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Durable concrete in sewerage using non-grinded rice husk ash and water-permeable mould

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Abstract. There are increasing interests in using natural pozzolans as partial replacements for ordinary Portland cement (OPC) in concrete due to the benefit to the environment, low-carbon footprint, and durability improvement potentials. In the present research, open-air-burnt non-grinded rice husk ash (RHA) samples from Ganawuri-Plateau State, Nigeria, were used as a partial replacement for OPC in concrete. A water-permeable form (controlled permeability formwork – CPF) was utilized to counter the adverse effects of high-water demand. The combined effects of CPF and RHA on the cover-zone microstructure/porosity were analysed by the mercury intrusion porosimetry (MIP) test. Water sorptivity and sulphuric acid resistance properties were measured by Surface Water Absorption Test (SWAT) and accelerated sulphuric acid resistance test, respectively, to study the suitability of the concrete mixtures for sewerage concrete structures. Compared to Portland cement concrete, the RHA with CPF samples had relatively low permeability and low water sorptivity while the RHA without CPF samples showed the highest resistance to sulphuric acid attack, exhibiting no weight loss, no gypsum formation at the surface with the least surface discolouration.

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1. Introduction

The quest for sustainability has led to the massive utilization of natural pozzolans and various agricultural by-products in concrete production. Rice husk ash (RHA) is a good partial replacement for ordinary Portland cement for reducing permeability and increasing the acid resistance properties of concrete [1–3]. In several ways, it has been shown that RHA can be used to improve the strength and durability of concrete [2, 4, 5]. It has also been established by several researchers that due to the high siliceous content of RHA, it can resist acid attack to a significant degree [6].

Sulphate attack is the major cause of concrete deterioration in the sewerage environment. The attack generates microbial induced corrosion [7]. The formation of sulphuric acid in a sewerage environment can simply be expressed as:

$$\text{sewage + sulphur reducing bacteria (SRB) + anaerobic condition= hydrogen sulphide } (H_2S) \tag{1}$$

$$H_2S \text{ + aerobic condition + oxygen= sulphuric acid } (H_2SO_4) \tag{2}$$

Microbial induced corrosion that results from the interaction of hydrogen sulphide and thiobacillus bacteria [2, 3] damages concrete structures, especially sewerage collection systems [9]. Olmstead and Hamlin (1900) wrote the first literature that brought the knowledge of this form of concrete corrosion to the limelight [9]. Wei et al (2010) explained the understanding that they termed the biological and physiochemical processes associated with microbiologically induced concrete corrosion. Sulphur-oxidizing microbes colonize the concrete in the presence of sufficient moisture and nutrient [10]. Sewerage provides the sulphur-oxidizing microbes with sufficient moisture and nutrients in wastewater treatment plants. Several microbes participate in this early stage of colonization due to the high pH value (pH 12) of the surface of a non-deteriorated concrete. Alkaline-tolerant microbes are one of the early participant microbes. The biogenic oxidation of sulphur on the concrete lowers the surface pH creating more palatable conditions for further microbial colonization [10]. According to Ramezanianpour (2010), “sulphate attack is one of the most aggressive environmental deteriorations that affects the long term durability of concrete structures” [11]. The SO_4^{2-} ions from H_2SO_4 reacts with Ca $(OH)_2$ and transform into gypsum ($CaSO_4.2H_2O$). It then attacks the cement matrix leading to expansion and further cracking of the interior concrete structure [2]. This leaves the concrete more susceptible to direct and further attacks by H_2SO_4 [2].

An increase in pozzolanic reaction and fixing of $Ca(OH)_2$ by RHA yields additional C-S-H [12], increases density and resistance to penetration of deleterious substances [2]. Nonetheless, RHA decreases the workability of concrete due to high surface area [3] and require more water for proper mixing and adequate compaction than Portland cement concrete. This effect could harm the durability properties of concrete if not controlled. Chindaprasirt et al. (2007) could only attain the same workability in OPC mortar of 0.55 water-to-cement ratio using 0.68 water-to-binder ratio when 20 % OPC was replaced with RHA [3]. Moreover, investigations have shown that RHA increases water demand in fresh concrete [1–4]. A greater percentage of the water is primarily for workability, placement, and compaction. It is important to seek a method for expelling excess water from fresh RHA concrete after achieving proper placement and compaction.

The production of high-performance concrete by blending RHA has been widely established and adopted in construction but not yet appreciated by many agricultural-based developing countries like Nigeria because of requirements in technology and cost for controlled incineration and grinding. Location, type of burning and grinding affect the chemical composition of RHA and the resultant properties of concrete. Therefore, advances in research for the utilization of RHA for different purposes are localized. “In particular, durability of concrete structures depend primarily on the permeability of the outer concrete cover, which generally is a result of production standard, casting conditions, compaction in heavily reinforced areas and curing conditions”[13]. Therefore, several studies are being carried out on how the resistance to mass transfer at the cover zone of concrete can be improved.

Controlled permeability formwork (CPF) - first developed in Japan in the early 1980s [14] is one of the techniques that could be used to improve the quality of concrete at the cover zone [13–15]. CPF is primarily composed of a textile liner that is tensioned on a conventional formwork allowing the drainage of excess water while retaining the cement paste and at the same time expelling air bubbles from freshly placed concrete[13, 15]. CPF can reduce up to 20 % water-to-cement ratio at the cover zone [14], thus, improving the durability of the concrete by reducing the permeability [14, 16]. Nonetheless, this does not prevent the problem resulting from poor compaction where applicable or aggregate nesting induced by lack of paste [13].

The objective of the paper is to evaluate the effects of RHA and CPF on the microstructure, resistance to water ingress, and sulphuric acid resistance properties of the cover-zone of concrete blended with different amounts of an open-air-burnt non-grinded rice husk ash from Nigeria (RHA) for possible utilization in the durability and sustainability designs of sewerage concrete structures.

2. Materials and Methods

2.1. Materials and specifications

A total of 48 samples consisting of 12 concrete types were prepared with OPC blended with RHA at 0 %, 10 % and 20 % by weights of the OPC. The 12 mix designs were selected from among the commonly used concrete in Nigeria and the mix proportions are shown in Table 1a.

The rice husk was obtained from a local rice mill in Ganawuri town, Riyom Local Government Council of Plateau State, Nigeria. It was dried in the open air and burnt under atmospheric conditions. RHA that passed through a 6 µm laboratory sieve with chemical properties shown in Table 1b was utilized for the concrete. The steps for processing the RHA are simplified in Fig. 1a. ED-XRF chemical analysis showed that the RHA could be classified as Type N pozzolanic material as per ASTM C618 classification because the percentage sum of SiO_3, Al_2O_3 and Fe_2O_3 components is greater than 70. Also, the SO_3 component of the RHA is not higher than 4 %.

Fine aggregate used was natural silica pit sand. It was sieved with a 5 mm aperture experimental sieve and washed to remove impurities it might contain. Surface dry density of the sand is 2.635 gcm^3. Crushed granite coarse aggregate was used. The aggregate size ranges from 5 mm to 20 mm with a surface dried density ~ 2.690 gcm^3, an absolute dried density ~ 2.663 gcm^3 and a water absorption rate of 1.00. The aggregate was used at a surface saturated condition and the water used was tap water.

Two types of moulds – conventional metal mould (N) and controlled permeability formwork (CPF) measuring 100 mm×100 mm×100 mm (Fig. 1b) were used. Three w/b ratios – 0.40, 0.50 and 0.60 were used and denoted as 40, 50 and 60 respectively. Water permeable sheets were used to construct the 100 mm×100 mm×100 mm CPF mould (Fig. 1b).

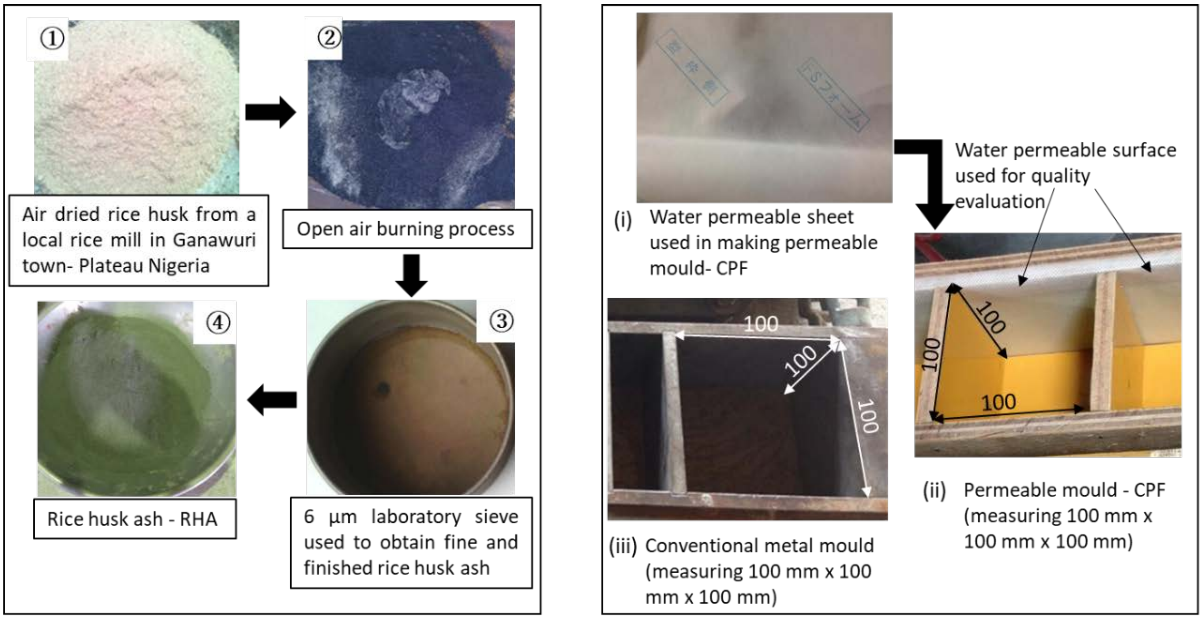
Table 1a. Concrete mix proportion.

Name of specimen	Binder type [1]	Mould type [2]	W/B (%)	RHA (%)	Mix composition (kg/m^3)			
					OPC	RHA	Coarse aggregate	Fine aggregate
40N *****	O	N	40	–	437	–	977	767
50N *****	O	N	50	–	437	–	911	716
50CPF	O	CPF	50	–	437	–	911	716
50RHA1-N	O-RHA	N	50	10	393.3	43.7	910	714
50RHA1-CPF	O-RHA	CPF	50	10	393.3	43.7	910	714
50RHA2-N *****	O-RHA	N	50	20	349.6	87.4	908	713
50RHA2-CPF *****	O-RHA	CPF	50	20	349.6	87.4	908	713
60CPF	O	CPF	60	–	437	–	847	665
60RHA1-N	O	N	60	10	393.3	43.7	845	663
60RHA1-CPF	O-RHA	CPF	60	10	393.3	43.7	845	663
60RHA2-N	O-RHA	N	60	20	349.6	87.4	844	662
60RHA2-CPF	O-RHA	CPF	60	20	349.6	87.4	844	662

Binder type [1] – O: ordinary Portland cement, RHA: open-burnt non-grinded rice husk ask
Mould type [2] – N: conventional metal mould, CPF: controlled permeability formwork
Asterisk (*): Samples used for MIP test

Table 1b. ED-XRF Chemical analysis of the properties of OPC and RHA.

Binder	SiO_2 (%)	Fe_2O_3 (%)	Al_2O_3 (%)	SO_3 (%)	CaO (%)	NiO (%)	CuO (%)	MnO (%)	K_2O (%)	Na_2O (%)
OPC	20.36	3.04	5.33	2.13	64.09	–	–	–	0.36	0.28
RHA	67.75	2.75	–	0.53	4.51	0.01	0.14	1.46	7.62	–
	MgO (%)	ZnO (%)	P_2O_5 (%)	TiO_2 (%)	Rb_2O (%)	BaO (%)	V_2O_5 (%)	Eu_2O_3 (%)	Cr_2O_3 (%)	Re_2O_7 (%)
OPC	1.50	–	–	–	–	–	–	–	–	–
RHA	–	0.10	11.70	0.39	0.09	0.01	0.01	0.08	0.04	0.03
	Specific gravity		Surface area (g/cm^2)							
OPC	3.16		3310							
RHA	2.11		–							



(a) (b)

Figure 1. Preparation of RHA material and moulds.
(a) Open-air burning and processing of RHA (b) metal and water permeable (CPF) moulds.

The curing condition applied was sealed curing for 28 days in the mould after casting the concrete. After curing, five sides of the specimen except for the side for measurements were sealed with aluminium tape and epoxy resin. Then, the specimens were kept in a controlled room condition (temperature – 20 °C and relative humidity – 60 %) and monitored until the time for measurements.

2.2. Test methods

Non-destructive and destructive tests were performed to measure porosity and transport properties of cover-zone of concrete and evaluate the durability in sewerage environment.

2.2.1 Surface moisture content

Surface moisture content was measured with a non-destructive electric-resistance type of moisture meter (model HI-100 of Kett Electric Laboratories Tokyo Japan) by pressing the device on the surface of the concrete for outputs expressed as count values. Appropriate initial surface moisture content has previously been confirmed by the author [17] and the upper threshold for the count values of the HI-100 moisture meter was established as 210 count values.

Moisture contents at the surfaces of the concretes that were not sealed with aluminium tape and epoxy resin were monitored until sufficiently dried cover zones that will ensure proper evaluations of the resistance to water ingress were attained. All the concrete samples (kept in a controlled room environment of a temperature of 20 °C and 60 % relative humidity) showed proper initial surface moisture contents at the age of 84 days.

2.2.2 Surface water absorption test (SWAT)

Surface water absorption test - SWAT (by Yokohama National University/Maruto Testing Company, Japan) [18] was used to measure the resistance to surface water ingress. SWAT measurements were conducted on the specimens 84 days after casting. The measurements were conducted in 20 °C and 60 % RH room condition. Coefficient of Surface Water Absorption- *CSWA* is the water sorptivity index used in the analysis. *CSWA* is the slope of an approximate linear regression between water absorption amount and the square root of time. The average value from four samples was used for the analysis.

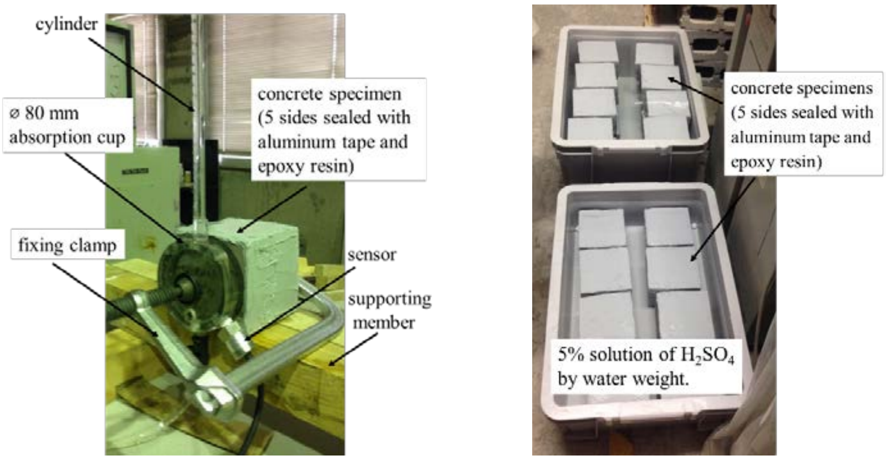
SWAT (Fig. 2a) is a simple non-destructive device that evaluates the quality of the covercrete at 10 minutes under natural dominant water suction [18, 19]. SWAT has proven to be effective in detecting the influence of curing conditions and the effects of microcracks in covercrete quality within 10–20 mm, which is the most affected by concreting works [19–24]. The rate of surface water absorption at 10 minutes (in which the time for pouring water is 10 seconds) by SWAT is termed p_{600} (in ml/m^2/s). Moreover, the new SWAT index (CSWA in ml/m^2/s$^{1/2}$) was recently introduced by the author and was found to be correlated with the JSCE standard sorptivity test results [23].

2.2.3 *Mercury intrusion porosimetry (MIP) test*

Mercury intrusion porosimetry (MIP) test was conducted on selected concrete specimens to evaluate the porosity and pore size distribution at the cover zone. Samples from the specimens marked with asterisks (*) in Table 1a were carefully selected for the MIP tests to evaluate pore size changes relating to CPF, metal mould, w/b ratio and RHA. After the SWAT test, a 100 mm concrete cube was used for the MIP test. The test was conducted at Denki Kagaku Kogyo (Co.) laboratory, Tokyo – Japan. The specimens were cut at a depth of 15 mm – 20 mm from the exposed surface. The cut samples were coarsely pulverized and subjected to acetone to stop further hydration of un-hydrated particles. Final test samples 2.5 mm – 5 mm squares weighing 2.3025 g, 2.3023 g, 2.5004 g, and 2.5008 g for 40N, 50N, 50RHA2-N and 50RHA2-CPF respectively were used. The volume of cement pastes and cement + RHA pastes in the test samples were 0.300658 ml for 40N, 0.354783 ml for 50N, 0.40139 ml for 50RHA2-N, and 0.40146 ml for 50RHA2-CPF. An AutoPore IV 9500 V1.09 was used for the test.

2.2.4 *Accelerated sulphuric acid resistance test*

Accelerated sulphuric acid resistance tests were conducted on 3 samples for each mix design to study the effect of partial replacement of OPC with RHA. Besides, the test measured the effect of CPF on the resistance to H_2SO_4 attack. Immediately after SWAT, the 3 samples used for the accelerated sulphuric acid resistance test were immersed in tap water for 4 days to avoid possible effects of concrete dryness on the test and to simulate real conditions in sewerage structures where water ingress occurs before H_2SO_4 forms and the later acid attack. After, the specimens were transferred into a 5 % solution of H_2SO_4 by water weight (Fig. 2b). The H_2SO_4 used was 98.08 % concentration. The parameters investigated were the time and weight changes of fully immersed concrete specimens with only one surface exposed to the acid solution. Weight changes of the concrete specimens were measured at 3, 7, 14, 21, 28, 56 days and the depth of scaling from the surface after 56 days following immersion.



(a) (b)

Figure 2. Test setups (a) Surface water absorption test (b) Accelerated H2SO4 test.

3. *Results and Discussion*

3.1. *Water sorptivity of concrete*

The influence of types of mould on time-dependent cumulative water absorption per unit area for the concrete cubes is presented in Fig. 3a while the water sorptivity at 10 minutes (CSWA in $ml/m^2/s^{1/2}$) is presented in Fig. 3b. Also, the initial surface moisture contents before the water absorption tests are shown in Fig. 3b.

Initial surface moisture contents measured by HI-100 moisture meter for the tested concrete samples range from 175 to 206 count values. As earlier mentioned, 210 count value of the HI-100 moisture meter has previously been established by the author as the upper threshold moisture content for effective evaluation of the resistance to water ingress property of concrete by SWAT [17]. It can be seen in Fig. 3b that before SWAT measurement, the surface moisture contents are slightly higher for samples cast with CPF than those cast with metal moulds. This indicates that CPF improved the quality by densifying the cover zone, reducing both the total pore volume and the pore diameters, thereby inhibiting rapid loss of moisture at the cover zone. Significant improvement in the resistance to water ingress for concrete cubes produced with CPF is observed as can be seen in Fig. 3b. A fifty-two per cent reduction in water sorptivity was observed for 50 % w/c OPC concrete. The water sorptivity results for 50N and 60-CPF specimens showed that CPF could effectively eliminate the 10 % w/c difference. Even with an additional 9 % reduction in sorptivity resulting from the use of CPF. For the RHA blended specimens, the maximum and minimum reduction in water sorptivity resulting from the use of CPF was 38 % and 19 % respectively.

The addition of RHA improved the resistance to water ingress properties of the concrete. This could be evidence that RHA yielded more C-S-H leading to an increase in the density and producing lower permeability. Just as expected, 50 % w/b contents revealed lower surface water absorption than 60 % w/b contents. Twenty per cent RHA showed slightly higher resistance to water absorption than 10 % RHA while combining RHA and CPF significantly improved the resistance to water absorption.

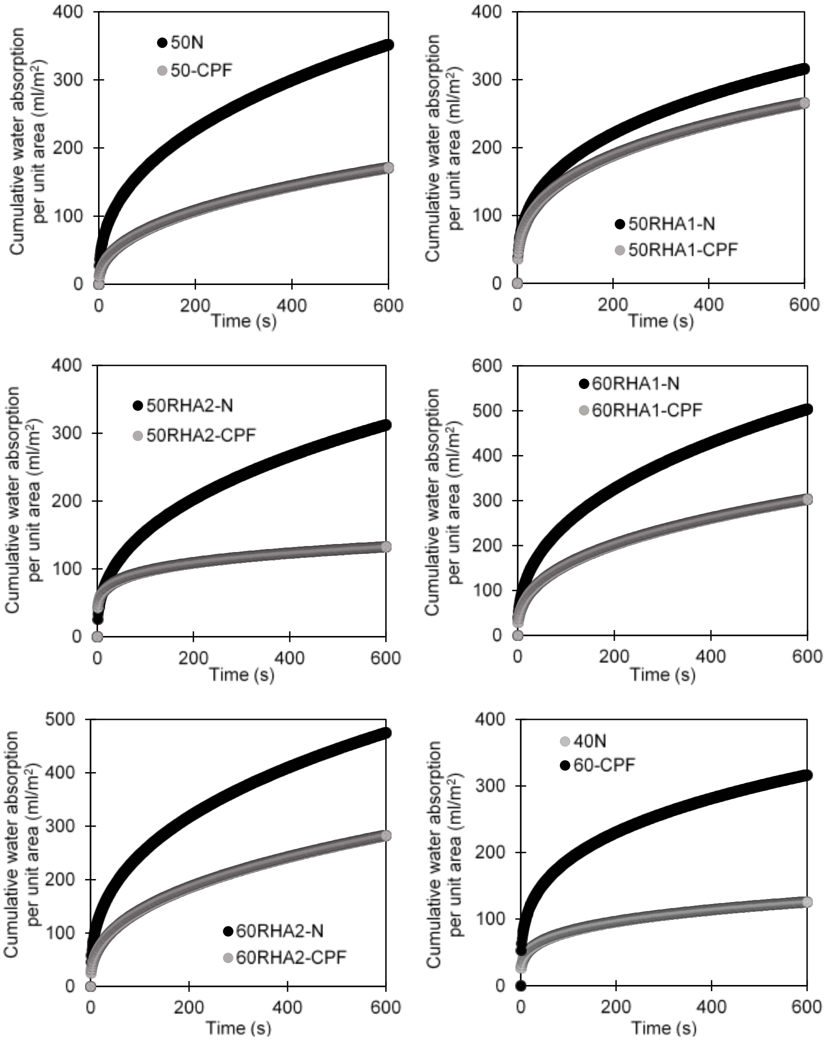


Figure 3a. Influence of type of mould on time-dependent cumulative water absorption per unit area for the concrete cubes.

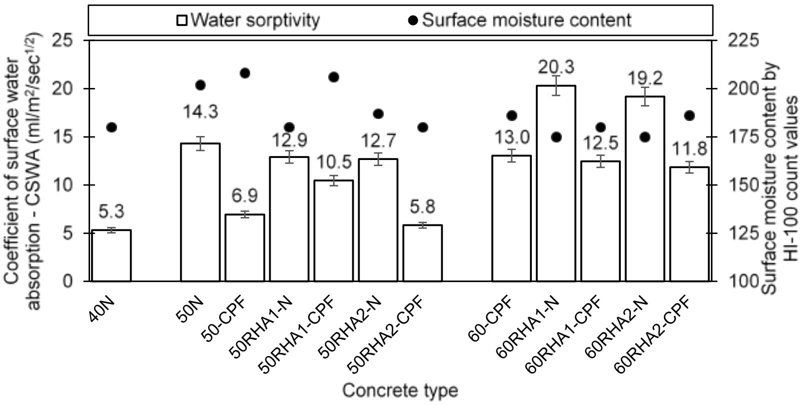
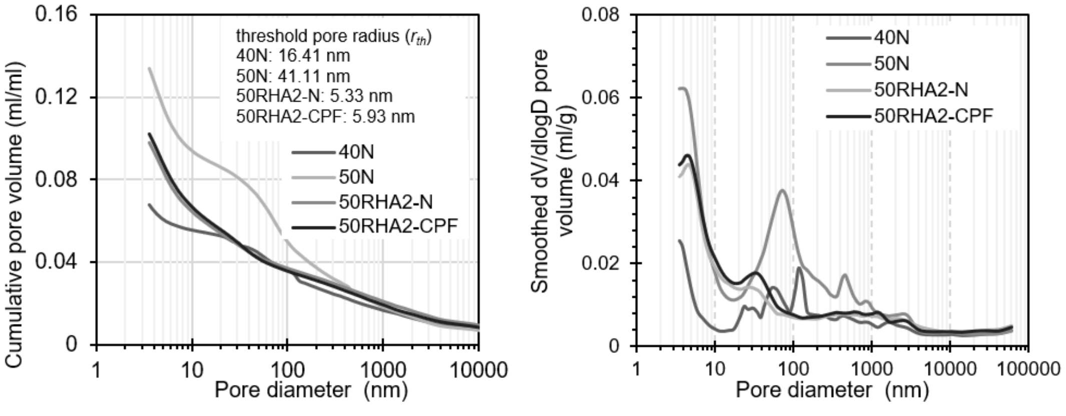


Figure 3b. Water sorptivity and initial surface moisture content of the concrete cubes.

3.2. Porosity and pore size distribution of concrete

A plot of cumulative pore volume against the pore diameter from the MIP measurements is presented in Fig. 4a while the pore size distribution is presented in Fig. 4b. It can be inferred from the results that intrudable porosity for the four concrete types is nearly the same pore diameter. They are 3.556 nm for 40 N, 50 N and 50RHA2-CPF and 3.889 nm for 50RHA2-N. Intrudable porosity (ϕ_{in}) is obtained as the smallest equivalent pore diameter that corresponds to the highest point on the cumulative porosity curve [24]. The quantitative threshold pore diameter (d_{th}) is calculated using the popular tangent method and confirmed with the Sakai C. et al novel method that derived its "basis on the empirical critical volume fraction for percolation"[25]. The method assumes that when 16 % of the cement paste is filled with mercury during the MIP test, the corresponding pore radius is the threshold pore radius [25]. Quantitative threshold pore diameter (d_{th}) is the largest pore diameter above which a comparatively little mercury intrusion occurs and below which a large volume of mercury intrusion starts [24]. The threshold pore radius for the concrete samples is 16.41 nm for 40 N, 41.11 nm for 50 N, 5.33 nm for 50RHA2-N and 5.93 nm for 50RHA2-CPF. The total porosity (in %) for 40N is 6.7888, 50N is 13.3998, 50RHA2-N is 9.8040 and 50RHA2-CPF is 10.2262. Threshold pore radius is defined as "the minimum pore size through which a mass has to pass for penetration to be possible" [25].

The addition of RHA significantly reduced the porosity and the threshold pore radius obtained from the cover zone of the concrete. Replacing 20 % of OPC with RHA reduced the total porosity by ~ 27 % for 50RHA2-N and ~ 24 % for 50RHA2-CPF compared to 50N. Also, the threshold pore radius improved about seven times. It is seen that the porosity for the 50RHA2-CPF sample increased by ~ 5 % because of CPF. Likewise, the threshold pore radius for the 50RHA2-CPF sample increased by ~ 10 %. Nevertheless, the increase in porosity and threshold pore radius are insignificant owing to heterogeneity of concrete and other major factors such as pore continuity/connectivity that affects permeability. The shift in the pore size distribution for the RHA blended specimens is reflected in the calculated threshold pore radius (r_{th}). The range of capillary pores that define continuity/connectivity of the pore system (ϕ10 nm to ϕ100 nm), which is the main indicator of the durability of concrete [26, 27], are dominant in 50N specimen. When the volume of pores in this range is high, it could indicate more interconnectivity and continuity of the pores and could translate into a higher permeability. Furthermore, large capillary pores (above ϕ100 nm) that have a direct influence on mass transfer [26–28] are seen more in specimens without RHA. These are evident in the water sorptivity values obtained from these specimens.



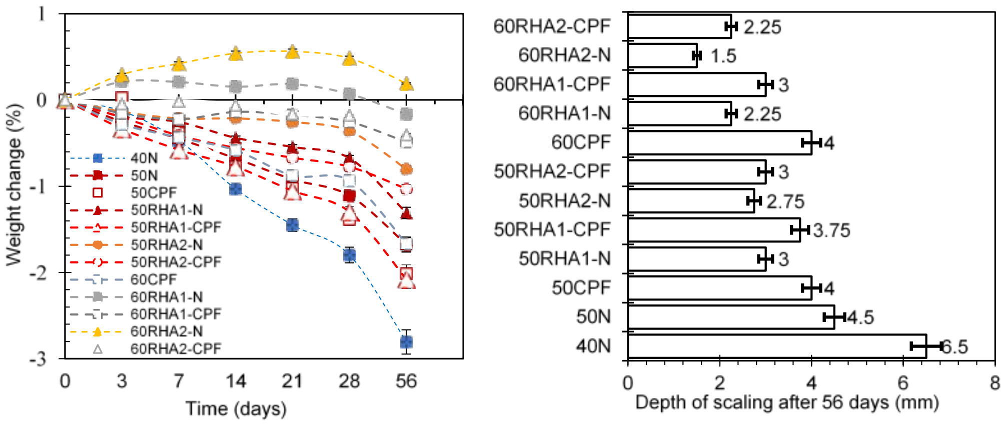
(a) (b)

Figure 4. (a) Cumulative pore volume vs pore diameter (b) Pore size distribution.

3.3. Accelerated sulphuric acid resistance

The results of the accelerated H_2SO_4 resistance tests are presented in Fig. 5. Fig. 5a shows the time-dependent weight change of concrete in 5% H_2SO_4 solution while Fig. 5b shows the depth of scaling after 56 days of immersion. A value of $R^2 = 0.8537$ was obtained in the linear regression analysis between the two indices.

As the percentage change in weight of the concrete specimens is analysed, it is inferred that the addition of RHA influences this parameter. The first clear trend is the reduction in the percentage of weight loss shown in Fig. 5a. Second, the inclusion of RHA reduced the depth of scaling (Fig. 5b). Regardless of the RHA dosage, its addition significantly improved the resistance to sulphate attack. Specimens with 60 % w/b contents showed the lowest weight loss. The lesser attack of H_2SO_4 on RHA blended concrete specimens could be resulting from a reduction in tricalcium aluminate (C_3A) and tricalcium silicate (C_3S) contents of the concrete and the lower sulphate adsorption by C-S-H. High C_3A in cement proves less resistance to sulphate attack, evident in the British Standards (BS 4027) stipulating a maximum of 3.5 % C_3A content in Portland cement for sulphate resistance. Besides, the reaction that causes concrete deterioration occurs between the sulphate and hydrates from calcium hydroxide (CH). CH is significantly generated by tricalcium silicate (C_3S) [11], and it is known that RHA reduces the C_3S content, which is the main cause of the low early age strength of RHA blended concretes. Visual evaluations revealed no gypsum formation on the surfaces of the RHA blended concretes. This suggests that the RHA countered to a significant value, the sulphate attack that reduces the stiffness of the hydrated pastes that could have been followed by large expansion and scaling due to ettringite formation.



(a) (b)

Figure 5. (a) Time-dependent weight change of concrete in 5% H_2SO_4 solution (b) Depth of scaling after the 56 days of immersion

4. Conclusions

In this study, durability performance of OPC concretes blended with 10 % and 20 % open-air-burnt non-grinded rice husk ash (RHA) and cast with conventional metal mould and water permeable mould (CPF) was investigated to evaluate potential utilization for sewerage concrete structures. From the result of the ED-XRF chemical analysis of the RHA, the MIP test, water sorptivity and accelerated acid resistance results of concretes with 40 %, 50 % and 60 % water-to-binder contents, the following conclusions are obtained:

1. The open-air burnt non-grinded RHA in this research could be classified as a Type N pozzolanic material as per ASTM C618 classification because the percentage sum of SiO_3, Al_2O_3 and Fe_2O_3 components is greater than 70. In addition, the SO_3 component of the RHA is not higher than 4 %.

2. The combination of 10 % OPC replaced with RHA and conventional metal mould did not significantly improve the water-resistance properties of the cover concrete, like for the combination of 20 % OPC replaced with RHA and a conventional metal mould. However, the water resistance properties of the 10 % and 20 % RHA concrete could be significantly improved by introducing CPF.

3. The replacement of 10 % and 20 % OPC with RHA provided significant resistance to sulphuric acid attack, to a greater extent than the conventional OPC concretes. These were applicable when cast with both conventional metal mould and CPF. Nonetheless, RHA concretes produced with conventional metal mould could provide greater resistance to H_2SO_4 attack than the ones produced with CPF.

4. A significant reduction of porosity and threshold pore diameter of concrete could be achieved by replacing OPC with 10 % or 20 % open-air-burnt non-grinded rice husk ash (RHA).

5. Open-air-burnt non-grinded rice husk ash from Nigeria (RHA) is a good Supplementary Cementitious Material (SCM) and could effectively be utilized in durability and sustainability designs of sewerage concrete structures.

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