PERFORMANCE ASSESSMENT OF MILLET HUSK ASH AS ALTERNATIVE SUPPLEMENTARY CEMENTITIOUS MATERIALS FOR INTERNALLY CURED HIGH PERFORMANCE CONCRETE

BY

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MTECH / SET / 2019 / 9686

DEPARTMENT OF BUILDING
FEDERAL UNIVERSITY OF TECHNOLOGY
MINNA

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A THESIS SUBMITTED TO THE POSTGRADUATE SCHOOL, FEDERAL UNIVERSITY OF TECHNOLOLGY, MINNA, NIGERIA IN PARTIAL FULFILMENT O F THE REQUIREMENT FOR THE AWARD OF THE DEGREE OF MASTER OF TECHNOLOGY (MTECH) IN BUILDING (CONSTRUCTION TECHNOLOGY OPTION)

DECLARATION

I, hereby declare that the research work titled "**Performance assessment of millet husk ash as alternative supplementary cementitious materials for internally cured high performance concrete**" is original and was carried out by me. Information from other sources (published or unpublished) has been duly acknowledged.

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CERTIFICATION

The project titled "**Performance assessment of millet husk ash as alternative supplementary cementitious materials for internally cured high performance concrete**" **by Onogwu Christian Majanwone (MTech/SET/2019/9686)** meets the regulations governing the award of the degree of masters of Technology (MTech) of the Federal University of Technology, Minna, and it is approved for its contribution to the scientific knowledge and literary presentation.

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DEDICATION

This research is dedicated to my family for their unlimited support throughout the course of my study.

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ABSTRACT

Challenges of deterioration and premature failure of concrete structures made with normal concrete (NC) has led to the development of high-performance concrete (HPC) which is a low water-binder and very dense concrete. However, lack of adequate internal water for proper curing in HPC are noted to result in autogenous shrinkage and micro-cracking for which existing literature showed are often addressed by incorporating internal curing (IC) agents such as superabsorbent polymers (SAP) and pre-saturated lightweight aggregate (LWA). Also of note is that HPC production requires additional supplementary cementitious materials (SCM) especially Silica fume – a material not readily available in Sub-Saharan Africa like Nigeria. This study thereby is a report of attempt at utilizing Millet husk ash (MHA) as SCM in HPC as an alternative to Silica fume. The HPCs were internally cured with pre-soaked pumice as LWA and SAP respectively with the view to establish the effectiveness of Nigeria supplementary cementitious material (MHA) and IC-agent (pre-soaked pumice) for production of HPC. This research presents results of 28, 56 and 90 days compressive strength and durability properties (Sulphate resistance and sortivity) of HPC mixtures having 7.5% silica fume content in comparison with MHA based HPC of varied (2.5%, 5%, 7.5%, 10% and 15%) MHA contents. The SAP and Pre-soaked pumice contents were maintained as 0.2% by weight of binder (b_{wob}) *and 5% by weight of coarse aggregate* (b_{woca})*. The curing of the samples for compressive strength and sortivity test was carried out in water while samples for sulphate resistance test was carried out in* $MgSO_4$ *solution for 28, 56 and 90 days before testing. HPC mix with 2.5%MHA performs best with compressive strength valves of 53.58, 66.78 and 69.13N/mm*^{2} *for 28, 56 and 90 days of age. For sulphate resistance and Sortivity test, the mix with 2.5%MHA has the best performance. MHA content of 2.5% and 5% pre-soaked pumice are recommended for use as Nigeria local SCM and IC-agent in HPC*

TABLE OF CONTENTS

LIST OF TABLES

Table **Page**

LIST OF FIGURES

LIST OF PLATES

LIST OF ABBREVIATIONS

ACI	American Concrete Institute
BET	Brunauer–Emmett–Teller
B_{wob}	by weight of binder
B_{woc}	by weight of cement
$Ca(OH)_2$	Calcium hydroxide
Cc	Coefficient of gradation
CEM II	Cement type II
CO_2	Carbon dioxide
C-S-H	Calcium-silicate-hydrate
Cu	Coefficient of uniformity
D_{10}	Cumulative 10% passing
D_{50}	Cumulative 10% passing
D_{90}	Cumulative 90% passing
FM	Fineness moduluss
HPC	High-performance concrete
HSC	High strength concrete
IC	Internal curing
IC agent	Internal curing agent
LOI	Loss on ignition
LWA	Lightweight aggregate
Na_2SO_4	Sodium sulphate
NSC	Normal strength concrete
MHA	Millet husk ash

PC	Portland cement
PCE	Polycarboxylate Ether
NMR	Neural magnetic resonance
PSD	Particle size distribution
RH	Relative humidity
SAP	Superabsorbent Polymers
SCMs	Supplementary cementitious materials
SF	Silica fume
S.G	Specific gravity
UHPC	Ultra-high-performance concrete
W/B	Water: binder ratio
W/C	Water: cement ratio
XRF	X-ray fluorescence

CHAPTER ONE

1.0 INTRODUCTION

1.1 Background to the Study

Concrete is a composite material made by mixing cement, water and aggregates (Mudashiru *et al*. 2021). The foundation of the material is cement which when mixed with water forms a paste that binds aggregates together thereby setting 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 the most vital element of structural design which is specified for compliance purpose (Olawuyi *et al.*, 2020, Mudashiru *et al.*, 2021). Many materials are being added to concrete to improve its properties in both fresh and hardened state, but still serve the same purpose as cement and with good workability (Olawuyi *et al*., 2020).

Challenges associated with deterioration and premature failure of concrete structures made with normal concrete (NC) has led to the development of high-performance concrete (HPC) (Mudashiru *et al*. 2021).Nowadays, high performance concrete (HPC) is mostly used in the construction industries for constructing tunnels, bridges, tall buildings because of its high durability, high strength, low water: binder (W/B) ratio, high modulus of elasticity (Aïtcin, 2004 and Orosz, 2017).

With HPC, Slimmer structural members can be constructed thereby giving rise to an aesthetically appealing structure (Nduka *et al.* 2020). The construction of structural member using HPC will help to reduce the amount of steel to be used, reduce the entire structure pressure, and increase functional spaces in buildings. Hence, greater architectural freedom, nearly unlimited structural shapes, forms, and near free reinforcement bars which results to lower labour and cost can easily be achieved by architects and designers (Wang *et al.*, 2015).

Concrete usually experiences autogenous shrinkage (uniform reduction of internal moisture in concrete as a result of hydration of cement) as a result of self-desiccation (a phenomenon caused by chemical reaction of cement and water) in the pore system of the hydrated cement paste when water is consumed during the hydration process of cement (Wu *et al.*, 2017). One of the most effective and efficient way of providing solution to these challenges in concrete production is by using a curing method called internal curing (IC). IC method has been reported to be one of the most effective and efficient way of reducing risk associated with autogenous shrinkage since low permeability of HPC renders external curing not to be sufficient enough for water to penetrate into the concrete (Olawuyi *et al.*, 2017, Mudashiru *et al*. 2021).

Researches has been carried out on the use of different IC agents in HPC production with the use of LWA and superabsorbent polymer (SAP) being the most commonly used in literature (Mudashiru *et al*. 2021). Hence, this research focuses on the use of the locally available pre-soaked pumice as the LWA and millet husk ash (MHA) as the SCM in HPC production.

1.2 Statement of the research problem

With the advancement of construction technology, concrete structures becomes more slimmer and taller than before requiring high performance in strength and durability of concrete. Increased structural performance has led to increase in reinforcement volume and need for closely spaced small diameter bars (RILEM report 23, 2000).

High performance concrete has many advantages, such as reduction in size of the columns, high workability and pump ability, improved durability in aggressive environment, and low maintenance cost. However, high performance concrete also has disadvantages, such as high self-desiccation, high autogenous shrinkage, and high early age crack tendency. Desiccation is the reduction of relative humidity due to consumption of capillary water in the progress of cement hydration. Autogenous shrinkage on the other hand is the macroscopic volume reduction of cement based materials when cement hydrates after initial setting. Self-desiccation is one of the main reasons of autogenous shrinkage and early crack of concrete (Mejihede & Hansen, 1996).

As a means of mitigation of self-desiccation and autogenous shrinkage and early age crack, wet curing, internal curing methods using admixtures such as super absorbent polymers and light weight aggregates are effective in reducing self-desiccation and autogenous shrinkage of high performance concrete.

In the light of this development, it is feasible to replace SF, SAP (which are expensive) with an alternative supplementary cementations material, Millet Husk Ash (MHA) and pre-soaked pumice as an internal curing agent for high performance concrete with a view to assessing its effect on the durability properties of high performance concrete containing Millet husk ash.

1.3 Aim and Objectives

The aim of the research is to assess the effects of millet husk ash (MHA) and pre-soaked pumice (as internal curing agent) on the HPC with a view of determining their suitability.
The specific objectives of this study are to:

i. Characterize the constituent materials (PC, MHA, SF and Aggregates)
ii. Evaluate the effect of the SCM on the fresh properties of the class 1 (C55/67) HPC.
iii. Asses the performance of Millet Husk Ash (MHA) based HPC internally cured.
iv. Assess the effectiveness of pre-soaked pumice as compared to SAP as IC -agents in MHA based HPC.

1.4 Scope of the Study

The research is experimental in nature and it involved performance assessment of MHA based HPC internally cured with pre-soaked pumice. Tests of different type were conducted to characterize the physical and chemical properties of HPC materials. The proportion of the binders were 92.5%PC+7.5%SF, 97.5%PC+2.5%MHA, 95%PC+5%MHA, 92.5.5%PC+7.5%MHA, 90%PC+10%MHA and 85%PC+15%MHA. The pre-soaked Pumice was added to each mix proportions at 5% contents by weight of coarse aggregate (b_{woca}). The SAP was also added to the control at 0.2% (b_{wob}). The water-cement ratio used was 0.3. The superplasticizer (SP) was administered at 1.5% by weight of binder (b_{wob}). 100 mm cubes were produced for the compressive strength (cured both in water and 5% $MgSO_4$ solution). For sorptivity test, 100ϴ x 50 mm concrete cylinders was used to produce the specimens and cured in water. All the tests were conducted at 28, 56 and 90-days curing ages. All the tests were conducted in accordance with British standard (BS) and American Society for Testing and Materials (ASTM).

1.5 Significance of the Study

Research on the utilization of local available SCM (MHA) and IC-agent (pumice) in HPC is less in literature. This research is an attempt to fill the gap identified in the literature with a view to determining the effect of MHA and pre-soaked pumice on the performance of MHA based HPC internally cured with pre-soaked pumice.

This research will also increase the awareness of construction industry stakeholders on the effectiveness of using these new materials for construction of high-rise-building and other civil engineering works in developing countries like Nigeria.

This research work focuses on the use of MHA as supplementary cementitious materials (SCM) in HPC production towards addressing some environmental problems associated with

CO_2 emission from Portland cement production process and as a means of turning agricultural wastes to useful products.

The adoption and utilisation of MHA in HPC projects might help built environment professionals (architects, builders, designers etc) to take decision that will increase effective usage of space by designing slimmer members.

The idea that the strength of HPC can be compromised through self-desiccation and the mitigation through internal curing using pre-soaked pumice is an innovation.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Concrete

Concrete is one of the widely used construction material for various types of structures in the construction industry due to its structural stability and strength and it account for about 25 million metric tons of global annual energy consumption (Mudashiru *et al*. 2021). Concrete is a composite construction material that is made by mixing cement, aggregates and water.

2.1.1 Classification of concrete

Different classification of concrete exists based on type of application, required performance, and on the admixture used. Neville (2012), gave different classification of concrete based on 28days curing age to include normal strength concrete (NSC) with compressive cube strength less than or equal to 50N/mm^2 ($f_{cu\ cube28} \leq 50$ N/mm^2), high performance concrete (HPC) / high strength concrete (HSC) with cube compressive strength greater than 50 N/mm^2 ($f_{cu\ cube28} > 50$ N/mm^2). Rajeev (2017) gave the compressive cube strength at 28days of age for different types of concrete to include Normal strength ($f_{cu\ cube28} = 20 - 50$ N/mm^2), high strength or high-performance ($f_{c,\ cube28} = 50 - 100$ N/mm^2), ultra-high-strength ($f_{cu\ cube28} = 100 - 150$ N/mm^2), special ($f_{c,\ cube28} > 150$ N/mm^2), and Reactive powder ($f_{c,\ cube28}$ =>200 N/mm^2). Table 2.1 shows the different classes of concrete based on strength criteria.

Table 2.1: Strength classes for normal weight concrete according to BS EN 2061:2001 and classification according to fib Model Code 2010.

Concrete type	**Strength class $f_{ck,\ cyl}$ / $f_{ck,\ cube}$**						
Low strength concrete	C8/10	C12/15	C15/20				
Normal strength concrete	C20/25	C25/30	C30/37	C35/45	C40/50	C45/55	C50/60
High strength concrete	C55/67	C55/67	C70/85	C80/95	C100/115	C100/115	
Ultra-High strength concrete				C110/130, C120/140			

Source: Dehn (2012)

Concrete can also classified according to different literatures on the basis of water to binder (W/B) proportioning as concrete with low W/B ratio below 0.4 (functions differently from NSC) and concrete with high W/B ratio of ≥ 0.4 (Aītcin, 1998; Neville, 2012). This study is focused basically on HPC.

2.1.2 High Performance Concrete

High-performance concrete (HPC) is defined as that type of concrete that is designed to be more durable and stronger than the normal conventional concrete with cube compressive strength in the range of 50-100 N/mm^2 at 28days of age (Rajeev, 2017). High-performance concrete (HPC) usually has an improved performance in terms of strength, durability, workability when compared with the NSC and the amount of cement in HPC is more than that in NSC with inclusion of one or more SCMs like SF, fly ash, RHA etc (Olawuyi, 2016). The production of HPC requires special materials which are combined in right proportion for the required performance of the structure (Rajeev, 2017). To achieve a HPC mix, the W/B is usually reduced, the pore space and microstructure of cement paste is refined using SCMs while workability is improved by incorporating a large amount of superplasticizer (Faleschini *et al*., 2015; Harbec *et al*., 2017).

2.1.3 Application of High-performance concrete (HPC)

The utilization of HPC in building construction has now gotten popular in the recent years and use widely in different productions (such as many long spans bridge girders, shotcrete repair, poles, parking garages, and agricultural applications (Mehta & Monteiro, 2014). The following are the structures found in Aïtcin (1998) constructed through HPC adoption reported by Olawuyi (2016).

- Water Tower Place built in 1970 in Chicago, Illinois, USA. This involved concrete of Strength 60 N/mm^2 at 28 days having a lingosulfonate based water reducer added.
- Norway's Gullfaks offshore platform built in 1981. It is made of concrete with average strength of 79 N/mm^2 at 28 days and a slump of 240 mm in the fresh state.
- Sylans and Glacières viaduct built in France in 1986. This utilized concrete of 60 N/mm^2 and 50 N/mm^2 at 28 days strength at different portions of the structures as against the initial design strength of 40 N/mm^2.
- Scotia Plaza, built in 1988 in Toronto, Canada, made of concrete with average tested strength of 93.6 N/mm^2 at the 91 days.
- Two Union Square, built in 1988 in Seattle, Washington, USA. Constructed with a concrete having a W/C of 0.22, utilizing naphthalene-based superplasticizer; outstanding aggregates of 10 mm maximum size and coarse graded sand of 2.80 fineness modulus; its strength at 28 days is 131 N/mm^2.
- Joigny bridge, built in 1989 in France. The concrete used for construction of the bridge deck had tested compressive strengths of 3 days = 26.2 N/mm^2; 7 days = 53.6 N/mm^2; 28 days =78.0 N/mm^2. The design was actually for 28 days strength = 60 N/mm^2 against the usual 35 – 40 N/mm^2.
- Montée St-Rémi Bridge built near Montreal, Canada in 1993. The bridge has two continuous 40 m spans made of HPC with 28 days strength of 60 N/mm^2.
- The 'Pont de Normandie' bridge, completed in 1993. This was the longest cable-stay bridge in the world when it was built having an overall length of 2141 m and a centre span of 856 m. It has approximately 35, 000 m^3 of HPC of 28 days strength = 60 N/mm^2 used for construction of the pylons and cantilever beams.
- Hibernia offshore platform, completed in Newfoundland, Canada in1996. It was built with a 'modified normal weight' HPC having W/B ratio of 0.31 for the splash zone

and 0.33 for the Submerged zone for a design of 28 days strength of 69 N/mm^2. The design constraints included unit mass of between 2200 and 2250 kg/m^3 for buoyancy and meeting an elastic modulus of $\geq$ 32 GPa.

- Confederation Bridge completed between Prince Edward Island and New Brunswick in Canada in 1997. It consists of 44 main spans of 250 m length each and massive main pier shaft and foundation elements fabricated on land. The approach pier foundation and some mass concrete sections, requiring control of thermal cracking, were built with Class C (fcu_{cube28} = 30 N/mm^2 and fcu_{cube28} = 40 N/mm^2) concrete which contained cement partially replaced with approximately 32% fly ash (FA) in addition to 7.5% silica fume (SF) by weight of cement content. The Class A concrete in this work had the strength at 28 days = 55 N/mm^2 and a maximum of 1000 coulombs chloride permeability at 28 days. Some piers with an abrasion resistance shield were built with strength at 28 days = 80 N/mm^2 concrete (Mehta & Monteiro, 2014).

In recent times, Olawuyi (2016) reported the following worldwide high-rise buildings that adopted the utilization of HPC in the construction of critical structural members and such structures include:

- 85-storey Tungtxt and Chintia Towers, Taiwan.
- 423.2 m Trump International hotels and Towers in Chicago, Illinois, USA, used 69 MPa concrete strength.
- 307 m Infinity Towers in Makkah, Mecca, used 70 N/mm^2 concrete strength commissioned in 2009.
- 541.3 m Tower One, World Trade Centre, built in 2014, used 97 N/mm^2 concrete strength.

- 451.9 m Petronas Twin Towers 1998.
- 508 m Taipei 101 2004 Taipei Taiwan.
- 91 storey Eureka Tower Melbourne Australia was completed in 2006.
- 90 storey Baiyoke-2 Tower Bangkok, Thailand, utilised 60 N/mm^2 concrete strength
- 47 storey Trianon Building in Frankfurt, Germany, built in 1992, used 112 MPa concrete strength.

2.1.4 Characteristics of high-performance Concrete (HPC)

The material constituents of HPC are water, cement, fine aggregate, coarse aggregate, mineral admixtures and one or two types of superplasticizers. HPC is characterized by special performances in term of workability, strength and durability. Mudashiru *et al*. (2021) mentioned two fundamental principles should be applied to get high compressive strength of HPC. These are low w/b ratio and high workability. Mostly it is designed with very low water binder ratio (w/b), from 0.35 down to 0.2. The low w/b ratio will affect the cement particle being close to each other due to less free water and then particles of cement will be denser. Consequently, this concrete is less permeable and more resistant to aggressive solution (Aïtcin, 1998).

HPC usually contains about 450-550 kg/m^3 of ordinary Portland cement (OPC) with the cementitious material ranged from 5% to 15%. By using superplasticizer, workability of concrete ranged from 400-600 mm slump with a very low w/c ratio can be achieved (Aïtcin, 1998; Neville, 2012). However, it is sometimes difficult to control the rheological properties of HPC, with a slump of 200 mm, and to maintain its workability at an hour or more after mixing (Aïtcin, 1998). The interaction between the cementitious materials and superplasticizers are more complex when incorporating some mineral admixtures such as blast furnace slag, fly ash, silica fume or natural pozzolans. Therefore, the selection of the compatible Pozzolanic material and superplasticizer requires further consideration (Lura *et al.*, 2008).

The information on the properties of cement needed in producing HPC are the fineness (Lura *et al.*, 2008), particle size distribution and the chemical composition of cement (Mudashiru *et al*. 2021). The quantity of chemical compounds in cement also affects the rate of strength development. Chemical compounds of cement are dicalcium silicate (C_2S), tricalcium silicate (C_3S), tricalcium aluminate (C_3A), and tetra calcium aluminoferrite (C_4AF). It will hydrate and gain early strength at a faster rate if the amounts of C_3A and C_3S contain is high in cements.

Workability is an important part in HPC because concrete with low w/b in the range of 0.2 to 0.3 and including mineral admixture cannot be placed easily. Superplasticizer has to be added during mixing to achieve high workability for that type of concrete. Also aggregate could affect the workability of HPC since aggregate is the most dominant quantity, which is about 70% of concrete volume. Its properties such as the fineness modulus, the maximum size and the type of aggregate influence the characteristic of HPC. The fineness modulus of fine aggregate of 3, which is a coarse one, is more favourable for HPC because there is a huge part of fineness material already in mix proportion of HPC. By increasing the fine modulus of fine aggregate, the surface area of the aggregate in the mixture is decreasing and free water is more available and increasing the workability (Neville, 2012).

Patel *et al*. (2013) which also reported and detailed in Mudashiru *et al*. (2021) explained that HPC having high strength can be produced with coarse aggregate size within the range of 20-25 mm. Lura *et al.* (2008) observed high strength concrete with different maximum size of coarse aggregates, cement content and w/b ratio. They mentioned that aggregate size of 10 mm gave higher strength than concrete that used higher aggregate size. In addition, Aïtcin (1998) reported that the type of coarse aggregate is also a significant factor in producing high strength HPC. Concrete containing basalt or limestone as coarse aggregate exhibit about 10% to 20% higher strength compared to granite and when comparing to limestone, concrete containing basalt exhibited higher compressive strength.

HPC can be produced with compressive strength of 80 N/mm^2 at 28 days inclusion of ultra-fine particles and superplasticizer in concrete with the w/c ratio of 0.3. It is expected that some particles react with lime and the other fill the voids between the cement and the silica. This effect contributes to the strength development of concrete. Also, compressive strength of concrete can be increased by decreasing w/c ratio, using efficiently cement/superplasticizer combination and using the best material (Aïtcin, 1998). There are different characteristics of HPC in terms of their application as detailed by many researchers in the literature (Aïtcin, 1998; Beushausen & Dehn, 2009; Mehta & Monteiro, 2014; Neville, 2012) but may range between the following properties:

a. High workability
b. High-early strength
c. High-strength
d. High-elastic modulus
e. High-durability and long life in severe environments
f. Low permeability and diffusion
g. Resistance to chemical attack
h. High resistance to frost and de-icer scaling damage
i. Toughness and impact resistance
j. Volume stability
k. Ease of placement
l. Compaction without segregation
m. Inhibition of bacterial and mould growth

2.1.5 Materials used in High-performance Concrete (HPC) production

The required performance of HPC is totally dependent on materials selection of the constituents. Materials selection will help to achieve consistent, high quality concrete while giving attention to handling, mixing and production techniques of the constituent materials. The constituent materials of HPC includes the following

2.1.5.1 Portland cement (PC)

Portland cement is a binder made by mixing together argillaceous and calcareous, or other alumina, iron and silica oxide materials with addition of clinker at a temperature of 1400^{o}C (Neville, 2012). In the word of Neville, 2012, the crude materials used for PC production comprise of mixtures of iron (Fe_2O_3), silica (SiO_2), lime (CaO), and alumina (Al_2O_3) and two minor mixes; magnesia (MgO) and gypsum ($CaSO_4 . 2H_2O$). The calcareous (CaO) materials can be found in calcium carbonate (limestone), marl, calcite, or shale and the argillaceous (SiO_2 and Al_2O_3) materials can be found in sand, shale, and mud.

According to Beaushausen and Dehn (2009), the Portland cement must be in accordance to BS EN 197 – 1(2000) specification. However, in the word of Olawuyi (2016), for workability and strength development to a long-term limit of ≤ 105 MPa, CEM I 42.5 N is recommended for use for the production of HPC. Olawuyi (2016) further asserts that CEM I 52.5 R will provide a higher strength but will demand more water and higher heat of hydration. Hence, the use of a retarding agent to prevent rapid hardening is recommended.

Table 2.2: Oxide composition of Portland cement from existing literature

Oxides	**SiO2**	**Al2O3**	**Fe2O3**	**CaO**	**MgO**	**SO3**	**K2O**	**Na2O**	**M2O5**	**LOI**
CEM II (%)	15.38	4.14	3.19	56.92	2.44	1.59	0.21	0.04	0.04	15.59

Source: Nduka *et al;* 2020

Properties of Portland cement

Characteristics of consideration of Portland cement as outlined in Neville (2012) include:

- Fineness
- Chemical composition
- Hydration
- Setting and hardness
- Strength Development
- Soundness

Fineness

This is one of the important characteristics of cement as it influences the rate of cement reaction with water. Fineness affects the rate of reaction because of the fact that finely ground cement has more exposed surface area than a coarsely ground one (Neville, 2012). By affecting the rate of reaction, the rate of setting and hardening of cement are also affected. Thus, fineness assists in completing the chemical reaction between cement and water within short time (Neville, 2012).

Fineness of cement is measured in terms of the specific surface of the cement which is determined in an air permeability apparatus (Olawuyi, 2016). A typical value of specific surface for ordinary Portland cement is 300m^2/kg (Mudashiru *et al*. 2021). Fineness could also be determined or estimated by sieving 0.11kg of cement sample through no. 170 sieve (0.09mm) for 15 minutes (Shetty 2004).

Chemical Composition

This affects all properties of cement apart from the fineness. Chemical composition is the basis for characterization of cement (Mudashiru *et al*. 2021). Cement with higher content of certain constituting compound relative to others could cause retardation or acceleration of the rate of setting and hardening (Olawuyi, 2016). The BS EN 196-1:2016 states basic requirements in relation to the ratios of lime, silica, alumina, and iron oxide. It also sets the limit for alkali and sulphate contents. Chemicals like calcium chloride and calcium sulphate (gypsum) are used as additives to accelerate or retard the rate of setting of cement paste (Olawuyi, 2016).

Hydration

Hydration can simply be defined as the chemical reaction between cement and water. Hydration of cement is an exothermic process; the heat released is called "heat of radiation" (Neville, 2012). For ordinary Portland cement, the heat released could be as high as 85-100 cal./g. for low heat cement, the heat of hydration is about 60-70 cal./g (Neville, 2012). Excessive release of heat during hydration could be detrimental (sometimes advantageous) to concrete especially when the concrete structure is massive (Neville, 2012). It causes cracking and disintegration of the structure due to the high insulation properties of concrete. It does not allow heat to dissipate easily thereby causing restrained thermal expansion (Olawuyi, 2016).

The hydration of cement can be thought of as a two-step process. In the first step, called dissolution, the cement dissolves releasing ions into the mix water. The mix water is thus no longer pure H_2O, but an aqueous solution containing a variety of ionic species, called the pore solution. The gypsum and the cement minerals C_3S and C_3A are all highly soluble. Therefore, the concentrations of ionic species in the pore solution increase rapidly as soon as the cement and water are combined. Eventually the concentrations increase to the point that the pore solution is supersaturated, meaning that it is energetically favourable for some of the ions to combine into new solid phases rather than remain dissolved (Neville, 2012).

The second step of the hydration process is called precipitation. The new precipitated solid phases, called hydration products, are different from the starting cement minerals. Precipitation relieves the super saturation of the pore solution and allows dissolution of the cement minerals to continue. Thus cement hydration is a continuous process by which the cement minerals are replaced by new hydration products, with the pore solution acting as a necessary transition zone between the two solid states (Neville, 2012).

The following are factors affecting hydration of cement as highlighted by (Neville, 2012)

- **C_3A content:** Increase in C_3A content leads to increase hydration of cement because higher percentage will react in short time.
- **Cement fineness**: Increase in cement fineness increases the hydration of cement.
- **Alkali content:** Increase in alkali content will increase reaction of gypsum that leads to increase hydration of cement.
- **Curing temperature:** Increase in temperature of curing increase the reaction of C_3A with gypsum which increase hydration of cement
- **Free CaO and MgO:** Increase of these oxides lead to decrease in optimum gypsum content (O.G.C.)
- **Chlorides:** Increase of chlorides lead to decrease in hydration of cement

Setting and Hardening

Setting is the stiffening of cement paste with time (Olawuyi, 2016). As soon as water is poured unto cement, hydration process begins and at the same time cement start to stiffen

(Olawuyi, 2016). The paste first loses its plasticity to become a bit rigid at this stage the cement is said to have set (Olawuyi, 2016).

Setting of cement is divided into two stages (Neville, 2012):

- Initial setting
- Final setting

The initial setting of the cement is reached when the needle (rod or square with 1mm^2 cross section) weighing 300 grams can no longer pierce the cement paste within 5mm from the bottom and the time taken to reach this stage is called the initial setting time (Olawuyi, 2016). The Final setting of the cement is attained when after gently lowering the needle it makes an impression but the circular cutting edge does not. The time taken to reach this stage is called the final setting time (Olawuyi, 2016). After the final setting; the paste continues to be more rigid and strong. The process after final setting is called "hardening" (Olawuyi, 2016).

The setting time of cement is determined by the use of the Vicat apparatus (Shetty, 2004). The Vicat apparatus is used to find the setting times of cement by filling the cylindrical mould with cement paste of known consistency. The final setting of cement is determined by using similar apparatus but with the needle fitted with a circular hollow cutting edge attachment of 5mm in diameter (Shetty, 2004).

The setting times of cement depend on the type of cement (Mudashiru *et al*. 2021; Shetty, 2004). The setting time should neither be too slow nor too fast. The setting times are highly variable property of cement. The initial setting time of cement should be long enough to enable mixing, transporting, placing and compaction of concrete or mortar to be completed before final setting starts (Mudashiru *et al*. 2021; Shetty, 2004).

Strength development of cement

Cement develops strength as a result of intertwining of the cement gel during hydration process (Neville, 2012). Strength test for cement is performed with mortar made by mixing cement and pure silica (or very clean sand) in the ratio of 1:3 and a water/cement ratio of 0.4 (or 10 % mixing water) (Mudashiru *et al*. 2021). These test results may be affected by grading, water content, thoroughness of mixing, degree of compaction, temperature and humidity during mixing and curing, condition of test equipment etc. (Mudashiru *et al*. 2021; Shetty, 2004).

Soundness

Soundness can simply be defined as the ability of hardened cement paste to retain its volume. In the soundness test a specimen of hardened cement paste is boiled for a fixed time so that any tendency to expand is speeded up and can be detected (Neville, 2012; Shetty, 2004).

2.1.5.2 Aggregates

Aggregates mechanical properties such as the shape, and grading of the aggregate in addition to the chemical reaction plays a vital role with respect to the strength and durability of high performance concrete (HPC) (Olawuyi, 2016). It is also very important to note that control of the amount of water demanded by the aggregate in HPC mix is very important. To achieve this, rounded aggregate should be selected and incorporated for HPC production (Beushausen & Dehn, 2009). The fineness nature of the fine aggregate should be as low as ≤ 150 μm (Beushausen & Dehn, 2009). As recommended by Neville, (2012) the fineness modulus of the sand should be in the range of 2.7-3.0. ACI 363R-92 recommends smooth texture and rounded shape fine aggregate. Beushausen and Dehn (2009) also recommend that coarse aggregate with maximum size of 8 to 16mm preferably crushed stone or granite should be use so as to avoid stress concentration due to lack of homogeneity in the concrete matrix. Rajeev (2017) stated that when a higher targeted compressive strength is desired, smaller maximum size of coarse aggregate is used. He went further to state that for concrete with compressive target strength of 70MPa, coarse aggregate with maximum size of 20 to 28mm is recommended. Similarly, for 125MPa target compressive mean strength, coarse aggregate with maximum size of 10 to 14mm is recommended for use

Properties of aggregates

The properties of aggregates include the following (Neville, 2012)

- Particle Size distribution
- Bulk density
- Water absorption capacity
- Moisture content
- Specific gravity
- Fineness modulus
- Particle shape
- Texture

Particle Size Distribution

This is the distribution of the various particle sizes in an aggregate sample (Neville, 2012). The particle size distribution or grading is very important as it affects many properties of the finished mortar. Properties like workability, compaction, density, durability, creep, shrinkage and cost of the mix are affected by the aggregate are grading (Olawuyi, 2016).

The particle size distribution is determined by the method of sieve analysis as described in BS EN 933-1:2012). It involves passing a sample of the aggregate through a series of sieves each having an aperture half the size of that in the previous sieve.

Bulk Density

Bulk density is defined as the weight of aggregate held by container of unit volume when filled or compacted under defined conditions. It is expressed in kg/m^3 or g/cm^3 (BS EN 933-1:2012).

Bulk density of a given aggregate sample depends on grading, texture, moisture content, degree of compaction, aggregate shape etc. bulk density is determined by weighing amount of aggregate required to fill an appropriate container of known volume and computed using the relationship as stated in Equations 2.1, while Equation 2.2 gives that for the specific gravity.

$$\text{Bulk Density (D)} = \frac{\text{mass of aggregate required to fill the container}}{\text{volume of the container}} \qquad (2.1)$$

$$\text{Specific Gravity} = \frac{\text{bulk density}}{1000} \qquad (2.2)$$

Aggregate’s bulk density is usually specified as loose or compacted bulk density depending on whether there is compaction or not. An aggregate of given specific gravity having high bulk density means that there is dense packing (probably due to good grading or compaction) and thus, less voids (Mudashiru *et al.* ,2021).

Water Absorption Capacity of aggregates

The internal pore characteristics are very important properties of aggregates. The size, the number, and the continuity of the pores through an aggregate particle may affect the strength of the aggregate, abrasion resistance, surface texture, specific gravity, bonding capabilities, and resistance to freezing and thawing action (Neville, 2012). Absorption relates to the particle's ability to take in a liquid. Porosity is a ratio of the volume of the pores to the total volume of the particle. Permeability refers to the particle's ability to allow liquids to pass through. If the rock pores are not connected, a rock may have high porosity and low permeability (Mudashiru *et al*., 2021).

Moisture Content

Moisture content is defined as the amount of water present in an aggregate sample. Properties like workability, compaction, density, durability, creep, shrinkage are affected by the aggregate's moisture (Neville, 2012).

Specific gravity

Specific gravity of aggregates can simply be defined as the ratio of weight of a given volume of aggregates to the weight of an equal volume of water (Neville, 2012). Specific gravity is dependent on the density of the material being studied, temperature, weight and pressure.

2.1.5.3 Water

Water (in quality and quantity) is an important factor to the mortar/concrete output from the production processes. Hence a good study of the requirements for good mortar and concrete. The mixing water initiates hydration process (responsible for strength development) and assists in workability (which enables concrete to be easily mixed, transported and placed) (Olawuyi *et al.,* 2017).

The quality of water must be drinkable. Water should be free from substances like clays, silts, algae, oils, acid, alkalis, salts, sugars and sugar derivatives, organic matter and sewage (Olawuyi *et al.,* 2017). Generally, any material that can retard hydration process and affect durability of concrete should be avoided. Thus, theoretically, the best water quality is distilled water. Practically, the best water quality is clean fresh water, fit for drinking (Olawuyi, *et al.,* 2017).

The quantity of water used for mixing is one of the most important factors that determine the quality of hardened concrete. Too little water produces a mortar that is harsh, difficult to mix, difficult to transport, difficult to place, difficult to compact and ultimately leading to a hardened concrete that is weak, easily eroded, porous and non-durable (Neville, 2012). On the other hand, too much water leads to a concrete that is prone to segregation, settlement, bleeding, excessive shrinkage and excessive cracking (Neville, 2012). The quantity of mixing water should be adequate to initiate hydration and provide workability. The quantity of mixing water is specified in terms of water/cement ratio (w/c) by weight (Neville, 2012).

2.1.5.4 Admixtures

According to ACI 212, 2010, define admixture as any material other than water, cement, aggregate and fibre reinforcement that is added to cementitious mixture aimed at modifying the freshly mixed, setting, or hardened properties of concrete. Cement admixtures include super plasticizers, accelerators, retarders, air-entraining admixtures, water resisting admixtures, foaming agents, pumping agents, pigments etc. (Rajeez, 2017). He went further and said that the compatibility of the admixture to be used is dependent on the nature and type of the binder.

Superplasticizer is the most commonly used admixture (Rajeez, 2017). Polycarboxylate Ether (PCE) which is a derivative of Polynapthalene Sulfonate (PNS), Polymelamine Sulfonate (PMS), and Acetone Formaldehyde Sulphite (AFS) is considered as the most effective and efficient superplasticizer to be used to disperse the calcined clay blended particles in Portland cement (Olawuyi, 2016). The dosage of superplasticizer used in the production of HPC plays an important in the workability of concrete. Usually, between 1.5% to 2% of binder weight is recommended as the maximum dosage of superplasticizer for HPC (Olawuyi, 2016). He stated further that the addition of superplasticizer is effective in terms of cost than adding cement that would otherwise necessitate an increase in water demand. However, Olawuyi,

(2016) further reported that an increase in the dosage of superplasticizer will cause a reduction in hydration of the cement paste, bleeding, excessive entrained air, increase in plastic shrinkage of the concrete. To avoid this, manufacturer guide with respect to the dosage must be strictly adhere to.

2.1.6 Properties of concrete at fresh state

Fresh or plastic concrete is a freshly mixed material which can be moulded into any shape. The relative quantities of cement, aggregates and water mixed together, control the properties of concrete in the wet state as well as in the hardened state (Neville, 2012).

The properties of concrete at fresh state include the following:

- Workably
- Segregation
- Bleeding

2.1.6.1 Workability

The fluidity of concrete is known as workability (Neville, 2012). Generally, workability is defined as the ease with which a given set of materials can be mixed into concrete and subsequently handled, transported and placed with minimum loss of homogeneity (Neville, 2012). Workable concrete is one which exhibits very little internal friction between particles (Neville, 2012)

Factors affecting workability of mortar includes (Neville, 2012):

- Water cement ratio(W/C)
- Size of aggregates
- Surface texture of aggregates
- Mix proportions

- Shape of aggregates
- Grading of aggregates

The key factor which affects workability is the free-water content of the mix after any water is absorbed into the aggregates (Neville, 2012). This quantity is defined by the water/cement ratio as stated in Equation 2.3.

$$\text{Water/cement ratio} = \frac{\text{weight of free water}}{\text{weight of cement}} \qquad (2.3)$$

The free water in a mix is the quantity remaining after the aggregates have absorbed water to the saturated surface-dry condition. The free water is used to hydrate the cement and to make the mix workable (Neville, 2012). With low water/cement ratios below 0.4, some of the cement is not fully hydrated. At a water/cement ratio of 0.4, the hydrated cement just fills the space previously occupied by the water, giving a dense concrete. As the water/cement ratio is increased above 0.4, the mix becomes increasingly workable but the resulting cured concrete is more porous owing to the evaporation of the excess water leaving void spaces (Neville, 2012).

The following tests are commonly employed to measure workability (Neville , 2012).

- Slump test
- Compacting factor test
- Flow table test
- Kelly Ball test

2.1.6.3 Segregation

Segregation according to Neville (2012) is the separation of the mixed constituents so that their distribution is no longer uniform due to insufficient cohesiveness of concrete because of the constituents used.

2.1.6.4 Bleeding

Bleeding is encountered when some of the water from the mortar mixes comes to the surface of newly placed mortar. It is sometimes known as water gain and is a form of segregation which is caused by squeezing out of some water from the concrete mixing (Neville, 2012).

2.1.7 Properties of concrete at hardened state

Concrete begins to harden immediately after mixing. Hardening is the process whereby water is lost from the mix and it occurs in three main ways, (Neville, 2012) namely:

- Absorption of water by the fine aggregate
- Evaporation of water from the mix due to exposure to the atmosphere
- Removal of water by cement when it begins to hydrate, i.e. the reaction between water and cement.

The study and understanding of the properties of hardened concrete is very important in predicting the performance of concrete in service condition. Because of the heterogeneous nature of concrete, its properties might be remarkably affected by minor variation in the mix proportions, curing methods, testing methods and/or the prevailing climatic condition (Neville, 2012)

The followings are key hardened properties of concrete (Neville, 2012):

- Crushing strength
- Tensile and flexural strength
- Density
- Permeability
- Durability

2.1.7.1 Crushing (compressive) strength

The ultimate crushing strength of concrete is of major importance as it is directly related to many other properties. Concrete having high compressive strength is likely to have a good durability when subjected to weathering and chemical attack (Neville, 2012).

The strength of concrete is influenced by certain factors. The factors are type and quality of cement, degree of compaction, water/cement ratio, age, curing condition, etc, (Mudashiru *et al*. 2021).

2.1.7.2 Density

Neville, (2012) defined the density of mortar and concrete as mass per unit volume. Density is also known as unit weight or unit mass in air. Density can be ascertained in the laboratory via the procedure offered in Neville, (2012). Hypothetically, density is the quantity of masses of all the constituents of a batch of concrete divided by the volume occupied by the mortar. Concrete samples with higher density than 2400 kg/m3 are known as normal weight concrete Neville, (2012).

2.1.7.3 Water absorption

Absorption (as a measure of permeability) is defined as a process through which liquid penetrates into and fill porous medium within a solid body, such as paste, mortar or concrete

(Neville, 2012). The strength of cement presented to cruel conditions depends generally on transport properties, which are affected by the pore framework. Water ingestion chiefly relies upon the aggregate volume of pores, filler composes, thickness and saturation systems of the solid (Mudashiru *et al*. 2021). Lausten *et al*. (2011) described water absorption as the ability of concrete samples to take in water by means of capillary suction.

2.2 Supplementary cementitious materials /pozzolan

Supplementary cementitious materials (SCMs) otherwise called Pozzolan are siliceous and aluminous materials, which in themselves possess little or no cementitious value, but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide liberated on hydration, at ordinary temperature, to form compounds possessing cementitious properties (Shetty, 2004). Neville, (2012) also defined a Pozzolan as a siliceous or aluminosiliceous material that, in finely divided form and in the presence of moisture, chemically reacts with the calcium hydroxide released by the hydration of Portland cement to form calcium silicate hydrate and other cementitious compounds.

2.2.1 Types of supplementary cementitious materials (SCMs) Pozzolan

Pozzolanic materials can be divided into two groups: natural Pozzolan and artificial Pozzolan (Shetty, 2004)

Natural Pozzolan are essentially volcanic ashes from geologically recent volcanic activities. Natural Pozzolan includes clay and shales, opalinc cherts, diatomaceous earth, volcanic tuffs and pumicites.

Artificial Pozzolan results from various industrial and agricultural processes, usually as by-products. Artificial Pozzolan includes fly ash, blast furnace slag, silica fume, metakaoline, surkhi, burnt clay, millet husk ash, sorghum husk ash, rice husk ash.

2.2.2 Classification of natural Pozzolan

Neville, (2012): classifies natural Pozzolan as: Class N, Class F, and Class C.

Class N—Raw or calcined natural Pozzolan that comply with the applicable requirements for the class as given herein, such as some diatomaceous earths; opalinecherts and shales; tuffs and volcanic ashes or pumicites, calcined or un-calcined; and various materials requiring calcination to induce satisfactory properties, such as some clays and shales.

Class F—Fly ash normally produced from burning anthracite or bituminous coal that meets the applicable requirements for this class as given herein. This class of fly ash has Pozzolan properties.

Class C—Fly ash normally produced from lignite or sub bituminous coal that meets the applicable requirements for this class as given herein. This class of fly ash, in addition to having Pozzolan properties, also has some cementitious properties.The basic requirements of various codes and standards for Pozzolan are presented in the Table 2.3 and 2.4

Table 2.3: ASTM Physical and Chemical Requirement for Pozzolan

S/NO	Property of Pozzolan	ASTM Requirement (%)
i)	Water-soluble solution	10.00
ii)	Fines: Amount retained when wet sieve	
	Number 30 Sieve (600ɥm) Max	2.00
	Number 200 Sieve (75 ɥm) Max	30.00
iii)	Drying shrinkage (Max)	0.15
iv)	Increase in drying shrinkage of Portland Pozzolan cement mortal bars at 28days (Max)	0.03
v)	Water requirement (Max % of control)	115
vi)	Silicon dioxide (SiO_2) + Aluminum oxide (Al_2O_3) + Iron oxide (Fe_2O_3)	70
vii)	Pozzolanic activity index with Portland cement at 28days(Max % of control)	75
Viii)	Magnesium oxide (MgO) Max	5.00
ix)	Loss of ignition (LOI) Max	12.00

Source: Neville, (2012)

Table 2.4: Chemical Composition of Pozzolan according to Indian Standard

S/N	Characteristics	Requirements (%)
i)	Silicon dioxide (SiO_2) + aluminum oxide (Al_2O_3) + iron oxide (Fe_2O_3) percent by mass, Min	70.00
ii)	Silicon oxide (SiO_2), percent present by mass, Min	35.00
iii)	Magnesium oxide (MgO), percent present by mass, Max	5.00
iv)	Total sulphur trioxide (Na_2O), percent present by mass, Max	2.75
v)	Available alkalis, as sodium oxide (Na_2O), percent present by mass, Max	1.50
vi)	Loss on ignition, percent by mass, Max	12.00

Source: Shetty (2004)

2.2.3 Millet Husk Ash as SCMs

Millets are cereal crops or grains grown for food and fodder. They are prevalent in semi-arid tropics of Asia and Africa. They form a diverse group of small grains cultivated in diverse and adverse environments, mostly in the dry, semi-arid to sub humid drought-prone agro ecosystems. Worldwide, there are nine species of millets with total production of 28.38 million tons, out of which 11.36 million tons (40 %) are produced in Africa. The two most important millets cultivated in Africa are: Pearl millet- Pennisetumglaucum, finger millet – Eleusinecoracana (Jimoh *et al.,* 2015).

2.2.3.1 Uses of millet and millet husk ash

Millet husk is the outer jacket of the grain of millet with high concentration of silica. Generally, this silica concentration is more than 70 % after burning. (Olawuyi *et al.,* 2017).

Millet is used as animal feeds and a variety of traditional foods, most common of these are “akamu, kunu” obtained through milling process, the flour produced are used for preparing “tuwo”, “dakkere”, “kunu”, “fura” and “waina”. The stalks are used for fencing walls, huts, roof, basket making, mat and “argawa”. The grains are mostly fermented to alcohol drinks such “burukutu”, “peto” (Jimoh *et al.*, 2015).

MHA is obtained by burning millet husk in a large open drum. The combustion of millet husk removes the organic matter and produces, in most cases, a silica-rich ash (Jimoh *et al.*, 2015). It is this high silica content that gives the ash its Pozzolanic properties (Olawuyi *et al.*, 2017).

MHA has been used for stabilizing black cotton soil in a number of countries including Columbia, Thailand and India (Ramasamy and Biswas, 2008).

Table 2.4 shows the chemical composition of MHA as presented by Jimoh *et al*; (2015).

Table 2.5: Chemical composition of MHA

Oxides	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**CaO**	**MgO**	**K_2O**	**BaO**	**MnO**	**SO_3**	**L.O.I**	**$SiO_2+Al_2O+Fe_2O_3$**
MHA (%)	72.4	14.2	1.5	6.4	1.0	0.2	0.1	0.1	0.1	2.0	88.1

Source: Jimoh *et al*; 2005

2.2.3.2 Summary of previous studies on MHA

Studies have been carried out on the adoption and utilization of MHA as SCM in construction. Some of the work in the respect includes but not limited to:

Uche *et al (*2012**)** studied the influence of MHA on the properties of plain concrete when replaced partially at varying percentage and concluded that the workability of fresh concrete decreases with increase in MHA content and this may not be unconnected with the water absorption capacity of MHA which has greater specific surface area than cement. Linear shrinkage of MHA concrete also increased with increase in percentage of MHA addition. The result of compressive strength increased at 5% MHA addition but decreases at higher percentage addition. As MHA increases up to 15 %, the concrete compressive strength was still within the structural concrete grade of 20 N/mm^2.

Tsado *et al (*2014) studied the compressive strength of MHA when it is partially replaced with cement at 5% and 20% with cement: water ratio of 0.65 in concrete production and found out that at 35 days of curing age the compressive strength for 0%, 10% and 20% replacement are 32.00 N/mm^2, 25.56 N/mm^2 and 23.18 N/mm^2 respectively and that MHA as a pozzolanic materials can be incorporated upto 10% replacement in order to develop a good and hardened concrete.

Jimoh *et al* (2015) studied the strength characteristics of MHA blended cement laterized concrete with the view to determining its suitability as an alternative building material at different percentage of replacement of sand with laterite and cement with MHA in 0%, 10% and 20% replacement and cured for 7, 21 and 28 days respectively with the view of establishing the ash and laterite content that will be used in cement and sand matrix. The results obtained showed that the best strength performance was obtained at 10% MHA and 0% LAT and 10% LAT and 10% MHA with strength of 19.04N/mm^2 and 20.01N/mm^2 which when compared with the control result (normal concrete) which is 32.98N/mm^2 at 28 days for compressive strength. The flexural strength at 28 days is 5.03 for 0% LAT and 10% MHA, 5.21N/mm^2 for 10% LAT and 0% MHA and 5.10N/mm^2 for 20% LAT and 0% MHA respectively. MHA laterized concrete was thereby adjudged by Jimoh *et al* (2015) to hold a promise in the quest for alternative building material, and as such can be used in light weight structures such as masonry walls, walk ways etc.

2.2.4 Silica fume as SCMs

Silica fume (SF) is a by-product of the manufacture of silicon metal and ferro-silicon alloys. SF is a very fine powder consisting mainly of spherical particles or microspheres of mean diameter about 0.15 microns, with a very high specific surface area (15,000–25,000 m^2/kg) (Vinayagam, 2012). SF has been successfully used in the production of HPCs and it is generally known to improve the cohesiveness and decrease the segregation and bleeding (EFNARC, 2005). It also increased the compressive strength, modulus of elasticity, flexural strength and enhances the durability at all ages.

Silica fume according to Mehta and Monteiro (2014) improves the rheology and strength properties of HPCs and enhance stability when used at low dosages --typically less than 4-7% by replacement of cement but have detrimental effects on the fresh property of the concrete at higher dosages. Any reduction in workability is generally due to silica fume's high fineness which is offset at least partially by its spherical particle shape. According to Aitcin (1998), silica fume can improve workability and hardened property of concrete because the spherical particles displace water molecules from the vicinity of cement grains so that entrapped water molecules between flocculated cement particles are freed.

Park *et al*. (2005), the high reactivity of silica fume particles can increase adsorption which reduces the amount available in solution and on cement particles and thereby decreases the workability. Mehta and Monteiro (2014) found that spherical carbon black with a similar grain size as silica fume resulted in similar workability. For pastes designed for HPCs, Vikan and Justnes (2003) found that adding silica fume at up to a 10% volume replacement increased yield stress. Plastic viscosity was however reduced when a polycarboxylate-based superplasticizer was used and increased when a naphthalene-based superplasticizer was used. The decrease in plastic viscosity was attributed to the displacement of water between cement grains and the spherical shape of the silica fume particles.

For superplasticized pastes, Park *et al.* (2005) found that the use of silica fume at cement replacement rates of 10 and 15% increased yield stress and plastic viscosity and decreases the strength properties of HPCs significantly and it was suggested that the silica fume be used to increase plastic viscosity to prevent segregation and that the sharp increase in yield stress be offset by the use of a ternary cementitious system with either fly ash or slag. For conventionally placed concrete, Vikan and Justnes (2003) found that adding silica fume to concrete at replacement rates up to 7% significantly reduced plastic viscosity but had little effect on yield stress. Higher dosages of silica fume increased yield stress substantially but increased plastic viscosity more gradually.

The principal effects of silica fume on the microstructure of concrete are due to the pozzolanic reaction and to the filler-effect due to particle dimension, being the average silica-fume particle size (0.1 m) one order of magnitude smaller than the cement particle size. By adding silica fume to the fresh mix, it is also possible to improve the workability and decrease or avoid bleeding (Neville, 1995; Aitcin, 1998). The actual mechanisms by which silica fume improves the concrete behavior are still under discussion. Certainly, an improvement of the microstructure, i.e., pore refinement, leads to an increase of the concrete strength. If silica fume is added to the cement paste, the total porosity is reduced (Khan *et al.*, 2010), and the pore structure as measured by Mercury Intrusion Porosimetry (MIP) is refined (Li *et al.*, 2014).

Furthermore, the effect of silica fume was also found experimentally and confirmed by numerical simulation affirmed by Bentz and Garboczi (2018) as the improvement of the interfacial transition zone (ITZ) between the aggregates and cement paste. Some problems are connected to the use of silica fume as an admixture. The reaction of silica fume with calcium hydroxide has a high chemical shrinkage; about 0.22 ml/g of silica fume reacted (Jensen & Hansen, 2018). Silica fume modified cement pastes show much greater self-desiccation and autogenous shrinkage than Portland cement (Jensen & Hansen, 2019). This fact leads both to greater bulk shrinkage of silica fume concrete and to microcracking of the cement paste restrained by the aggregate (Li *et al*., 2014). Early-age cracking of HPC occurs due to the fact that the deformations are restrained. Restraint can be external which caused as a result of the adjoining structures and or the internal to the concrete member caused by reinforcement or by non-shrinking aggregates (Li *et al*., 2014). External restraint may cause through cracking of a concrete member while internal restraint may result in micro-cracking.

2.3 Internal curing of HPC

Internal curing of concrete in the sixties according to Mudashiru *et al*. (2021) is labelled for and it significantly affects the rate and magnitude of expansion. Internal curing is one of the causes of micro-cracking eradication and lightweight aggregate concrete (LWAC) durability improvement stated by Mudashiru *et al*; (2021) as exposed to aggressive environments, such as bridge decks or concrete ships. The benefits of using LWA in concrete to help reduce cracking in slabs and bridge decks had been conjecturing known for decades by the LWA industry but the reasons were not examined (Norbert *et al*., 2007). It was generally believed according to the literature (Beushausen *et al*; 2009, Xiao *et al*; 2018) that the improved properties of LWAC were due to elastic compatibility between LWA and matrix and to improved ITZ. It was suggested that incorporating a saturated lightweight fine aggregate at the beginning of the nineties into the concrete mixture to provide an internal source of water to replace that consumed by chemical shrinkage during hydration of the paste could be a good idea. The suggestion opened the way to extensive research in the field.

Several studies have dealt with the experimental evaluation of the presence of LWA on autogenous shrinkage in which most assessed the use of coarse aggregates, evaluating the influence of their amount and degree of saturation. Recently, Mudashiru *et al*. (2021) explored the use of LWA as fine aggregate has been examined in the literature and demonstrated that autogenous shrinkage was practically eliminated by the use of wet LWA and also showed that the compressive strength did not suffer significantly from internal or external drying when a sufficient replacement of NWA with pre-wetted LWA was used. Khan *et al*; (2010) also used expanded clay LWA, Liapor and Leca of 4-12 mm grain size with about 600 kg/m^3 content and the absorbed water exceeded 40 kg/m^3 when soaked. The LWA were employed in three different states: oven-dried, moist, and water-impregnated.

In concrete with w/cm ratio of 0.3, autogenous shrinkage was eliminated even in the case of dry LWA, suggesting that LWA can absorb part of the mixing water and release it afterwards to the self-desiccating matrix. Yang *et al*;, (2019) suggested replacing part of the coarse aggregates by LWA of similar size as a means for providing internal curing in HPC to optimize for strength and the method was named autogenous curing in which the concrete obtained improved in mechanical properties regardless of the curing conditions as a result of a continuous hydration for months after casting as observed with x-ray diffraction (Yang *et al*; 2019). The mechanism of water transport from the LWA to the hydrating cement paste based on capillary suction was proposed. However, the issue of early-age deformations was not addressed (Yang *et al*; 2019).

Sickert *et al.* (2011) measured the expansion on LWAC with different amounts of LWA until more than 1 year after casting; measurements started at 24 hours. The mixtures contained from 500 to 800 kg/m3 of Liapor 9.5 mm maximum particle size that was moisturized with 1/3 of the mixing water for 45 sec prior to mixing (Zhutovsky *et al.*, 2001a; Zhutovsky *et al.*, 2001b). Van Breugel *et al*. (2014) also studied the effect of replacing a small part of the NWA by saturated LWA using mainly pumice sand of different size fractions. Expansion was measured in the first hours, followed by some shrinkage in the first week of hydration.

The use of pumice sand of different fractions led to different shrinkage reductions, with the largest grains being more effective. This was attributed to the different pore structures of the different sizes. According to Bentz and Snyder (2015), the spatial distribution of the ‘water reservoirs’ in the mixture is of primary importance in the internal curing process. The distance of the saturated LWA from the point in the cement paste, where the RH-drop takes place, determines the efficiency of the internal curing. They estimated the maximum transport distance of water, as a consequence of the depercolation of the pores in a low w/c ratio paste, as few hundreds of micrometers. If the water-reservoirs are well distributed within the matrix, shorter distances have to be covered, and the efficiency of the internal-curing process is increased. These considerations lead to the choice of small LWA. Bentz and Snyder (2015) calculated the percentage of cured paste with analytical formulas and numerical simulation as a function of the distance of water penetration and of the particle size distribution of the LWA.

2.3.1 Superabsorbent polymer (SAP)

Superabsorbent polymer is cross-linked polyelectrolytes which has the ability to absorb or retain large quantity water and release the water when the condition changes or need arise (Siriwatwechakul *et al*. 2010). They also have the ability to swell when in contact with an aqueous and begin to for a hydrogel (Mechtcherine & Reinhardt, 2012). Two classification of SAP exist. The thermoset or cross-linked polymers and thermoplastic or linear polymers. The thermoplastic polymers or three- dimensional polymers are polymers having a large molecular weight with its polymer’s chains overlapping, forming pseudo-three-dimensional structures with the junctions acting as physical cross-links (Siriwatwechakul *et a*l. 2010). While the thermoset or cross-linked polymers are polymers with chemical cross-links which hold the chain together so as to prevent being dissolved when immersed in solvents and or solution (Siriwatwechakul *et a*l. 2010).

2.3.1.1 Absorption and desorption of SAP in pore solutions

The absorption of SAP in pore solutions of the cement paste is dependent on the concentration of the ions present in that solution. Studies have been done on influence of SAP on the cement paste pore solution's ions concentration to be a function of time. From the report of the studies, it shows that potassium (K^+), sodium (Na^+), sulphate ions (SO_4^{2-}) and hydroxyl ions $(OH)^-$ are the highest ions concentration that are present in the pore solution of the cement paste and the highest concentrations is usually developed as soon as the hydration commence and remain constant until after the setting time (Lura *et al*; 2006). At the later age the ions concentration will decrease as a result of formation precipitation. The concentration of ions present in the pore solution of each cement paste usually differs depending on the contents of the alkali present in each Portland cement, and also the incorporation of supplementary cementitious materials (SCM) was further affirmed to affect the concentration of the ions (Lura *et al*; 2006).

From the study of Dehn (2012) on the use of Hundred to eight hundred (100-800 µm) SAP particle size, it was observed that after 24 hours, the water absorbed by SAP was higher than 500 g/g but after 300 seconds the water absorption was about 130 g/g.

From the study of Jensen and Hansen (2006) in respect to SAP desorption isotherm, it was observed that the free liquid absorbed by SAP in a distilled water as well as in a synthetic pore fluid was 350 g/g and 37 g/g respectively, the remaining pore fluid retained by the SAP was about 3 g/g at 98% relative humidity (RH) and less than 1 g/g at 86% RH (Jensen and Hansen, 2001). It was later affirmed that at the early stage of the hydration in low w/c concrete as a result of the rapid self-desiccation almost all the pore fluid absorbed by the SAP will be released.

Also in the work of Monnig (2009) on SAP desorption rate as a function of mass loss at different relative humidity of layers of saturated particles of SAP and it was observed that the water loss was rapidly close to the surface of the polymer but the water closer to the core of the polymer needs to overcome more side-chains in the polymer that interact with the molecules of water via the use of Van-der-Waal's forces.

2.3.1.2 Impact of SAP on the concrete microstructure and hardening of binder

The free water present in saturated SAP which desorbed when the condition changes and led to a RH reduction and cement hydration promotion is the mechanism that contributed to self-desiccation reduction in HPC incorporated SAP (Olawuyi 2016). Due to the inclusion of SAP, the hydration process and microstructure of the concrete were influenced. Therefore, as reported by Mechtcherine and Reinhardt (2012), the issues of considerations are the impact of SAP inclusion on cement degree of hydration and microstructure development characteristics which included porosity, pore size distribution, morphology and connectivity of bulk cement paste, interfacial transmission zone (ITZ) amid the cement pastes and SAP and the voids created by the SAP.

2.4 Concrete Mix Design

According to Neville (2012), the following are the procedure used for the selection of mix of concrete.

- Step 1: Choice of a slump
- Step 2: Choice of maximum aggregate size
- Step 3: Estimate of water content and air content
- Step 4: Selection of water/cement ratio
- Step 5: Calculation of cement content
- Step 6: Estimate of coarse aggregate content
- Step 7: Estimate of fine aggregate content
- Step 8: Adjustment to mix

However, in the British mix design method as presented by Neville (2006) comprises of the following five (5) steps:

- Step 1: Compressive strength to determine the water-cement/ratio. This method thereby brought to fore the concept of target strength
- Step 2: Determination of water content based on required workability recognising the influence of the maximum aggregate size and type
- Step 3: Determination of cement content
- Step 4: Determination of total aggregate content
- Step 5: Determination of fine aggregate content from the total aggregate content base on maximum size of aggregate, workability required, W/B ratio, and the percentage of fine aggregate passing through 600 µm sieve.

In the work of Aitcin (1998), as cited in Olawuyi (2016), presented a mix procedure for HPC to include the following five steps:

- No. 1 – choice of W/B;
- No. 2 – determination of water content;
- No. 3 – estimate for superplasticiser dosage;
- No. 4 – estimate for coarse aggregate content and
- No. 5 – estimate for entrapped air content (assume value).

This research, therefore, draws from (Olawuyi, 2016; Nduka *et al*., 2020; Olawuyi *et al*., 2020) suggested an approach for the modification of the British Mix Design (DOE method) to cover HPC with typical HPC mixes found in works of literature as reference. The specified cement content in literature for HPC is adhered to while compatibility and the manufacturer's guide on superplasticiser determine its choice and dosage. The W/B became the first guiding principle for this design, as suggested in the work of Aitcin (1998). The natural sand available was sieved to reduce the high dust content, and the quantity retained on 300 µm sieve was used in the mixes.

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 Research design

The research design used was experimental research design. The physical and chemical properties of the constituent materials, the characteristics of the concrete in the fresh state, the response of the hardened HPC mixes to the effects of applied force in compression, uniformity of the particles, movement of water by capillary and the impact of chemical ions on the hardened HPC mixes investigated gave a proper understanding of the strength and durability behaviours of the HPCs.

3.2 Materials

The materials that was used for this research work are binders (PC, SF and MHA), fine aggregates (sand), coarse aggregate, superplasticizer, pumice, and water.

3.2.1 Binders (PC, MHA and SF)

The PC for this study was Dangote brand (3X) of Portland cement (CEM 42.5N) whose properties conform to the requirements of BS EN 197-1: 2000. It was purchased from a cement store in Gidan-Kwano; Minna, Niger State.

The MHA which was used as (SCM) was obtained from the incineration of the husk using the locally fabricated incinerator available at the Concrete Laboratory of the Department of Building, Federal University of Technology, Minna, Niger State. The burning took place in an open air for about 24 hrs with a temperature below or equal to 700^{o}c and then allow to cool before harvesting and milling with grinding machine. The milled MHA was sieved with 75µm in accordance to ASTM C430- 2014 before storing in an airtight polythene bag.

The SF used as the second SCM for this study was purchased from a Construction Chemical company in Lagos manufactured by PURECHEM manufacturing LTD. The X-ray florescence (XRF) and particle size distribution (PSD) tests were conducted on the PC, MHA and SF for the actual information on their physical and chemical properties and to understand better, their possible influence on the cement hydration process and its products on strength and durability characteristics of various hardened HPC mixtures. Plate I shows the photographs of the binders.

Plate I: Silica fume (left), Millet husk ash (middle) and Portland cement ((Right)

3.2.2 Aggregates

The fine aggregate used for this research is natural sand with minimum particle size of 300 µm which is the requirement specification for HPC production (Shetty 2004, Neville, 2012, Nduka *et al*., 2020; Olawuyi *et al*., 2020). The sand's physical characteristics (i.e., specific gravity (SG); fineness modulus (FM); coefficient of uniformity (Cu); coefficient of curvature (Cc); and dust content) were analysed using the sieve analysis.

Crushed granite stone which passes through 13.50 mm sieve size and retained on at least 9.50 mm sieve size was used as coarse aggregate in compliance with typical HPC mixes found in the literature (Nduka *et al*., 2020; Olawuyi *et al*., 2021). The coarse aggregate was washed to remove dust impurities for less water demand by the mixture. Plate II shows the fine and coarse aggregates used for this study.

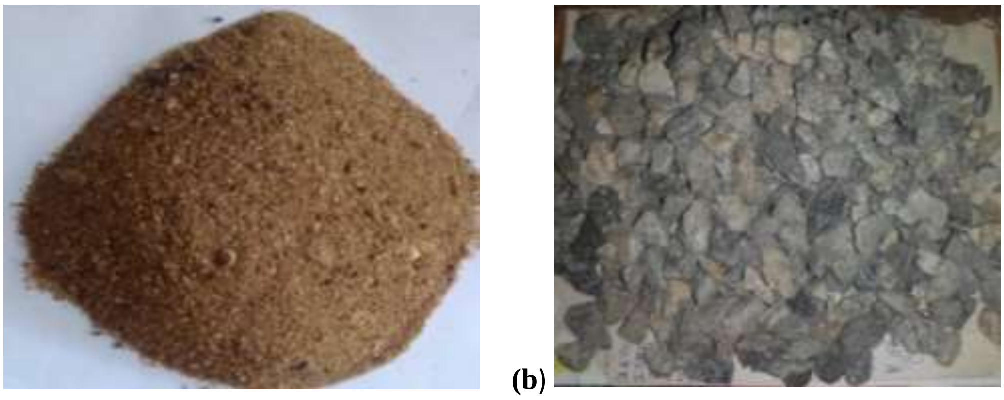
(a) (b)

Plate II: Fine aggregate (a) and coarse aggregate (b)

3.2.3 Internal curing agents

A Superabsorbent polymer (labelled FLOSET 27CS) of ≤ 600 μm grain size produced in France by SNF Floerger was added at 0.2% by weight of binder (b_{wob}) as detailed in Olawuyi and Boshoff (2017) considering 12 g/g as the SAP absorption capacity conforming to the requirement of SAP specification for the production of HPC determined by tea-bag test (Olawuyi *et al*., 2021). The SAP type used is a thermoset polymer specifically the covalently cross-linked polymers of acrylamide and acrylic acid obtained from bulk solution polymerization and neutralized by alkali hydroxide.

The pre-soaked Pumice used for this study was a porous igneous rock formed as a result of explosives volcanic eruptions which was later crushed and 12.5 mm maximum size was incorporated. The pumice was soaked for 24hrs in water after which the water was drained. An increase in the weight of the pumice was noticeable (Olawuyi *et al*., 2020).

3.2.4 Water

The water used for this study was portable water from the tap behind the convocation Square of Federal University of Technology, Minna, Niger State in accordance with the specification of BS EN 1008 (2002) was used for the mixing at 0.3 W/B (Ogunbayo *et al*. 2018).

3.2.5 Superplasticizer

A sky 504 Masterglenium polymer-based polycarboxylic ether (PCE) superplasticizer supplied by Armorsil Manufacturing Incorporation was used as the chemical admixture (superplasticizer) and was administered at 1.5% concentration by weight of binder (b_{wob}) as used in the typical HPC mixtures. As recommended in the work of Aitcin (1998) reported in Olawuyi *et al*; (2021), the water content of the superplasticizer was removed from the volume of water used in mixing in order to maintain the original W/B designed for (i.e., not to cause an increase in the designed W/B). Plate III shows the superplasticiser- Masterglenium Sky 504 used in this study.

Plate III: MasterGlenium sky 504 superplasticizer

3.3 Methods

Materials characterization, proportioning of the materials, production of the specimens, strength and durability properties determination was achieved using laboratory experiments in Building Department laboratory, Federal University of Technology Minna.

3.3.1 Materials Characterization

Materials characterization comprises of various tests carried out in order to determine the physical and chemical properties, chemical compositions, particle size distribution of the binders and aggregates and specific gravity of the aggregates, binders and consistency test, setting time and soundness test for the binders.

3.3.1.1 Chemical analysis

The chemical compositions of binders (MHA, SF & OPC) were studied using X-ray Fluorescent (XRF) analyser connected to a computer system for data acquisitions. About 100g of these binders were packaged in sealed polythene bags and sent to the chemical laboratory for the determination of the oxide compositions in accordance with BS EN 196-1 (2016).

3.3.1.2 Particle size distribution of aggregates and binders (sieve analysis)

According to Neville (2012), particle size distribution is defined as the grading material in to fractions. In accordance to British Standards (BS EN 933-1, 2012), the analysis of the aggregates particle size distribution as well as the binder was carried out using a sieving approach. The standard sieves set used for the coarse aggregate in descending order are 12.5 mm, 10 mm, 6.3 mm and 4.75 mm while 4.75 mm, 2.36 mm, 1.18 mm, 600 µm, 300 µm, 150 µm and 75 µm were used for the fine aggregate. The outcomes from particle size distribution of the sample were plotted on a semi-log graph with sieve particle diameter or size on the X-axis with the logarithmic axis while the Y-axis indicates the percentage passing.

As stated in Mudashiru *et al*; (2021), D60 is the particle diameter passing 60%; D30 is the particle diameter passing 30% and D10 is the particle diameter passing 10% were determined for further analysis to obtain the coefficient of uniformity (Cu) and coefficient of curvature (Cc) using equation 3.1 and 3.2.

$$\text{Uniformity Coefficient (Cu)} = \frac{D60}{D10} \tag{3.1}$$

$$\text{Coefficient of Curvature (Cc)} = \frac{D30xD10}{D60} \tag{3.2}$$

3.3.1.3 Specific Gravity Test of Binders and aggregates

The specific gravity of the binders and aggregates was determined in accordance to BS EN 933-1 – 2012. The apparatus are: rubber bottle, weighing balance (electronic scale), Glass plate, Weighing plate, 2 number specific gravity tubes (25ml). The following procedure was adopted:

Two (2) empty tubes were weighed on the scale and their weights were recorded as w_1, the tubes were then filled with water up to the 25ml mark, weighed and recorded as w_4. A small quantity of the materials was placed in each of the tubes and weighed as w_2, water was added to the materials and filled up to the 25ml mark and weighed as w_3. All the results obtained are recorded and specific gravity calculated using Equation 3.3.

The specific gravity equation = $$\frac{W2-W1}{(W4-W1)(W3-W2)} \qquad (3.3)$$

Where:

W_1= weight of empty bottle

W_2= weight of bottle + material

W_3= weight of bottle + material + water

W_4= weight of bottle + water

3.3.1.4 Moisture content of materials

The Moisture content of the materials was determined in accordance to BS EN 933-1, (2012). The apparatus are: BS sieves, wide metallic container, weighing balance and electric oven.

Fine aggregates were sieved using the standard sieves. Fine aggregates passing 5mm and retained on 300 µm were used. Then 500g was weighed out using the weighing balance. The fine aggregate was then spread in a wide metallic container which was put in an electric oven for 24 hours and then reweighed. The new weights were then recorded and the moisture content was calculated using the formula below

$$\text{Moisture content (w)} = \frac{M2-M1}{M3-M1} \times 100 \qquad (3.4)$$

Where M_1 = Weight of empty cans

M_2 = weight of can + wet sample

M_3 = Weight of can + dry sample

3.3.1.5 Consistency of the binders

Normal consistency is a test that is used to determine the amount of water required to produce binders’ paste of standard consistency. According to Neville (2012), standard consistency is described as the consistency which permitted a Vicat plunger having 10 mm diameter and 50 mm length to penetrate to a depth of 33-35 mm or 5-7 mm from the top and bottom of the mould respectively. The test was carried out in accordance with Neville (2012) using a vicat apparatus with the following procedures:

- About 300 g of cement was measured.
- The cement paste is placed in a vicat mould after mixing and the excess cement is removed by means of trowel
- Once the mould is filled completely, the surface of the mould is smoothly levelled and placed on a glass plate under the vicat apparatus
- The plunger was then attached to the apparatus and released quickly to find out the depth of the penetration
- Once the standard penetration (33-35 mm from the top) has been achieved then the setting time test begins

3.3.1.6 Setting time

The experiment begins immediately after the consistency has been achieved and was carried out on the cement and also on the combination of both binders (PC, SF & MHA), for all the mixes. The test was conducted in accordance with ASTM 1585 (2014) using a vicat apparatus. The initial setting time as described by Neville (2012) is the time period elapsed between the time when water is added to the cement and the time at which the cement paste starts losing it plasticity (i.e. the time at which 1 mm^2 needle make a penetration in such a way that the distance between the bottom of the vicat mould to the needle is between 4-7 mm and or 5 mm from the top of the mould) and was specified by the code to be minimum of 45 minutes, but 90 minutes was preferred on the field. Also, the final setting time as described by Neville (2012), is the time period elapsed between the time when water is added to the cement and the time at which the cement paste completely loses its plasticity (i.e., the time at which 5 mm diameter circular attachment fails to make an impression).

3.3.1.7 Soundness test

The soundness test was conducted in accordance with Neville (2012) using a Le-Chatelier mould in order to measure the quality of the binders with respect to expansion effect. The apparatus used in carrying out the experiment were the Le-Chatelier mould, cement, glass sheet, mixing pan, trowel and weighing balance. It was performed under a room temperature of.27 ± 2^oc and 65 ± 5% relative humidity. The procedures used are as follow:

i. About 300 g of binders was measured and 0.78P was added as the water (where P is the water required for standard consistency in percentage)
ii. The mould was oiled together with the base glass sheet and the paste of the binders was well mixed and placed inside the Le-Chatelier mould
iii. The mould was later covered at the top with a glass sheet and a small weight was placed over the cover plate

iv. Then this assemblage was later immersed in water at a temperature of 27 ± 2^{o}c for 24 hrs
v. The distance between the indicator points was later measured after removing from the water at 24 hrs and recorded as L1
vi. The assemblage was later immersed in boiling water for about 30 minutes and left there for about 3 hrs
vii. The mould was removed after boiling and the distance was taken and recorded as L2
viii. The soundness of cement was then calculated from L2 - L1

3.3.1.8 Slump test

According to Neville (2012), for the slump test, flow table test was used to measure the workability of HPC when a spread between 400-600mm is ascertained. The specimen is then cast and de-moulded after 24hrs.

3.3.2 Proportioning of HPC materials

In accordance with the work of Aitcin (1998) as cited by Nduka *et al.*, 2020, and Olawuyi *et al.*, 2021, mean target strength of C55/67 at 28 days was adopted as the mix design procedure for material proportioning for HPC production. Table 3.1 below shows materials proportioning of HPC with 5% of pre-soaked pumice by weight of coarse aggregate (b_{woca}), 0.2% SAP by weight of the binder, W/B of 0.3.

Table 3.1: 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

3.3.3 Production of concrete specimen

In order to achieve the required quality performance requirements of HPC according to the literature (Aitcin, 1998; Neville, 2012, Mehta & Monteiro, 2014, Olawuyi *et al*., 2021) the procedures used in the production of HPC have a significant effect. Concrete mixer in Building Laboratory Department, Federal University of Technology, Minna was used to for mixing of the materials of the HPC. The fine aggregate was first poured in to the mixer followed by the binders (PC, SF & MHA) after which the binders have been thoroughly mixed manually with each other and a homogeneous mixture achieved. After mixing for about 30 seconds, the granite and the pre-soaked pumice was added and the mixing continues for another 1 minute before adding about 75% of the mixing water. The remaining 25% of the mixing water was pre-mix with masterglenium (sky 504) and added to the mixture and the mixing continues for about 3 minutes until a homogenous mixture is obtained. Subsequent mixes followed the same procedure.

3.3.4 Compressive Strength

The examination of the HPC compressive strength in this study was carried out in accordance with British Standard (BS EN 12390-3: 2019). A total of 108 concrete cube specimen was produced using 100 mm cube and cured in ordinary water for 28, 56 and 90 days. The compressive strength test was conducted in Concrete Laboratory, Building Department using a compressive testing machine with a maximum loading capacity of 2000 kN with a model number (JYS-2000A Class 1). The setup for the test is shown below in Plate V. It was ensured that the face in touch with the loading platens is not the same as the cube's cast face. An average of triplicate gave the compressive strength for various curing ages as calculated using Equation 3.5.

$$\text{Compressive strength F} \quad = \quad \frac{P}{A} \tag{3.5}$$

Where; F is the compressive strength in N/mm^2

P is the maximum load at failure, in N

A is the cross-sectional area, in mm^2

3.3.5 Sulphate attack

Sulphate attack test was carried out in line with Mudashiru *et al*. (2021). A 100mm cube was used to produce 108 concrete specimen and cured in 5% $MgSO_4$ solution for 28, 56 and 90 days to determine the resistance in an aggressive sulphate environment as $MgSO_4$ is known to cause the most detrimental attack on concrete. The influence of sulphate attack on the HPCs was asses in terms of visual or physical assessments of the specimens, mass loss, residual compressive strength and strength loss factor.

3.3.6 Sorptivity

Sorptivity is defined as the ability of a material to absorb and transmit water through it via capillary suction. It is a durability test used to determine the rate of water absorption of concrete per unit time. The test was carried out in accordance to ASTM 1585 (2014) standards on cylindrical specimens. A total of 54 Ø100 x 50 mm cylindrical specimens were produced and cured in water for 28, 56 and 90 days. The specimen was removed from the curing tank and placed in the oven at a temperature of 50 ± 2 °C for 72 hrs. A suitable sealing material (aluminium tape) was used to seal the side and top of the specimen. The mass of the sealed specimens was measured and record as the initial mass for the water absorption calculations. Support device (preferably, steel rod) was placed at the bottom of the pan and the pan will be filled with tap water up to1 to 5 mm above the top of the support device. The specimen surface was placed on the support device and the time starts immediately. The time and date of the initial contact with water was recorded. The test was carried out at intervals of 1, 5, 10, 20, 30 minutes and every 1hr up to 6hrs.

For each mass determination, the specimens were removed from the pan at their respective intervals and the time was stopped while the mass is taken (the specimens was inverted so the wet surface are not in contact with the balance pan) after cleaning the surface water by means of a dampened paper towel. The absorption, I, is the change in mass divided by the product of the cross-sectional area of the test specimen and the density of water.

$$I = \frac{Mt}{A\ X\ D} \tag{3.7}$$

Where:

I = rate of absorption,

M_t = the change in specimen mass in grams, at the time t,

A = the exposed area of the specimen, in mm^2, and

D = the density of water in g/mm^3.

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION

4.1 Characterization of the constituent Materials

The strength and durability of the concrete is wholly dependent on characteristics of the constituent materials. Therefore, there is need for appropriate data collection with respect to physical and chemical properties of the concrete constituent materials.

4.1.1 Sieve analysis /particle size distribution (PSD) of aggregates

Figure 4.1 and table 4.1 presents the sieve analysis/ particle size distribution of the fine and coarse aggregates and the summary of sieve analysis of aggregates used for this study.

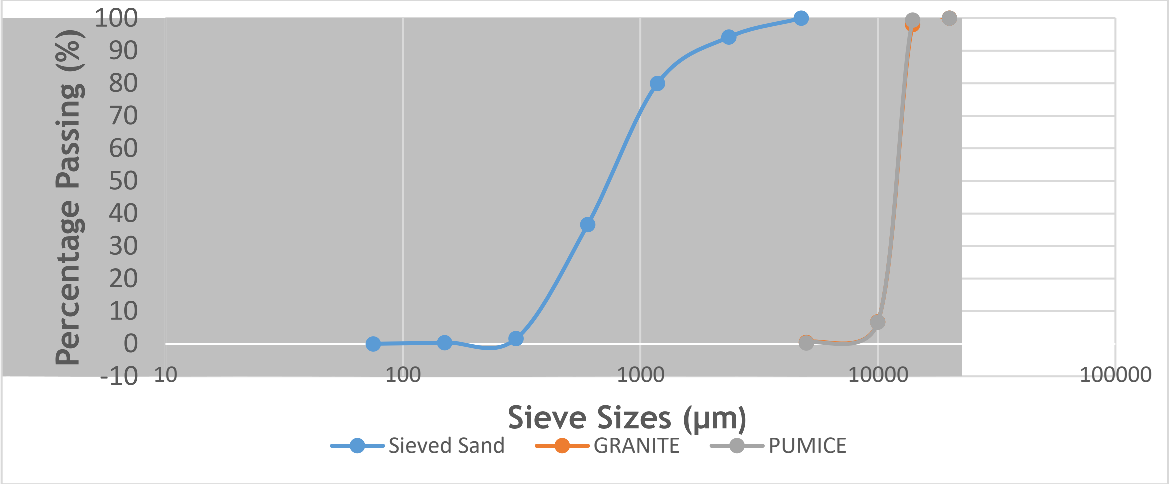

Figure 4.1: sieve analysis/particle size distribution of aggregates

Table 4.1: 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		

The results above show that the fine aggregate used for the study is in conformity to the medium sand classification of Shetty (2004) having a uniformity coefficient (C_u) of 2.39, coefficient of curvature (C_c) of 0.94 and fineness modulus (FM) of 2.88. The coarse aggregates used for the study have a coefficient of uniformity (C_u) of 1.32 and coefficient of curvature (C_c) of 0.92 and belong to the class of uniformly graded stone. Table 4.1 explain further that both the fine and coarse aggregates (i.e., crushed granite and pre-soaked pumice) used for the study are appropriate for the production of HPCs.

4.1.2 Particle size distribution (PSD) and BET specific surface area of binders

Illustration of particle size distribution and specific surface area of CEM II of grade 42.5N and MHA used for this research are shown in Figures 4.2, 4.3, 4.4, 4.5 and Table 4.2. From the result of BET model's distribution analysis, the particle size of the binders are not the same as shown in Figures 4.2 and 4.3. The differences in particle size of the binder may be as a result of production technique used (Mudashiru *et al*. 2021). The production of CEM II followed the ball milling process while in MHA production; a 5 µm sieve is attached in the grinding compartment of the machine, which makes it possible to have particle size close to 5 µm size. According to Mudashiru *et al*. (2021), calcination and grinding technique has effect on the microstructure and pore structure of cementitious material. From figure 4.2 and 4.3 it was observed that 90% (D_{90}) for CEM II and MHA particles are smaller than 4000 and 3800 µm respectively. D_{50} (median particle size) CEM II and MHA are 48.8 and 32.0 µm respectively. Also, the particle size below 10% (D_{10}) are within 4.90 and 158 µm for CEM II and MHA. As shown in table 4.2, the BET surface area for CEM II and MHA are 256.84 m^2/g and 118.94 m^2/g respectively. From the BET analysis, the pore diameter of the binders are 2.89 and 1.31 µm for CEM II and MHA and based on these values, CEM II has the finest particle size. Comparing the DA BET analysis, MHA sample has the least pore size diameter and thus it is classified as a macro-mesoporous material (Vieira *et al*; 2020) and the specific surface area is in accordance to the work of Mudashiru *et al*. (2021).

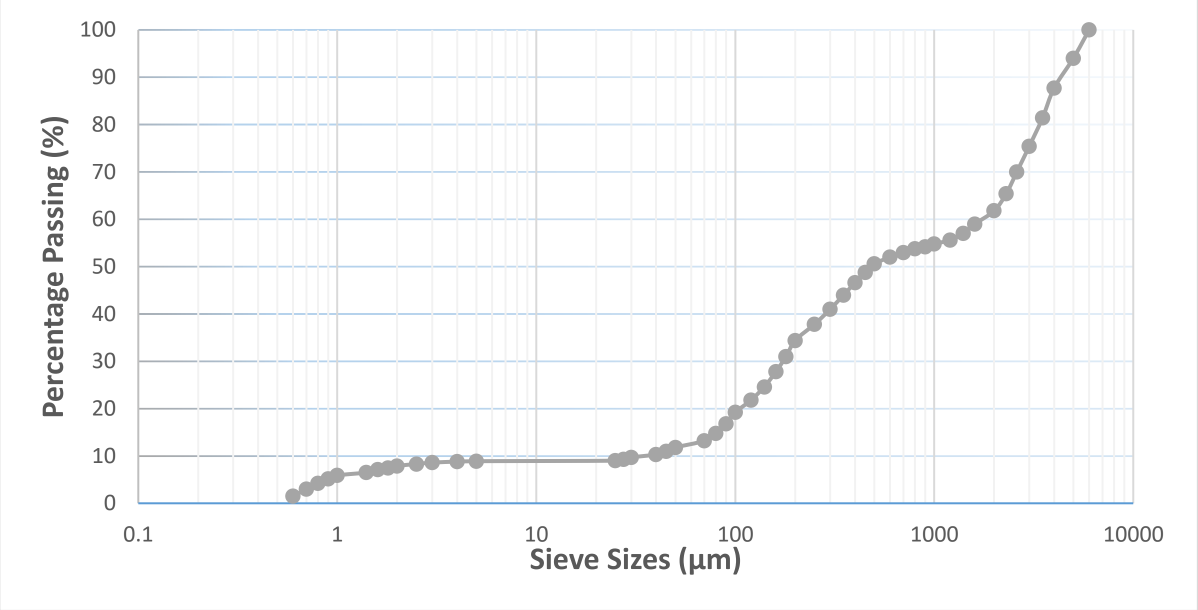

Figure 4.2: Particle size distribution of CEM II, 42.5N

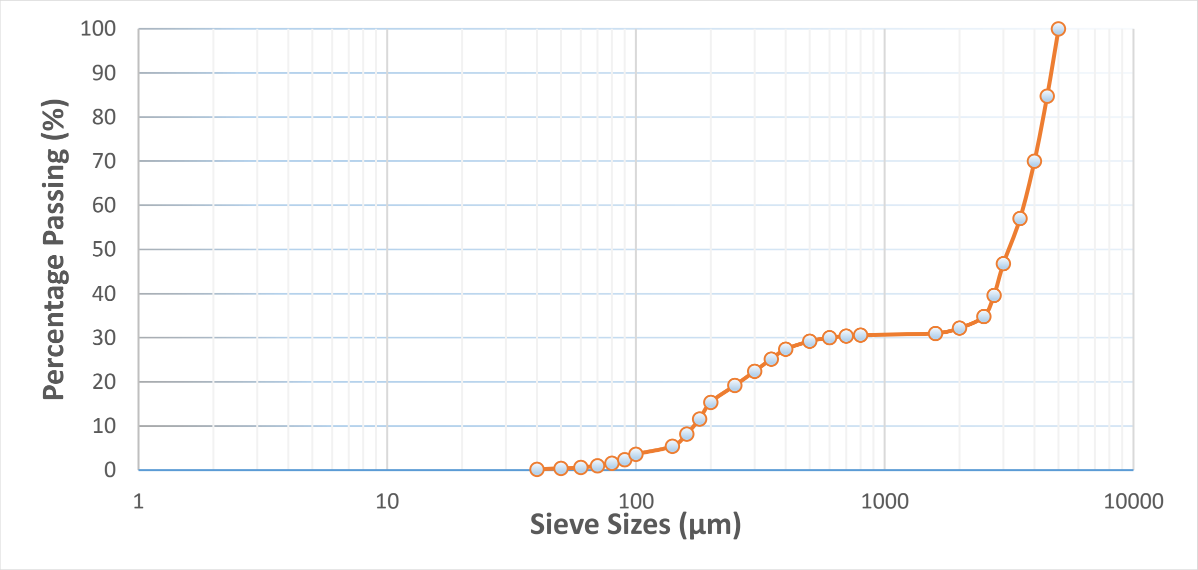

Figure 4.3: Particle size distribution of MHA

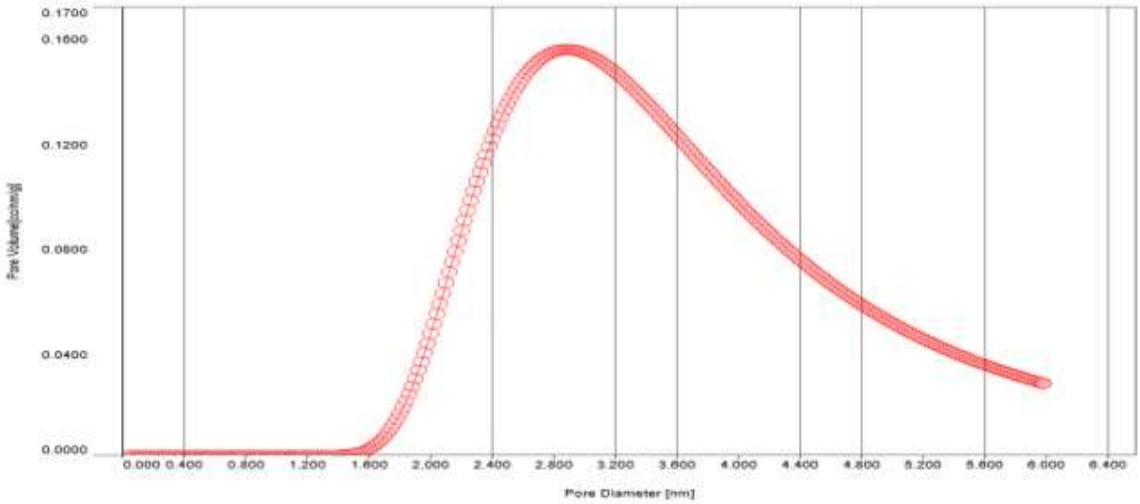

Figure 4.4: BET Distribution Analysis CEM II

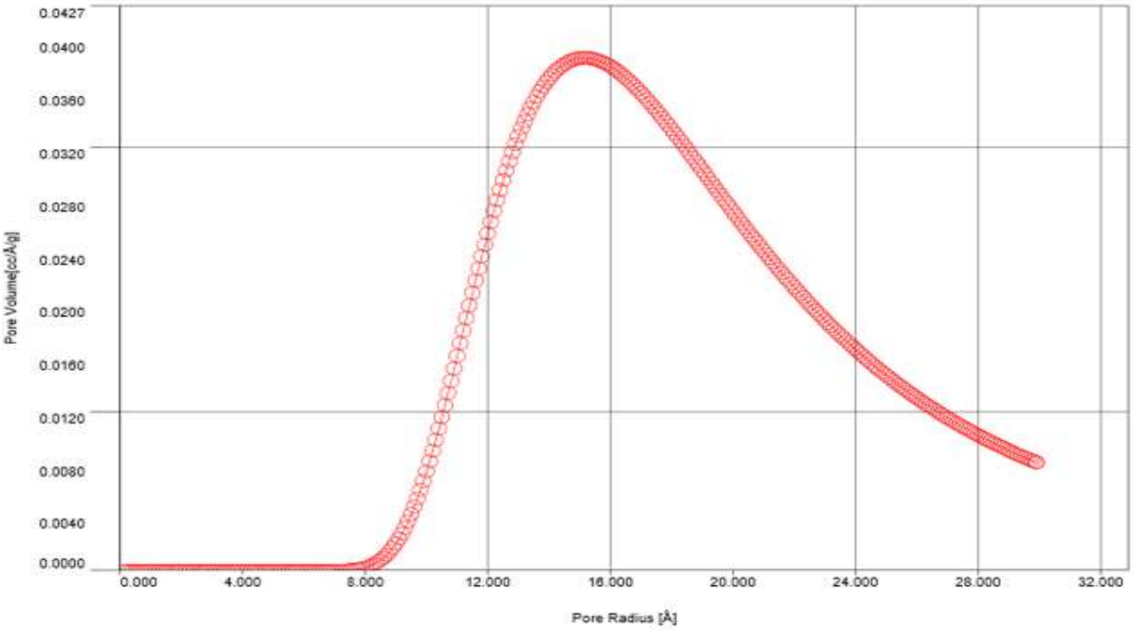

Figure 4.5: BET Distribution Analysis MHA

Table 4.2: Summary of Physical Properties of Binder

Binder	BET surface area (m^2/g)	Micropore Volume cm^3/g	D_{90} (µm)	D_{50} (µm)	D_{10} (µm)	Pore Diameter (µm)
CEM II	256.84	0.421	4000	48.8	4.90	2.92
MHA	118.94	0.688	3800	32.0	158	1.31

4.1.3 Specific gravity of Constituent Materials

Table 4.3 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

Table 4.3: Specific Gravity of PC, MHA, CCW and aggregates

Test	PC	MHA	SF	F/Agg.	Granite	Pumice
Empty cylinder (g)w_1	85	85	85	85	85	85
Empty cylinder + 1/3full of sample (g)w_2	107	96	100	119	119	112
Wt. of empty cylinder + 1/3full of sample + water full (g) w_3	169	161	162	176	176	165
Wt. of bottle + water only to full (g) w_4	154	154	154	154	154	154
SG $= w_2 - w_1 / (w_4 - w_1) - (w_3 - w_2)$	3.14	2.63	2.24	2.85	2.85	1.77

4.1.4 Chemical analysis of binder

The oxide composition of PC, MHA and SF used for this study was examined in accordance with the requirements of oxides composition specification for the production of HPC.

4.1.5 XRF characterization of binders

Table 4.4 present the result of XRF analysis of the binders (MHA, SF & PC) powder. The result shows that The MHA is a class N Pozzolan because the sum of the main oxides (SiO_2 + Al_2O + Fe_2O_3) gives 81.57% which is above 70% minimum limit as specified in Neville (2012). The SF major content is SiO_2 (94%) and this implies that it is a very strong and reactive Class F Pozzolan (Olawuyi *et al*; 2021). The sum of silica, alumina and ferric oxides (SiO_2+Al_2O_3+Fe_2O_3) for the SF (96.35%) is above the 70% (Olawuyi *et al*; 2021). The PC on the other hand is major content is calcium oxide (CaO – 60.35%). This conforms to oxides composition for CEM II Portland cement found in literature (Neville, 2012; Mehta and Monteiro, 2014).

Table 4.4: Oxide Composition of Binder Constituents

Oxides	MHA (%)	SF (%)	CEM II (%)
SiO_2	77.04	96.35	25.64
Al_2O_3	2.66	0.47	5.24
Fe_2O_3	4.87	0.28	7.15
CaO	1.55	0.05	60.35
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	2.86	1.52	0.67
$SiO_2 + Al_2O_3 + Fe_2O_3$	81.57	96.35	38.03

4.2 Fresh Properties

The setting time and the workability of the mix used in HPC production were explored to know the flow of the slump and the effect of the SCM utilized on the setting time of blended mix.

4.2.1 Consistency, setting time, soundness of binder (PC, MHA& SF) and workability

Table 4.5 present the results of standard consistency, setting time and soundness tests for the binary binders (PC/SF and PC/MHA) at constant 7.5% SF with 92.5%PC and varying of MHA (2.5, 5, 7.5, 10 and 15%) with PC (97.5, 95, 92.5, 90 and 85%). It was observed that the water demand for M1, M2, M3, M4 and M5 was higher than M0. This could be as a result of hygroscopic nature of Portland cement (Olawuyi *et al*; 2021). Also, it was observed that the effect of the chemical admixture (superplasticizer) aid the setting time of the binary binder with M0 having initial and final setting time of 45 and 135 minutes while M1, M2, M3, M4 and M5 have initial and final setting time of 280 305, 300 330, 310 400, 340 450, and 360 510 minutes respectively. Also, increase in the expansion value of all the mixes obtained from soundness test was observed to be in the range of 11 mm to 12 mm but the values are within the limits (Mudashiru *et al*. 2021).

Table 4.5: consistency, setting time and soundness test of HPCs

Test	Consistency					Setting time		Soundness (mm)		
Mix ID	Binder wt (g)			Water (%)	Penetration depth (mm)	Initial (mins)	Final (mins)	Before boiling (L_1)	After boiling (L_2)	Expansion (L_2-L_1)
	PC	SF	MHA							
M0	462.5	37.5		105	28	45	135	10.5	11	0.5
M1	487.5		12.5	110	26	280	305	11	12	1.0
M2	475		25	112	30	300	330	10.5	11	0.5
M3	462.5		37.5	116	32	310	400	10.5	11	0.5
M4	450		50	117	36	340	450	11	11.5	0.5
M5	425		75	120	38	360	510	11	12	1.0

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

4.2.2 Slump flow test

Workability test for each of the HPC mixture with 5% pre-soaked pumice and 0.2% SAP was examined using slump flow test as described in BS EN 12350-5 (2009) before the production of the specimens. From figure 4.6, the slump flow value for the controls M0a (0.2% SAP content) and M0b (5% pre-soaked pumice content) and M1, M2, M3, M4 and M5 (5% of pre-soaked pumice) are 510, 490, 505, 520, 530, 545 and 560 mm respectively. From the result, the flow values of all the HPCs with MHA and pre-soaked pumice (IC-agent) has a higher values of 505, 520, 530, 545 and 560 mm when compared with the HPC having SF and same content of pre-soaked pumice having a value of 490mm. Also, from the result it was observed that HPC with 7.5% of SF and 0.2% SAP content has a higher slump flow value (510mm) when compared with HPC having 7.7% of SF and 5% of pre-soaked pumice (490mm). It was also observed that the slump flow values for all the HPC mixtures are in the range of 460-600 mm (Mudashiru *et al*. 2021).

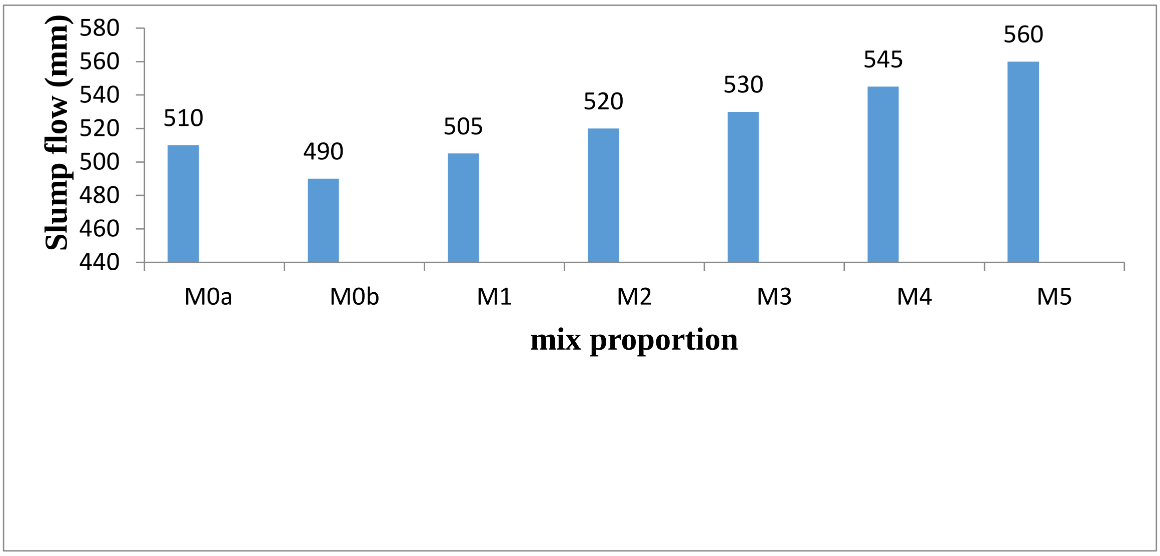

Figure 4.6: Workability of HPCs
M0=92.5%PC+7.5%SF; **M1**=97.5%PC+2.5%MHA; **M2**=95%PC+5%MHA; **M3**=92.5%PC+7.5%MHA; **M4**=90%PC+10%MHA; **M5**=85%PC+15%MHA **a**=0.2% SAP **b**=5% pre-soaked pumice;

4.3 Hardened Properties of the HPCs

The compressive strength, sulphate attack, and sorptivity test of the various mix HPCs are discussed below

4.3.1 Compressive strength of the HPCs

Figure 4.7 present the compressive strength of the HPCs cured in water at 28 days of age. From the figure, the values obtained for the controls (i.e M0a and M0b) are; 59.86 and are 58.62 N/mm^2 while the values for M1, M2, M3, M4 and M5 are; 53.58, 52.04, 50.21, 47, 73 and 45.90 N/mm^2.

Figure 4.8 show the result of compressive strength of internally cured HPC cured in water at 56 days of age. From the figure, the values for the controls M0a and M0b are; 73.18 and 72.15 N/mm^2 while for M1, M2, M3, M4 and M5 are; 66.78, 64.54, 61.41, 59.75 and 57.60N/mm^2

Figure 4.9 present the values of compressive strength of internally cured HPC at 90 days. From the figure, the values for M0a and M0b (controls) are; 75.23 and 74.08 N/mm^2 while that for M1, M2, M3, M4 and M5 are; 69.13, 65.92, 63.02, 60.31 and 58.82 N/mm^2.

From the results, it also shows that 97.5PC+ 2.5MHA (M1) internally cured with pre-soaked pumice has a close compressive strength values of 53.58, 66.78 and 69.13 N/mm^2 when compared with the control M0b (92.5PC+7.5SF) having a compressive strength value of 58.62, 72.15 and 74.08 N/mm^2 internally cured with pre-soaked pumice at 28, 56 and 90 days of age respectively. also from the result, M0b (92.5PC+7.5SF) internally cured with 5% pre-soaked pumice content has a lower compressive values of 58.62, 72.15 and 74.08 N/mm^2 when compared with M0a (92.5PC+7.5SF) having a compressive strength values of 59.86, 66.78 and 75.23 N/mm^2 internally cured with 0.2% SAP content at 28, 56 and 90 days of age respectively. From the figures it also reveals that from 28 to 56 days of curing, 20% increase in compressive strength was observed while from 56 to 90 days of curing 3% increase in compressive strength was observe but the increase in compressive strength is at a decreasing rate. The reason for the continual increase in strength with ages was due to continual hydration of the binders (Olawuyi *et al.,* 2020).

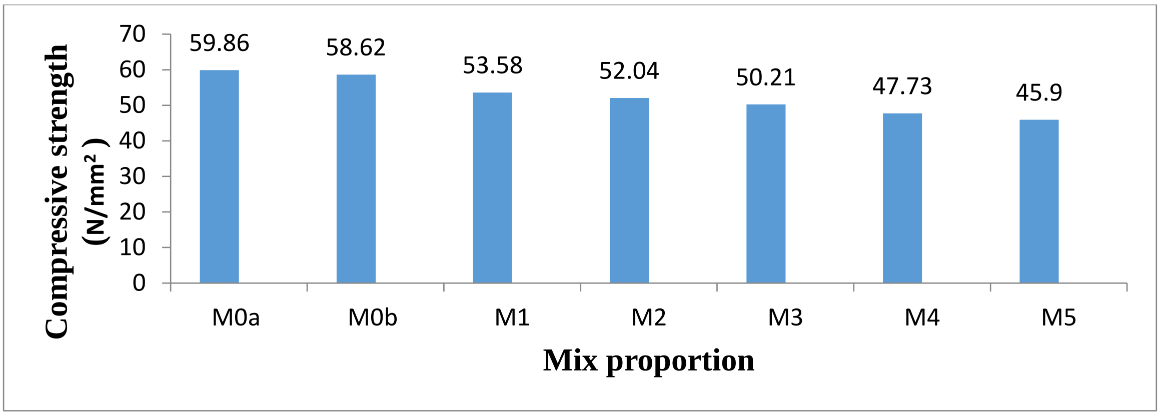

Figure 4.7: compressive strength of the HPCs at 28 days
M0=92.5%PC+7.5%SF; **M1**=97.5%PC+2.5%MHA; **M2=**95%PC+5%MHA; **M3**=92.5%PC+7.5%MHA; **M4**=90%PC+10%MHA; **M5**=85%PC+15%MHA
a=0.2% SAP 5%; **b=** pre-soaked pumice

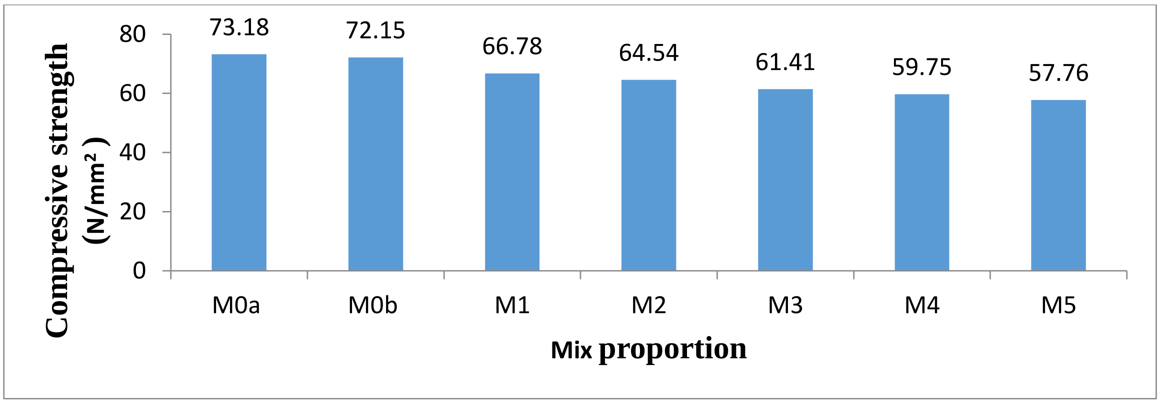

Figure 4.8: compressive strength of the HPCs at 56 days
M0=92.5%PC+7.5%SF; **M1**=97.5%PC+2.5%MHA; **M2=**95%PC+5%MHA; **M3**=92.5%PC+7.5%MHA; **M4**=90%PC+10%MHA; **M5**=85%PC+15%MHA
a=0.2% SAP 5%; **b=** pre-soaked pumice

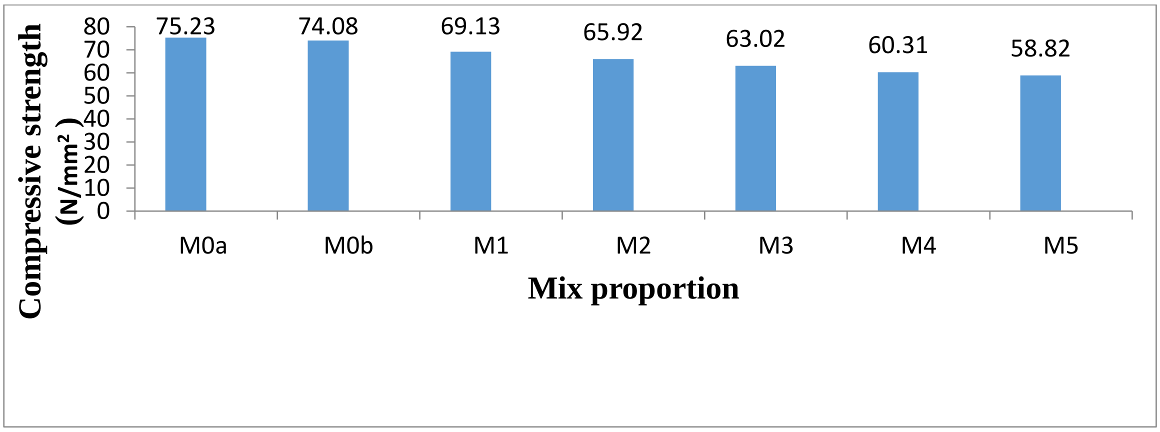

Figure 4.9: compressive strength of the HPCs at 90 days
M0=92.5%PC+7.5%SF; **M1**=97.5%PC+2.5%MHA; **M2=**95%PC+5%MHA; **M3**=92.5%PC+7.5%MHA; **M4**=90%PC+10%MHA; **M5**=85%PC+15%MHA
a=0.2% SAP 5%; **b=** pre-soaked pumice

4.3.2 Influence of sulphate attack on the compressive strength of the HPCs

Physical or 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.

4.3.2.1 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 4.6. 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 fines 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 (PC + SF, PC + MHA). 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

Table 4.6: Physical characteristics of internally cured f 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

4.3.2.2 Mass loss

Figure 4.10, 4.11 and 4.12 provide an illustration of the mass loss of various HPCs mix cured in $MgSO_4$ at 28, 56 and 90 days of age.

From figure 4.10, the mass loss values obtained for the controls (i.e M0a and M0b) at 28 days in $MgSO_4$ solution are: 1.89% and 1.49% while the values for M1, M2, M3, M4 and M5 are; 1.51%, 1.56%, 2.16%, 2.32% and 2.84%.

From figure 4.11, 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 4.12, 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.

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-absorbing of the sulphate particles 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 presoaked 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 presoaked 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 presoaked 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 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)

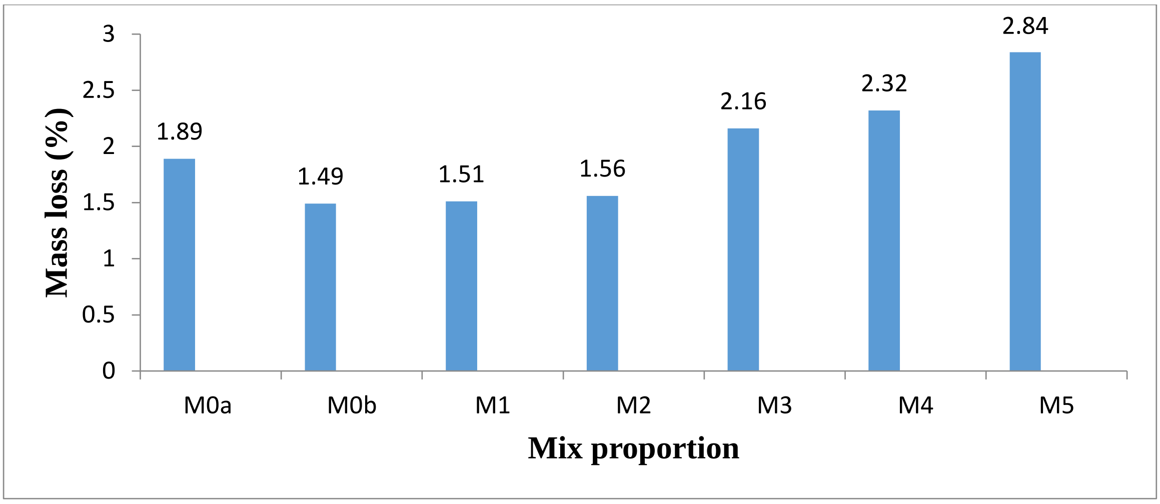

Figure 4.10: Mass loss of internally cured HPCs subjected to $MgSO_4$ at 28 days
M0=92.5%PC+7.5%SF; **M1**=97.5%PC+2.5%MHA; **M2**=95%PC+5%MHA; **M3**=92.5%PC+7.5%MHA; **M4**=90%PC+10%MHA; **M5**=85%PC+15%MHA **a**=0.2% SAP; **b**=5% pre-soaked pumice

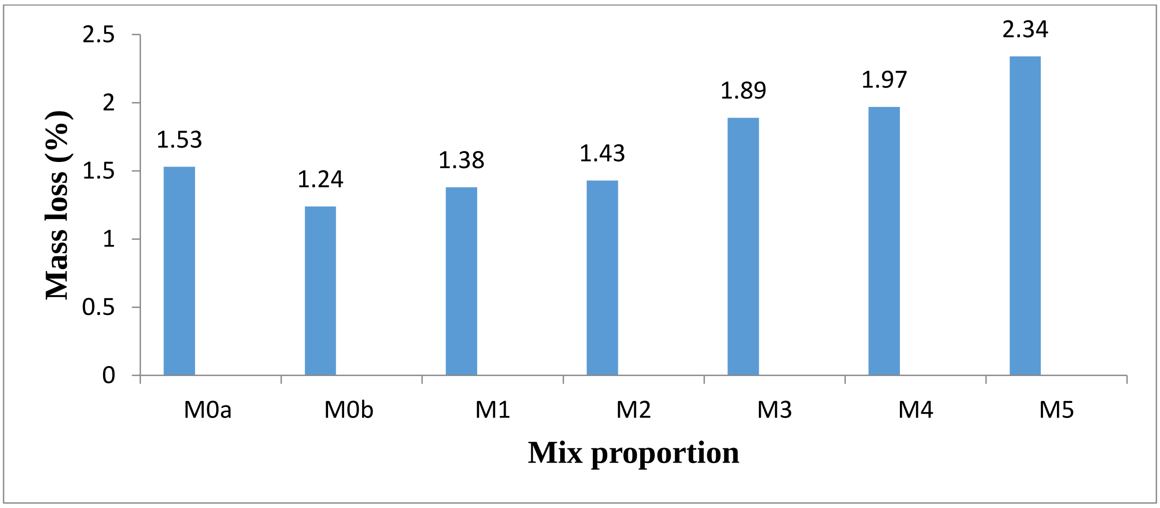

Figure 4.11: Mass loss of internally cured HPCs subjected to $MgSO_4$ at 56 days
M0=92.5%PC+7.5%SF; **M1**=97.5%PC+2.5%MHA; **M2**=95%PC+5%MHA; **M3**=92.5%PC+7.5%MHA; **M4**=90%PC+10%MHA; **M5**=85%PC+15%MHA **a**=0.2% SAP; **b**=5% pre-soaked pumice

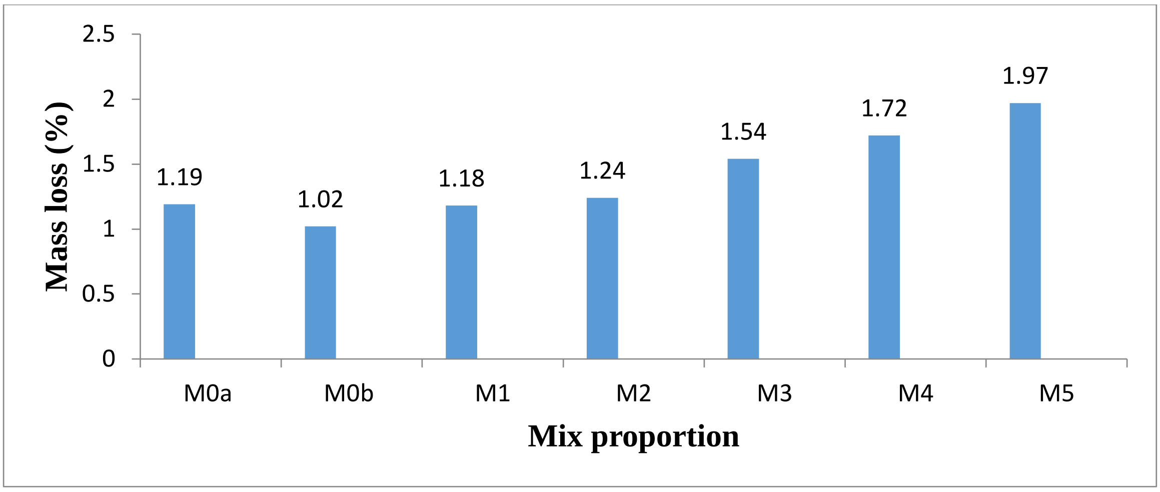

Figure 4.12: Mass loss of internally cured HPCs subjected to $MgSO_4$ at 90 days
M0=92.5%PC+7.5%SF; **M1**=97.5%PC+2.5%MHA; **M2**=95%PC+5%MHA; **M3**=92.5%PC+7.5%MHA; **M4**=90%PC+10%MHA; **M5**=85%PC+15%MHA **a**=0.2% SAP; **b**=5% pre-soaked pumice

4.3.2.3 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 4.13, 4.14 and 4.15.

From figure 4.13, the strength loss factors for the controls (i.e 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 4.14 for the controls (i.e 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 4.15, for the controls (i.e 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%.

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 presoaked 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.

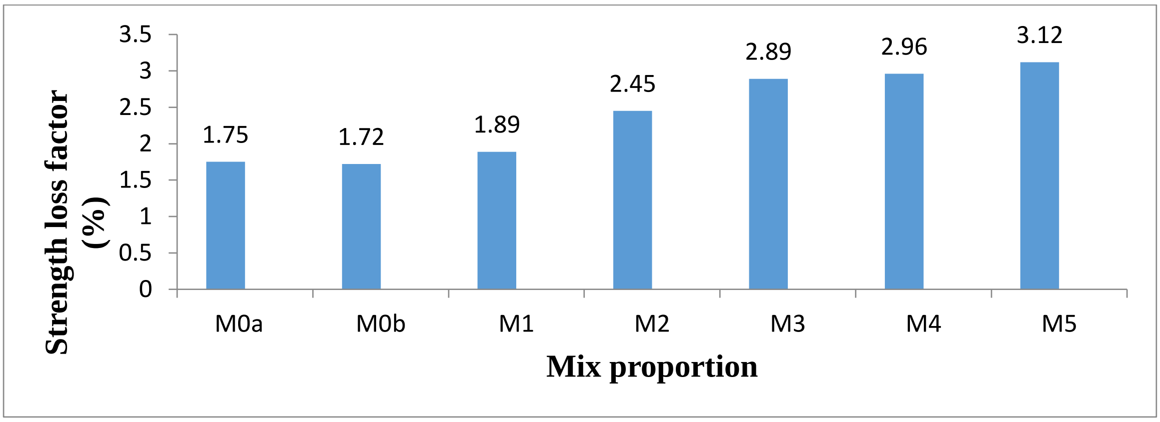

Figure 4.13: Strength loss factor of internally cured HPCs subjected to $MgSO_4$ at 28 days
M0=92.5%PC+7.5%SF; **M1**=97.5%PC+2.5%MHA; **M2**=95%PC+5%MHA;
M3=92.5%PC+7.5%MHA; **M4**=90%PC+10%MHA; **M5**=85%PC+15%MHA
a=5% pre-soaked pumice; **b**=0.2% SAP

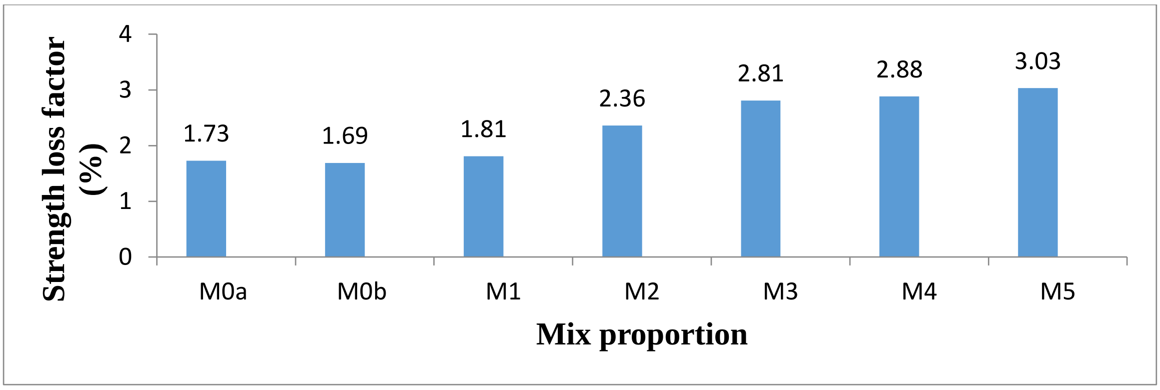

Figure 4.14: Strength loss factor of internally cured HPCs subjected to $MgSO_4$ at 56 days
M0=92.5%PC+7.5%SF; **M1**=97.5%PC+2.5%MHA; **M2**=95%PC+5%MHA;
M3=92.5%PC+7.5%MHA; **M4**=90%PC+10%MHA; **M5**=85%PC+15%MHA
a=5% pre-soaked pumice; **b**=0.2% SAP

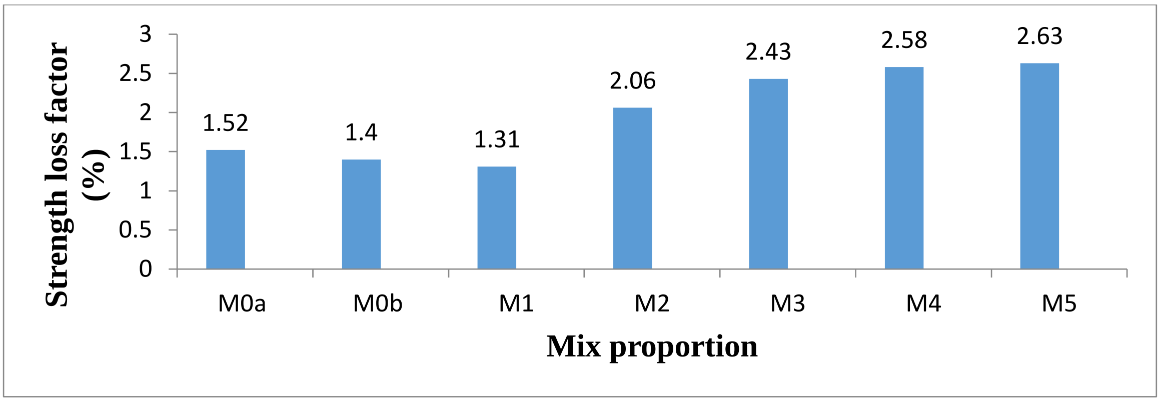

Figure 4.15: Strength loss factor of internally cured HPCs subjected to $MgSO_4$ at 90 days
M0=92.5%PC+7.5%SF; **M1**=97.5%PC+2.5%MHA; **M2**=95%PC+5%MHA; **M3**=92.5%PC+7.5%MHA; **M4**=90%PC+10%MHA; **M5**=85%PC+15%MHA
a=5% pre-soaked pumice; **b**=0.2% SAP

4.3.3 Sorptivity

Figure 4.16, 4.17 and 4.18 illustrate the results of sorptivity versus the square root of time for all the internally cured HPC at 28, 56 and 90 days. From the figures, it was observed that all the mixes shows similar sorptivity at square root of 8, 14 and 24 seconds but from 35 to 147 sec square root, the sorptivity for all the mixes excluding that of 7.5%SF, 2.5MHA, 5%MHA and 7.5MHA show is higher for all the ages. From the figures, the sorptivity of MHA is more comparable to SF. This might be because of the difference in density. From the figures, it can also be seen that increase in MHA content lead to increase sorptivity for all the ages with 2.5%MHA being less than 15%MHA. However, with increase in curing ages, sorptivity for all the HPC decreases. This might be due to a reduction in the void and capillary porosity within the concrete as hydration continues (Olawuyi 2017).

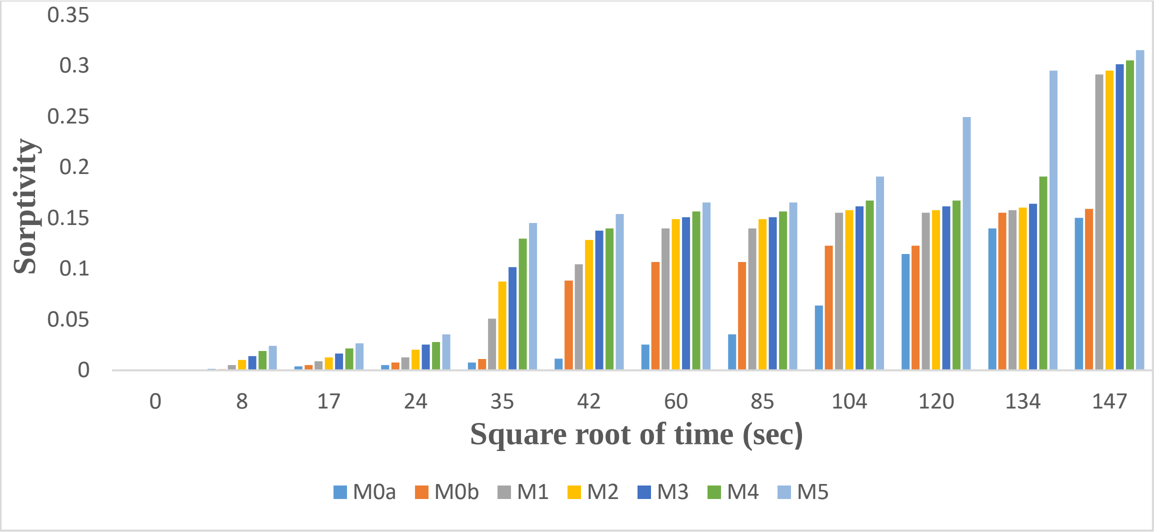

Figure 4.16: Sorptivity vs square root of time for internally cured HPCs at 28 days

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% presoaked pumice

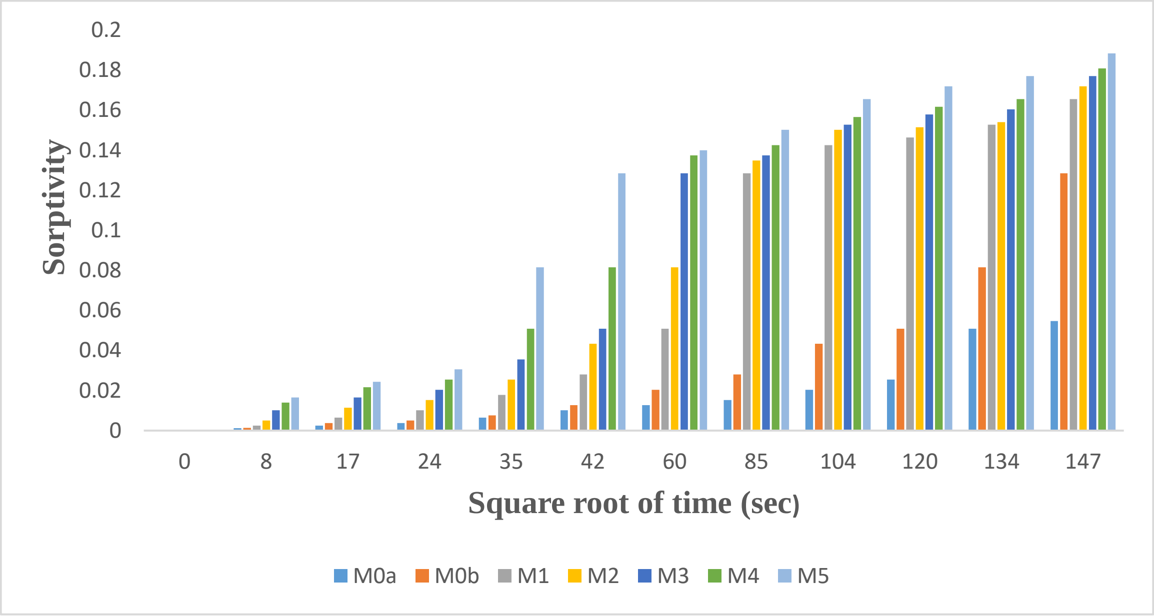

Figure 4.17 Sorptivity vs square root of time for internally cured HPCs at 56 days

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% presoaked pumice

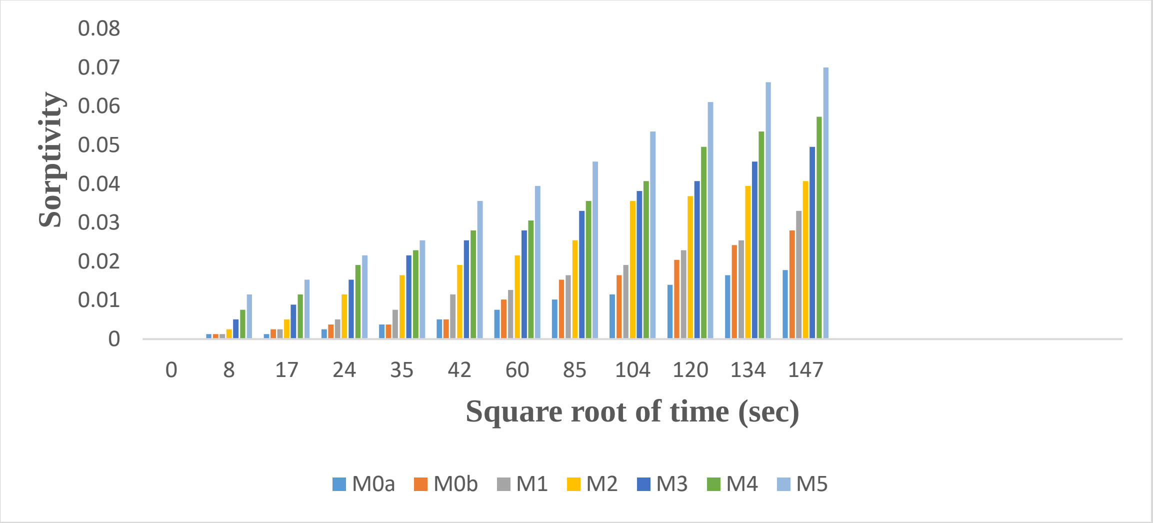

Figure 4.18: Sorptivity vs square root of time for internally cured HPCs at 90days

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%MH
a= 0.2% SAP; **b=** 5% presoaked pumice

4.4 Summary of findings

The inferences deduced from the study can be summarized below

i. The SF and MHA used for the study were a good Class F and N Pozzolan (Olawuyi *et al*; 2021).

ii. 2.5%MHA HPC cured internally with presoaked pumice has a close value compressive strength when compared with 7.5%SF HPC cured internally with presoaked pumice.

iii. The compressive strength of HPCs internally cured with 0.2% SAP is more than the compressive strength of HPCs internally cured with 5% presoaked pumice.

iv. The compressive strength of HPC decreases as the MHA content increases for HPC internally cured with 5% presoaked pumice with 2.5%MHA having the highest value of compressive strength of 53.58Mpa.

v. A whitish magnesium sulphate crystal, 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 precipitation was noticed.

vi. 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.

vii. 2.5%MHA performed best when compare with the control (i.e 7.5%SF) in resisting $MgSO_4$ attack and it exhibit the highest residual strength. Hence it has better resistance to chemical attack.

viii. 0.2%SAP incorporated IC-agent has a higher residual strength (smaller strength loss) when compared with 5%pre-soaked pumice incorporated IC-agent and thus has a higher resistance to chemical attack.

ix. The IC-agents incorporated increases the rate of water absorption (sorptivity) of the HPCs but decreases as the curing age increases.

x. MHA content of 2.5% and 5% pre-soaked pumice are recommended for use as Nigeria local SCM and IC-agent in HPC.

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The studies explored the effectiveness of SCM and IC-agent (MHA and pre-soaked pumice) in comparism with SF and SAP in HPC with administration of Masterglenium sky 504 superplasticizers. From the study it was seen that the IC-agents (SAP and pre-soaked pumice) has effect on the strength and durability properties of the HPC but the impact on the strength and durability properties as seen from the result presented is not drastically detrimental. 2.5%MHA perform best when compared with the control (7.5%SF) than any other mixes (5%MHA, 7.5%MHA, 10%MHA and 15%MHA). 0.2% SAP content perform more than 5% pre-soaked pumice.

4.5 Recommendation

The following recommendation is made from the study:

i. MHA content of 2.5% and 5% pre-soaked pumice are recommended for use as Nigeria local SCM and IC-agent in HPC because they performed well when compared with the control (7.5% SF and 0.2%SAP).

4.6 Areas for further studies

i. Further exploration of the effects of sulphate attack on the strength and durability properties of MHA based HPC internally cured with presoaked pumice at a period longer than 90 days of age.

ii. Further processing of MHA use in HPC should be done so as to give a finer particle to enhance the reactions and hydration process.

4.7 Contribution to knowledge

I. The use of MHA SCM in HPC production will help addresses some environmental problems associated with CO_2 emission from Portland cement production process and as a means of turning agricultural wastes to useful products.

II. The use of the investigated Nigeria local SCM (MHA) and IC-agent (pre-soaked pumice) will provide awareness for construction industry stakeholders on the effectiveness of using these new materials for construction of high-rise-building and other heavy civil engineering works.

III. The adoption and utilization of MHA as SCM and pre-soaked pumice as IC-agent in HPC will provide an innovative way for built environment professional to take decision that will increase effective usage of space and lower cost of construction for concrete elements.

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APPENDIX

A: RESULTS OF SORPTIVITY

A1: Sorptivity of internally cured HPCs at 28 days

Time (sec)	0	60	300	600	1200	1800	3600	7200	10800	14400	18000	21600
$\sqrt{Time}$	0	8	17	24	35	42	60	85	104	120	134	147
						Mass (g)						
C0a	1190.10	1190.11	1190.14	1190.18	1190.24	1190.33	1190.53	1190.81	1191.31	1192.21	1193.31	1194.49
C0b	1188.46	1188.47	1188.51	1188.57	1188.65	1188.04	1188.75	1189.59	1190.69	1191.89	1193.11	1194.36
C1	1154.42	1154.46	1154.53	1154.63	1155.03	1155.63	1156.45	1157.55	1158.73	1159.95	1161.19	1163.48
C2	1152.01	1153.31	1153.41	1153.57	1154.26	1155.14	1156.15	1157.32	1158.52	1159.76	1161.02	1163.34
C3	1151.82	1151.93	1152.06	1152.26	1153.06	1154.03	1155.11	1156.30	1157.54	1158.81	1160.10	1162.47
C4	1139.08	1139.23	1139.40	1139.62	1140.64	1141.72	1142.82	1144.05	1145.33	1146.65	1148.15	1150.55
C5	1138.76	1138.95	1139.16	1139.44	1140.52	1141.66	1142.87	1144.17	1145.67	1147.63	1149.95	1152.43
						∆Mass(g)						
C0a	0.00	0.01	0.03	0.04	0.06	0.09	0.20	0.28	0.50	0.90	1.10	1.18
C0b	0.00	0.01	0.04	0.06	0.08	0.10	0.71	0.84	1.10	1.20	1.22	1.25
C1	0.00	0.04	0.07	0.10	0.40	0.60	0.82	1.10	1.18	1.22	1.24	2.29
C2	0.00	0.08	0.10	0.16	0.69	0.88	1.01	1.17	1.20	1.24	1.26	2.32
C3	0.00	0.11	0.13	0.20	0.80	0.97	1.08	1.19	1.24	1.27	1.29	2.37
C4	0.00	0.15	0.17	0.22	1.02	1.08	1.10	1.23	1.28	1.32	1.50	2.40
C5	0.00	0.19	0.21	0.28	1.08	1.14	1.21	1.30	1.50	1.96	2.32	2.48
						Sorptivity						
C0a	0.00	0.0013	0.0038	0.0051	0.0076	0.0115	0.0255	0.0356	0.0637	0.1146	0.1400	0.1502
C0b	0.00	0.0012	0.0051	0.0076	0.0120	0.0127	0.0883	0.1069	0.1400	0.1228	0.1553	0.1590
C1	0.00	0.0051	0.0089	0.0127	0.0509	0.0764	0.1044	0.1400	0.1502	0.1553	0.1579	0.2915
C2	0.00	0.0102	0.0127	0.0204	0.0874	0.1120	0.1286	0.1489	0.1558	0.1579	0.1604	0.2953
C3	0.00	0.0140	0.0165	0.0255	0.1018	0.1235	0.1375	0.1508	0.1579	0.1617	0.1642	0.3017
C4	0.00	0.0191	0.0216	0.0280	0.1299	0.1375	0.1400	0.1566	0.1630	0.1674	0.1910	0.3055
C5	0.00	0.0242	0.0267	0.0356	0.1375	0.1451	0.1540	0.1655	0.1910	0.2495	0.2953	0.3157

A2: Sorptivity of internally cured HPCs at 56days

Time (sec)	0	60	300	600	1200	1800	3600	7200	10800	14400	18000	21600
$\sqrt{Time}$	0	8	17	24	35	42	60	85	104	120	134	147
						Mass (g)						
C0a	1180.10	1180.11	1180.13	1180.16	1180.21	1180.29	1180.39	1180.51	1180.67	1180.87	1181.27	1181.70
C0b	1178.20	1178.21	1178.24	1178.28	1178.34	1178.44	1178.60	1178.82	1179.16	1179.56	1180.20	1181.21
C1	1177.32	1177.34	1177.39	1177.47	1177.61	1177.83	1178.23	1179.24	1180.36	1181.51	1182.71	1184.01
C2	1162.01	1162.05	1162.14	1162.26	1162.46	1162.80	1163.44	1164.50	1165.68	1166.87	1168.04	1169.43
C3	1161.80	1161.88	1162.01	1162.17	1162.45	1162.85	1163.86	1164.94	1166.14	1167.38	1168.64	1170.03
C4	1159.08	1159.19	1159.36	1159.56	1159.96	1160.60	1161.68	1162.80	1164.03	1165.30	1166.60	1168.02
C5	1148.76	1148.89	1149.08	1149.32	1149.96	1150.97	1152.07	1153.25	1154.55	1155.90	1157.29	1158.77
						∆Mass(g)						
C0a	0.00	0.01	0.02	0.03	0.05	0.08	0.10	0.12	0.16	0.20	0.40	0.43
C0b	0.00	0.01	0.03	0.04	0.06	0.10	0.16	0.22	0.34	0.40	0.64	1.01
C1	0.00	0.02	0.05	0.08	0.14	0.22	0.40	1.01	1.12	1.15	1.20	1.30
C2	0.00	0.04	0.09	0.12	0.20	0.34	0.64	1.06	1.18	1.19	1.21	1.35
C3	0.00	0.08	0.13	0.16	0.28	0.40	1.01	1.08	1.20	1.24	1.26	1.39
C4	0.00	0.11	0.17	0.20	0.40	0.64	1.08	1.12	1.23	1.27	1.30	1.42
C5	0.00	0.13	0.19	0.24	0.64	1.01	1.10	1.18	1.30	1.35	1.39	1.48
						Sorptivity						
C0a	0.00	0.0012	0.0025	0.0038	0.0064	0.0102	0.0127	0.0153	0.0204	0.0255	0.0509	0.0547
C0b	0.00	0.0013	0.0038	0.0051	0.0076	0.0127	0.0204	0.0280	0.0433	0.0509	0.0815	0.1285
C1	0.00	0.0025	0.0064	0.0102	0.0178	0.0280	0.0509	0.1285	0.1426	0.1464	0.1528	0.1655
C2	0.00	0.0051	0.0115	0.0153	0.0255	0.0433	0.0815	0.1349	0.1502	0.1515	0.1540	0.1719
C3	0.00	0.0102	0.0165	0.0204	0.0356	0.0509	0.1285	0.1375	0.1528	0.1579	0.1604	0.1770
C4	0.00	0.0140	0.0216	0.0255	0.0509	0.0815	0.1375	0.1426	0.1566	0.1617	0.1655	0.1808
C5	0.00	0.0165	0.0243	0.0306	0.0815	0.1285	0.1400	0.1502	0.1655	0.1719	0.1770	0.1884

A3: Sorptivity of internally cured HPCs 90days

Time (sec)	0	60	300	600	1200	1800	3600	7200	10800	14400	18000	21600
$\sqrt{Time}$	0	8	17	24	35	42	60	85	104	120	134	147
						Mass (g)						
C0a	1175.18	1175.19	1175.20	1175.22	1175.25	1175.29	1175.35	1175.43	1175.52	1175.63	1175.76	1175.90
C0b	1179.10	1179.11	1179.13	1179.16	1179.19	1179.23	1179.31	1179.43	1179.56	1179.72	1179.91	1180.13
C1	1170.12	1170.13	1170.15	1170.19	1170.25	1170.34	1170.44	1170.57	1170.72	1170.90	1171.10	1171.36
C2	1172.41	1172.43	1172.47	1172.56	1172.69	1172.84	1173.01	1173.21	1173.49	1173.78	1174.09	1174.41
C3	1171.50	1171.54	1171.61	1171.73	1171.90	1172.10	1172.32	1172.58	1172.88	1173.20	1173.56	1173.95
C4	1179.78	1179.84	1179.93	1180.08	1180.26	1180.48	1180.72	1181.00	1181.32	1181.71	1182.13	1182.58
C5	1176.81	1176.90	1177.02	1177.19	1177.39	1177.67	1177.98	1178.34	1178.76	1179.24	1179.76	1180.31
						ΔMass(g)						
C0a	0.00	0.01	0.01	0.02	0.03	0.04	0.06	0.08	0.09	0.11	0.13	0.14
C0b	0.00	0.01	0.02	0.03	0.03	0.04	0.08	0.12	0.13	0.16	0.19	0.22
C1	0.00	0.01	0.02	0.04	0.06	0.09	0.10	0.13	0.15	0.18	0.20	0.26
C2	0.00	0.02	0.04	0.09	0.13	0.15	0.17	0.20	0.28	0.29	0.31	0.32
C3	0.00	0.04	0.07	0.12	0.17	0.20	0.22	0.26	0.30	0.32	0.36	0.39
C4	0.00	0.06	0.09	0.15	0.18	0.22	0.24	0.28	0.32	0.39	0.42	0.45
C5	0.00	0.09	0.12	0.17	0.20	0.28	0.31	0.36	0.42	0.48	0.52	0.55
						Sorptivity						
C0a	0.00	0.0013	0.0013	0.0025	0.0038	0.0051	0.0076	0.0102	0.0115	0.0140	0.0165	0.0178
C0b	0.00	0.0013	0.0025	0.0038	0.0038	0.0051	0.0102	0.0153	0.0165	0.0204	0.0242	0.0280
C1	0.00	0.0013	0.0025	0.0051	0.0076	0.0115	0.0127	0.0165	0.0191	0.0229	0.0255	0.0331
C2	0.00	0.0025	0.0051	0.0115	0.0165	0.0191	0.0216	0.0255	0.0356	0.0369	0.0395	0.0407
C3	0.00	0.0051	0.0089	0.0153	0.0216	0.0255	0.0280	0.0331	0.0382	0.0407	0.0458	0.0496
C4	0.00	0.0076	0.0115	0.0191	0.0229	0.0280	0.0306	0.0356	0.0407	0.0496	0.0535	0.0573
C5	0.00	0.0115	0.0153	0.0216	0.0255	0.0356	0.0395	0.0458	0.0535	0.0611	0.0662	0.0700

B: COMPRESSIVE AND RESIDUAL STRENGTH

With 5% presoaked pumice					
Mix ID	**CS (water)**	**CS ($MgSO_4$)**	**∆ CS**	**Loss (%)**	**Residual CS**
compressive and residual strength at 28 days					
C0a	59.86	58.83	1.03	1.75	98.25
C0b	58.62	57.63	0.99	1.72	98.28
C1	53.58	52.59	0.99	1.89	98.11
C2	52.04	50.80	1.24	2.45	97.55
C3	50.21	48.80	1.41	2.89	97.11
C4	47.73	46.36	1.37	2.96	97.04
C5	45.90	44.51	1.39	3.12	96.88
compressive and residual strength at 56 days					
C0a	73.18	71.94	1.24	1.73	98.27
C0b	72.15	70.95	1.20	1.69	98.31
C1	66.78	65.59	1.19	1.81	98.19
C2	64.54	63.05	1.49	2.36	97.64
C3	61.41	59.73	1.68	2.81	97.18
C4	59.75	58.08	1.67	2.88	97.12
C5	57.76	56.06	1.70	3.03	96.97
compressive and residual strength at 90 days					
C0a	75.23	74.10	1.13	1.52	98.48
C0b	74.08	73.06	1.02	1.40	98.60
C1	69.13	68.24	0.89	1.31	98.69
C2	65.92	64.59	1.33	2.06	97.94
C3	63.02	61.52	1.50	2.43	97.57
C4	60.31	58.79	1.52	2.58	97.42
C5	58.82	57.31	1.51	2.63	97.37

C: INFLUENCE OF SULPHATE ATTACK ON MASS OF HPCS

With 5% presoaked pumice

Mix ID	W1$_{water}$(g)	W2$_{MgSO4}$(g)	W1- W2(g)	Loss (%)
Influence of sulphate attack on mass of HPCs at 28 days				
C0a	2532.13	2485.16	46.97	1.89
C0b	2584.21	2546.27	37.94	1.49
C1	2536.06	2498.34	37.72	1.51
C2	2498.34	2459.96	38.38	1.56
C3	2578.90	2524.37	54.53	2.16
C4	2498.79	2442.13	56.66	2.32
C5	2560.03	2489.33	70.70	2.84
Influence of sulphate attack on mass of HPCs at 56 days				
C0a	2648.31	2608.40	39.91	1.53
C0b	2614.17	2582.41	32.06	1.24
C1	2621.63	2585.94	35.69	1.38
C2	2541.19	2505.36	35.83	1.43
C3	2592.24	2544.16	48.08	1.89
C4	2501.92	2453.58	48.34	1.97
C5	2663.02	2602.13	60.90	2.34
Influence of sulphate attack on mass of HPCs at 90 days				
C0a	2694.10	2662.42	31.68	1.19
C0	2687.05	2659.92	27.13	1.02
C1	2644.04	2613.20	30.84	1.18
C2	2607.01	2575.08	31.93	1.24
C3	2514.38	2476.25	38.13	1.54
C4	2609.18	2565.06	44.12	1.72
C5	2658.12	2606.77	51.35	1.97

Plate IV: Display of constituent (left) and weighing of MHA (rright)

Plate V: soaking of pumice (left) and weighing of fine aggregate (rright)

Plate VI: weighing of coarse aggregate (left), mixing of concrete (middle) and casting of HPC samples (rright)

Plate VII: compressive strength test ssetup, withdrawal of samples from the curing tank (left), loaded sample (middle), and sample at failure state (right)

Plate VIII: Sorptivity test setup, drying of samples (left), samples exposed to capillary action in the water (middle), and sample weighing (right)

Plate IX: Visual appearance of HPCs after 28-days (left), 56-days (middle) and 90-days (right) of age