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Research article

Carbonation Resistance of Rice Husk Ash Concrete: Interplay Between Replacement Level, Transport Properties, and Carbonation Depth

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A B S T R A C T

A systematic parametric experimental investigation was conducted to evaluate the influence of rice husk ash (RHA) incorporation on the durability performance of concrete composites. Six RHA replacement levels, namely 5%, 10%, 15%, 20%, 25%, and 30%, were considered to assess the effects of increasing RHA content on key durability-related properties. Particular emphasis was placed on water permeability and carbonation resistance, which are critical indicators governing the long-term durability and service performance of cement-based composites. The experimental results demonstrated that the incorporation of RHA considerably enhanced the resistance of concrete to water penetration and carbonation. With increasing RHA replacement, the depth of water penetration and carbonation depth progressively decreased. These reductions, together with SEM observations of a denser cementitious matrix, are consistent with refinement of the transport network and reduced pore connectivity. Because pore-size distribution and pore connectivity were not directly quantified, pore refinement is interpreted as a plausible mechanism inferred from the combined permeability, carbonation, and microstructural evidence. These improvements may be attributed to the highly reactive silica present in RHA, which promotes pozzolanic reactions and the formation of additional cementitious hydration products, together with the physical filler effect of the fine RHA particles. Overall, the findings indicate that appropriately proportioned RHA can serve as an effective supplementary cementitious material for improving the durability and long-term performance of concrete composites.

1. INTRODUCTION

Concrete durability is the ability of concrete structures to sustain mechanical performance, structural integrity, and functional qualities when exposed to environmental and service-related deterioration processes over their specified service life [1]. Water permeability and carbonation are particularly important aspects in determining concrete durability because they have a direct impact on the movement of aggressive chemicals and the degradation of the protective environment surrounding steel reinforcement [2]. Water permeability represents the ability of water to penetrate and migrate through the interconnected pore network of concrete [3]. A highly permeable concrete generally contains a more connected capillary pore structure, facilitating the ingress of moisture and potentially harmful dissolved substances [3-5]. Reducing water permeability through pore refinement and improved microstructural densification is therefore essential for enhancing concrete durability.

Carbonation is another significant degradation mechanism in reinforced concrete. It happens when ambient carbon dioxide penetrates the concrete and combines with alkaline hydration products, specifically calcium hydroxide, gradually lowering the internal pH [6-8]. When the carbonation front approaches the reinforcement, the passive protective layer around the steel may become unstable, increasing the risk of corrosion [9, 10]. As a result, carbonation depth is commonly utilized as an essential metric for evaluating durability and Long-term performance of concrete. Rice husk ash (RHA) is an agricultural byproduct produced by the controlled combustion of rice husks and is increasingly recognized as a promising supplementary cementitious material (SCM) for long-term concrete production [11-14]. When formed under the proper combustion circumstances, RHA has a high silica content and is primarily amorphous. Its performance is significantly impacted by silica concentration, amorphous phase, specific surface area, particle size, carbon content, and combustion temperature [13, 15, 16]. RHA's highly porous and irregular morphology also provides a large reactive surface area, which might improve its pozzolanic activity under appropriate processing conditions [16, 17]. The incorporation of RHA into concrete can considerably alter both fresh and hardened-state properties [13, 18, 19]. From a durability standpoint, the pozzolanic reaction between reactive silica in RHA and calcium hydroxide formed during cement hydration favors the production of additional calcium–silicate–hydrate (C–S–H) phases [20, 21]. This reaction, together with the physical filler effect of tiny RHA particles, can refine the pore structure, diminish capillary pore connectivity, and increase the compactness of the cement-based matrix. As a result, RHA has been extensively studied as a useful compound for enhancing concrete resistance to water and harsh environmental agents. Water permeability is very susceptible to changes in pore structure and connectivity.

Previous research suggests that using properly processed RHA can limit water penetration by filling gaps and increasing secondary hydration reactions [22-25]. The ensuing refinement of the capillary pore network minimizes the paths available for water transport, hence limiting the entrance of dissolved hostile chemicals. However, the positive effect is highly reliant on RHA replacement levels and physicochemical properties [22]. Excessive RHA content may increase water demand and cause poor workability or incomplete compaction, thus offsetting the durability benefits [13, 26]. RHA has also shown significant promises for enhancing carbonation and reinforcement-corrosion resistance, albeit the link is more complex [27-29]. A denser microstructure can limit the rate at which carbon dioxide and moisture infiltrate the concrete [28]. At the same time, the pozzolanic reaction may consume calcium hydroxide, reducing the cement paste's alkaline reserve. RHA affects carbonation resistance by balancing pore-structure refinement, CO_2 transit, hydration development, and reducing calcium hydroxide content. At optimal replacement levels, the microstructural benefits may take precedence and contribute to increased resistance to carbonation-induced deterioration [13, 30].

Carbonation is directly connected with the danger of reinforcement corrosion because it gradually reduces pore-solution alkalinity and can destabilize the passive film that protects embedded steel [31]. When the carbonation front reaches the reinforcement, corrosion starts more likely in the presence of enough moisture and oxygen. Thus, the usage of RHA can indirectly affect corrosion durability through its impacts on permeability, carbonation kinetics, moisture transport, and microstructural development [13, 19, 28]. However, according to the literature, the durability performance of RHA concrete cannot be generalized solely on the basis of replacement percentage; it is strongly governed by RHA reactivity, particle characteristics, burning conditions, fineness, replacement level, water-to-binder ratio, curing regime, and concrete maturity [26, 32, 33]. Overall, present research reveals that RHA can make a significant contribution to the creation of more durable and sustainable concrete by improving pore structure and minimizing water and aggressive agent movement. However, more systematic research is needed to demonstrate the relationship between RHA properties, replacement level, water permeability, carbonation depth, and the resulting potential for

2. EXPERIMENTAL DETAILS

2.1 Cement

This study employed ordinary Portland cement (Tasluja) that met Iraqi Standard IQS 5:2019 Type I and EN 197-1:2011, with a CEM I 42.5R strength class. The chemical compositions supplied by the factory are shown in Table 1.

2.2 Rice husk ash (RHA)

Rice husk was gathered from a local rice mill in Babylon Governorate, Iraq, and burned in a controlled kiln to create rice husk ash (RHA). After cooling, the ash was processed with a 2-kg capacity ball mill grinder to produce a fine, somewhat uniform powder. The ground RHA was then sieved and graded in accordance with applicable supplemental cementitious material requirements, including BS EN 450-1:2012. The RHA's chemical composition was determined using X-ray fluorescence (XRF) analysis. The XRF analysis showed that SiO_2 was the main oxide component of the RHA, accounting for 91.97% of its composition. The high SiO_2 content indicates a substantial potential for pozzolanic reactivity, although the phase state of the silica was not directly determined in the present study. The ash was also evaluated in terms of its physical qualities, such as fineness and particle-size distribution, which are critical factors influencing its reactivity and dispersion in the cement matrix. The produced RHA was dried before being added to the cementitious mixes. Table 1 shows the RHA's complete chemical composition and physical properties.

Table 1. Physical and chemical properties of cement and RHA.

Physical properties		
Specific gravity	3.17	2.61
Specific surface (cm^2/kg)	320	-
Specific surface area m^2/g	-	32.58
Mean grain size (μm)	20.1	2.98
Chemical Compositions	**Cement**	**RHA**
SiO_2	19.3	91.97
Al_2O_3	5.21	1.09
Fe_2O_3	2.97	0.31
CaO	63.81	0.94
MgO	2.85	0.23
Na_2O	0.10	0.18
K_2O	0.51	1.37
SO_3	2.47	0.09
LOI	2.81	3.80

2.3 Superplasticizer

Sika ViscoCrete-1681(Hardening accelerator, high range water reducing & super plasticizing admixture for Concrete & Mortar) was used based on the dosage required to enhance the performance of concrete mix. The specific gravity of SP was 1.070 ± 0.02 g/cm^3, while the pH-Value: 4.5 – 6.5 conforming to EN.

2.4 Mix proportions

Seven concrete mixtures were prepared, comprising a control mixture (OPC) and mixtures with 5%, 10%, 15%, 20%, 25%, and 30% cement replacement by RHA. The mix proportions of all concrete mixtures are presented in Table 2.

Table 2. Mix proportions of concrete mixtures.

Mix %RHA	Cement	RHA	FA*	CA**	Water	SP***
	(kg/m^3)					(%)
OPC	400	0	700	1100	160	1.50
5%	380	20	700	1100	160	1.50
10%	360	40	700	1100	160	1.50
15%	340	60	700	1100	160	1.50
20%	320	80	700	1100	160	1.50
25%	300	100	700	1100	160	1.50
30%	280	120	700	1100	160	1.50

FA*: fine aggregate, CA**: coarse aggregate, SP***: superplasticizer

3. WATER PERMEABILITY TEST

The flow table test, also known as the flow test, was used to determine the consistency and workability of a fresh concrete mixture. Before casting the concrete specimens for the subsequent test, the flow test was performed to check that the mixes were workable. Before testing, the flow table and its surrounding surface were properly cleaned to eliminate dust, concrete residue, and other impurities from prior tests. This cleaning method was required to avoid exterior debris from interfering with test findings and to provide accurate and consistent measurements of concrete flow.

Cleaning the testing surface was critical to keep dust, leftover concrete, and other impurities from interfering with the test results. The flow table test was performed on freshly made concrete mixtures in line with ASTM C230-97. The flow numbers were calculated using the static free-flow approach, in which the concrete spreads only due to its own weight and gravitational forces, without the need of extraneous mechanical forces. Depicts a set of concrete cube specimens measuring 150 × 150 × 150 mm used to assess the water permeability of the concrete composite. The depth of water penetration into the specimen under applied water pressure was measured and used to evaluate the water permeability of the concrete composite. The shorter length of water permeability shows that the concrete composite is more water impervious. For the water permeability of concrete composite test, each set of six specimens per composition was used, and the average value of the six data points was calculated as the result.

Specimens were cured for 28 days at 23 ± 2 ∘C and 100% relative humidity. The water permeability of concrete composites was tested using water permeability equipment in accordance with BS EN 12390-8. The specimens' side faces were attached to a layer of sealant created by combining grease and cement. The sealant ensures a tight fit between the specimen's side face and the inwall of the specimen sleeve. The specimen was placed from the smaller end to the larger end of the specimen sleeve, with the specimen and sleeve making close contact with the hand. Next, the specimen with the sleeve was placed on the pad of the hydraulic pressure testing machine. Under the pressure of the testing machine, the specimen was entirely inserted into the sleeve. After the excess sealant squeezed out of the specimen sleeve was removed, the specimen and sleeve were placed on the water permeability instrument and secured with numerous screws. The water pressure was maintained at 3.5 MPa, but the floating pressure did not exceed 0.05 MPa.

The test should last 24 hours before the specimens are removed from the water permeability device. After the test, the specimen was cut in half along its longitudinal portion on the pressure testing equipment. A marking pen was used to draw the contour line of penetrated water across the specimen's longitudinal section. The specimen's longitudinal segment included measurement points every 1 cm along the bottom. The length of water permeability at each measurement point was measured with a steel ruler. Finally, the average length of water permeability for the specimen can be measured.

4. CARBONATION TEST

Concrete composite's carbonation resistance was evaluated with cube specimens sized 100 × 100 × 100 mm. The carbonation resistance of a concrete composite can be calculated by measuring the specimen's carbonation depth under CO_2 pressure. The shorter carbonation depth indicates that the concrete composite is more carbonation resistant. For the carbonation resistance of concrete composite test, each composition set contained 21 specimens, and the result was determined as the average of 21 specimens. Specimens were cured for 28 days at 23 ± 2 °C with 100% relative humidity. The concrete carbonation test was carried out in a concrete carbonation box in accordance with the EN 12390-12:2020 standard. Prior to the carbonation test, specimens were cooked for 48 hours in an oven set to 60 °C. To maintain the concrete carbonation box, keep the CO_2 content at 20 ± 3%, humidity at 70 ± 5%, and temperature at 20 ± 5 °C. The carbonation test should last 24 days. Parallel lines were drawn 10 mm ahead of time on the two side faces that needed to be carbonized to serve as measuring points. Following the test, the specimen was cut in half, and a 1% phenolphthalein alcohol solution was sprayed over the fracture surface. After around 30-60 seconds, the color of the specimen's carbonation changes. The carbonation depth of the specimen was measured at each measuring point using a steel ruler. Finally, the average carbonation depth of the material can be calculated.

4.1 Phenolphthalein measurement of carbonation depth

The carbonation depth of the concrete specimens was evaluated using the phenolphthalein spray method. The indicator solution was prepared by dissolving 1 g of phenolphthalein in 95% ethanol and subsequently diluting the solution to a total volume of 100 mL. The prepared indicator was sprayed uniformly onto the freshly fractured surface of the concrete specimen. Following application, the fully carbonated region remained colorless because carbonation reduces the alkalinity of the cementitious matrix, whereas the uncarbonated region developed a purplish-red coloration owing to its relatively high alkaline pH. The color was allowed to develop for approximately 30 s before measurements were taken. The carbonation depth was then measured using a steel ruler. Measurement points were established at 10-mm intervals along both sides of the specimen, and the distance from each marked point to the carbonation front, identified by the transition between the colorless and purplish-red regions, was recorded.

5. RESULTS AND DISCUSSION

5.1 RHA ratio impacts concrete permeability

The water-permeability data show a distinct and progressive reduction in water penetration length as the RHA replacement rate increases. Figure 1 and Table 3 shows that the penetration length dropped from roughly 19.9 mm for the control mixture (0% RHA) to 16.0, 13.4, 11.2, 9.4, 7.9, and 6.8 mm for 5, 10, 15, 20, 25, and 30% RHA, respectively. Thus, including 30% RHA reduced the water penetration depth by roughly 13.1 mm, resulting in a 65.8% reduction compared to the control. The response was nonlinear, with a significant improvement between 0 and 20% RHA, followed by a decreasing incremental reduction at higher replacement levels. This result suggests that increasing RHA content gradually reduces water transport through the cementitious matrix, most likely due to the combined effects of particle packing, pore filling, and pozzolanic reaction. RHA has a highly porous structure and contains a high proportion of silica; under suitable processing conditions, this silica may contribute to pozzolanic reactivity; its fine particles can fill voids between cement grains, while the subsequent reaction of reactive silica with calcium hydroxide promotes additional C-S-H formation and refinement of the capillary pore network.

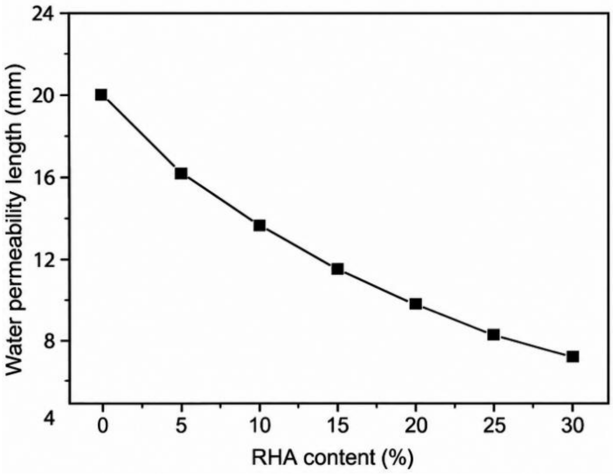

Fig. 1. Effect of RHA content on water penetration depth.

Table 3. Water permeability specimen results.

RHA	Specimen						Mean	SD
	1	2	3	4	5	6		
0%	19.2	20.2	19.8	19.9	20.7	19.6	19.9	0.51
5%	15.5	16.2	15.9	16.6	16.1	15.7	16	0.39
10%	13.4	12.9	13.9	12.8	13.6	13.4	13.4	0.42
15%	11.9	11.4	10.7	11.3	10.7	11.2	11.2	0.45
20%	9	9.4	8.9	10.2	9.5	9.3	9.4	0.46
25%	8.2	7.5	7.8	8.4	8.1	7.6	7.9	0.35
30%	6.9	6.5	7.3	6.8	6.5	6.7	6.8	0.29

A comprehensive analysis by Xu et al. [20] highlighted the pozzolanic and internal-curing capabilities of RHA as essential mechanisms influencing the formation of cement-based materials and their transport properties. Experimental evidence also supports the current trend. Vishavkarma and Harish [34] found that increasing RHA replacement reduced permeability, water absorption, and permeable pore space, with the magnitude of improvement varying with RHA dosage. More crucially, a study particularly examining the water permeability of RHA concrete revealed that 30% RHA reduced permeability by approximately 44% compared to the control and identified 30% is a suitable replacement level under various cement-content situations. The far bigger reduction observed in the current study (65.8%) supports the same fundamental mechanism but indicates that the specific RHA and combination tested here may have resulted in a more efficient modification of the transport routes. However, such differences should not be interpreted as evidence of inherently superior RHA performance because permeability is highly dependent on RHA fineness, amorphous silica content, burning history, particle morphology, water-to-binder ratio, cement content, curing regime, degree of hydration, and the specific permeability test procedure. This diversity is clear in the literature: whereas several studies demonstrate increased permeability with increasing RHA, others have found that the response can reverse at greater replacement levels or in other mixed configurations.

For example, Kumar and Devi [35] discovered that RHA-containing concrete had a beneficial permeability response at modest replacement levels, but that raising RHA above 10% could have a negative impact on carbonation behaviour under their specific settings. Likewise, Nisar and Bhat [27] reported carbonation depth increased with RHA replacement under specific curing and relative humidity conditions, emphasizing the necessity of discriminating between physical transport resistance and carbonation resistance. Conversely, the current results demonstrate that the decline in water penetration remains continuous up to 30% RHA, showing that, within the examined range and under the curing and testing circumstances used, the favourable effects of pore refinement and reduced pore connectivity prevail. This conclusion is corroborated by the carbonation results provided in this study, which show that carbonation depth fell from about 2.30 to 1.60 mm, a 30.4% reduction, as RHA grew from 0 to 30%.

The simultaneous decrease in water penetration and carbonation depth gives more evidence for progressive refining of the transport network than either metric viewed separately. Carbonation is influenced by several factors, including CO_2 transport through the pore system, calcium hydroxide availability, pore-solution alkalinity, relative humidity, curing history, and carbonation reaction kinetics, resulting in a significant reduction in water penetration compared to carbonation depth. As a result, the current findings indicate that RHA predominantly increased the physical barrier against fluid and gas transport, whereas the total carbonation response represents the competing effects of reduced transit and lower alkaline reserve.

This conclusion is consistent with previous RHA investigations that show that the interplay of microstructural densification and cementitious system chemical properties has a considerable influence on durability performance. For example, studies on RHA and silica-fume concretes have linked improved water-transport characteristics to microstructural densification and increased reactivity of the extra cementitious elements. As a result, within the 0-30% replacement range investigated, 30% RHA produced the lowest water-permeability length and thus the highest resistance to water ingress, but this should be considered the optimum level only in terms of the measured permeability response within the tested range, rather than a universal optimum RHA replacement level. Overall, the results reveal that RHA can significantly increase the transport-related durability of cementitious composites, while also proving that its performance is governed by the physicochemical properties of the ash as well as the design and curing conditions of the cementitious system.

5.2 Scanning electron microscope (SEM) test result

SEM analysis was performed on the control and RHA-containing concretes to provide supplementary microstructural observations and to assist in interpreting the durability results. The SEM micrograph of the control mixture after 28 days of curing is shown in Figure 2, while the corresponding micrograph of the RHA-containing concrete is shown in Figure 3. The SEM image of the control concrete shows a relatively heterogeneous and loosely packed cementitious matrix, with small and large calcium hydroxide (CH) crystals dispersed throughout the hydrated matrix. In comparison, the formation of C-S-H gel appears less evident, resulting in a less compact microstructure. The RHA-containing concrete exhibits a comparatively denser and more compact matrix, with hydration products distributed around the RHA particles.

The micrograph also shows CH crystals and needle-like ettringite phases embedded in a C-S-H-rich matrix. The presence of finely divided RHA may provide a physical filler effect, facilitating particle packing and reducing the size and continuity of voids within the cementitious matrix. In addition, the reactive silica in RHA can participate in pozzolanic reactions with calcium hydroxide produced during cement hydration, contributing to additional C-S-H formation. These effects may explain the denser and more homogeneous appearance of the RHA-containing matrix. The SEM observations are consistent with the experimentally observed reduction in water penetration with increasing RHA content. However, SEM observations are considered supplementary qualitative evidence rather than direct quantitative evidence of pore refinement or transport resistance, since pore-size distribution, total porosity, and pore connectivity were not independently measured in the present study.

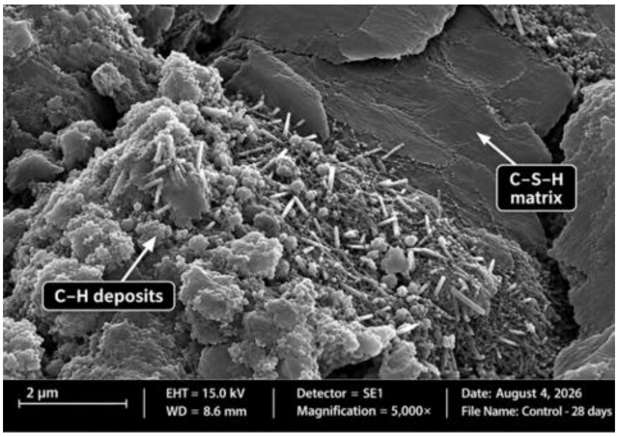

Fig. 2. SEM micrograph of control concrete after 28 days of curing.

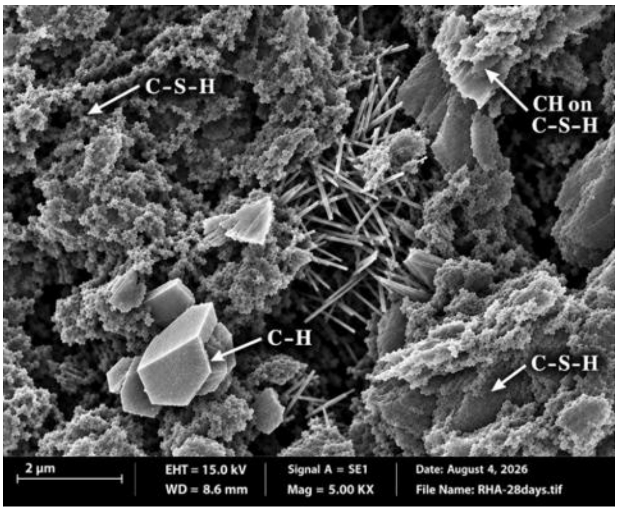

Fig. 3. SEM micrograph of RHA-containing concrete after 28 days of curing.

5.3 Effect of RHA content on the carbonation depth

Carbonation data as shown in Table 4 reveal that when RHA replacement increases, the carbonation depth decreases gradually (Figures 4 and 5).

Table 4. Carbonation specimen's specimen results.

RHA (%)	S1	S2	S3	Mean (mm)	SD
0	20.5	19.7	19.8	20.00	0.43
5	16.5	15.7	16.1	16.10	0.40
10	14.1	13.4	13.5	13.67	0.37
15	10.9	11.7	12.3	11.63	0.70
20	10.0	10.1	9.4	9.83	0.37
25	8.7	8.2	8.0	8.30	0.36
30	6.9	7.7	7.3	7.30	0.40

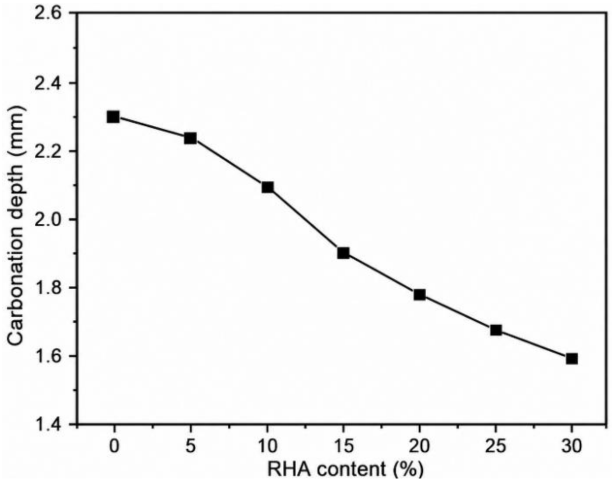

Fig. 4. Effect of RHA replacement level on carbonation depth after carbonation exposure.

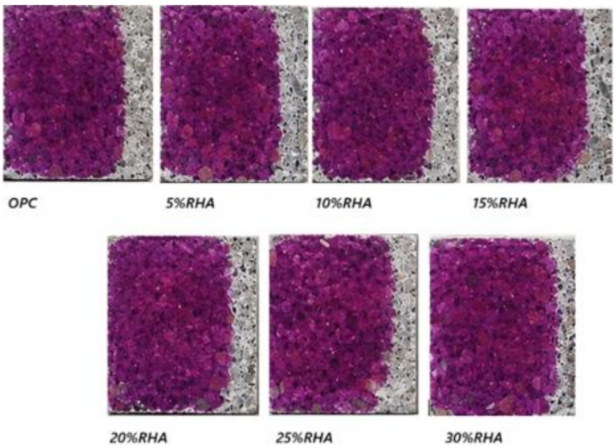

Fig. 5. Effect of RHA content on carbonation depth.

Carbonation depth reduced from about 2.30 mm in the control mixture (0% RHA) to 2.24, 2.09, 1.90, 1.79, 1.68, and 1.60 mm at 5, 10, 15, 20, 25, and 30% RHA, respectively. Thus, 30%

of RHA reduced carbonation depth by about 0.70 mm (30.4%) when compared to the control. The reduction was minimal at 5% RHA but grew more evident between 10 and 20%, with a steady decrease at higher replacement levels. The observed improvement can be attributed to the combination filler, particle-packing, and pozzolanic effects of RHA. Fine RHA particles can fill holes in the cementitious matrix, whereas the silica-rich RHA may react with $Ca(OH)_2$ to contribute to additional C-S-H formation, leading to a refined pore structure and reduced CO_2 transit. Similar increases in transport-related durability have been found for RHA-containing cementitious materials. However, the carbonation response of RHA is still reliant on its reactivity, fineness, burning circumstances, water-to-binder ratio, curing regime, and exposure humidity. These findings are consistent with prior research findings from Ma et al. [36], Nisar and Bhat [27], Kumar et al. [35], Amran et al. [13], and Zhang et al. [24]. On the other hand, Ma et al. [36] discovered that RHA increased the carbonation coefficient in natural-aggregate concrete. Similarly, Vishavkarma et al. [34], Kumar et al. [35], and Hu et al. [30] discovered that carbonation resistance improved up to 10% RHA but degraded with higher replacement levels. These disparities demonstrate that RHA content alone does not alter carbonation performance. The study indicated that the carbonation depth has reduced by up to 30% RHA, indicating pore refinement and decreased CO_2 transport. dominated the potential adverse consequences of cement dilution and $Ca(OH)_2$ consumption [28]. This interpretation is corroborated by the water-permeability measurements, which show a drop in penetration length from 19.9 to 6.8 mm (65.8%) at 30% RHA. The simultaneous decreases in water penetration and carbonation depth reflect a gradual refining of transport channels. Carbonation is influenced by multiple factors, including physical transport, CO_2 diffusion, pore-solution chemistry, $Ca(OH)_2$ availability, and exposure circumstances [31, 37]. Overall, 30% of RHA had the lowest carbonation depth within the analyzed range, indicating better carbonation resistance under the conditions tested. However, this should be regarded as the best-performing amount within the measured range rather than a universal optimum, because the optimal RHA content is determined by the physicochemical properties of the ash and the mixture, as well as the exposure conditions.

6. CONCLUSIONS

This article detailed the experimental results of durability tests on concrete composites incorporating RHA. The outcomes of this investigation allow us to conclude the following conclusions can be drawn:

1. RHA considerably reduced water penetration. The water penetration length gradually dropped from roughly 19.9 mm for the control concrete to 6.8 mm at 30% RHA, a reduction of approximately 65.8%. This improvement shows a significant reduction in capillary pores' connection and increased resistance to water intrusion.
2. Carbonation depth reduced as RHA content increased. The carbonation depth was lowered from around 2.30 mm for control.

   mixture to 1.60 mm for the 30% RHA mixture, a reduction of approximately 30.4%. Under the exposure settings used in this study, RHA increased the apparent carbonation resistance of the concrete.
3. Reduced water penetration and carbonation depth indicate that RHA improved the cementitious pore structure. The fine RHA particles and their reactive silica content are thought to contribute through particle packing, pore filling, and pozzolanic reactions, albeit the current work did not quantitatively measure the individual contribution of these mechanisms.
4. RHA's positive benefits gradually grew within the tested replacement range. 30% RHA produced the shortest water penetration length and carbonation depth, making it the best-performing replacement level among the combinations evaluated. This value, however, should be viewed as the optimum within the tested range and under the specific curing and exposure conditions, rather than as a universally optimal RHA substitution level.
5. The study found that using RHA as a supplementary cementitious material can enhance the durability of concrete while also utilizing an agricultural byproduct. Nonetheless, the durability performance of RHA concrete is likely to be affected by RHA physicochemical properties, replacement level, mixing proportions, curing circumstances, and environmental exposure.

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