

Carbonation Performance of Mortar Cube Incorporating Rice Husk as Sustainable Supplementary Cementitious Material

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Abstract

Carbonation reduces the alkalinity of mortar by converting calcium hydroxide into calcium carbonate, leading to a significant drop in pH. Prolonged carbonation may result in long-term deterioration of mechanical performance and reduced service life of mortar structures. Increased carbonation depth enhances permeability, facilitating the ingress of moisture and aggressive agents. The carbonation performance of mortar cubes incorporating rice husk ash (RHA) as a sustainable supplementary cementitious material is shown in this paper. RHA was used as a partial replacement of cement at 0%, 5%, 10%, 15%, and 20% by weight. Cement mortar cubes (without RHA) were also tested to evaluate long-term carbonation behaviour. All specimens were exposed to a controlled accelerated carbonation chamber after standard curing. Carbonation depth was measured using the phenolphthalein indicator method. The results showed that RHA significantly influenced the pore structure and carbonation resistance of mortar. Mortars with 5% and 10% RHA exhibited lower carbonation depths than the control mix. Higher replacement levels (15–20%) resulted in increased carbonation depth due to dilution of cementitious phases. Aged cement mortar cubes showed deeper carbonation than RHA-modified mortars. The optimum carbonation resistance was observed at 10% RHA replacement. The study confirms that RHA improves durability while promoting sustainable cement use.

Keywords Carbonation depth, Accelerated carbonation chamber , mortar cubes, rice husk ash(RHA)

Introduction

Carbonation is one of the most critical durability concerns affecting reinforced concrete structures, as it leads to the reduction of alkalinity and potential steel corrosion. The growing use of sustainable supplementary cementitious materials (SCMs) has significantly altered the chemical and microstructural characteristics of mortar and concrete.

Structures commonly made with or extensively using mortar include: Masonry buildings – load-bearing walls constructed with brick, stone, or concrete blocks bonded by mortar. Brickwork and blockwork walls – used in residential, commercial, and boundary structures. Stone masonry structures – such as historical monuments, retaining walls, and heritage buildings. Plastered and rendered surfaces – walls and ceilings where mortar acts as a protective and finishing layer. Masonry arches, piers, and

columns – where mortar provides load transfer and bonding between units. Chimneys and fireplaces – requiring mortar for bonding and thermal resistance. This study focuses on evaluating the carbonation performance of concrete incorporating various sustainable SCMs. Sustainable supplementary cementitious materials (SCMs) significantly influence the carbonation depth of concrete by modifying its pore structure and alkalinity. The use of materials such as fly ash, slag, and silica fume reduces calcium hydroxide content, which affects the buffering capacity against carbonation. Higher SCM replacement levels generally lead to increased carbonation depth due to reduced pH levels.

However, optimized SCM combinations can improve microstructural density and enhance resistance to carbonation.

Fly ash and GGBS affect concrete carbonation under accelerated conditions. It finds that higher SCM percentages generally increase carbonation depth, but optimized FA+GGBS combinations enhance performance. XRD and thermal analyses explain underlying mechanisms[1].

A landmark critical review describing how SCMs change hydration products, pore structure, and carbonation mechanisms. Highlights differences between blended cement and pure OPC carbonation. Provides durability guidelines and phase chemistry insights[2].

carbonation rates under various SCMs, humidity, and temperature conditions, finding slag mixes often perform better than pozzolanic SCMs. Alkali chemistry and reserve alkalinity correlate more with carbonation than strength or porosity[3].

Ternary concrete with fly ash and silica fume under simultaneous carbonation and chloride exposure. Shows improved resistance when SCMs are used correctly[4].

Develops carbonation resistance factors based on resistivity and pore structure, indicating SCM blends generally show higher carbonation than OPC mixes [5].

Review on carbonation processes and Highlights mix design and environmental effect on carbonation depth[6].

Evaluates durability parameters for fly ash, GGBS, and silica fume SCMs, including carbonation performance among other deterioration mechanisms. Highlights balance of sustainability and durability[7].

Examines the impact of SCMs (silica fume, low- and high-Ca fly ash) on concrete carbonation depth. It observed that carbonation depth increases with higher cement replacement but decreases with SCM replacing aggregate. The findings highlight how SCM type and placement influence diffusion properties[8]. carbonation depth with microstructural changes under freeze-thaw cycles. Results show freeze-thaw significantly alters pore structure, increasing CO₂ penetration. It provides insights on durability behavior under combined environmental stresses[9]. Effects of low-volume fly ash on concrete carbonation depth under 20% CO₂ with varying humidity and temperature. Shows that environmental conditions significantly influence carbonation progression. Predictive carbonation models are validated against experimental results[10].

carbonation depth in high-Ca fly ash blended concrete using numerical hydration and carbonation reaction models. It demonstrates complex interaction between hydration and carbonation processes due to fly ash chemistry. The model helps estimate carbonation penetration in blended mixes[11].

Hybrid ML framework (LSSVM + metaheuristics) to predict carbonation depth in fly ash concrete. Shows high prediction accuracy ($R^2 > 0.96$) and identifies key factors (w/b, exposure time) influencing carbonation. This improves over traditional empirical models[12].

Compares SCM concretes under accelerated and natural carbonation. It finds reserve alkalinity more predictive of carbonation resistance than strength or porosity. Slag mixes show generally better carbonation resistance than other SCM blends[13]. Critical review discusses carbonation mechanisms in blended cements, emphasizing how SCMs alter pore solution chemistry and microstructure. It synthesizes many experimental carbonation depth studies across different SCMs. Provides foundational understanding for SCM carbonation behavior[14].

Comparative study applies ANN, SVR, and RF models to predict carbonation depth using a large synthetic dataset. ANN achieved the highest predictive accuracy, while SVR proved robust for linear and nonlinear trends. The study highlights key influential input variables like compressive strength and exposure time.

Novel MOEA/D-ANN feature selection combined with ANN to predict carbonation depth with high accuracy. Shows that optimizing feature selection greatly enhances prediction performance and reduces training time. The approach is tested on multiple ML models including SVM, RF, and DT[16].

Predicts carbonation depth of recycled aggregate concrete using ensemble methods and tree-based models. It identifies key mix and environmental parameters influencing carbonation. The model demonstrates strong potential for use in sustainable concrete performance prediction[17].

Employs eight different ML algorithms—including XGB, GB, RF, and SVM—to analyze carbonation depth in fly ash concrete mixtures. Hybrid ML models combining feature reduction with regression deliver higher accuracy. Provides detailed parametric sensitivity to material and environmental factors[18].

Develops and compares hybrid ensemble models for carbonation depth prediction, improving accuracy versus single ML models. SHAP and sensitivity analysis reveal the relative importance of concrete mix features. Results support use of ensemble learning for durability modelling[19].

Integrates machine learning with optimization to predict carbonation depth under varied environmental and material conditions. The model identifies key influencing variables like w/c ratio and exposure time. High accuracy metrics indicate practical applicability for durability design[20].

Compares ML algorithms (ANN, SVM, RF) with AJ and FEM models for predicting carbonation progression. ML techniques show superior flexibility in capturing complex behavior of carbonation depth. Provides a basis for merging physics-based and data-driven predictions[21].

Combines ANN with swarm intelligence to improve robustness in carbonation depth prediction. The hybrid model outperforms traditional ANN and enhances generalization. Useful for sustainable concrete frameworks using recycled aggregates[22]. Develops an ANN-based long-term carbonation depth prediction model for natural exposure conditions. Model training uses extensive environmental and material data. Shows strong predictive capacity compared with traditional empirical models[23].

Carbonation depth results from phenolphthalein, XRD, and FTIR for OPC and PPC mixes under accelerated carbonation. Advanced methods showed nearly double the carbonation depth of the conventional indicator. Findings also reveal increased carbonation with higher W/C ratios[24].

Standard techniques (phenolphthalein, pH methods) and a novel surface field area-based average depth calculation. The comparison underlines how technique choice influences measured carbonation depth. Alternative approaches yield different insights into carbonation front geometry[25].

Experimentally compares five carbonation detection techniques (PI, CA, IR, XRD, TGA). Quantitative CA, IR, and TGA report depths 2–3× larger than phenolphthalein, showing indicator limitations. Highlights the trade-offs between complexity, accuracy, and cost[26].

An early experimental investigation using TGA, XRDA, and FTIR to measure carbonation depth beyond phenolphthalein. Identifies three carbonation zones and shows advanced methods detect deeper carbonation fronts. Reinforces the need for multi-method measurement in durability studies[27].

Experimental–modeling work introduces stress influence coefficients into traditional carbonation depth models. It shows stress (tensile/compressive) systematically affects carbonation penetration. Modified equations provide better fit to experimental data[28].

A comprehensive review of prediction models linked to experimental data, including Fick-type, exponential, curve-fit, and hybrid models. Discusses how experimental measurements feed into models and ML approaches. Details strengths and limitations of each method class[29].

Experimental carbonation of reinforced concrete and a validated diffusion-reaction simulation model. Experimental depth measurements validate numerical predictions. Demonstrates how cover thickness and mixture affect carbonation depth and corrosion onset[30].

Experimental carbonation depth data and time-series (AR) analysis to model depth over time. Shows the model's predictive power improves as more carbonation measurements are incorporated. Offers a statistical method linking experimentation and forecasting[31].

Surface protective materials modified with hybrid nano-SiO₂ (HNS) can reduce carbonation depth by up to 79.0%. The HNS refines the pore structure by decreasing large and capillary pores by over 48%, creating a superior physical barrier against CO₂ ingress[32].

Ordinary Portland Cement (OPC) provides significantly better carbonation resistance than Portland Pozzolana Cement (PPC) across various water-to-cement (W/C) ratios. It identifies that minimizing the W/C ratio is the most effective internal technique for reducing carbonation depth in both cement types[33].

Establishes that Self-Compacting Concrete (SCC) offers superior resistance compared to normally vibrated concrete due to its higher inherent density. It also confirms that adding Ground Granulated Blast Furnace Slag (GGBS) and Silica Fume (SF) reduces porosity and increases strength, thereby maximizing carbonation resistance[34]. Cement replacement by SCMs can sometimes increase carbonation risk by reducing alkaline reserve, using SCMs as an aggregate replacement actually decreases carbonation depth. It provides a new perspective on using SCMs to enhance durability without compromising the chemical buffer of the cement[35].

Explores "active carbonation" as a curing technique to create a pre-carbonated, dense surface layer that protects the concrete core. It shows that 72 hours of accelerated carbonation curing can improve compressive strength by up to 111%, effectively locking the surface against future CO₂ penetration.[36].

Applying waterproofing and anticorrosive materials at the initial stage of service life can suppress carbonation rates by 46.4% to 74.3%. It emphasizes that early intervention is far more effective for long-term depth minimization than applying coatings after years of exposure[37].

Advanced Artificial Neural Networks (ANN), this study identifies compressive strength as the primary predictor for carbonation resistance. It validates that increasing concrete strength is the most reliable physical strategy to reduce predicted carbonation depth across various environmental conditions[38]. Composite binders (e.g., GGBS combined with Fly Ash) perform better against carbonation than single SCM blends. Specifically, S95 grade GGBS combined with grade I Fly Ash achieved the best resistance by optimizing the pore structure while maintaining better alkaline stability than Fly Ash alone. Compressive stress level of approximately 0.2 to 0.35 times the peak compressive strength can actually minimize carbonation depth. The moderate loading densifies the concrete's internal structure, though exceeding 0.5 times the strength increases carbonation due to micro-cracking.

Mix combination of 20% Silica Fume (PC20SF) is the most optimum for decreasing carbonation depth. This specific dose optimizes the balance between pore-filling pozzolanic reactions and the preservation of the concrete's pH level[41].

Evaluates phenolphthalein against quantitative methods (TGA, XRD, FTIR, CA) for carbonation depth measurement. Phenolphthalein is shown to provide intuitive and economical results but often underestimates carbonation relative to more precise techniques. The paper discusses phenolphthalein's limitations in partial carbonation zones and its influence on reported depths[42].

Carbonation depths in OPC and PPC concrete were measured with phenolphthalein and advanced techniques (XRDA, FTIR). Phenolphthalein results were nearly half of those from advanced techniques, highlighting its underestimation issue. The study confirms phenolphthalein's usefulness but suggests caution in interpreting results for durability assessment[43].

Develops a digital image-processing algorithm to automatically detect carbonated vs. non-carbonated zones on phenolphthalein-treated concrete surfaces. It demonstrates improved objectivity and reproducibility over manual visual inspection, addressing a key limitation in conventional phenolphthalein tests. The algorithm provides a basis for systematic carbonation depth extraction[44].

An automated measurement system using machine vision and CNNs to classify phenolphthalein-indicated carbonation in concrete. It shows strong correlation with standard EN 13295 measurements while reducing operator subjectivity. The work is significant for advancing digital carbonation assessment tools[45].

Phenolphthalein as the most common method for carbonation depth estimation and notes its limitations in detecting partial carbonation zones. It emphasises combining phenolphthalein with other methods to enhance measurement quality. The analysis provides a modern overview of carbonation detection techniques[46].

Carbonation depth of concrete with partial cement replacement by RHA (5–15%) under controlled conditions. Results show that carbonation depth decreased most at up to 10% RHA replacement, indicating improved resistance at moderate RHA content, while higher RHA levels resulted in increased depths. The research also correlated carbonation behaviour with permeability and microstructural features, using SEM/XRD analyses[47].

Experimental work investigated carbonation penetration in brick–aggregate concrete blended with different percentages of rice husk ash. Carbonation depth tests in a concentrated CO₂ environment showed that RHA contributed to reduced CO₂ ingress compared to control mixes, with optimum performance at certain replacement levels. [48].

Methodology

3.1 Materials

Ordinary Portland Cement (OPC) conforming to IS 269 / IS 12269 was used as the primary binder. Rice husk ash (RHA), obtained from controlled combustion of rice husk followed by grinding, was used as a sustainable supplementary cementitious material. Standard graded river sand conforming to IS 650 was used as fine aggregate. Potable water was used for mixing and curing.

3.2 Mix Proportions

Mortar mixes were prepared by partially replacing cement with RHA at 0% (control), 5%, 10%, 15%, and 20% by weight of cement. A constant binder-to-sand ratio of 1:3 was maintained for all mixes. The water-to-binder ratio was kept constant for all mixes to ensure consistency and isolate the effect of RHA on carbonation performance.

3.3 Specimen Preparation

Mortar cubes of size 70.6 mm × 70.6 mm × 70.6 mm were cast for each mix. Dry ingredients were mixed thoroughly to achieve uniform distribution of RHA, followed by gradual addition of water. The fresh mortar was placed into oiled moulds in two layers and compacted using a standard tamping rod. Specimens were demoulded after 24 hours.

3.4 Curing Regime

After demoulding, all specimens were water-cured at a temperature of $27 \pm 2^\circ\text{C}$ for a curing period of 28 days. On completion of curing, specimens were air-dried for 24 hours to attain a uniform moisture condition before carbonation exposure.

3.5 Application of Accelerated Carbonation Chamber

Accelerated carbonation testing was performed using a carbonation chamber to simulate long-term atmospheric carbonation under controlled laboratory conditions. The chamber was operated with the following parameters:

- CO₂ concentration: 3–5%
- Relative humidity: 60–70%
- Temperature: $27 \pm 2^\circ\text{C}$

The specimens were placed inside the chamber with sufficient spacing to ensure uniform exposure of CO₂ on all surfaces. Carbonation exposure was carried out for 28, 56, and 90 days, depending on the test program.

The accelerated carbonation chamber enhances CO₂ diffusion into the cementitious matrix, allowing assessment of carbonation resistance within a reduced time frame.

3.6 Carbonation Depth Measurement Using Phenolphthalein Indicator

At the end of each carbonation exposure period, specimens were removed from the chamber and split into two halves using a compression testing machine. A 1% phenolphthalein indicator solution was sprayed uniformly on the freshly fractured surface.

The uncarbonated region, with pH greater than 9.5, turned pink, while the carbonated region remained colourless. The boundary between the pink and colourless zones was identified as the carbonation front.

3.7 Measurement of Carbonation Depth Using Vernier Caliper

Carbonation depth was measured from the exposed surface to the carbonation front using a digital Vernier caliper with an accuracy of 0.01 mm. Measurements were taken at three different locations on each specimen to minimize local variability.

The average carbonation depth was calculated using Equation (1):

$$d_{\text{avg}} = \frac{d_1 + d_2 + d_3}{3}$$

3.8 Data Analysis

The experimentally obtained carbonation depths were analysed to evaluate the influence of RHA replacement levels on carbonation resistance. Results from RHA-incorporated mixes were compared with control OPC mortar to identify the optimum replacement level for improved durability performance.

where d_1, d_2, d_3 are individual carbonation depth .

3.9 Reliability and Repeatability of the Method

The use of controlled chamber conditions, standardized specimen preparation, and multiple depth measurements ensured repeatability and accuracy of the experimental results.

Carbonation depth is generally governed by the diffusion of CO₂ through the pore structure of concrete and follows a square-root-of-time relationship, expressed as:

$$X = k\sqrt{t}$$

where

X = carbonation depth (mm),
k = carbonation coefficient (mm/ $\sqrt{\text{time}}$), and
t = exposure time.

Fig. 1 Sample Placement in Accelerated Carbonation Chamber



Fig. 2 Carbonation of Concrete Samples



Fig. 3 Accelerated Carbonation Chamber Apparatus



Fig. 4 CO₂ supply setting in ACC



Fig. 5(a) Mixing of cement, sand and rice husk ash.



Fig. 5(b) Mortar Cube Sample Preparation with binary combination of cement and replacement of rice husk ash with variation of 5% from 0 to 20%.

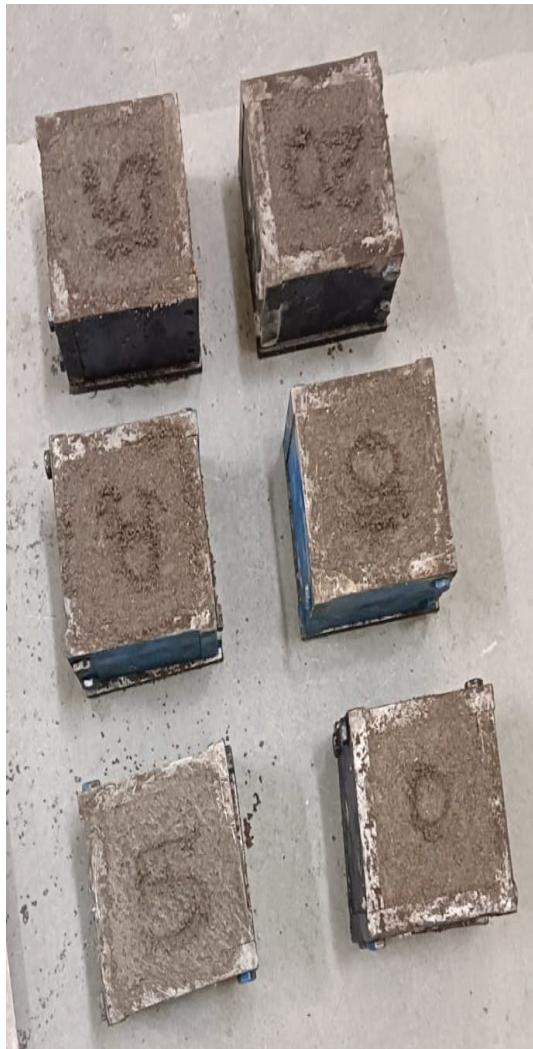


Fig. 6(a) Carbonation test using phenolphthalein indicator



Fig. 6(b) Carbonation depth measurement using Vernier Caliper



Fig.6(c) Carbonation depth measurement using Vernier Caliper



Result and Discussion

Table 1: Carbonation Depth Measurement for Mortar cube Specimens

Specimen ID Mortar Cube(MC)	Mix Type	Exposure Time (days)	1 st Measured Carbonation Depth (mm)	2 nd Measured Carbonation Depth (mm)	3 rd Measured Carbonation Depth (mm)	Average Carbonation Depth (mm)
MC1	OPC	56	11.8	12.5	12.2	12.1
MC2	OPC	56	11.5	12.3	12.4	12.07
MC3	OPC	56	11.6	12.7	12.3	12.2
MC4	OPC	56	11.9	12.1	12.7	12.23
MC5	OPC	56	11.3	11.9	12.2	11.8
MC6	OPC	90	13.4	13.2	13.4	13.33
MC7	5% RHA	28	6.4	6.7	6.8	6.63
MC8	10% RHA	28	6.2	6.3	6.5	6.97
MC9	15% RHA	28	6.4	7.4	7.3	7.033
MC10	20% RHA	28	7.1	7.6	7.8	7.5

Mortar incorporating 5% and 10% RHA shows significantly lower carbonation depths (6.63–6.97 mm) at 28 days compared to OPC mixes. OPC mortar specimens exposed for 56 days exhibit average carbonation depths in the range of 11.8–12.23 mm, indicating relatively higher CO₂ penetration. Results indicate that partial replacement of cement with RHA up to an optimum level improves carbonation resistance, while excessive replacement adversely affects performance. An increase in RHA content beyond 10% leads to a gradual rise in carbonation depth, reaching 7.50 mm for 20% RHA, due to reduced calcium hydroxide availability.

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Data availability Experiment work. The data used to support the findings of this study are included within the article.

Declarations Competing interests The authors declare no competing interests.

Conclusion

Based on the experimental results, it can be concluded that the incorporation of rice husk ash significantly influences the carbonation resistance of concrete. Ordinary Portland cement concrete exhibits higher carbonation depths, which increase with exposure time. Partial replacement of cement with RHA up to 10% effectively reduces carbonation depth due to improved microstructural densification and pozzolanic activity. However, higher replacement levels (15–20% RHA) lead to increased carbonation depth, attributed to the dilution effect and reduced alkalinity. Therefore, an optimum RHA replacement level of 5–10% is recommended to enhance carbonation resistance while maintaining durability performance. These tools allow accurate prediction of durability performance under varying conditions. footprint of the construction industry. Future studies may focus on evaluating carbonation depth at extended exposure periods (180, 365 days and beyond) to better understand the long-term durability performance of RHA-incorporated concrete. Future research may investigate carbonation resistance of concrete incorporating sustainable SCMs such as agricultural ashes (bagasse ash, bamboo leaf ash, palm oil fuel ash), industrial by-products (red mud, copper slag, steel slag), and recycled materials (waste glass powder, ceramic waste powder). The combined use of multiple sustainable SCMs (e.g., waste glass powder with GGBS or calcined clay) can be studied to optimize pore structure refinement and carbonation resistance. Advanced microstructural analysis techniques (SEM, XRD, FTIR, TGA, MIP) can be employed to correlate carbonation depth with hydration products, pore size distribution, and CO₂ binding capacity of sustainable SCM-based concretes.

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