figure 7, for the controls (M0a and M0b), the strength SLF at 90 days are: 1.52% and 1.40% while for M1, M2, M3, M4 and M5 the SLF are; 1.31%, 2.06%, 2.43%, 2.58% and 2.63%.

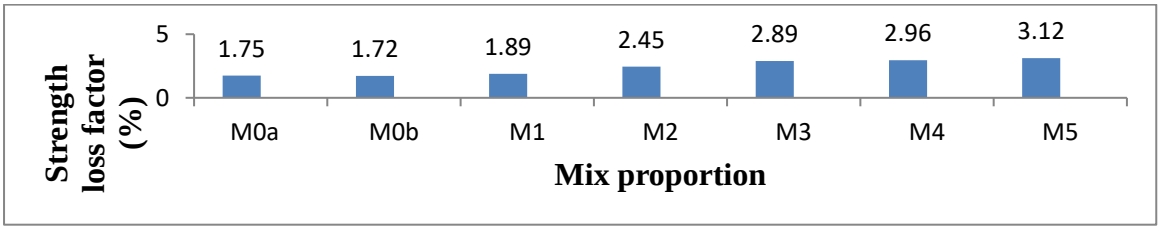

Figure 5: Strength loss factor of internally cured HPCs subjected to $MgSO_4$ at 28 days

M0=92.5%PC+7.5%SF; **M1**=97.5%PC+2.5%MHA; **M2**=95%PC+5%MHA; **M3**=92.5%PC+7.5%MHA; **M4**=90%PC+10%MHA; **M5**=85%PC+15%MHA
a=5% pre-soaked pumice; **b**=0.2% SAP

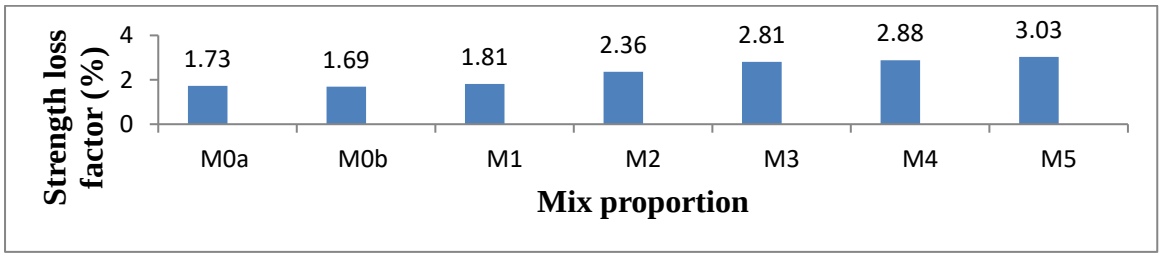

Figure 6: Strength loss factor of internally cured HPCs subjected to $MgSO_4$ at 56 days
M0=92.5%PC+7.5%SF; **M1**=97.5%PC+2.5%MHA; **M2**=95%PC+5%MHA; **M3**=92.5%PC+7.5%MHA; **M4**=90%PC+10%MHA; **M5**=85%PC+15%MHA
a=5% pre-soaked pumice; **b**=0.2% SAP

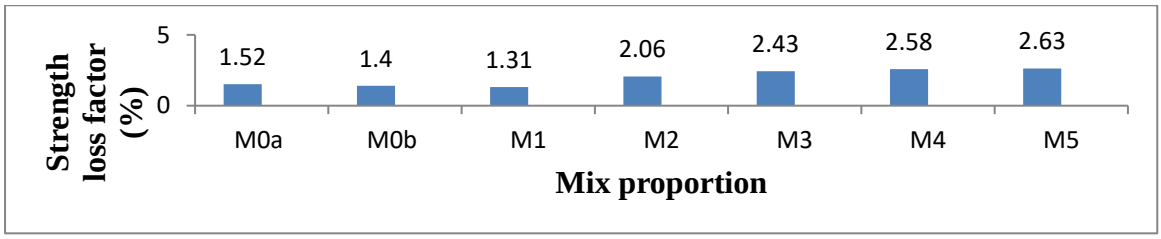

Figure 7: Strength loss factor of internally cured HPCs subjected to $MgSO_4$ at 90 days
M0=92.5%PC+7.5%SF; **M1**=97.5%PC+2.5%MHA; **M2**=95%PC+5%MHA; **M3**=92.5%PC+7.5%MHA; **M4**=90%PC+10%MHA; **M5**=85%PC+15%MHA
a=5% pre-soaked pumice; **b**=0.2% SAP

From the figures generally, it was observed that all the mixes were affected by $MgSO_4$ solution. From the figures, at 28, 56 and 90 days of age, SF based HPC internally cured with SAP (M0a) suffer more attack when immersed in sulphate solution with a SLF of 1.75%, 1.73% and 1.52% when compared with SF based HPC internally cured with pre-soaked pumice with SLF of 1.72%, 1.69% and 1.40%. From the figures, it was also observed that as the proportion of MHA increases, SLF increases at a decrease rate from 2.5%MHA to 7.5%MHA and at an increasing rate from 10%MHA to 15%MHA for all the curing ages with 2.5% of MHA internally cured with pre-soaked pumice having the lowest SLF of 1.89%, 1.81% and 1.31% while 15% MHA content has the highest SLF of 3.12%, 3.03% and 2.63% for pre-soaked pumice internally cured HPCs at 28, 56 and 90days

Conclusion

From the study, The SF and MHA used for the study were a good Class F and N Pozzolan with physical and chemical properties that conforms to ASTM C618 (2012) specifications.
Whitish magnesium sulphate crystals, micro-crack pattern and a frosted surface was observed on all the mixes when cured in 5% $MgSO_4$ solution at all the curing ages with the exception of 28 days where a grey colour with few precipitations was noticed. Curing in 5% $MgSO_4$ lead to loss in the mass and compressive strength values for all HPCs. But the loss in mass and compressive strength decreased with increase in curing ages. 2.5%MHA performed best when compared with the control (7.5%SF) in resisting $MgSO_4$ attack and it exhibited the highest residual strength. Hence it has better resistance to chemical attack.
MHA content of 2.5% and 5% pre-soaked pumice are recommended for use as SCM and IC-agent in HPC.

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