Influence of Magnesium Sulphate on the Compressive Strength of Internal Cured (IC) Rice Husk Ash based High Performance Concrete

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Abstract

The incorporation of superabsorbent polymers (SAP) and pre-saturated lightweight aggregate (LWA) as internal curing agents (IC) have been proven to be more effective and acceptable in autogenous shrinkage and self-dessication mitigation. However, the incorporation of the internal water reservoirs (SAP & LWA) increases the porosity of the concrete matrix which can injure the strength and durability properties of the HPC. This paper explores the use of the two IC agents as internal curing agents and the comparative study was investigated on their influence on compressive strength of RHA based high performance concrete in aggressive $MgSO_4$ *environment. HPC mixtures of designed C55/67 target minimum cube strength at 28-day were studied. Concrete cubes of 100 mm were cast and cured in both water and* $MgSO_4$ *solution for 28-day hydration period. The result reveals that the mix with LWA (*$PHPC_1$*) is better with the highest residual compressive strength compared to the SAP mixes in terms of MgSO4 resistance but other way round in terms of the compressive strength of specimens cured in water.***

Keywords: *Internal Curing, High-performance Concrete, Superabsorbent Polymers, Rice Husk Ash, Pre-soaked Pumice*

INTRODUCTION

The declination in giving up the trend in the usage of High-Performance Concrete in building construction in the recent years is getting wider because of their unique properties of high strength, high durability, high modulus of elasticity and construction of tunnels, bridges, precast pylons, tall buildings and parking garages with the possibility of having relatively thin sections (Aïtcin, 2004 and Orosz, 2017). The HPC production had also known to require the use of other supplementary cementitious materials (SCM) and most especially the silica fume (SF) which is much cost and not available in some Sub-Saharan Africa especially in Nigeria. Therefore, the non-availability and the high cost of the SF geared up the researchers towards the local sourcing of the alternative materials and as a result necessitated the incorporation of the Rice Husk Ash (RHA) which has been reported as a very reactive good pozolanic activity and as a good, effective and efficient substitution to silica fume (SF) (Olawuyi *et al.*, 2020).

The high-performance concrete may not turn out to be durable and maintenance free because these concrete mixtures may generate too much heat and are characterized by high autogenous and drying shrinkage, and therefore is prone to cracking due to restrained shrinkage at early age (Schrofl *et al.*, 2012). Autogenous shrinkage could be described according to Jensen and Hansen (2001) reported by Olawuyi (2016) as the measurable change in external volume of a concrete mixture due to chemical shrinkage. High-performance concrete mixtures were developed due to a growing focus on durability and are created by the use of lower water-cementitious materials (w/c) ratios, chemical admixtures and supplementary cementitious materials (SCMs) (Hoff, 2002). However, such performance can only be achieved if the cementitious system is well hydrated, meaning that it has to be well cured and concrete with w/b ratios less than about 0.42 do not have enough water to fully hydrate the cement in the mixture (Schrofl *et al*., 2012).

According to Byard and Schindler (2010), as the cement in a concrete mixture hydrate, water in the capillary pores is consumed. This process decreases relative humidity in the mixture and increases the temperature and internal stresses, resulting in an increased risk of drying shrinkage and cracking (ACI (308-213) R-13 2013). In order to reduce the risk of the autogenous shrinkage, it is necessary to mitigate the decrease in relative humidity in the mixture during hydration and this can be achieved by using internal curing approach as the low porosity characteristic of HPC had hindered the possibility of rapid water percolation into the cement matrix by means of external curing and thereby called for an intensive studies in internal curing (Bentur *et al*., 2001), Internal Curing (IC) according to ACI (213-03R) is defined as the process whereby the hydration of cement continues because of the availability of the internal water that is not part of the mixing water and could be achieved through the incorporation of IC agents that have the ability and capability to absorb and retain water in the system then release when the condition changes (Byard and Schindler, 2010). Hence, reduces the capillary stresses and provide additional water for cement hydration.

Many researchers have incorporated different IC agents in concrete but with pre-saturated lightweight aggregate (LWA) and superabsorbent polymers (SAP) been the most acceptable (ACI (308-213) R13, 2013; RILEM Rep 041 - Jensen and Lura (eds.), 2006). According to the literature (Olawuyi and Boshoff, 2017) that studied the micro-structure and porosity of the concrete matrix using Computer Tomography (CT) Scanning and Scanning electron Microscope (SEM) discovered that the incorporation of these IC agents caused a change in the concrete's micro-structure which resulted in to an increase in the porosity of the concrete. As a result, the incorporation of these IC agents (SAP and LWA) which led to an increase in the porosity of the concrete might allow the penetration of aggressive chemicals in to the concrete matrix which may affect the concrete strength and durability because most of durability problems in cementitious composites involve the ingress of water and aggressive chemicals (Taylor, 2014). As a result, the incorporation of these IC agents (SAP and LWA) which led to an increase in the porosity of the concrete might allow the penetration of aggressive chemicals most especially the sulphate ions in to the concrete matrix which may affect the concrete strength and durability because most of durability problems in cementitious composites involves the ingress of water and aggressive chemicals such as sulphate attack.

The concrete's sulphate attack is one of the problems of durability facing the reinforcing steel and other materials using in the production of concrete. The negative effects of the sulphate attack on concrete as identified by Neville (2012) encompasis cracking, spalling, loss of strength and other effects because most concrete structures are having direct contact with the ground (soil) and the water beneath (i.e. the underground water) which possible that the sulphate ions might be present. The sulphate ions from the seawater, ground water and soil are discovered to be mixed together with some other ions such as magnesium, potassium, calcium and sodium ions. The aggressiveness of sulphate environment was classified in accordance with the Durability Guide Act 201 Committee based on the $SO_4{}^{2--}$ (g/l) concentrations and was later affirmed that $MgSO_4$ environment is the most aggressive environment. When this $MgSO_4$ reacts with cement compounds most especially the C_3A and the lime $\{Ca(OH)_2\}$ including the CSH, they decomposed and formed gypsum and ettringite in detriment to cement property and produce softening and expansive deterioration types. The mechanical properties of concrete were affected under the attack of sulphate as a result of the delayed ettringite crystal effect enhancement and weakening effect of damage evolution (Syamsul, 2017; Olonade, 2016).

The development and nucleation of delayed ettringite crystal fills the pores of samples and led to a decrease in the porosity of the concrete in the first stage of sulphate attack but resulted in to an increase in the surface hardness of samples. On the other hand, the surface hardness decreases owing to the damage in surface of the concrete caused by by the expansion force of the delayed ettringite (Syamsul, 2017). The blended cement incorporated RHA as a mineral admixture led to a reduction in the ettringite formation owing to a decrease in the quantity of lime $\{Ca(OH)_2\}$ and C_3A as a result of the additional CSH produced by the RHA reactivity. Thus, enhanced concrete resistance against sulphate attack (Neville, 2014; Ramezanianpour *et al.*, 2009). The severe affected structures built in marine environment and concrete sewers hydraulically (Olonade, 2016). Many concrete structures affected by sulphate often prone to maintenance or complete rehabilitation that attract substantial expenditure that could have been directed for new structures production. Furthermore, man-hour is lost while operation would be grossly affected during repair and thus, the cost incurred as a result of maintaining the sulphate attack on concrete is substantial to bear.

Internal curing (IC) agents of different types have been incorporated in different studies as said earlier by many researchers but with SAP and pre-soaked saturated LWA being the most acceptable (ACI (308-213) R13, 2013; RILEM Rep 041 - Jensen and Lura (eds.), 2006). The SAP is somehow expensive due to its availability in just some limited places and as a result geared the researchers towards the local sourcing of the alternative materials that led to the development and incorporation of the pre-soaked saturated LWA that is locally available (Schrofl *et al.*, 2012). It is now of particular importance to check the influence of these IC agents as a result of a change in environment on the strength and durability performance of RHA based HPC since the first yardstick in measuring the performance of the concrete is by checking its strength and extremely, high durability properties are the main benefits of HPC. The study will therefore be filling the gap by examining the comparative study of the effectiveness of these two IC agents on strength and durability performance of RHA based HPC as the study incorporated these two IC agents as a comparative study are scarce in literature.

EXPERIMENTAL INVESTIGATION

Materials

The constituent ingredients utilized in this research work are namely: IC agents (SAP & LWA), Natural Sand, Crushed Stone, Binder (PC & RHA), Water and Superplasticizer (SP). Superabsorbent polymers (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 (2018) 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 LWA incorporated was a porous igneous rock formed as a result of explosives volcanic eruptions which was later crushed and 13 mm maximum size was incorporated (Olawuyi *et al.*, 2020).

The river sand (natural) conforming to medium sand classification was used according to Shetty (2004) as the fine aggregate (i.e. the requirement specifications for the production of HPC) with a minimum particle size of 300 µm (Nduka *et al.*, 2020; Olawuyi *et al.*, 2020; Aitcin, 1998; Beushausen and Dehn, 2009; Neville, 2012). A greywacke crushed stone of 13 mm maximum aggregate size was used as the coarse aggregate in accordance with typical HPC mixtures reported in the literature (Beushausen and Dehn, 2009; Neville, 2012; Olawuyi *et al.*, 2021). In order to achieve a reduction in the dust content of the aggregate for less water demand by the mixtures, the crushed stone was washed and surface dried before the usage.

Also, in accordance with BS EN 197-1 (2011) and NIS 444-1 (2003), CEM II/A-LL, 42.5N was used as the main binder in compliance with requirement for PC specification for the production of HPC. The supplementary cementitious material (SCMs) used was the RHA obtained from rice husk acquired from a rice mill at Minna, Niger State, Nigeria and incorporated at 10% by weight of the PC. The RHA was calcined using a locally fabricated incinerator in a controlled environment at a temperature below or equal to 700^{o}c. A sky 504 masterglenium polymer-based polycarboxylic ether (PCE) superplasticizer supplied by Armorsil Manufacturing Incorporation was used as the chemical admixture (superplasticizer) and constantly administered at 1.5% concentration by weight of binder (b_{wob}) as used in the typical HPC mixtures (Olawuyi, *et al.*, 2020; Nduka *et al.*, 2020). 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) and a portable cleaned water in accordance with the specification of BS EN 1008 (2002) was used for the mixing at 0.3 W/B reported by Ogunbayo *et al.* (2018).

The production of the specimens was achieved through the use of a concrete mixer in Building Laboratory Department, Federal University of Technology, Minna. The fine aggregate was first poured in to the mixer followed by the binders (PC & RHA) after which both (PC & RHA) have been thoroughly mixed manually with each other and a homogeneous mixture had been achieved. The granite was added after mixing for about 30 seconds and the mixing continues for another 1 minute before adding about 75% of the mixing water. A masterglenium (sky 504) superplasticizer was pre-mixed with the remaining 25% of the mixing water, this is added to the mixture and mixed further for 3 minutes. The other mixes also followed the above steps just that the saturated lightweight aggregate (LWA) was added together with the granite before the addition of water for the mixes incorporated LWA. Furthermore, for mix incorporated SAP, the dry SAP particles was added immediately after the 30 seconds addition of binders and sand. Hence, all the fine contents (sand, binders & SAP) were allowed to be mixed together for about 30 seconds as well for even dispersion of the SAP particles before the addition of granite and water respectively as discussed before and recommended in the literature (Aitcin, 1998; Mehta & Monteiro, 2014; Neville, 2012).

Methods

Properties of Constituents Materials

The particle size distribution of the aggregate's samples (sand, granite and LWA) was determined by wet sieving while the specific gravity of the aggregates and binders were also determined in the Building Concrete Laboratory of Federal University of Technology, Minna. Section 3.3 further present and discuss the result of the physical properties of the constituent materials.

HPC Specimens Production

A target strength of 60 MPa at 28-day characteristic strength was adopted as the reference mixture (CHPC) which was designed in accordance with the work of Aitcin (1998) reference method of HPC mixtures as reported in the literature (Nduka *et al.*, 2020, Olawuyi *et al.*, 2021) showing in table 3.1 below. Furthermore, other HPC mixtures incorporating SAP at 0.2% ($S^{0.2}$) by weight of binder (b_{wob}) and 5% (P^5) and 7.5% ($P^{7.5}$) of pre-saturated lightweight aggregate by weight of coarse aggregate (b_{woca}) at constant W/B of 0.3 were also produced. The binder comprises of 90% Portland cement and SCM of 10% (PC+RHA).

Fresh Property

The flow table test was used in carrying out the slump flow measurement as described in BS EN (12350 - 5 - part 1) as a measure of workability of the HPC mixtures. When the required workability and cohesion (400-600 mm slump flow spread) for the specified design mix had been met, the specimens for both the curing regimes (i.e. curing in water and $MgSO_4$ solution) were cast as explained in section 2.2.1 and 2.2.2 and de-moulded after 24h of casting.

Table 1: Materials mix proportioning

Materials	Mixture types (kg/m^3)			
	Reference	HPC with SAP	HPC with pre-soaked pumice	
	CHPC*	SHPC*	$PHPC_1$*	$PHPC_2$*
CEM II 42.5 N	485	485	485	485
Rice Husk Ash (10%)	55	55	55	55
Superplasticizer (1.5% b_{wob})	8.1	8.1	8.1	8.1
Fine sand	700	700	700	700
Coarse aggregate	1050	1050	997.5	971.25
Pre-soaked pumice			52.5	78.75
SAP (0.2%)		1.08		
Water	156	156	156	156
Additional water for SAP		12.5g/g of SAP		
W/B	0.3	0.3	0.3	0.3

*CHPC is Control HPC, SHPC is SAP HPC (containing 0.2% SAP b_{wob}); $PHPC_1$ is Pumice HPC (containing 5% pumice b_{woca}) and $PHPC_2$ is Pumice HPC (with 7.5% pumice b_{woca})

Specimen Production and Testing

A total of 72 (100 mm) specimens were tested in which 36 were cured in ordinary water and the other 36 were cured in 5% $MgSO_4$ solution to determine the resistance in an aggressive sulphate environment. The specimens were tested for compressive strength in accordance with BS EN 12390-3 (2019) at 28-day and the strength was determined by dividing the crushing load with the area of the cube specimen. Three concrete specimens were tested for each mix and average reported.

RESULTS AND DISCUSSION

Physical properties of Constituents Materials

Table 2 reveals the fine aggregate to have a coefficient of uniformity (C_u) of 2.39, coefficient of curvature (C_c) of 0.94 and fineness modulus (FM) of 2.87 conforming to standard specification stated by Shetty (2004) to be a medium sand classification while the coarse aggregate used for the study is a uniformly graded stone. The outcome of the results of the physical properties shown in Table 2 makes it crystal clear that both the fine and coarse aggregates are suitable for HPC production.

Table 2: Physical Properties of Materials

Item	Sand	Granite	Pumice	RHA	PC
D_{10}	360	10000	10000		
D_{30}	540	11000	11000		
D_{60}	860	13000	13000		
C_u	2.39	1.30	1.3		
C_c	0.94	0.93	0.93		
FM	2.87				
SG	2.80	2.8	1.80	2.10	3.14

Fresh Properties of HPC

The results of the flow table test conducted on the mixes before casting revealed that the mixes comply with the target limits of 400-600 mm slump flow for all. This implies that irrespective of the IC-agents incorporated, the consistency and the workability were similar.

Compressive Strength of HPCS

The compressive strength test was carried out at 28th day with water and $MgSO_4$ solution as the curing media. Table 3 and Figure 1 presents the various HPCs compressive strength with the same binder proportions. The result shows that the compressive strength of the HPCs having IC-agents incorporated are slightly higher than the control in the two-curing media. The loss in compressive strength as influenced by $MgSO_4$ was observed as 3.84%, 1.26%, 0.36% and 1.62% for CHPC, SHPC, $PHPC_1$ and $PHPC_2$ of HPCs respectively as compare to the specimen cured in water. Furthermore, the result also revealed that the HPC incorporated SAP (SHPC) has the highest compressive strength of 59.14 MPa in water and 58.40 MPa in MgSO4 while the HPCs incorporated LWA with compressive strength of 58.02 MPa ($PHPC_1$) and

57.89 MPa ($PHPC_2$) in water but 57.81 MPa ($PHPC_1$) and 56.95 MPa ($PHPC_2$) in MgSO4.

Table 3: Influence of Curing Media on Compressive Strength

Specimen Number	Curing Media Water	 MgSO4	CS28w	CS28c	Loss%	Residual CS
CHPC	54.96	52.85	100	96	3.84	96.16
SHPC	59.14	58.40	108	106	1.26	98.74
PHPC1	58.02	57.81	106	105	0.36	99.64
PHPC2	57.89	56.95	105	104	1.62	98.38

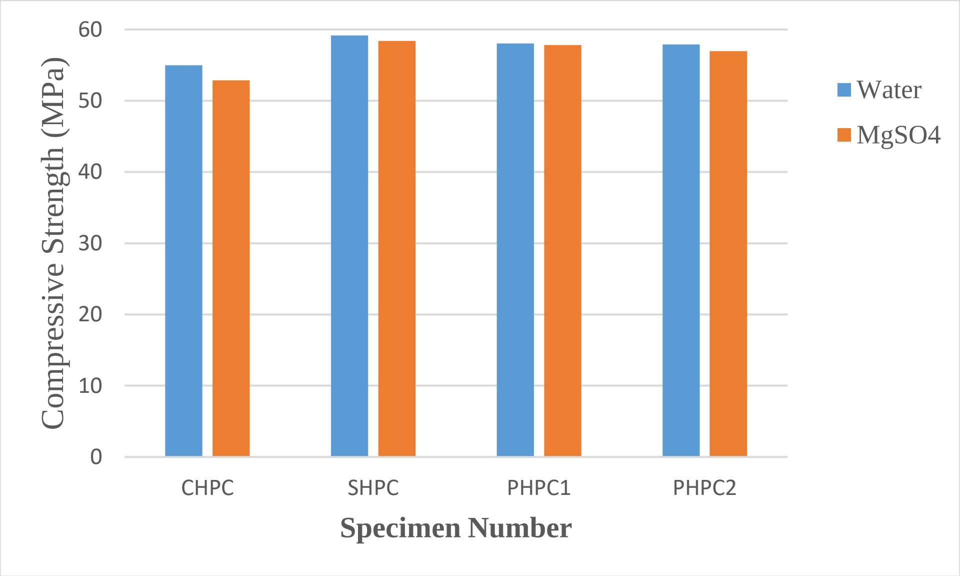

Figure 1: Influence of Curing Media on Compressive Strength

SUMMARY OF FINDINGS

The following are the inferences deduced from the study

i. The RHA used as the SCM is a good alternative to silica fume

ii. Use of SAP and saturated Pumice (i.e. LWA) as IC-agents improved the 28-day compressive strength of the HPCs.

iii. MgSO4 as a curing media resulted in slight loss in compressive strength values for all HPCs studied.

iv. The HPC incorporated SAP shows higher compressive strength in both curing media in relation to other HPCs mixtures (i.e. $PHPC_1$ &$PHPC_2$).

v. HPCs containing SAP as IC-agent had better performance than those containing presoaked Pumice in compressive strength values but were noted to exhibit lower residual strength values.

CONCLUSIONS AND RECOMMENDATION

The present research work explored the effectiveness of the SAP and presoaked Pumice as IC-agents in C55/65 RHA based HPC made from binary binder consisting of cement and 10% RHA with administration of Masterglenium sky 504 superplasticizers. The study revealed that the two IC-agents have effect on the strength and durability properties of the HPC but more pronounced in SHPC. The SHPC has a residual strength of 98.74 %; $PHPC_2$ has 98.38 % while $PHPC_1$ revealed the highest residual strength of 99.64 %.

The study thereby recommends the use of presoaked Pumice of Nigeria origin at 5% b_{woca} for the Class of RHA based HPC studied.

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