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INVESTIGATION ON THE DURABILITY OF CONCRETE WITH RICE HULL ASH (RHA) USING SORPTIVITY AND RAPID CHLORIDE MIGRATION TESTS

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ABSTRACT: Rice hull ash (RHA) is an agricultural residue that typically exhibits pozzolanic properties. In this study, RHA was used as a partial cement replacement in concrete. Physical and chemical characterization tests using X-ray Fluorescence (XRF) and X-ray Diffraction (XRD) revealed that the RHA is an amorphous material with over 90% SiO_2. Concrete specimens with 5% and 10% RHA percentage replacement by weight were prepared. After 28 and 56 days of curing, the water sorptivity, and chloride migration were evaluated. Results showed that for the sorptivity tests, all specimens containing RHA exhibited lower sorptivity values after 28 and 56 days of curing relative to the control. The rapid chloride migration test showed that the concrete specimens with RHA consistently outperformed the standard concrete, further indicating that the addition of RHA increased the durability of concrete. From the results, RHA can be used as a viable partial cement replacement up to 10 wt%, with a marked improvement in durability properties. The use of RHA as partial cement replacement can result in a more environmentally friendly concrete.

Keywords: *Rice hull ash, durability, Supplementary cementitious material (SCM), Sorptivity, Chloride migration*

1. INTRODUCTION

Concrete is one of the most widely used materials in the world. Its use in construction is approximately twice that of other construction materials combined, finding use in various structures such as roads, buildings, dams, and harbors [1,2]. Cement, the main component of concrete, has a predicted compound annual growth rate (CAGR) of 5.1% from 2022 to 2029 in the Asia Pacific alone [3]. Cement production, however, brings about several environmental damages, as it involves raw materials being mined, transported, crushed, and burned at high temperatures (1200°C–1450°C), with the whole process requiring a significant amount of fossil fuels and energy [4]. The cement industry is responsible for 7% of the global carbon dioxide emissions [5]. Therefore, the use of alternative materials to lessen the use and production of cement, without sacrificing performance, should be explored.

Pozzolanic materials are siliceous and aluminous materials that are not cementitious but form cementitious compounds when mixed with the calcium hydroxide [$Ca(OH)_2$] of cement during the hydration process. $Ca(OH)_2$ or CH, in cement chemistry notation, is one of the by-products of the hydration of tricalcium silicate (C_3S) and dicalcium silicate (C_2S), but it is relatively weak and soluble in water, thus providing little contribution to the overall strength of the concrete product. However, its mixture with siliceous materials (i.e., silicon dioxide or silica) and water results in the formation of calcium silicate hydrates (C-S-H), which is beneficial to the properties of concrete [6]. Therefore, the use of pozzolanic materials as partial cement replacement can compensate for the partial loss of concrete strength due to the reduced cement content. In many cases, it can even enhance the strength and durability properties of concrete.

Many studies have investigated the use of pozzolanic materials or supplementary cementitious materials (SCM's) as partial cement replacements [7]. One such SCM that can be widely utilized in the field is rice hull ash (RHA), a byproduct of rice hull combustion. It is rich in silica content, containing around 85-96% [8]. RHA can be amorphous when produced in controlled combustion with burning temperatures ranging from 500°C–700°C or crystalline when produced through uncontrolled combustion or open-field burning with temperatures greater than 700°C [9]. Generally, amorphous RHA performs better compared to crystalline RHA due to its higher reactivity with cement [9]. Furthermore, the utilization of amorphous RHA in concrete can provide several advantages, such as higher strength, improved durability properties, and reduced materials cost [10]. Nevertheless, crystalline RHA can still be utilized as pozzolanic material if the particle size can be made smaller, down to the micrometer range, which can result in higher surface area per unit volume which, in turn, may accelerate the pozzolanic reaction and the filler effect [11].

In this study, the performance of concrete with RHA was assessed by evaluating the sorptivity, and rapid chloride migration of concrete specimens. Sorptivity is one of the primary measures of the durability of concrete. A lower sorptivity means that concrete is considered more resistant to water absorption. In contrast, a high sorptivity coefficient suggests the presence of a well-linked porous structure or a low tortuosity of the pore network [12]. Rapid chloride migration is another measure of the permeability and penetrability of concrete. In general, a less porous concrete is less susceptible to chloride attack [13]. In a way, the rapid chloride migration test complements the sorptivity test.

2. RESEARCH SIGNIFICANCE

Various methods of producing RHA have been conducted in previous studies [14,15,16]. In the study of Quiring [14], the crystalline RHA with 89% silica content was produced through uncontrolled burning. The study of Chao-Lung and co-workers [15] produced RHA using a steam boiler. The RHA was found to be mainly amorphous with a partial crystalline character. In the study of the group of Marthong [16], the RHA which was produced using a furnace has a low silica content of only 75%. Fig. 1-3 show images of RHA that are typically obtained from uncontrolled burning and from steam boilers.

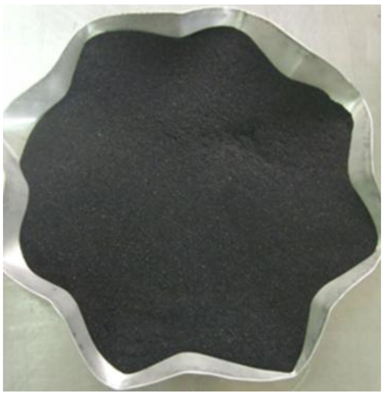

Fig. 1. RHA produced by Quiring [14]

Fig. 2. RHA from a local waste-to-energy plant.

Fig. 3. RHA produced by Elarde and co-workers [17].

In this study, RHA was used to test its effects on the durability properties of concrete, specifically the sorptivity and rapid chloride migration. As compared to the RHA used in other studies, the RHA produced in this study has a lighter color due to its low carbon content.

3. METHODS

3.1 Materials

ASTM standards were followed in the preparation and determination of the properties of concrete components. The cement used is Type I Ordinary Portland Cement (OPC). The specific gravity of produced RHA is 1.91. Gravel was used as coarse aggregate, with a specific gravity (SG), absorption capacity (AC), and dry-rodded unit weight of 2.47, 3.13%, and 1,492.5 kg/m^3, respectively. It was sieved to attain a maximum aggregate size (MAS) of 19 mm. ASTM C127 was followed for the SG and AC of coarse aggregate while ASTM C29 was followed for the dry-rodded unit weight.

Sand with a fineness modulus (FM) of 2.36, SG of 2.51, and AC of 3.61% was used as fine aggregate. ASTM C136 was followed for the FM while ASTM C128 was followed for the SG and AC of fine aggregate.

3.2 Methods

Fig. 4 illustrates the collected rice hull ash obtained after the combustion process. In order to ensure that the particles of the ash meet the appropriate size requirement for use as a partial cement replacement, the RHA was carefully sifted through Sieve No. 100 which allowed only the finer particles to pass through. This step was necessary to allow uniformity of the ash when incorporated into the mixture. To determine its material composition and classification, the RHA was characterized through X-Ray Fluorescence (XRF) and X-Ray Diffraction (XRD) analyses.

Fig. 4. Representative picture of RHA used in this study.

3.1.1 Mix proportion and casting of concrete specimens.

ACI 211.1 (Standard Practice for Selecting Proportions for Normal, Heavy Weight, and Mass Concrete) was followed in determining the appropriate proportion of concrete mixture components. The mix design was assumed to have a slump of 75-100 mm, water-to-binder ratio of 0.5, and non-air-entrained concrete. Table 1 shows the proportions of raw materials for the mix design, in kg per m^3 of concrete with varying replacement percentages of RHA.

Table 1. Proportions of raw materials for the mix design, in kg per m^3 of concrete.

	Control	5 wt% RHA	10 wt% RHA
RHA, kg	0	21	41
Cement, kg	410	390	369
Water, kg	201	201	201
Fine aggregate, kg	505	575	564
Course aggregate, kg	1016	1016	1016

The molding of the specimen was done after the acceptable value of the slump was achieved. Vibrator was used to consolidate the concrete and minimize the air pockets in the fresh concrete. The fresh concrete samples were then allowed to harden for 24 hours, cured underwater for 28 and 56 days, and tested. A total of 25-Ø100x200mm cylindrical concrete specimens were cast. The specimens were cured by immersion in a curing tank after one day of casting. All these procedures were done according to ASTM C31: Standard Practice for Making and Curing Concrete Test Specimens in the Field.

3.1.2 Sorptivity

A total of 20-Ø100x50mm concrete disk specimens extracted from 10-Ø100x2000mm parent concrete were tested for sorptivity following ASTM C1585-04 or Standard Test Method for Measurement of Rate of Absorption of Water by Hydraulic Cement Concretes. The specimens were oven-dried at 50 ± 2˚C for 3 days and preconditioned for 15 days at 23 ± 2°C to achieve moisture equilibrium. Before subjecting the specimens to a sorptivity test, their weight and dimensions were measured, and they were covered by duct tape and plastic sheets to ensure that only one surface was in contact with water. A support device was also used to ensure that the exposed surface was 1-3 mm below the water level. Shown in Fig. 5 is the actual setup of the experiment.

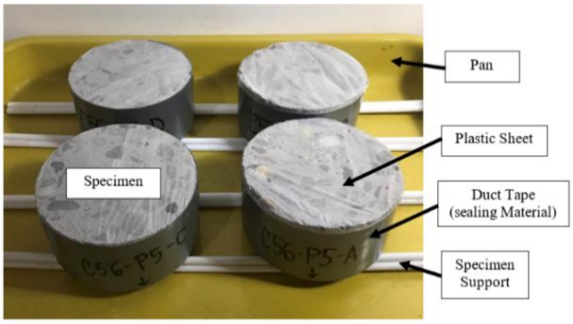

Fig. 5. Actual set-up of the sorptivity test.

Specimens were weighed at time intervals given in ASTM C1585-04. The weight of specimens was recorded and tabulated to compute the rate of absorption (I) through Eq. (1). Sorptivity was determined as the slope of the best-fit line through plotted absorption (mm) against the square root of time ($sec^{0.5}$). It was also made sure that the dataset for each trial or specimen followed a linear relationship with a correlation coefficient of not less than 0.98. Initial sorptivity was measured during the first 6 hours, and secondary sorptivity was measured from the first 24 hours until day 7 when specimens and water came into contact.

$$I = \Delta m/(A \times \rho) \qquad (1)$$

where I is the absorption (mm), Δm is the change in mass(g), ρ is the density of water (0.001 g/mm^3), and A is the exposed cross-sectional area of the specimen (mm^2).

3.1.3 Chloride Migration

The chloride ion penetration resistance was measured in terms of the chloride migration coefficient using the rapid migration test as per NT build 492. Each Ø100x50mm concrete specimen was fitted in a fabricated PVC pipe with the application of elastomeric sealant to avoid significant leakage. The sleeve was filled with 300 ml of 0.3 M NaOH solution (anolyte), and the initial temperature was recorded before placing it inside a catholyte reservoir. The said reservoir contains around 12 liters of 10% NaCl solution (catholyte). A DC potential of 30V was applied through the anode and cathode by submerging them within the solutions. The migration setup was left connected to the power supply for 24 hours as shown in Fig. 6.

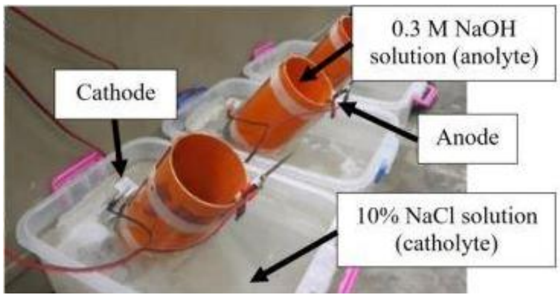

Fig. 6 Chloride migration set-up.

After the set period, the final temperature measurement was conducted. The specimens were removed from the reservoir and fractured for the determination of penetration depth. A 0.1 M silver nitrate solution was sprayed over the section and after several minutes, a white silver chloride precipitation was visible. This was utilized for the measurement of the penetration depth.

Recorded data were substituted to the simplified version of the Nernst-Einstein equation as per NT build 492 for the computation of the non-steady migration coefficient as shown in Eq. (2).

$$\boldsymbol{D} = \frac{\mathbf{0.0239(273+}\boldsymbol{T}\mathbf{)}\boldsymbol{L}}{\mathbf{(}\boldsymbol{U}\mathbf{-2)}\boldsymbol{t}}\left(\boldsymbol{x_d} - \mathbf{0.0238}\sqrt{\frac{\mathbf{(273+}\boldsymbol{T}\mathbf{)}\boldsymbol{Lx_d}}{\boldsymbol{U}\mathbf{-2}}}\right) \quad (2)$$

where D is the non-steady state migration coefficient (m^2/sec), U is the absolute value of the applied voltage (V), T is the average value of the initial and final temperature in the anolyte solution (°C), L is the thickness of the specimen (mm), x_d is the average value of the penetration depths (mm), and t is the test duration (h).

4. Results and Discussion

4.1 Physical and Chemical Properties of RHA

From the XRF results shown in Table 2, the RHA is 90.59% silica. This is within the typical silica content of RHA [8]. Furthermore, the combined weight percentage of silica, aluminum oxide, and iron oxide at 95.57% is much higher than the 70% required minimum weight percentage of Class N pozzolan set by ASTM C618 (Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete).

Table 2. Chemical composition of RHA.

Chemical formula	Percent
SiO_2	90.59
Al_2O_3	4.66
Fe_2O_3	0.32
CaO	0.92
P_2O_5	0.07
K_2O	2.88
Others	0.56
LOI (750˚C)	3.914

XRD analysis of the RHA revealed that the RHA is amorphous, as evidenced by the broad hump at 20-30° (Fig. 7). This broad peak is characteristic of amorphous silica [18]. Although the temperatures during combustion most likely exceeded 700°C, these temperatures probably did not last long enough to transform the ash into its crystalline phase, preserving its pozzolanic potential [19]. Also, the amount of K_2O, a known catalyst of phase transition from amorphous silica to cristobalite, is not that high. However, it is significant enough that it may have contributed to the grayness of the ash. K_2O is known to dissociate at around 350 ℃ creating a blanket over the surface of the rice hull (RH) during combustion, trapping some of the carbon [20].

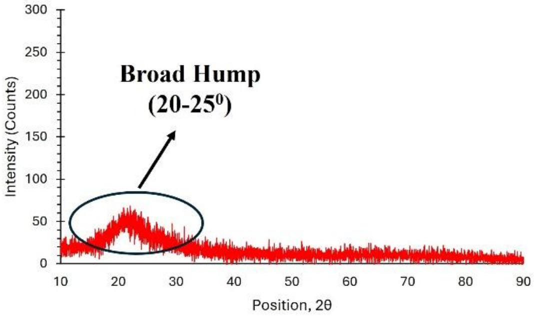

Fig. 7. Raw diffraction pattern of RHA Sample

The LOI of the RHA is also low at 3.9%. This is lower than the RHA of Chao-Lung [15] (LOI: 8.5%) and the locally procured RHA from a waste-to-energy plant (LOI: 7.5%). The low LOI of the RHA in this study is indicative of low carbon content which because of the porosity of carbon results in higher demand for water during mixing.

4.2 Sorptivity

Table 3 presents the average initial and secondary sorptivity values for varying RHA wt % and curing days, while Fig. 8-12 show the sorptivity plots for both initial and secondary sorptivity. The plots consist of the absorption I vs. the square root of time and are the average of 3-4 specimens. Almost all of the specimens exhibited linear behavior (r=0.98). For the secondary sorptivity values, saturation was observed at 6 or 7 days and therefore these absorption values were not included in the plots. For the specimens with RHA cured for 56 days, a transition from initial to secondary sorptivity was observed and secondary sorptivity measurements were started at 3 days onwards.

Table 3. Average sorptivity results of concrete with RHA at different wt % at 28- and 56-day curing periods.

Specimen	Sorptivity (mm/sec$^{0.5}$) Initial	 Secondary
Control – 28d	0.01030^{a}	0.0024^{a}
5 wt% RHA – 28d	0.00614^{b}	0.0010^{b}
10 wt% RHA – 28d	0.00666^{b}	0.0013^{b}
5 wt% RHA – 56d	0.00786^{b}	0.0007^{c}
10 wt% RHA – 56d	0.00860^{b}	0.0012^{d}

*In a column, sorptivity values followed by the same letter are not significantly different. Values are averages of 3-4 specimens.

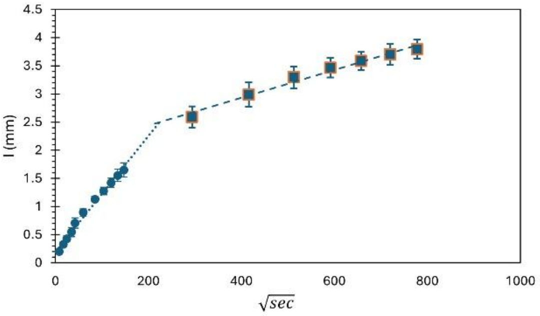

Fig. 8. Sorptivity plot for control concrete specimens. Error bars represent the standard deviation of the absorption values.

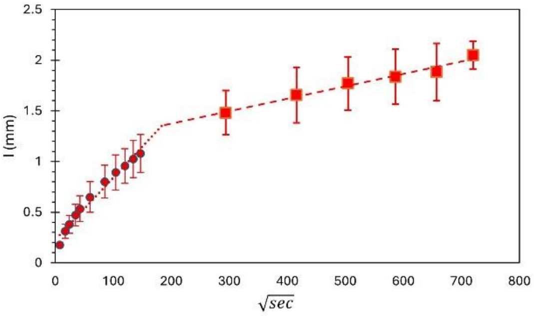

Fig. 9. Sorptivity plot for 5 wt% RHA-28d concrete specimens. Error bars represent the standard devation of the absorption values.

For initial sorptivity, the control specimen yielded the highest value of 0.0103 mm/sec$^{0.5}$. The initial sorptivity lowered by 16-42% as RHA replaced cement for both the 28- and 56-day-cured specimens, with 5 wt%-28d having the lowest value at 0.00614 mm/sec$^{0.5}$.

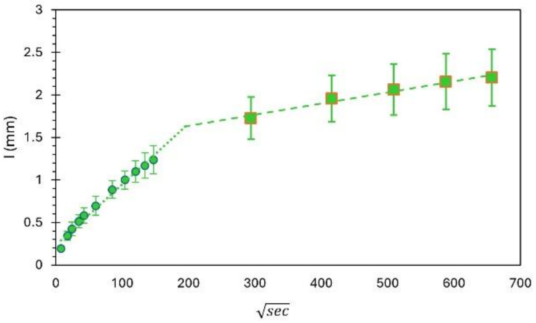

Fig. 10. Sorptivity plot for 10 wt% RHA-28d concrete specimens. Error bars represent the standard deviation of the absorption values.

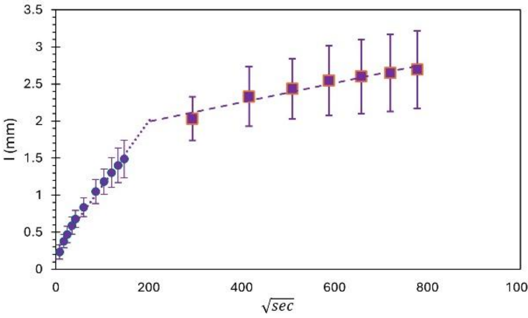

Fig. 11. Sorptivity plot for 5 wt% RHA-56d concrete specimens. Error bars represent the standard deviation of the absorption values.

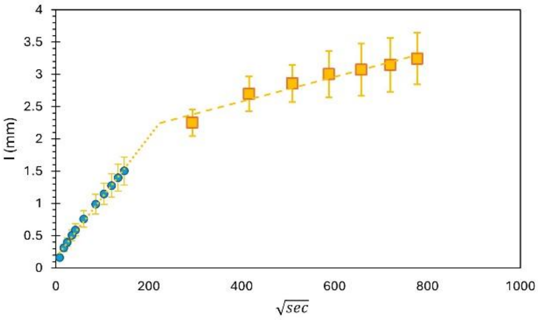

Fig. 12. Sorptivity plot for 10 wt% RHA-56d concrete specimens. Error bars represent the standard deviation of the absorption values.

All sets of specimens are significantly lower from the control specimen ($p<0.05$) except the initial sorptivity of 10 wt% RHA-56d which was nevertheless equivalent to the control. The 5 wt% RHA replacement was found to be the optimum RHA replacement in terms of sorptivity due to it exhibiting the lowest sorptivity values. However, concrete with 10 wt% RHA replacement is more economical and with similar sorptivity properties.

It was also observed that considering the same RHA percentage replacement, there was an increase in sorptivity value as specimens were cured for 56 days. These changes were found to be insignificant for 5 wt% RHA but significant for 10 wt% RHA for initial sorptivity. For secondary sorptivity, these increases were found to be insignificant for both 5 and 10 wt% RHA. This could be because of the difference in the initial saturation level of the specimens. When the saturation level is low at the start of the experiment, the initial sorptivity tends to be higher. But in the secondary sorptivity, the capillary pores near the surface are already saturated, thereby resulting in no significant difference in the secondary sorptivity.

Moreover, an increase in both initial and secondary sorptivity was observed from 5 wt% to 10 wt% RHA; however, these differences were found to be statistically insignificant except for the secondary sorptivity at 56 days wherein there was a slight increase in secondary sorptivity from 5-10 wt% RHA ($p>0.05$) but nevertheless was still significantly lower than the control. The 5% increase in RHA replacement does not substantially affect the sorptivity of concrete. The results are in general agreement with those found in the literature [13, 21].

The lower sorptivity values of concrete specimens with RHA suggest lower porosity of the specimens. These suggest that RHA particles underwent pozzolanic reaction as well as exhibited the filler effect, where the RHA particles were able to significantly fill up the pores of the concrete and form additional C-S-H gels [13, 22]. These additional CSH gels, besides filling up the pores, make the concrete more durable even with less cement present in the mix.

4.3 Rapid Chloride Migration

Shown in Table 4 are the computed chloride migration coefficient of the concrete specimens based on the varying RHA replacement and curing periods. Groups with a 28-day curing period produced migration coefficients of 19.3582×10^{-12} m^2/sec, 10.1609×10^{-12} m^2/sec, and 10.9896×10^{-12} m^2/sec for 0 wt%, 5 wt% and 10 wt% RHA, respectively. Moreover, groups with a 56-day curing period have 5.2499×10^{-12} m^2/sec, and 8.4882×10^{-12} m^2/sec for 5 wt% and 10 wt% RHA, respectively. It was observed that the control group has the highest migration coefficient produced. With the addition of the RHA replacement and longer curing period, the value tends to decrease with these factors. The obtained values can be interpreted using the established criteria as shown in Table 5 [23].

From the criteria, the control group was categorized as “Not suitable for aggressive environment”. Specimens from 5 wt%-28d, 10 wt%-28d, and 10 wt%-56d were considered to have “moderate resistance against chloride ingress”. Lastly, the 5 wt%-56d showed the highest resistance among the groups having a “good resistance against chloride ingress”. It is clear from the results that the addition of RHA significantly enhances resistance of concrete against chloride ingress, particularly at 56 days curing. These results are in general agreement with those stated in the literature [13,21,24,25,26,27], wherein incorporating 5-20 wt% RHA was found to improve the resistance of concrete against chloride ingress.

Table 4. Chloride migration coefficients of concrete with and without RHA at different 28- and 56-days curing.

Specimen	Chloride Migration Coefficient, m^2/sec, Average (SD)
Control – 28d	19.36 (0.61)
5 wt% RHA – 28d	10.16 (2.48)
10 wt% RHA – 28d	10.99 (0.75)
5 wt% RHA – 56d	5.25 (1.58)
10 wt% RHA – 56d	8.49 (0.97)

Table 5. Interpretation of migration coefficient.

Computed Migration Coefficient	Remarks
$D < 2 \times 10^{-12}$ m^2/sec	Very good resistance against chloride ingress
$D < 8 \times 10^{-12}$ m^2/sec	Good resistance against chloride ingress
$D < 16 \times 10^{-12}$ m^2/sec	Moderate resistance against chloride ingress
$D > 16 \times 10^{-12}$ m^2/sec	Not suitable for aggressive environment

4.4 Mechanism of action of RHA and implications on durability

It has been well established from the literature [10] that the silica in RHA lowers the porosity of concrete by increasing the packing density and enhancing the interstitial transition zone (ITZ) between the matrix and the aggregate [27]. The silica then reacts with C-H to form C-S-H to further fill up the pores among the cement grains [28]. These reactions, the filler effect, and the pozzolanic reaction improve the pore structure of both the cement paste and the ITZ [29] as well as reduce the width of the ITZ [30].

The pore refinement and ITZ enhancement of the RHA results in an increase in the mechanical properties of concrete, particularly compressive strength. It has been found that for normal concrete, the optimum wt% replacement of RHA is 10-15% [31]. For durability properties, 20-30% wt% replacement are claimed [13]. However, it should be noted that beyond 10 wt% RHA, the workability of concrete is reduced such that the use of superplasticizers becomes necessary [13]. Hence, for this study, only 5-10 wt% RHA was evaluated. It was desired to produce a highly durable concrete without sacrificing the mechanical properties as well as its workability.

Performing sorptivity and rapid chloride migration tests in tandem gives a more complete picture of the durability of concrete. Sorptivity is a measure of pore conditions on the surface, while chloride migration is a measure of pore conditions within the bulk of the material. The larger and more interconnected the pores are, the greater the transport properties will be. Concrete with good durability properties should not be prone to shrinkage cracking, due to high porosity, and corrosion, induced by chloride ingress. Given that the results of this study show that concrete with RHA exhibited lower sorptivity relative to the control and the rapid chloride migration results range from "moderate resistance" to "good resistance" to chloride ingress, it can be reasonably inferred that not only that porosity was reduced but the pore network was also somewhat disrupted. However, whether this disruption is due to less interconnectivity of the pores or binding of chloride to the C-S-H gels in the pores or a combination of both remains to be seen.

4 CONCLUSIONS

This study was conducted to determine the effect of varying parameters on the durability of concrete specimens with RHA as partial cement replacement and comparing it with OPC concrete. In conclusion, it has been found that:

1. RHA was characterized as an amorphous and light gray powder consisting of 90.59% silica with low carbon content (LOI: 3.9%).
2. Both initial and secondary sorptivity decreased significantly with 5 wt% and 10 wt% RHA for both 28- and 56-day-cured specimens relative to the control, indicating lower porosity and consequently, higher durability for concrete with RHA. This is further confirmed by the chloride migration coefficients obtained from the chloride migration test.
3. The optimum replacement in terms of durability for both the pozzolanic reaction and filler effect appears to be 5 wt% RHA although 10 wt% RHA is more economical. In terms of sorptivity and chloride migration, the use of RHA showed a marked increase in durability of concrete relative to OPC concrete. The study was also able to demonstrate that for concrete with up to 10 wt% RHA replacement, standard curing is sufficient.

An important key takeaway in this study that can be applied in practice and in research is that up to 10% RHA as cement replacement can be used in concrete to improve its overall durability while also making it more sustainable.

For future studies, it is recommended that SEM imaging be done on concrete specimens with RHA to definitively assess the pore refinement due to RHA or if an unknown mechanism is behind the observed results.

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