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Performance of reactive MgO concrete under increased CO₂ dissolution

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Abstract

The strength gain of reactive MgO cement (RMC) samples depends on carbonation, which is limited by the formation of an initial carbonate layer and the low dissolution of CO₂. This study investigates the use of seeds and NaHCO₃ (SBC) to extend the surface area for carbonation and increase CO₂ dissolution, respectively. The influence of seeds and SBC on the hydration kinetics of RMC was evaluated by isothermal calorimetry and pH measurements. Mechanical performance results were supported by XRD, TG-DTG and SEM, which identified the amount and morphology of final phases. The introduction of well-dispersed seeds within the initial mix design enabled the enhanced nucleation of carbonates, while the dissolution of CO₂ was improved by the increased initial pH and HCO₃⁻ provided by SBC. The simultaneous use of seeds and SBC led to dense microstructures composed of interconnected carbonate networks, resulting in 142% increase in 28-day strengths (24 vs. 58 MPa).

Keywords: MgO (D); Carbonation (C); pH (A); Compressive strength (C); Microstructure (B)

1 Introduction

The production of Portland cement (PC) is associated with a high energy demand and carbon dioxide (CO₂) emissions [1, 2], which has led the cement industry to search for alternative binders that can reduce the overall CO₂ emissions emitted by PC production. Reactive magnesium oxide (MgO) cement (RMC) has gained interest due to its lower production temperatures than PC (i.e. 700-1000 vs. 1450 °C) [3-5] and ability to gain strength via its reaction with CO₂. The hardening of RMC formulations under carbonation has paved the way for the sequestration of atmospheric CO₂ as stable carbonates in cement-based mixes.

The strength development of RMC-based mixes involves a two-step process that initiates with the hydration of MgO into Mg(OH)_{2(aq,s)}. This step is accompanied with an increase in the pH of the pore solution, which stimulates the diffusion of CO₂ for the second step that is based on carbonation. When subjected to carbonation, Mg²⁺ reacts with CO₃²⁻, resulting in the formation of a range of hydrated magnesium carbonates (HMCs). The formation of HMCs enables strength gain in RMC mixes by reducing the porosity via their expansive formation and provides binding by establishing an interconnected network. While the simultaneous formation of various HMCs in carbonated RMC samples has been reported in previous studies [6, 7], some of the most common ones observed include the needle-like nesquehonite (MgCO₃·3H₂O), rosette-like hydromagnesite (4MgCO₃·Mg(OH)₂·4H₂O) and dypingite (4MgCO₃·Mg(OH)₂·5H₂O), and acicular artinite (MgCO₃·Mg(OH)₂·3H₂O) [7, 8]. Since the rate and degree of carbonation, as well as the morphology of the carbonate phases are the main factors controlling strength development [9-12], the progress of the carbonation reaction determines the final performance of RMC formulations.

Several studies have looked into the improvement of strength via the enhancement of hydration within RMC mixes. These have included the adjustment of mix design via the use of a hydration agent (HA) or the curing conditions via the use of high temperature pre-curing, which has led to an increase in the hydration of RMC by 54% after 1 day [12-14]. In addition to hydration, the use of elevated (i.e. accelerated) concentrations of CO_2 ($\geq 5\%$) in the curing of RMC samples has led to a rapid strength gain by increasing the rate of the carbonation reaction and the associated formation of HMCs [6, 10, 11, 15-20]. For instance, RMC-based concrete samples involving the use of a HA (magnesium acetate $((\text{CH}_3\text{COO})_2\text{Mg})$ at a concentration of 0.1 M) cured under 10% CO_2 for 28 days revealed higher strengths than the corresponding PC-based samples (58 vs. 38 MPa) [11]. The simultaneous introduction of HA and 24 hours of high temperature pre-curing under 60 °C also resulted in 40% higher 28-day strengths in comparison to the control in RMC samples (56 vs. 40 MPa) [12].

In spite of these advancements in the reaction mechanisms associated with the use of HA [11], high temperature pre-curing [12] and accelerated carbonation curing [10], the resulting RMC formulations still revealed large amounts of residual uncarbonated $\text{Mg}(\text{OH})_{2(s)}$ (brucite). Previous research [10, 11] reported that up to 55% of the final binder phase is composed of uncarbonated brucite after 14 days of curing. These high contents of unreacted brucite highlight the inefficiency in the use of RMC as a binder, which not only limits the strength development of concrete formulations it is incorporated in, but also increases the overall cost. These low carbonation degrees were linked with the solid barriers that limit the contact area between the carbonate ions and $\text{Mg}(\text{OH})_{2(aq,s)}$ and the low solubility of CO_2 in the pore solution. On one hand, the precipitation of carbonation products on the outer surface of $\text{Mg}(\text{OH})_{2(aq,s)}$ prevents its further contact with carbonate ions. On the other hand, the formation of brucite and precipitation of HMCs lower the pH of the solution, during which

the overall conversion of magnesium ions into carbonate phases is limited by the extent of the dissociation of carbonic acid into bicarbonate or carbonate ions [21].

Previous research [15] studied the incorporation of dypingite seeds into RMC formulations to provide additional nucleation sites within the prepared mixes, with the overall goal of increasing the surface area for carbonate formation. While the dispersion of seeds within the mix was not optimum due to the high seed content used in this study (i.e. 50% of the binder by mass), improvements in the carbonation reaction were limited as other factors, such as the dissolution of CO_2 , controlled the overall process. To overcome these issues and enhance the overall strength development of RMC mixes, this study focused on the improvement of carbonation via two main initiatives: (i) the inclusion of carbonate seeds (i.e. hydromagnesite ($4\text{MgCO}_3 \cdot \text{Mg}(\text{OH})_2 \cdot 4\text{H}_2\text{O}$)) to provide additional nucleation sites that can facilitate further carbonation within the prepared mixes and (ii) the increase of initial pH through the use of sodium bicarbonate (NaHCO_3) to improve the solubility of CO_2 within the pore solution.

The introduction of seeds can provide additional nucleation sites within the pore space and allow hydrate and carbonate phases to nucleate and grow on seed surfaces away from the original $\text{MgO}/\text{Mg}(\text{OH})_2$ grains. The increased area can facilitate further access to the unhydrated MgO and uncarbonated $\text{Mg}(\text{OH})_2$ particles. Accordingly, as carbonation proceeds, the nuclei of carbonation products do not only form on the surface of the $\text{MgO}/\text{Mg}(\text{OH})_2$ grains, but also around the seeds dispersed within the pore space. This dispersed formation of hydrate and carbonate phases can enhance the carbonation process, leading to an increase in the amount of HMCs that fill in the initially available pore space.

In addition to increasing the pH, the incorporation of NaHCO_3 in RMC systems provides HCO_3^- ions, enabling the continuation of the carbonation reaction and shifting the equilibrium towards the production of additional carbonate ions to react with the dissolved Mg^{2+} ions. The initially introduced HCO_3^- can also have a catalyzing effect on the dissolution of brucite at early stages due to the formation of multidentate mononuclear surface complexes that destabilize Mg-O bonds and the water coordination of Mg-atoms at the surface [22]. The reaction of the dissolved MgO with the additional NaHCO_3 can result in the formation of HMCs, thereby increasing the pH through the release of NaOH.

In line with these mechanisms, this study presents a detailed analysis on the performance and microstructural development of carbonated RMC concrete formulations involving the use of seeds and NaHCO_3 at various concentrations. Concrete samples were prepared and subjected to carbonation curing under a CO_2 concentration of 10% for up to 56 days. The influence of these additives on the hydration kinetics of RMC was evaluated by isothermal calorimetry and pH measurements. The performance of the prepared formulations was analyzed at different durations by compressive strength testing. X-ray diffraction (XRD), thermogravimetry-differential thermogravimetry (TG-DTG), and scanning electron microscopy (SEM) were employed to analyze the formation of hydration and carbonation products and investigate the microstructural development at the end of the curing process.

2 Materials and Methodology

2.1 Materials

The main cement binder used in this study was RMC (commercial name “calcined magnesite 92/200”) obtained from Richard Baker Harrison (UK), which was produced at around 1000 °C. The chemical and physical properties of RMC are shown in Table 1. The reactivity of RMC (i.e. measured by the time required for the neutralization of 0.25 M of acetic acid by 5 grams of RMC [23]) was recorded as 520 seconds. Sodium bicarbonate (SBC), obtained from VWR (Singapore), was used at a concentration of 0.05-0.15 M to promote the carbonation process. Hydromagnesite ($4\text{MgCO}_3 \cdot \text{Mg}(\text{OH})_2 \cdot 4\text{H}_2\text{O}$) seeds, obtained from Fisher Scientific (UK) with a specific surface area of $43.5 \text{ m}^2/\text{g}$, were used at a fixed amount corresponding to 0.5% of the total binder (by mass) to provide additional nucleation sites and stimulate the nucleation and growth of HMCs in the pore space. Fig. 1 shows the microstructure of hydromagnesite seeds, which possessed a round ball-like shape with a diameter of $\sim 1\text{-}5 \text{ }\mu\text{m}$, composed of $\sim 0.5 \text{ }\mu\text{m}$ diameter disks. Saturated surface dry (SSD) gravel with a particle size of 4.7–9.5 mm was used to form the aggregate profile in the prepared concrete samples. The compositions presented in this study did not include any fine aggregates to enable the extraction of carbonated paste from the concrete samples without any contamination and to ensure accurate quantification of the hydrate and carbonate phases via XRD and TG-DTG.

2.2 Methodology

Two sets of samples containing different concentrations of SBC (i.e. with and without 0.5% seeds), as listed in Table 2, were designed to investigate the influence of SBC and seeding on the hydration and carbonation of RMC formulations. The control sample (CS) was prepared without any additives to provide a comparison with samples including seeds (S) and SBC. Each set included SBC at a concentration of 0.05 M (samples SBC0.05 and SBC0.05-S), 0.1

M (samples SBC0.1 and SBC0.1-S) and 0.15 M (samples SBC0.15 and SBC0.15-S) along with samples without any SBC (CS and S). The solution to binder ratio was kept constant at 0.7 for all samples. The initial pH of water without any SBC was recorded as 7.80, whereas those of 0.05-0.15 M SBC ranged between 8.73-8.84. Before casting the concrete samples, corresponding pastes were prepared to study the effects of solutions involving SBC and seeds on the hydration of MgO via isothermal calorimetry and pH measurements.

For the preparation of concrete samples, seeds were dispersed in water/SBC solutions by a magnetic stirrer, which were then added to the dry mix as a part of the mixing solution. After mixing for 10 minutes, the concrete samples were cast into 50×50×50 mm cubic molds, consolidated by a vibrating table and trowel finished. All samples were then subjected to 10% CO₂, 30±2 °C and 80±5% relative humidity (RH) in an incubator. The CO₂ concentration of 10% was chosen to maximize the carbonation of samples, in line with the findings of a previous study [16]. After 24 hours, during which all samples reached initial strengths of > 1.5 MPa, they were demolded and subjected to 10% CO₂, 30±2 °C and 80±5% RH for up to 56 days.

2.2.1 Isothermal calorimetry

The heat flow and cumulative heat evolved due to the hydration of each mixture were studied at 30 °C by an I-Cal 2000 High Precision Calorimeter in accordance with ASTM C1702 – 15a [24]. To prepare the paste samples, RMC was added to water or SBC solutions with or without seeds, which were previously heated to 30 °C in order to produce mixes at the same

temperature as the measurement temperature. The prepared mixes were instantaneously placed into the isothermal calorimeter channel to measure the heat of hydration.

2.2.2 pH measurement

Paste samples were prepared and exposed to carbonation curing (10% CO₂, 30±2 °C and 80±5% RH) for up to 28 days for pH measurements. The pH values of each sample were determined at different ages by crushing and grinding the samples and dispersing the fragments in water. After 20 minutes of mixing, the solids were filtered through a vacuum and the pH was measured by using a Mettler Toledo pH meter with an accuracy of ±0.01. All the measurements were conducted in triplicates and the average values were recorded.

2.2.3 Compressive strength

The compressive strength of concrete samples was measured by uni-axial loading in triplicates at 3, 7, 14, 28 and 56 days. The equipment used for this purpose was a Toni Technik Baustoffprüfsysteme machine, operated at a loading rate of 55 kN/min.

2.2.4 XRD, TG-DTG and SEM analyses

Representative samples, extracted from the cubes crushed during strength testing and selected by quartering, were stored in acetone for 24 hours to stop hydration, followed by vacuum

drying for 24 hours in preparation for XRD, TG-DTG and SEM analyses. The vacuum dried samples were ground down to pass through a 75 μm sieve before they were analyzed under XRD and TG-DTG.

XRD was recorded on a Philips PW 1800 spectrometer using Cu K_{α} radiation (40 kV, 30 mA) with a scanning rate of 0.04° 2θ /step from 5 to 80° 2θ . The RIR technique [25-27] was applied for the quantitative analysis of the phases present by making use of a standard phase with a known proportion, whose integrated intensity was compared to that of the phase of interest. The internal standard used for quantification purposes was fluorite (CaF_2) included at 5 wt.%. Calibration curves for MgO and $\text{Mg}(\text{OH})_2$, the two main components used for quantitative analysis, are straight lines through the origin i.e. $y = kx$, where x is the RIR of the phase analyzed and y is the weight fraction of the component investigated. The value of k is 0.2886 for MgO and 0.3651 for $\text{Mg}(\text{OH})_2$ [7]. The RIR results were acquired by dividing the integrated intensity of the strongest line of the phase with that of the standard. All results were obtained by taking the average of 2 measurements with a standard deviation of $< 3\%$.

TG-DTG was conducted on a Perkin Elmer TGA 4000 equipment from 40 to 950°C with a heating rate of $10^{\circ}\text{C}/\text{min}$ under nitrogen flow. To quantify the amount of mass loss at each decomposition process, DTG curves were deconvoluted according to the Gaussian area deconvolution method using Origin 2017 software, which revealed the area corresponding to the decomposition process.

The vacuum dried samples were mounted onto aluminum stubs using double-sided adhesive carbon disks and coated with gold before SEM analysis. SEM was carried out with a Zeiss

Evo 50 microscope to investigate the morphologies of the hydration and carbonation products within the prepared samples.

3 Results

3.1 Isothermal calorimetry

Fig. 2 (a) and (b) present the heat flow and cumulative heat of RMC pastes during the first 24 hours of hydration, respectively. The heat flow results indicated that the dissolution of MgO and nucleation of brucite occurred a few minutes after mixing for all samples. When compared to the control sample (CS), the higher initial pH of SBC0.05, SBC0.1 and SBC0.15 samples resulted in lower exothermal peaks due to the slower dissolution of MgO. The introduction of seeds in the S sample led to a higher heat flow than CS at early stages (i.e. ≤ 5 hours) due to the increased hydration of MgO through the provision of additional nucleation sites within the pore space. These additional nucleation sites stimulated the nucleation and growth of brucite away from the MgO grains, thereby increasing the contact surface of MgO with water and enhancing the initial dissolution of MgO.

Samples involving the simultaneous use of seeds and SBC indicated the highest initial heat flow peaks amongst all samples. Within these formulations, the additional nucleation sites induced by the seeds enhanced the hydration reaction and stimulated the formation of carbonate phases that formed as a result of the reaction between Mg^{2+} and OH^- (i.e. from the dissolution of MgO) and HCO_3^- (i.e. from SBC), as shown in Equations 1 and 2, which may have contributed to the higher heat flow observed during the first 2 hours. The increase in the

heat flow was correlated with the SBC content within each sample, which corresponded to the concentration of HCO_3^- available for the carbonation reaction. The composition of $x\text{MgCO}_3 \cdot y\text{Mg}(\text{OH})_2 \cdot z\text{H}_2\text{O}$ depended on the concentration of $[\text{Mg}^{2+}]$, $[\text{OH}^-]$ and $[\text{CO}_3^{2-}]$ ions as the solubility product constant of HMCs varied from $10 \times 10^{-38.3}$ (i.e. hydromagnesite) to $10 \times 10^{-5.7}$ (i.e. nesquehonite) [28-30]. Although they had a notable influence on the initial reaction kinetics by stimulating dissolution and nucleation at early stages, the inclusion of seeds and/or SBC did not reveal a higher cumulative heat than CS during the first 24 hours. This was attributed to the increase in pH in the presence of SBC, which slightly lowered the overall hydration degree, while stimulating the subsequent carbonation reaction.

3.2 pH

Fig. 3 presents the pH values of all samples subjected to carbonation for up to 28 days. The initial pH values of fresh pastes containing SBC were higher than those of CS and S pastes (i.e. 10.50-10.59 vs. 10.18-10.22). These higher initial pH values of SBC pastes were in line with the trend observed in the pH values of the individual solutions (8.73-8.84 for SBC vs. 7.80 for CS) and could be attributed to the reaction between $\text{Mg}(\text{OH})_{2(\text{aq})}$ from the dissolution of MgO and SBC, as shown in Equation 3. The increase in the degree of this reaction, which led to the formation of HMCs and the release of NaOH, enabled the higher pH values observed in samples containing SBC. Alternatively, the slight reduction observed in the

initial pH values with the introduction of seeds could be associated with the increased nucleation and precipitation of $\text{Mg}(\text{OH})_{2(\text{s})}$, reducing the hydroxide ions.

When compared to the pH values of the fresh pastes, a notable decrease in pH was revealed during the first 3 days of carbonation in all samples. This was partially due to the precipitation of brucite when the concentration of Mg^{2+} and OH^- ions reached supersaturation (i.e. $[\text{Mg}^{2+}]x[\text{OH}^-]^2 > \text{solubility product constant of } \text{Mg}(\text{OH})_2$ (e.g. $K_{\text{sp}} = \sim 10 \times 10^{-11.2}$) [31, 32]). Furthermore, the pH value was reduced by the dissolution of CO_2 in the pore solution (Equations 4 and 5) to give H^+ and CO_3^{2-} ions (Equation 6), which neutralized NaOH (Equation 3) and reacted with $\text{Mg}(\text{OH})_{2(\text{aq})}$ to form HMCs (Equation 2). Despite their lower initial pH values, CS and S samples demonstrated a similar pH as those of SBC samples after 3 days of carbonation, which ranged between 10.10-10.19. As the duration of carbonation increased, a slight increase in pH was observed after 3 days, which could be attributed to the continuation of the hydration reaction [10, 11] and the increase in OH^- concentration as water was consumed in the hydration and carbonation reactions.

$\text{CO}_{2(\text{g})}$ dissolves in water and forms carbonic acid:

H_2CO_3 dissociates in high pH solution:

During the entire curing process of 28 days, samples containing SBC generally revealed higher pH values than others. A comparison of the initial pH values of the fresh pastes with the pH values reported after 28 days of carbonation revealed a noticeable reduction in the pH of samples containing SBC, whereas those of CS and S remained almost unchanged. This difference in the reduction of pH could be an indication of higher CO₂ dissolution and hence carbonation in SBC samples when compared to CS and S. The higher pH values of SBC samples due to the formation of NaOH, as shown in Equation 3, could be associated with their ability to carbonate even further at longer curing durations by improving the solubility of CO₂ (Equations 4 and 5), thereby continuing to gain strength after 28 days.

The pH of the pore solution was adjusted by the dissolution of MgO and the precipitation of Mg(OH)₂, which were neutralized by the dissolution of CO₂ and affected by the presence of seed particles. The higher initial pH of solutions containing higher contents of SBC inhibited the dissolution of MgO, reducing the release of OH⁻ and thereby resulting in a narrow range of pH. The presence of seed particles also stimulated the precipitation of brucite and HMCs. Therefore, the pH of seeded samples was generally lower than non-seeded samples. The higher initial pH in the presence of SBC also led to a higher dissolution of CO₂, which later neutralized the alkali solution. In this respect, no direct linear relationship between the SBC dosage and pH could be identified at early or later ages.

3.3 Compressive strength

The compressive strength development of all concrete samples over 56 days of curing is shown in Fig. 4. All samples demonstrated an increase in strength with time, which was an

323 indication of the continuous progress of hydration and carbonation during the curing process.
324 When compared with unseeded samples, the introduction of seeds into RMC formulations led
325 to a noticeable improvement in compressive strength. This was also apparent by the rate of
326 strength gain, which was much higher in seeded samples during the first 28 days of curing. In
327 comparison to CS, the simultaneous introduction of seeds and SBC had a significant
328 influence on compressive strength, which continued to increase until 56 days. The slower
329 strength development of CS that did not include any SBC was associated with the reduction
330 in the rate of carbonation at longer durations, in contrast to those of SBC samples that
331 demonstrated a continuous carbonation at longer durations (> 14 days), leading to
332 significantly higher strengths.

333
334 The first few days of carbonation revealed similar strengths in all samples, whereas the
335 influence of seeds and SBC on strength development was more apparent at ≥ 7 days. The
336 individual use of seeds (i.e. without any SBC) in the S sample led to 96% and 53% increase
337 in the strength of CS at 28 days (47 MPa vs. 24 MPa) and 56 days (55 MPa vs. 36 MPa),
338 respectively. This significant increase in the strength development of the S sample was
339 attributed to the active role of seeding in stimulating the nucleation of hydration and
340 carbonation products away from the original MgO particles within the pore space. This
341 improvement in the arrangement of particles facilitated the creation of extra contact surface
342 area for further carbonation within seeded samples. As a result, the compressive strength of
343 the S sample at 14 days (33 MPa) almost reached that of CS at 56 days (36 MPa), enabling a
344 reduction in the curing duration by 42 days.

345
346 A similar trend was observed in the strength development of SBC samples (i.e. with or
347 without seeds), where the influence of these additives was not as obvious during the early

stages of curing. For instance, the 3-day compressive strengths of SBC samples without any seeds were less than that of CS (7-9 MPa vs. 11 MPa) due to the reduction in the dissolution of MgO and the associated formation of brucite at higher pH values provided by the inclusion of SBC. However, SBC samples demonstrated a faster strength development than CS as the duration of curing increased, which was associated with the benefits of their higher pH under carbonation. The use of SBC not only increased the pH of the pore solution and thereby the dissolution of CO₂, but also provided further HCO₃⁻ that stimulated the carbonation reaction and enhanced the formation of HMCs (Equations 3-6). This increase in the amount of carbonates was reflected as higher strengths within SBC samples at longer durations. The individual use of 0.15 M SBC within the SBC0.15 sample translated into 61% higher strengths than CS at 56 days (58 vs. 36 MPa). Amongst unseeded samples, the amount of SBC introduced into the initial mix design was influential in strength development, which was apparent by the higher 56-day strengths obtained by the SBC0.15 and SBC0.1 samples in comparison to SBC0.05.

While the individual use of seeds (S sample) or SBC (SBC0.05, SBC0.1 and SBC0.15 samples) both had notable influences on the strength development of RMC formulations, the use of seeds by themselves increased the rate of strength gain and led to higher strengths than SBC samples during the first 28 days of curing. However, a different scenario was observed at longer durations, during which the use of ≥ 0.1 M SBC revealed an increase in strength gain and resulted in the same strength (~56 MPa) as the S sample at 56 days. The delayed strength development of SBC samples was due to the slowing down of the dissolution of MgO under the high pH provided by SBC during the initial stages of curing, whereas the presence of seeds enhanced the carbonation process starting with early ages.

The formulations involving the simultaneous use of seeds and SBC achieved the highest strengths amongst all samples investigated in this study, which was more apparent at later ages. For instance, the simultaneous use of seeds and SBC led to similar strengths as the S sample at ≤ 14 days, whereas these samples outperformed all others after 14 days. Similar to unseeded samples, the compressive strength of SBC0.1-S and SBC0.15-S samples was higher than that of SBC0.05-S at 56 days. When compared to CS, samples containing seeds and SBC demonstrated up to 142% and 100% higher strengths at 28 days (58 MPa vs. 24 MPa) and 56 days (72 MPa vs. 36 MPa), respectively. This significant enhancement in the strength development, which was much higher than the advancements offered by the individual use of seeds or SBC, was associated with the increase in the hydration and carbonation reactions enabled by the combined use of these additives. Within these formulations, the presence of seeds facilitated the increased formation of hydrate and carbonate phases via the presence of additional nucleation sites that provided extra surfaces for phase formations starting with early ages. Furthermore, SBC enhanced the dissolution of CO_2 under the high pH provided and increased the CO_3^{2-} concentration in the pore solution, thereby stimulating the continuous formation of HMCs at later ages. Therefore, the combination of these mechanisms not only led to higher initial strengths than CS, but facilitated the continuation of the carbonation reaction and associated strength development in the longer durations.

3.4 XRD

The XRD patterns of all samples subjected to carbonation for 28 days are shown in Fig. 5. In addition to the formation of HMC phases such as nesquehonite, dypingite and hydromagnesite, unhydrated MgO (main peak at $42.9^\circ 2\theta$) and uncarbonated brucite (main

peak at $38.1^\circ 2\theta$) was observed in samples with or without seeds and/or SBC. A comparison of these residual phases based on the intensity of the internal standard, fluorite (main peak at $28.2^\circ 2\theta$), revealed the lower intensities of uncarbonated brucite in samples including seeds and/or SBC than that of CS. The detailed quantification of unhydrated MgO and uncarbonated brucite within each sample after 28 days of carbonation is provided in Table 3.

When compared to CS, the sole inclusion of seeds in the S sample reduced the uncarbonated brucite content from 50% to 38%, also revealing lower unhydrated MgO contents (34% vs. 32%). This increase in the utilization of brucite confirmed the role of seeds in extending the reactive surface of brucite for carbonation by enabling its nucleation on seed surfaces, in addition to the MgO grains. A similar trend was also observed in SBC samples (without any seed addition). The uncarbonated brucite within SBC0.05, SBC0.1 and SBC0.15 samples was reduced from 50% in CS to 38-44%, whereas the MgO contents of these samples ranged between 30-36%, similar to that of CS (34%). This obvious improvement in the carbonation of SBC samples was due to the increased initial pH provided via the use of SBC, which enhanced the dissolution of CO_2 . Moreover, the presence of additional HCO_3^- ions within the initial mix design enabled the continuation of the carbonation reaction and shifted the equilibrium towards the increased formation of HMCs.

In line with the compressive strength results, the simultaneous inclusion of seeds and SBC in RMC formulations resulted in the lowest amounts of uncarbonated brucite in SBC0.05-S, SBC0.1-S and SBC0.15-S samples, whose brucite contents was reduced by up to 58% when compared with CS (21-35% vs. 50%). Considering the similar residual MgO contents of these samples with the CS (34-35% vs. 34%), the main contribution of these additives was attributed to the continuation of the carbonation reaction, which involved the conversion of

brucite into strength providing carbonate phases. The simultaneous use of seeds and SBC not only stimulated the dissolution of CO₂ within the pore solution, but also provided extra surface area for the carbonation of brucite and precipitation of HMCs, thereby leading to significantly higher strengths than all other samples presented in this study.

3.5 TG-DTG

Fig. 6 reveals the TG and DTG curves of all samples after 28 days of carbonation. Corresponding well with the compressive strength results, the higher mass loss of samples containing seeds and/or SBC in comparison to CS was an indication of their higher reaction degrees and hence, the amount of hydrate and carbonate phases within each formulation. The decomposition temperatures of each phase were determined based on the endothermic peaks on the DTG curves. In addition to the endothermic peak at 120 °C, another one corresponding to the dehydration of HMCs was observed at 220 °C in samples containing seeds and/or SBC. A strong endothermic peak responsible for the decomposition of uncarbonated brucite, accompanied with the dehydroxylation (e.g. hydromagnesite/dypingite) and decarbonation (e.g. nesquehonite) of HMCs was observed at around 380-400 °C. This was followed by the decarbonation of HMCs until 950 °C. Furthermore, samples containing seeds demonstrated a small peak at ~510 °C, which could be attributed to the decomposition of seeds [33, 34], in addition to the increased amount of HMC formation within these samples. In summary, the three main decomposition steps observed in all samples were consistent with the patterns presented in previous studies [8, 35-40], as listed below:

(i) 50 to 280 °C: Dehydration of water bonded to HMCs (e.g. nesquehonite, hydromagnesite and dypingite) [8, 35, 37-40].

(ii) 280 to 550 °C: Decomposition of uncarbonated brucite, dehydroxylation of HMCs (e.g. nesquehonite, hydromagnesite and dypingite) and decarbonation of HMCs (e.g. nesquehonite) [8, 36-38, 40].

(iii) 550 to 900 °C: Decarbonation of HMCs [8, 38].

Table 4 presents the mass loss associated with each decomposition process obtained by calculating the area under the corresponding deconvoluted DTG curves. In line with the strength and XRD results, the inclusion of seeds and/or SBC demonstrated an improvement in the overall carbonation degree. This was obvious from the mass loss due to the decarbonation, which was up to 39% and 69% higher in samples containing SBC or seeds in comparison to CS (4.2% and 5.1% vs. 3%), respectively. This increase in the amount of CO₂ absorbed was more pronounced in samples containing both seeds and SBC (SBC0.05-S, SBC0.1-S and SBC0.15-S samples). When compared to CS, the mass loss due to the decarbonation within these samples increased by up to 115% (5.6-6.5% vs. 3%). This increase was a clear indication of the enhancement of carbonation and corresponding mechanical performance demonstrated by RMC samples involving the simultaneous use of seeds and SBC.

3.6 Microstructural analysis

The microstructures of unseeded (CS, SBC0.05, SBC0.1 and SBC0.15) and seeded (S, SBC0.05-S, SBC0.1-S and SBC0.15-S) samples after 28 days of carbonation are shown in

Fig. 7 and Fig. 8, respectively. Along with the dominant presence of needle-like nesquehonite, disk/rose-like hydromagnesite/dypingite was observed in all samples. The formations of these HMC phases were the main source of strength development in RMC formulations. The binding strength provided by the dense network established via the interlocking of these carbonates, coupled with their pore filling effect, can explain the mechanical performance of each sample prepared in this study. In this respect, the two main parameters considered during this process were the amount and morphology of the carbonate phases, whose details are elaborated further below.

The microstructure of the control sample (CS) revealed the formation of disk-like hydromagnesite/dypingite with a diameter of $\sim 2\text{-}4\text{ }\mu\text{m}$, which were blended amongst nesquehonite needles ($\sim 0.5\text{-}1\text{ }\mu\text{m}$ diameter and $\sim 4\text{-}5\text{ }\mu\text{m}$ length), as seen in Fig. 7 (a). The introduction of SBC in the SBC0.05 sample led to a similar microstructure as CS, albeit with slightly larger hydromagnesite/dypingite disks with a diameter of $\sim 2\text{-}5\text{ }\mu\text{m}$, which formed on top of a solid layer of nesquehonite ($\sim 1\text{-}2\text{ }\mu\text{m}$ diameter and $\sim 6\text{-}15\text{ }\mu\text{m}$ length), as seen in Fig. 7 (b). The dimensions of nesquehonite needles grew with SBC content, reaching $1.5\text{ }\mu\text{m}$ in diameter and $> 10\text{ }\mu\text{m}$ in length within samples SBC0.1 (Fig. 7 (c)) and SBC0.15 (Fig. 7 (d)). When compared to CS, the densification of the microstructure via the formation of large carbonate crystals contributed to the improvement in the strength of SBC samples. This was associated with the role of SBC in enhancing the carbonation reaction by providing an increased concentration of CO_3^{2-} , thereby stimulating carbonation and the subsequent formation of carbonate phases.

In addition to common HMCs such as nesquehonite and hydromagnesite/dypingite, round ball-like hydromagnesite seeds were observed in the S sample (Fig. 8 (a)). The role of seeds

in providing additional nucleation sites within the prepared RMC formulations was revealed via the growth of nesquehonite around the seed surfaces. The microstructures of the seeded samples showed the seeds to be acting as micro-aggregates that facilitated the formation of nesquehonite needles that blended into larger crystals. Furthermore, the presence of seeds also contributed to the densification of the microstructure by filling in the available pores amongst the individual nesquehonite needles. The formation of a dense nesquehonite layer composed of crystals with a diameter of $\sim 0.5\text{-}1\text{ }\mu\text{m}$ and length of $> 10\text{ }\mu\text{m}$ enabled the S sample to reveal much higher strengths than CS.

The simultaneous inclusion of seeds and SBC revealed much denser microstructures than all other samples, which was linked with the higher carbonation degrees of these samples via the provision of additional nucleation sites, as well as higher pH and increased CO_3^{2-} concentration. The associated improvement in carbonation was observed by the formation of a dense wall-like structure composed of interconnected (i.e. without any visible gaps in-between) nesquehonite needles within SBC0.05-S (Fig. 8 (b)), SBC0.1-S (Fig. 8 (c)), and SBC0.15-S (Fig. 8 (d)) samples. The larger crystal size of nesquehonite within SBC0.15-S (Fig. 8 (d)) than SBC0.1-S (Fig. 8 (c)) could explain the slightly higher 28-day compressive strength of the former. The intermingling of individual nesquehonite needles (up to $2\text{ }\mu\text{m}$ diameter and $8\text{-}10\text{ }\mu\text{m}$ length) into a dense plate contributed to the improvement of the mechanical performance of these samples. In addition to the dense nesquehonite plates, rose-like hydromagnesite/dypingite crystals that formed on top of nesquehonite needles were seen in the SBC0.05-S sample (Fig. 8 (b)). These hydromagnesite/dypingite crystals could be distinguished from the hydromagnesite seeds that were introduced within the initial mix design, which possessed a round ball-like morphology (Fig. 1). The well-dispersed seeds served as micro-aggregates that filled in the initially available pore space, thereby reducing

porosity while facilitating the increased formation of HMCs, which were a product of the enhanced carbonation process enabled via the simultaneous use of seeds and SBC in RMC formulations.

4 Conclusions

The performance of carbonated RMC-based concrete samples primarily depends on the extent of the carbonation process as well as the morphology of the final carbonate phases. However, the progress of the carbonation reaction is limited by the formation of a carbonate layer around the unreacted particles, thereby creating a physical barrier that prevents further contact between unreacted MgO/Mg(OH)_2 with CO_3^{2-} for continuous carbonation. Moreover, the precipitation of brucite and HMCs lowers pH, thereby reducing the dissolution of CO_2 and the concentration of CO_3^{2-} available for carbonation. These limitations can lead to large amounts of uncarbonated Mg(OH)_2 , resulting in a limited strength development and an ineffective use of RMC as a binder.

With these limitations in mind, this study aimed to extend the amount of reactive surface and enhance the dissolution of CO_2 within the pore space to increase the extent of carbonation. The increase in reactive surface was enabled via the introduction of well-dispersed seeds into the pore space. The presence of seeds facilitated the nucleation of $\text{Mg(OH)}_{2(\text{aq,s})}$ on their surfaces in addition to the MgO grains, thereby increasing the total area for the formation of HMCs. Furthermore, the dissolution of CO_2 within pore solution was enhanced via the use of SBC, which led to a high initial pH that provided the necessary environment for CO_2 dissolution. SBC increased the carbonation of RMC not only by increasing the pH but also by

providing HCO_3^- ions that enabled the continuation of the carbonation reaction and shifted the equilibrium towards the increased production of HMCs.

The influence of seeds and SBC within RMC formulations was studied by investigating the strength and microstructural development of two sets of concrete samples prepared with (i.e. 0.5% of the total binder by mass) and without seeds, containing different concentrations of SBC (i.e. 0-0.15 M). The provision of extra surface via the introduction of seeds led to an increased area for the reaction of Mg-phases with the dissolved CO_2 . This improvement of the carbonation process at early ages reduced the uncarbonated brucite content and increased the formation of HMCs, meanwhile providing a 75% reduction in the total curing duration. In addition to their role as nucleation sites, seeds filled the available pore space and played the role of micro-aggregates, thereby forming a link in-between individual carbonate crystals that contributed to the formation of a dense microstructure. On the other hand, the use of SBC increased the initial pH and provided additional HCO_3^- , which enhanced the dissolution of CO_2 and facilitated the continuation of the carbonation reaction at later ages. The simultaneous use of seeds and SBC in RMC formulations not only stimulated the carbonation reaction at both early and later ages, but also led to the establishment of very dense microstructures composed of higher degrees of carbonate formation with larger crystal sizes. These improvements in the amount and morphology of carbonates translated into a significant enhancement in the mechanical performance of RMC-based concrete samples, resulting in up to 142% higher 28-day compressive strengths than the control sample.

Although the unreacted MgO particles were generally surrounded by brucite and HMC formations, which inhibited them from easily participating in any further reaction under ambient conditions, the slow diffusion of water over time could potentially result in the

further hydration of MgO, which can give rise to stability concerns in the long term. To mitigate any undesirable expansion caused by this extended hydration when the concrete has hardened, the prepared samples can be subjected to accelerated carbonation conditions for longer durations (e.g. 10-20% CO₂ for 56 days) to densify the microstructure via the formation of a thick carbonate layer, thereby reducing the permeability of water within the pore structure. Since the performance and durability of carbonated RMC formulations depend on many parameters such as the initial mix design and curing conditions, which control the hydration and carbonation degrees as well as the morphology of carbonates, the long-term dimensional stability of these samples should be evaluated in a further study investigating the influence of these factors.

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Table 1 Chemical composition and physical properties of RMC.

	Chemical composition (%)							Physical properties	
	MgO	SiO ₂	CaO	R ₂ O ₃	K ₂ O	Na ₂ O	LOI	Specific gravity (g/cm ³)	Specific surface area (m ² /g)
RMC	>91.5	2.0	1.6	1.0	-	-	4.0	3.0	16.3

Table 2 Compositions of concrete samples investigated in this study.

Mix label	Solution	Mix proportion (kg/m ³)			
	(type and concentration)	Solution	RMC	Seed	Aggregates
CS	H ₂ O				
SBC0.05	SBC 0.05 M		450	0	
SBC0.1	SBC 0.1 M				
SBC0.15	SBC 0.15 M	315			1360
S	H ₂ O				
SBC0.05-S	SBC 0.05 M		447.75	2.25	
SBC0.1-S	SBC 0.1 M				
SBC0.15-S	SBC 0.15 M				

Table 3 MgO and Mg(OH)₂ contents of all samples obtained by XRD after 28 days of carbonation.

Mix label	MgO (%)	Brucite (%)
CS	34.0	50.2
SBC0.05	36.3	39.7
SBC0.1	34.7	37.6
SBC0.15	30.3	44.1
S	32.3	38.2
SBC0.05-S	35.3	20.6
SBC0.1-S	33.5	35.4
SBC0.15-S	35.0	35.4

Table 4 Mass loss values of carbonated RMC samples obtained by TG-DTG after 28 days of carbonation.

Mix label	Mass loss (wt.%)			
	Dehydration	Dehydroxylation	Decarbonation	Total
CS	3.3	26.8	3.0	33.1
SBC0.05	7.3	26.1	4.0	37.4
SBC0.1	6.4	26.3	4.2	36.9
SBC0.15	8.4	26.8	4.0	39.3
S	8.6	25.9	5.1	39.6
SBC0.05-S	7.9	24.6	5.6	38.1
SBC0.1-S	9.8	25.4	6.5	41.7
SBC0.15-S	9.7	25.1	5.7	40.5

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(a)

(b)

(c)

(d)

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(a)

(b)

(c)

(d)

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