

# Sulphate Resistance of Internally Cured Rice Husk Ash Based High Performance Concrete

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*Abstract- High performance concrete is known with its unique property of low water-binder ratio and thereby reported to be susceptible to autogenous shrinkage while the previous research efforts directed at incorporation of superabsorbent polymers (SAP) and pre-saturated lightweight aggregate (LWA) as internal curing (IC) agents in autogenous shrinkage mitigation and the incorporation resulted into an increase in the porosity of the HPC. This study seeks to fill the existing gap on proper understanding of the effectiveness of Nigeria pre-soaked pumice in comparison with SAP being a foreign material not available in Nigeria. While rice husk ash (RHA) on the other hand, affirmed to be an effective silica fume (SF) alternative replacement, a major requirement in HPC production. The work studied the sulphate resistance of HPC containing RHA as the SCM with Nigeria pre-soaked pumice and SAP incorporated as IC agents. HPC mixtures of designed C55/67 characteristic cube strength at 28 days were produced incorporating SAP at 0.2% (SHPC) by weight of binder (bwob) and pre-soaked pumice at 5% (PHPC1) and 7.5% (PHPC2) by weight of coarse aggregate at constant 10% RHA bwob using 100 mm cubes cured in water and magnesium sulphate solution. The gradual deterioration and resistance to sulphate of these specimens were examined via visual observations, mass loss and strength loss. The results revealed that the concrete cured in sulphate solution showed white sulphate crystals on both the reference HPC (CHPC) and the HPC incorporated IC agents at all ages except at 28 days where the specimens had grey colour and few precipitate. All HPC specimen studied suffered slight mass and strength losses but the losses decreased as the curing age increases as a result of a progressive hydration. The losses were observed to be higher in CHPC as compared to the internally cured HPCs. The study revealed that the RHA is a very reactive pozzolan and can be used as alternative to silica fume and the IC agents have effect on the strength property of the HPC. Pre-soaked Pumice of Nigeria origin's utilization at 5% was observed to be better compared to other mixes in sulphate resistance behaviour and the study therefore recommend 5% Pre-soaked pumice as an IC agent in RHA based HPC production for good performance in sulphate environment.*

*Index Terms- High-Performance Concrete; Superabsorbent Polymer; Internal Curing; Rice Husk Ash, Presoaked Pumice.*

## I. INTRODUCTION

### 1.1 Background to the Study

Concrete is the most common construction material in use nowadays and accounts for a large part of the infrastructure system. It is recorded as most utilized material after water in the world according to Syamsul (2017). Compared with other construction materials, concrete is easy to form to any shape and its basic ingredients are available in the local area of the construction site, such as the fine aggregate, coarse aggregate, water and Portland cement (Neville, 2012). Like other building materials being used in the construction industry, concrete experiences the development of qualities in order to support the requisite needs. This has led to the developments from normal concrete (NC) to high strength or high-performance concrete (HSC/HPC) up to ultra-high strength concrete (UHSC). The International Federation for Structural Concrete (2012) and Model Code (2010) described NC as concrete with maximum compressive strength of 50 N/mm<sup>2</sup> at 28 days and the HSC/HPC up to UHSC are generally concrete with minimum compressive strength of 50 N/mm<sup>2</sup> at 28 days. The deterioration and premature failure of concrete structures such as marine structures and concrete bridge deck, led to the development of HPC which is the answer to weakness of NC.

The trend in the usage of HPC in building construction in recent years is getting better because of its unique properties of low water: binder (W/B), high strength, high durability, high modulus of elasticity and the possibility of having relatively thin sections for the construction of tunnels, bridges,

precast pylons, tall buildings and parking garages (Aitcin, 2004 & Orosz, 2017). Hence, Cement is one of the most vital ingredients in HPC production, providing binding capabilities to the mixture. However, the setbacks of cement lie in the high energy demand and clinker's calcination high temperatures of about 1450 °C with the attendant effect of greenhouse gases like carbon dioxide (CO<sub>2</sub>) emission of 0.85 ton per 1 ton of cement manufacturing (Tulashie *et al.*, 2021). Reports have estimated clinker's annual global CO<sub>2</sub> emissions to be 5-8% (Afroughsabet *et al.*, 2021), making it the second-largest contributor of CO<sub>2</sub> emissions after the power generation industry (Wi *et al.*, 2018). Owing to this, serious efforts have been made by researchers and practitioners toward the establishment of the supplementary cementitious materials (SCMs) for incorporation in the production of concrete. For instance, in the work of Sandhu & Siddique (2017), it was affirmed that SCMs in cement and concrete applications lowered heat of hydration, increased strength, decreased permeability at higher amounts, enhanced chloride and sulphate resistance/mild acids, lesser materials costs regarding cement savings, and ecologic benefits related to waste disposal and reduced CO<sub>2</sub> emissions which is one of the sustainable development goals (SDGs). The past literature on SCMs (Aitcin, 2004; Biskri *et al.*, 2017; Scrivener *et al.*, 2018; Jaskulski *et al.*, 2020; Nwankwo *et al.*, 2020) pointed out different demerit of using the SCMs within the world. Scrivener (2018) posit that over 80% of SCMs used in cement production come from limestone, FA, or GGBS. GGBS available worldwide is 5-10% compared to cement, while FA is about 30% higher. Biskri *et al.* (2017) pointed out that the inclusion of GGBS in HPC mixtures reduces hydration heat and increases permeability and porosity in binders paste with 20% maximum cement replacement. Aitcin (2004) affirmed that the production of HPC requires about 7.5–8% SF incorporation and hence, argued that SF's access, economics, and service factors are relevant concerns for HPC applications (Aitcin, 2004).

Ghafari *et al.* (2016) posited that aside the high-cost implications of the SF, the concrete appearance aesthetically is affected by its dark grey colour and also observed, was the increase in the water demand owing to its high specific surface area which also

having effect on the flowability. In addition, the inclusion of SF requires a higher dosage of superplasticizer that makes mixtures sticky. Previous studies (Zhang *et al.*, 2003; Mazloom *et al.*, 2004; Igarashi *et al.*, 2005) as reported in Ghafari *et al.* (2016), showed that the addition of SF in HPC increased autogenous shrinkage. The SF is a foreign material which is not readily available in Sub-Saharan Africa most especially in Nigeria and as a result, very expensive because of the import duty charges. The non-availability that led to the high cost of SF warrants research thoughts toward local sourcing of alternative materials and hence necessitated the incorporation of the Rice Husk Ash (RHA) which is considered a viable alternative with about 60% increase in rice production in Nigeria between 2013 and 2019 which gave rise to the need to improve the rice production byproduct requirements (Syamsul, 2017; Olawuyi *et al.*, 2020; Nduka *et al.*, 2020; Salisu *et al.*, 2021). Recent studies (Chindaprasirt *et al.*, 2007; Ganesan *et al.*, 2008; Salas *et al.*, 2009; Madaudoust *et al.*, 2011; Gastaldini *et al.*, 2014 and Bie *et al.*, 2015; Mudashiru *et al.*, 2021) incorporated RHA in HPC mix at between 5-20% binder content with improved compressive strength and durability. However, studies in Nigeria (Alhassan, 2008; Agbede & Obam, 2008; Oyekan & Kamiyo, 2008; Dabai *et al.*, 2009; Abalaka, 2012) replaced cement with RHA in either normal strength concrete or sandcrete block applications with little or no improvement in compressive strength recorded. However, the cement replacement with RHA in high-performance concrete and mortar is adjudged to improve concrete performances regarding strength, durability, and pore refinement (Mudashiru *et al.*, 2021; Nduka *et al.*, 2020; Olawuyi *et al.*, 2020).

HPC made from the proposed materials need to be ascertained for issues of high heat of hydration, high autogenous and drying shrinkage with cracking due to restrained shrinkage at early age being of concern here (Schrofl *et al.*, 2012). The requisite need of HPC with regards to durability and high modulus of elasticity performance can only be achieved if the cementitious system is well hydrated and this calls for special attention to the curing needs for this low W/B concrete towards providing enough water to fully hydrate the cement in the mixture (Schrofl *et*

*al.*, 2012). Internal curing (IC) methods is reported as the most effective method of reducing the risk of autogenous shrinkage as the HPC low permeability rendered the external curing not to be sufficient and could be achieved through the incorporation of IC agents that have the ability and capability to absorb and retain water in the system and then release when the condition changes (Byard & Schindler, 2010). Different studies have incorporated different IC agent in concrete but with lightweight aggregate (LWA) and superabsorbent polymers (SAP) been the most acceptable (ACI, 2013; Jensen & Lura, 2006).

The SAP is a foreign material and not available in Nigeria and resulted in to searching for alternative materials that led to the development of Nigeria locally available pre-soaked pumice. Furthermore, the incorporation of these IC agents, according to Olawuyi and Boshoff (2017), caused a change in the concrete's micro-structure resulted in an increase in the porosity of the concrete and allowed the penetration of aggressive chemicals most especially the sulphate ions into the concrete matrix. Therefore, as a result of the availability of both the presoaked pumice and RHA in Nigeria and limited studies using these materials in HPC, this paper therefore aimed at evaluating the sulphate resistance of HPC containing RHA as a partial replacement of cement internally cured with Nigeria presoaked pumice substituting the SAP.

## II. MATERIALS AND METHODS

The study incorporated different materials as mentioned earlier and an experimental program was designed for the production of high-performance concrete mix for the best protection in sulfate environment by adding several combinations of a local natural pozzolan such as RHA. Therefore, materials used and the experimental procedures are described in the following sections.

### 2.1 Materials

The research incorporated CEM II/A-LL, 42.5 N (3X) from Dangote Cement, Obajana Plant conforming to BS EN 197-1 (2011) as the main cementitious material. RHA was obtained from rice husk and calcined using fabricated local incinerator.

This RHA was calcined to ash at a temperature of about 600-700°C in a controlled environment then pulverized using a grinding mill machine and incorporated constantly at 10%  $b_{wob}$ . The chemical admixture used was a MasterGlenium SKY 504 superplasticizer supplied by Armorsil Manufacturing Incorporation and administered at constant concentration of 1.5%  $b_{wob}$ . The river sand used was at the air-dry condition with a minimum particle size of 300  $\mu\text{m}$  (i.e., all the particles smaller than 300  $\mu\text{m}$  were removed using the sieving method) in compliance with the requirement for fine aggregate specification for HPC production (Aitcin, 1998; Beushausen and Dehn, 2009; Neville, 2012; Nduka *et al.*, 2020; Olawuyi *et al.*, 2021). 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. Crushed granite stone that passed 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 (Aitcin, 2004; Beushausen and Dehn, 2009; Neville, 2012; Olawuyi & Boshoff, 2018; Nduka *et al.*, 2020; Olawuyi *et al.*, 2021). The greywacke crushed granite used was in saturated surface dry conditions after been washed to eliminate the fine content that will likely increase water demand. The characterization of these materials are given in Figure 1, 2, 3 and 4. A Superabsorbent polymer (labelled FLOSET 27CS) of  $\leq 600 \mu\text{m}$  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 outcome result was compared with the aftermath result of the RHA based HPC incorporated pre-soaked Nigeria Pumice as the IC agent at 5% and 7.5% varied contents by weight of the coarse aggregate after been soaked in water for about 24 hours. The pre-soaked Pumice used 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 with portable cleaned water.

### 2.2 Concrete specimen production

HPC samples were produced with 100 mm cubes for the physical characteristics, visual assessment, mass loss and strength loss following BS EN 12390-3 (2019) and RILEM Technical Recommendation TC14-CPC 4 (1975). The mix design of HPC specimens as per the British method is shown in Table 1. The water-to-binder ratio (W/B) and RHA were kept constant at 0.3 and 10% ( $b_{wob}$ ) respectively. A fixed SAP content of 0.2%  $b_{wob}$  and 1.5%  $b_{wob}$  superplasticizer content was introduced. Extra water of 12.5 g/g of SAP was added based on the SAP absorption properties established by Olawuyi's (2016). The HPC classifications were tagged with the name of HPC with the IC agents initials attached. For instance, the HPC containing 0.2% SAP and as well as 5% and 7.5% presoaked pumice were coded as SHPC, PHPC<sub>1</sub>, PHPC<sub>2</sub> respectively and CHPC for the reference samples. After casting, each sample was cured at room temperature for 24 h. After demoulding, specimens were cured in water as well as in 5% sulphate solution at  $21 \pm 2$  °C till the experiment date.

Table 1: Materials mix proportioning

Materials	Mixture types (kg/m <sup>3</sup> )			
	Referen ce	HPC with SAP	HPC with pre- soaked pumice	
	CHPC*	SHPC C*	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 W/B	12.5g/g of SAP			
	0.3	0.3	0.3	0.3

### 2.3 Test Methods

#### 2.3.1 Characterization of cementitious materials (CEM II & RHA)

The specific surface area and PSD of CEM II and RHA were analyzed using BET (Nova Station B Quantachrome Instrument, Boynton Beach, FL, USA) and laser PSD (Malvern Mastersizer 3000, Worcestershire, UK) respectively as well as their XRF (using Bruker AXS S4, Explorer, Germany).

#### 2.3.2 Workability

The slump flow measurement was performed as described in BS EN 12350-5 (2019) in determining the workability of the HPC mixtures. The specimen was prepared following Olawuyi's (2016) HPC preparation procedures after meeting Neville's (2012) recommendation on 400 - 600 mm slump flow spread on workability and cohesion for HPC. The HPC specimen produced were covered in the laboratory with a jute bag and allowed to harden for 24 h before demoulding and curing in a sulphate solution tank at  $21 \pm 2$  °C until testing.

#### 2.3.3 Sulphate Resistance Test

This test was performed to assess the physical characteristics and response of HPC to attack by immersing in sulphate solution with a total of 162 samples prepared base on three curing ages (28, 56 and 90) and triplicate samples per crush. Sulphate ions in the solution was combined with calcium hydroxide and gypsum to form an expansive reaction. Concrete cubes of 100 mm size were produced and examined for sulphate action. The assessment was made in terms of mass change, visual observation and strength deterioration factor.

##### 2.3.3.1 Mass change

Deterioration of the HPC mixtures immersed in 5% magnesium sulphate solution was carried out by measuring change in mass of the specimens after the respective curing ages. The initial weight of the

specimens and the weight after the immersion was measured for determination of mass loss due to the deterioration of the specimen by sulphate. The average values of three specimens were considered for assessment.

### 2.3.4 Strength loss

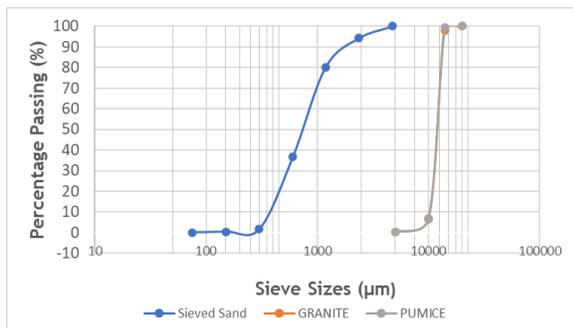
The deterioration of concrete specimens was investigated by evaluating the strength distortion factor expressed in percentage, The strength loss was measured at their respective ages of immersion and was calculated.

## III. RESULTS AND DISCUSSIONS

### 3.1 Materials Characterization

#### 3.1.1 Particle size distribution (PSD) of the aggregates

Figure 1 presents the particle size distribution of the fine and coarse aggregates examined in this study. The results revealed that the fine aggregate conformed to the medium sand classification of Shetty (2004) with uniformity coefficient ( $C_u$ ) of 2.39, coefficient of curvature ( $C_c$ ) of 0.94, specific gravity value of 2.82 and fineness modulus (FM) of 2.88. The coarse aggregates used was also observed to be a uniformly graded stone with coefficient of uniformity ( $C_u$ ) of 1.32 and 0.92 coefficient of curvature ( $C_c$ ). Figure 1 makes it crystal cleared that both the fine and coarse aggregates (i.e., crushed granite and pre-soaked pumice) are appropriate for the production of HPCs.



agents contributed to the reduction in mass loss which decreased as the curing ages increases. This is as a result of the continuous hydration enhanced by the RHA introduced as SCM, yielding additional C-S-H through its reactions with lime on one part and also, the IC agents incorporated providing the internal reservoir water for further hydration. The mass loss of HPCs with SAP as IC agent for instance gave percentage mass loss values of 2.75, 2.25 and 1.46 at 28-, 56- and 90-days and 1.92, 1.88 and 0.74 for the PHPC<sub>1</sub>, 4.0, 2.64 and 2.24 for PHPC<sub>2</sub> respectively. It is interesting to note that IC agents' addition positively contributed to resistance of MgSO<sub>4</sub> attack when compared to that reference mixture (CHPC). Application of the RHA in HPC is advantageous under sulphate environment due to the discontinuous pore structure of the ash concrete and amount of calcium hydroxide present in concrete

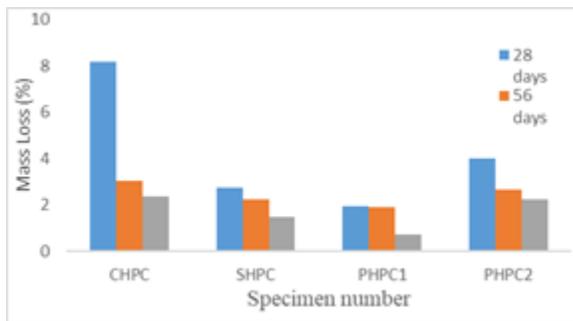


Figure 2: Mass loss of internally cured RHA based HPCs subjected to MgSO<sub>4</sub> attack

### 3.2.1.2 Strength loss

Comparisons between strength internally cured RHA based HPCs cured in both water and MgSO<sub>4</sub> media at various ages (28, 56 and 90 days) are shown in Figure 3. At the end of 90 days curing age, the strength of concrete specimens immersed in MgSO<sub>4</sub> solution is compared with that of HPCs immersed in water bath. The difference in the compressive strength between HPCs immerse in water bath and those immersed in MgSO<sub>4</sub> solution is regarded as the strength loss. From the results displayed in the Figure 3, it is obvious that the entire specimen suffered strength losses but the loss was not drastically detrimental.

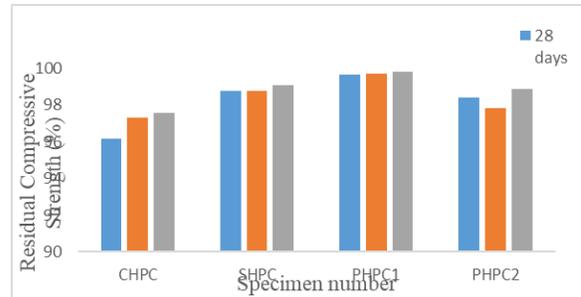


Figure 3: Strength losses as caused by MgSO<sub>4</sub> attack on internally cured RHA based HPC

The strength loss was observed to be more in reference HPC (CHPC) specimens when compared to those having IC agents incorporated (SHPC, PHPC<sub>1</sub> and PHPC<sub>2</sub>). The PHPC<sub>1</sub> exhibited the lowest loss in strength with the highest residual strength of 99.64%, 99.71% and 99.81% at 28, 56 and 90 days respectively followed by the SHPC and PHPC<sub>2</sub> with residual strength of 98.74%, 98.76%, 99.08% and 98.38%, 97.83% and 98.86% at 28, 56 and 90 days respectively. Strength losses values of 2.11 N/mm<sup>2</sup>, 1.80 N/mm<sup>2</sup> and 1.70 N/mm<sup>2</sup> were observed for CHPC mixes at 28, 56 and 90 days respectively, whereas, the strength loss value of 0.74, 0.62 and 0.50 N/mm<sup>2</sup> were recorded in SHPC; 0.21 N/mm<sup>2</sup>, 0.15 N/mm<sup>2</sup> and 0.12 N/mm<sup>2</sup> for PHPC<sub>1</sub> and 0.94 N/mm<sup>2</sup>, 0.90 N/mm<sup>2</sup> and 0.58 N/mm<sup>2</sup> for PHPC<sub>2</sub> at 28, 56 and 90 days respectively. The result clearly showed that the RHA incorporated as the SCM gave the required target strength and the IC agents improved the compressive strength of the concrete while the effect of the MgSO<sub>4</sub> solution on compressive strength decreases as curing age increases. This is understood to be the result of the RHA introduced as the SCM that served as the filler which produced additional C-S-H when reacted with calcium hydroxide and findings agrees well with reports presented by Kwan and Wong (2020) and Vieira (2020).

## IV. CONCLUSION

The outcome of this research work on the impact of Nigeria presoaked pumice and the RHA of Nigeria origin incorporated as the IC agent and SCM respectively on the sulphate resistance of HPC internally cured can be deduced as thus:

- Both SAP and presoaked pumice used in this study as IC agents improved the compressive strength of the HPCs at all ages (28, 56 and 90 day).
- MgSO<sub>4</sub> as a curing media resulted in slight loss in the mass and compressive strength values for all HPCs studied. The trend however decreased as the curing ages increased.
- PHPC<sub>1</sub> (i.e., RHA-based HPC with 5% presoaked pumice) performed best in resisting MgSO<sub>4</sub> attack by exhibiting the highest residual strength implying better resistance to chemical attack.

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