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Influence of Carbonated Calcium Generated from Waste Concrete Sludge on the Strength and Microstructural Properties of Portland Cement Mortar

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Abstract. It is imperative to identify solutions to lower the carbon footprint of construction materials. One of the ways is to recycle and reuse waste concrete materials through indirect CO2 mineral carbonation. This study replaces Portland cement (PC) in mortar composites with up to 15% carbonated calcium (CC) generated from waste concrete at 5% intervals (i.e, 0%, 5%, 10%, 15%) by mass of PC. The slump and flow of the mortar composites (MC) developed (MC0, MC5, MC10, and MC15) were determined using the mini-slump cone and flow table tests. 50 × 50 ×50 mm cubes and 160 mm × 40 mm × 40 mm beam specimens were cured in a climate-controlled room at 23 ± 2 °C and a relative humidity of 65 ± 5% for 24 hours before being water cured for 7 and 28 days, respectively. The bulk density (BD), dry density (DD), compressive strength (CS), and flexural strength (FS) were determined. Additionally, the morphology of the hardened mortar was examined using scanning electron microscopy (SEM). The slump decreases from 8 mm in MC0 to 4 mm in MC15, and the BD is 2272–2462 kg/m^3. The DD of the hardened mortar increases from 2104–2290 kg/m^3. At 28 days, MC0, MC5, MC10, and MC15 exhibit CS of 16.8, 19.2, 22.8, and 24.9 MPa, respectively, and FS of 3.4, 3.7, 3.9, and 4.3 MPa. The microstructural analysis shows that the rhombohedral-shaped CC matrix is formed around the cement particles, depicting a whitish hydration product termed calcium silicate hydrate, with minimal visible porosity as CC increases in the SEM images. Conclusively, 15% CC inclusion in MC exhibited a 50% decrease in slump. Also, BD, DD, CS, and FS increased by 7.7%, 8.1%, 33%, and 2.1%, respectively, at optimum CC content due to the dense microstructure revealed in the SEM.

1. Introduction

The environmental sustainability of cement production is under increasing scrutiny, especially as technological innovations continue to spring up across the construction and concrete industries. Despite significant advancements in concrete technology, cement manufacturing remains one of the most energy-intensive and CO_2-emitting industrial activities, contributing substantially to global environmental challenges such as climate change [1]. Consequently, alternative materials, including fly ash [2], silica fume [3], ground granulated blast-furnace slag (GGBS) [4], cassava peel ash [5], and rice husk ash [6] have been investigated for their potential to improve strength, durability [7], and sustainability [8] when used as supplementary cementitious materials in the development of Portland cement (PC) composites.

The cement industry also generates large volumes of solid waste—such as concrete debris, kiln dust, and waste ready-mix concrete sludge (i.e., by-product from washing concrete mixers, containing fine aggregates, cement, and water)—which can degrade land and water resources if

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not properly managed [9]. In South Africa and many developed nations, the disposal of such waste in landfills is prohibited, driving the need for alternative management strategies [10]. Conventional methods like stockpiling and incineration have significant drawbacks: stockpiling consumes valuable land resources, while incineration releases harmful pollutants [11]. Moreover, these practices fail to promote material recovery and reuse, underscoring the need for more sustainable solutions.

Recent research has increasingly focused on utilising solid waste from cement and concrete production by leveraging their chemical reactivity [12]. Many of these waste materials, such as kiln dust and demolished concrete, contain reactive calcium compounds that can be recycled into new products [13]. Waste ready-mix concrete, for instance, consists of excess or leftover concrete that is typically discarded [14]. A promising recycling approach is mineral carbon capture and utilisation (MCCU), which exploits the reactivity of these wastes to capture and store CO_2 while producing valuable by-products such as calcium carbonate (CC) that can be reused in construction [15]. MCCU can occur via direct or indirect carbonation: in direct carbonation, CO_2 reacts directly with the solid material, while in indirect carbonation, reactive species such as calcium are first extracted into solution before carbonation. Indirect carbonation is generally preferred because it yields more crystalline products and achieves greater process efficiency, making it suitable for industrial-scale production of high-quality CC [15].

An expanding body of literature has examined the incorporation of CC into sustainable concrete and mortar systems, demonstrating its potential to enhance environmental performance [16] as well as rheological, mechanical, and microstructural properties [17]. Antoni et al. [18] reported the use of FA (10%), SF (2.5%), natural fine size CC (0-15%), and PC (87.5%) in MC. The study combines pozzolanic materials (FA and SF) with natural CC up to 15% CC at 5% intervals. The result shows that 15% CC inclusion with pozzolans exhibits an increase in workability and improved CS by up to 5.2% (early age) and 4% (later age). Conversely, other studies observed reductions in compressive strength when using carbonated recycled concrete aggregates [19]. Safaei et al. [20] found that 2% nano-CC improved compressive strength and 1.5% enhanced tensile strength, with higher additions leading to a reduction in strength. Similarly, replacing PC with 1% nano-CC yielded an optimum compressive strength of 42 MPa compared to 35 MPa for the control concrete mix [21]. Nejad et al. [22] reported a concrete compressive strength of 54 MPa at 0.5% nano-CC, followed by decreases to 51 MPa and 48 MPa at 0.7% and 1%, respectively. Collectively, these findings indicate that the physico-structural and mechanical properties of CC are highly dependent on the synthesis conditions, which in turn influence their performance in construction applications.

In the present study, CC derived from the indirect mineral carbonation of waste concrete sludge was used as a partial substitute for cement in mortar production. A distinguishing feature of this work is the use of a 1000 L capacity concrete sludge recycling pilot plant for mineral carbonation, marking a significant step beyond laboratory-scale experimentation. Operating at this scale enables a more efficient extraction of calcium ions, overcoming kinetic and equilibrium limitations typically encountered in smaller systems. This pilot-scale approach demonstrates industrial feasibility while aligning with circular economy principles through waste valorisation, reduced landfill disposal, and lower CO_2 emissions. Accordingly, Portland cement (PC) was partially replaced with up to 15% CC by mass, obtained via indirect MCCU. The study investigated the fresh properties of the mortar pastes, evaluated the hardened mechanical performance, and performed microstructural characterisation using scanning electron microscopy (SEM) to elucidate the composite behaviour.

2. Materials characterisation

The materials utilised comprised CEM II/A-L 52.5N PC and CC derived from the indirect mineral carbonation of waste ready-mix concrete (WRMC) sludge. The WRMC was obtained from a mid-

tier mining and materials company based in Cape Town, South Africa. Deionized water free from soluble salts was used in all experimental procedures. High-purity carbon dioxide (99.95%) was supplied by a local vendor and administered at a controlled flow rate of 20 L/min. The chemical composition and morphology of the binders (PC and CC) were analyzed using Panalytical Axios X-ray fluorescence (XRF) equipment and a scanning electron microscope (SEM) JCM-7000 NeoScope, respectively, both available at the Chemical Engineering Department, Cape Peninsula University of Technology, South Africa. XRF (Table 1) indicates a high content of CaO in PC and CC. The SEM image at a magnification of 2000*X* (Fig.1) shows that particles of PC are angular and irregular, while CC exhibits a rhombohedral shape with a visible void between the particles.

Table 1: Chemical composition of PC and CC

	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	SO_3	LOI
PC	18.73	0.18	3.43	2.85	0.05	1.12	63.0	0.19	0.42	1.89	7.2
CC	1.08	0.01	0.13	0.08	-	-	55.23	-	-	0.72	42.01

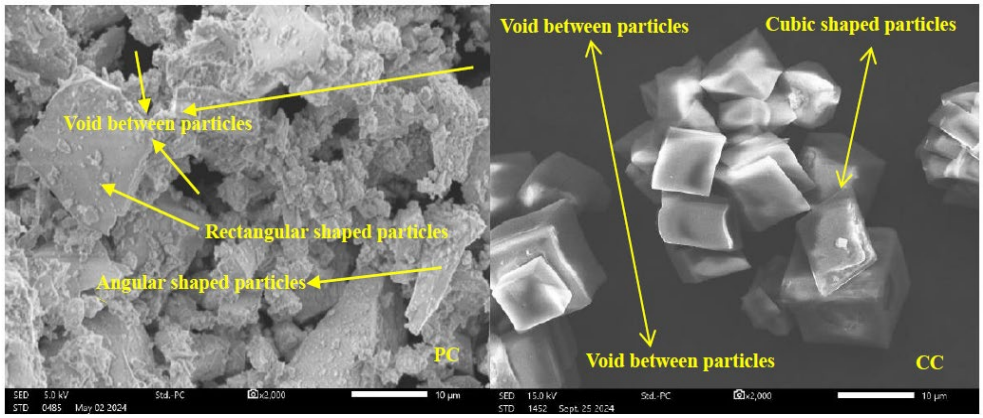


Fig 1: SEM images of PC and CC

2.1 Mortar composition, mixture procedure, and fresh properties testing.

The mixture composition of the mortar is presented in Table 2. The control mixture contains 100% PC, labelled MC0. Thereafter, PC was replaced with CC up to 15% at 5% intervals, and labelled MC5, MC10, and MC15, respectively. A constant binder to aggregate mass ratio of 1:3, and a water to binder ratio of 0.4, was utilised in line with previous studies [23][24]. A 1% by mass of binder Chryso Premia superplasticizer (SP) was adopted to tailor rheology [25][26].

Table 2. Mortar mixture proportion

MIX ID	PC [g]	CC [g]	Sand [g]	Water [g]	SP [g]
MC0 (Control)	1300	-	3900	520	13
MC5	1235	65	3900	520	13
MC10	1170	130	3900	520	13
MC15	1105	195	3900	520	13

For the control mortar, the fine aggregate was first weighed and transferred into the 10 L Pan mixer, as shown in Fig. 2a. The PC was then added and mixed thoroughly for 3 minutes, followed by the introduction of water and the SP, and an additional 3 minutes of mixing. For the other mortar mixtures, both CC and PC were added to the aggregate immediately after placing the fine aggregate in the mixer, mixed thoroughly for 3 minutes, after which water and SP were incorporated, and

mixing continued for another 3 minutes. Slump test was performed using a mini-slump cone on the fresh mortar mixtures following the procedures outlined in ASTM C0230/C0230M-20 [27]. The fresh mortar was placed into a mould with a height of 60 mm, a base diameter of 100 mm, and a top diameter of 70 mm, as illustrated in Fig. 2b. The cone was filled in two layers, each compacted with ten strokes of the tamping rod. After filling, the cone was carefully lifted and positioned beside the formed mortar, and the height difference was recorded as the slump value of the mortar. Additionally, the flow test was performed using the flow table test (Fig. 2c). The bulk density was determined by measuring the mass of mortar occupying a known volume, as illustrated in Fig. 2c. A 3-liter batch of fresh mortar was prepared for the test. The container was first half-filled with mortar using a scoop, then tilted about 30 mm to each side and dropped ten times onto a rigid surface to achieve compaction. The remaining portion of the container was filled, and the same procedure was repeated. The bulk density of the mortar was subsequently calculated using Eq. 1.

$$\rho_m = \frac{m_2 - m_1}{V_v} \tag{1}$$

where (ρ_m) in $\frac{Kg}{m^3}$ is the bulk density, m_1 is the mass of the empty container, m_2 is the mass of the container filled with mortar, and V_v is the volume of the container.

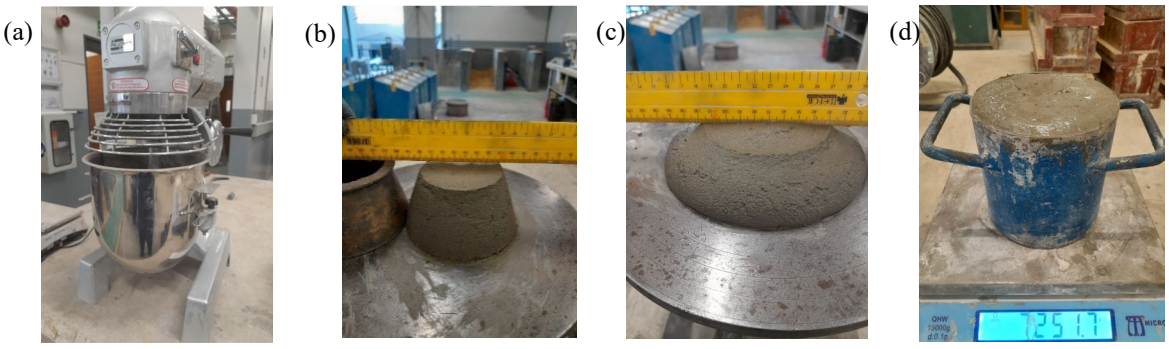


Fig. 2 (a) 10 L pan cake mixer, (b) slump, (c) slump flow, and (d) bulk density

2.2 Mechanical performance and microstructural properties testing procedures.

50 mm × 50 mm × 50 mm standard cubes and 160 mm × 40mm × 40mm beam moulds were filled with fresh mortar and compacted on a vibrating table in two layers for 3 min after each layer, as shown in Fig. 3a and placed in a climate-controlled room at 23±2 ℃ and a relative humidity of 65±5% for 24 h (Fig. 3b) before it was demoulded and subject to standard water curing. The Zwick Z250 Material Testing Machine was used to determine the compressive and flexural strengths of the cubes and beams after 7- and 28-day curing (Fig.3c and d). The compressive and flexural strengths of the cubes and beams were determined using Eq. 2–5.

$$f_c = \frac{F}{A_c} \tag{2}$$

where f_c is the compressive strength (MPa), F is the compressive load (N), and A_c is the compressive area of the cube (mm^3).

$$W = \frac{1}{6} bh^2 \tag{3}$$

$$M_{max} = F \times x \quad (4)$$

$$F_f = \frac{M_{max}}{W} \quad (5)$$

where F_f is flexural strength (MPa), M_{max} is the maximum moment (Nmm), W is the section modulus (mm^3), F is the maximum load (N), and x is the distance (mm).

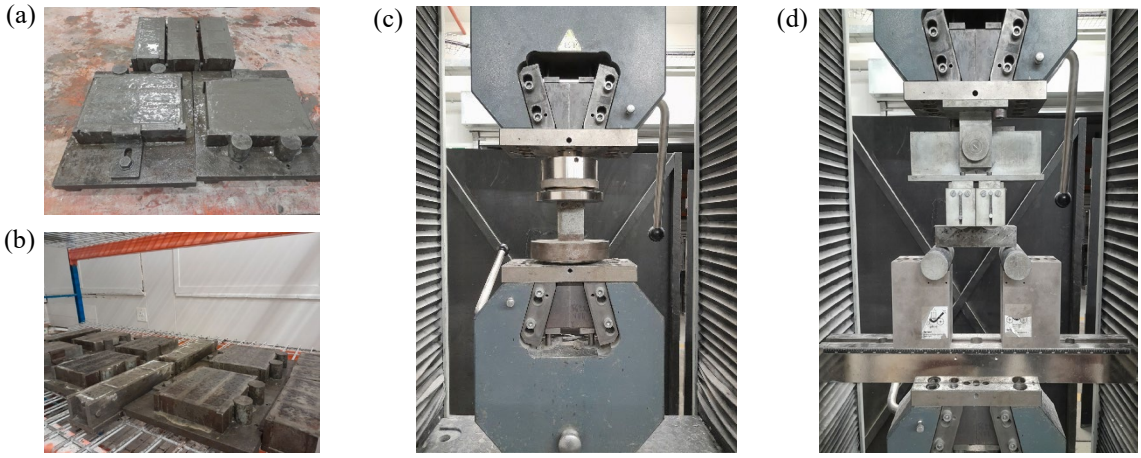


Fig. 3 (a) Specimens, (b) climate-controlled curing, (c) compression test, and (d) flexure test

3. Results and discussion

The results of slump and flow are shown in Fig. 4a. Both the slump and slump flow of MC decreased with increasing CC in a nearly linear proportion. In Fig. 4b, BD increases by 2.4, 7.4, and 7.7%, respectively, as CC content increases from 0 to 15%. A similar trend was exhibited with 2.6%, 7.3%, and 8.1% increase in DD at CC of 5%, 10% and 15% compared to the control. The increase is in agreement with what was obtained in another study [28].

The compressive and flexural strengths against mixture ID are shown in Figs. 4c&d. After 7 days of curing, compressive strengths of MC5, MC10, and M15 were enhanced by 18, 30, and 34%, respectively, compared to MC0. Also, after 28 days, 13%, 27%, and 33% increases were exhibited compared to the control. This result is supported by Gonnon and Lootens [29]. The MC kinetics reflect strength enhancement as CC increases up to 15%. The flexural strength against mixture identification is shown in Fig. 4d. After 7 days of curing, MC5, MC10, and MC15 outperformed the control samples by 4, 14, and 20%, respectively. Similarly, flexural strength was enhanced by 8, 13, and 21%, respectively, after 28 days. The improvement in flexural strength is due to the enhancement and accelerating effect of CC in the cement hydration process through its filler [30][31]. In addition, CC, as a cement filler material, was reported to have an incremental impact on flexural properties [32].

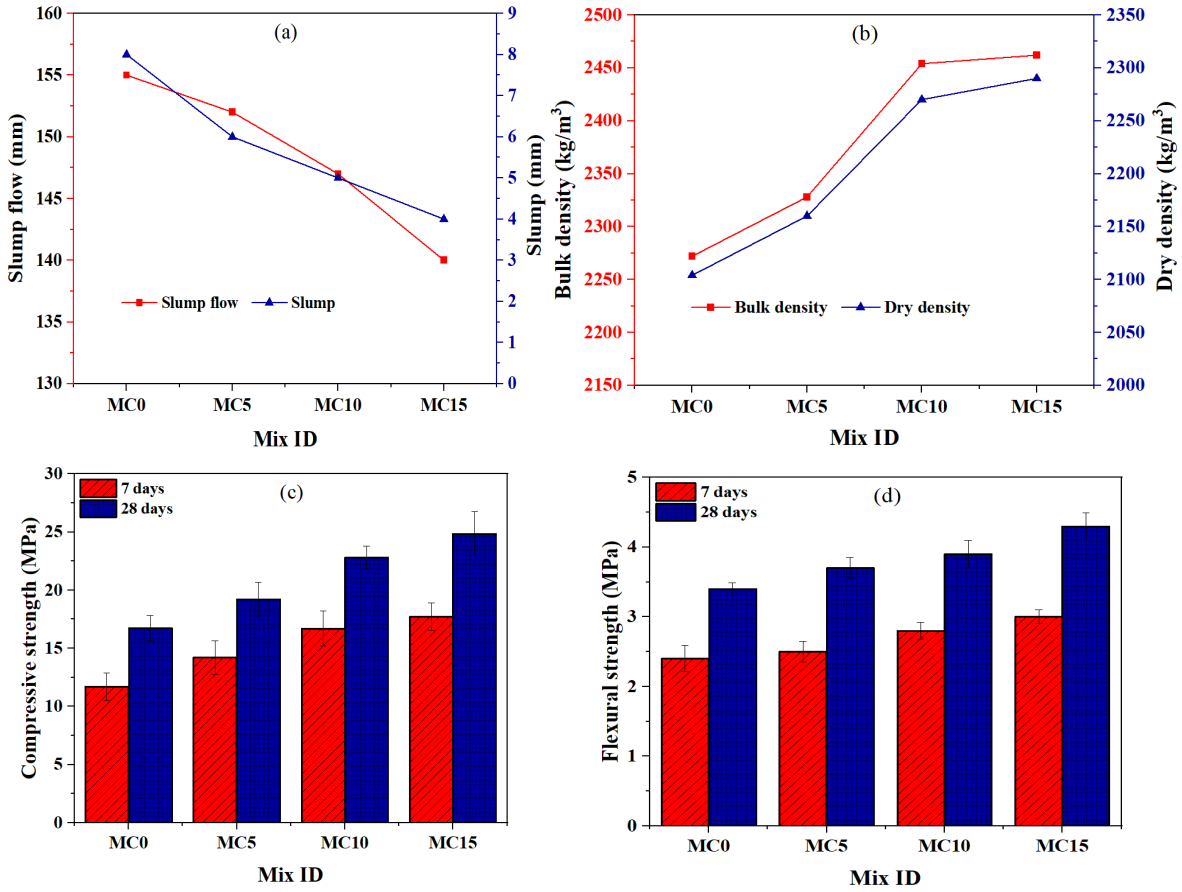


Fig. 4. (a) Slump and slump flow, (b) bulk and dry density, (c) compressive strength, and (d) flexural strength

The internal morphology of the MC using SEM images is depicted in Fig. 5a-d. The formation of hydration product, calcium silicate hydrate (C-S-H), is shown on the surface of MC in Fig. 5a. The CC matrix, exhibiting a rhombohedral shape, surrounds the cement particles and forms more whitish C-S-H (Fig. 5b), with more angular particles, which aid interlocking and improve mechanical properties. As CC increases, the MC-based structure offers high cohesion with only minor observable porosity and pore refinement (Fig. 5c). CC acts as a filler, refining the matrix and producing a relatively dense structure with improved particle packing, although some localised porosity persists. MC15 (Fig. 5d) exhibits a more refined microstructure, with the filler effect enhanced and CC particles more uniformly dispersed within the matrix. This improved particle packing substantially reduces voids, prevents susceptibility to crack propagation by filling the interstices of the MC, thereby enhancing the mechanical properties [33].

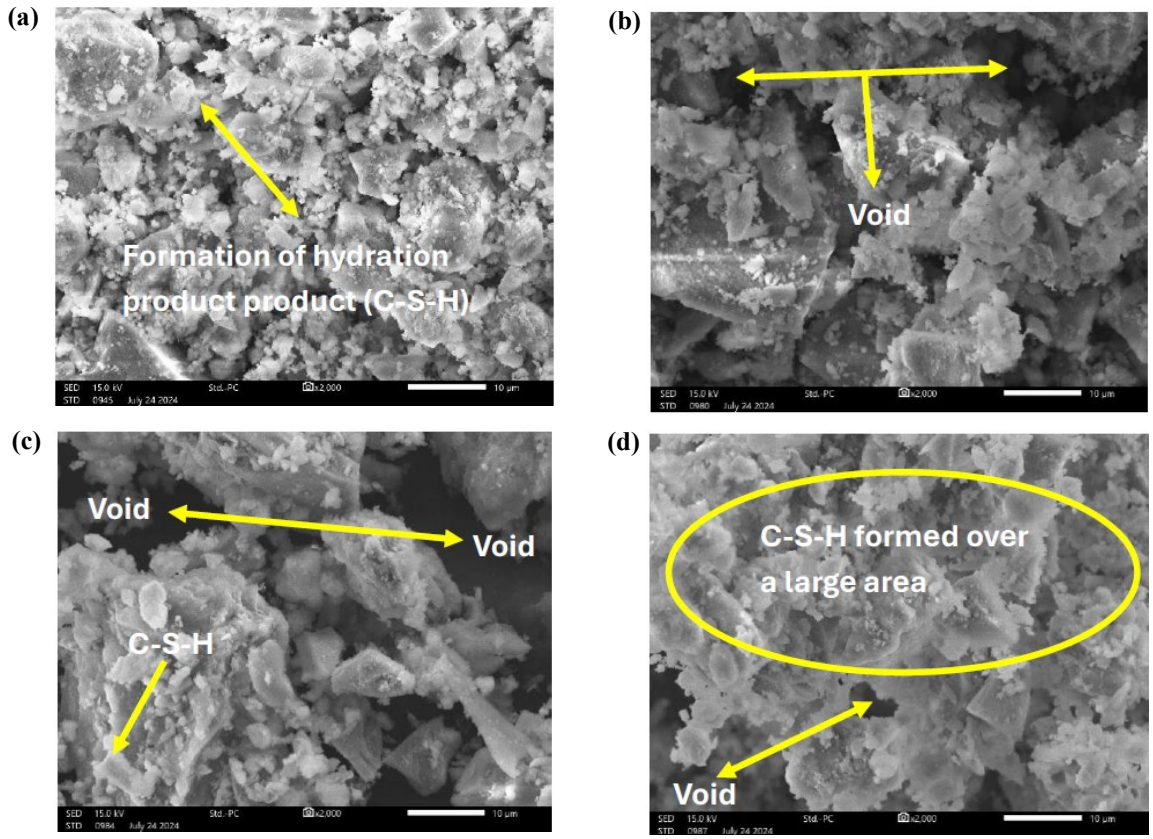


Fig. 5. SEM (a) MC0, (b) MC5 (c) MC10 and (d) MC15

4. Conclusion

The feasibility of employing CC, which is produced via indirect CO_2 mineral carbonation of waste ready-mix concrete, in mortar application was examined in this study. By encouraging circularity in building materials and lowering reliance on PC, the project benefits the environment. Up to 15% of Portland cement (PC) in mortar was substituted with CC at intervals of 5%. After examining the mortar's fresh, mechanical, and microstructural properties, the following conclusion can be drawn:

I. CC inclusion accelerates mortar setting times and reduces workability by up to 50%. Beyond 15% CC inclusion, the MC was not workable due to insufficient consistency. Hence, an optimum CC inclusion of 15% was adopted.

II. The slump of MC decreased up to 50% from MC0 to MC15. Additionally, BD and DD increased by 7.7 and 8.1%, respectively, at 15% optimum CC inclusion.

III. The CS and FS of MC15 outperformed the control (MC0) by 33 and 2.1%, respectively, after 28 days due to the densification of the composite matrix through C-S-H formation over a large area of the SEM images.

5. Recommendation

I. The long-term mechanical properties of MC incorporating CC are recommended for further studies.

II. The durability performance of MC incorporating CC when subjected to mild and aggressive environmental conditions is recommended for further studies.

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