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Mechanical and microstructural changes in reactive magnesium oxide cement-based concrete mixes subjected to high temperatures

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Abstract

This study investigated the mechanical and microstructural changes in reactive MgO cement-based concrete cured under ambient and accelerated carbonation conditions, followed by exposure to high temperatures. The compressive strength of ambient-cured samples increased from 10 to 30 MPa when subjected to up to 200 °C, which was achieved via increased hydration of the remaining MgO. The accelerated formation of brucite at 50 °C enhanced the compressive strength of carbonated samples (58 vs. 65 MPa). A relatively stable performance (~56 MPa) was observed at temperatures ranging between 100 and 300 °C for the carbonated samples, associated with the additional formation of brucite and the transition of nesquehonite and hydromagnesite to artinite. The hydrated magnesium carbonates (HMCs) forming around brucite acted as barriers and inhibited its dehydroxylation. The decomposition of brucite and HMCs at 400 °C caused a porous microstructure and a low residual strength (5-8 MPa) in both the ambient-cured and carbonated samples.

Keywords: Temperature; Microstructure; Carbonation; Stability; MgO; Reactive magnesium oxide cement

1 Introduction
In search of binders that are relatively more environmentally friendly than Portland cement (PC), researchers have identified reactive magnesium oxide (MgO) cement (RMC) as a promising candidate. The environmental benefits of the material include its ability to absorb and store atmospheric carbon dioxide (CO$_2$) permanently, its lower production temperatures (i.e., 700-1000 °C for PC) [1-5], and a possibility for full recyclability [6]. The limited global reserves of MgCO$_3$, which is used as the raw material to form RMC through calcination, could be overcome with seawater or reject brine from desalination as magnesium-rich sources in the production. In the process, alkali reagents such as lime, calcium hydroxide, dolime (CaO.MgO) [7, 8], hydrated lime (Ca(OH)$_2$·2H$_2$O) [9], sodium hydroxide (NaOH) [10, 11], and ammonia solution (NH$_4$OH) [12] are generally used to increase the pH of the feed solution and thus induce the precipitation of brucite (Mg(OH)$_2$). The precipitate can be separated by filtering and washing [7] and subsequently calcined to form MgO [11]. In comparison to the utilization of MgCO$_3$ as the raw material, the waste brine route would potentially help to offset the high embodied CO$_2$ content [13], of which 1.1 tonnes of CO$_2$ per unit tonne of MgO arises from the decomposition of MgCO$_3$ alone [14].

In addition to the sustainability consideration, one of the main engineering criteria for any material to be applied in construction is its ability to achieve the required mechanical properties. RMC-based composites gain strength through both hydration and carbonation processes. In the hydration process, MgO is converted into Mg(OH)$_2(aq,s)$ which provides a limited binding action for the matrix. The formation of Mg(OH)$_2(aq,s)$ is also associated with the release of OH$^-$ ions and the increase in pH value of the pore solution, thus stimulating the dissolution of CO$_2$. The dissolved CO$_2$ reacts with Mg(OH)$_2(aq,s)$ to form stable hydrated magnesium carbonates (HMCs) during the carbonation process. Needle-like nesquehonite (MgCO$_3$·3H$_2$O), disk/rosette-like hydromagnesite (4MgCO$_3$·Mg(OH)$_2$·4H$_2$O), dypingite (4MgCO$_3$·Mg(OH)$_2$·5H$_2$O), and acicular artinite (MgCO$_3$·Mg(OH)$_2$·3H$_2$O) are the most common HMCs formed in carbonated RMC concrete formulations [3, 4, 15]. The HMCs increase the matrix density as a result of their expansive nature and interconnected networks to promote the binding ability and the associated strength gain of RMC-based composites [16-19]. Thus, the degree of hydration and carbonation are the main factors in determining the performance of RMC samples.

Significant improvement in the performance of RMC-based composites has been achieved via the enhancement of the hydration and carbonation processes. The classical ball milling method
was used to modify the morphology and microstructure of reactive MgO, leading to an improved hydration reaction rate [20]. With the same water-binder ratio of 0.6, the use of hydrochloric acid (HCl) as a hydration agent (HA) at a concentration of 0.05 M enhanced the hydration rate of MgO and formation of Mg(OH)$_2(aq,s)$, resulting in an approximately two-fold increase in the 28-day compressive strength in comparison to a reference mix without the HA [21]. Similarly, the use of magnesium acetate ((CH$_3$COO)$_2$Mg) at 0.05 M concentration in RMC-based concrete mixes of a water-binder ratio of 0.55 was responsible for an increase in the hydration degree of MgO by 30% and in the compressive strength by 2.7 times after 14 days of curing [17]. Incorporation of nucleation seeding such as hydromagnesite was shown to accelerate the hydration of MgO and induce the formation of an amorphous carbonation phase to contribute to a significant strength enhancement of the composite [22]. By adding 3 wt. % of caustic magnesium oxide and chemical admixture to the RMC, 3D printing of complex structures with precise shape retention were achieved [23].

Carbonation via elevated CO$_2$ concentrations (i.e., 5-20%) stimulated the formation of HMCs and resulted in an increase of approximately 7 times in the 28-day compressive strength with respect to ambient carbonation [16, 17, 21, 24]. Moreover, the simultaneous implementation of the hydration agents and the accelerated carbonation increased the 28-day compressive strength by 14 times compared with the control RMC samples cured under the ambient condition (4 MPa vs. 56 MPa) [17]. Under a carbonation environment of 10% CO$_2$, simultaneous inclusion of (CH$_3$COO)$_2$Mg as a hydration agent and hydromagnesite as seeds improved the hydration and carbonation to result in a 64% improvement in CO$_2$ sequestration and a 46% increase in the 28-day compressive strength (48 MPa vs. 70 MPa) [18]. The HA or seeds alone led to a strength enhancement of only 42% and 33%, respectively, suggesting a synergy when the two techniques are implemented together. The significant enhancement in the mechanical performance within RMC samples with hydration agents and/or nucleation seeding under the accelerated carbonation was fundamentally attributed to an increase in the carbonation content and formation of HMCs of larger morphologies [5, 17, 18, 21, 25]. Supercritical CO$_2$ (scCO$_2$) was recently shown to lead to a mature strength development of RMC-based composites within hours of exposure and enhance the CO$_2$ sequestration content [26]. XRD phase quantification with Rietveld refinement showed that the HMCs were predominantly composed of nesquehonite and hydromagnesite under prolonged carbonation [27]. An investigation with SEM, transmission electron microscopy (TEM), electron energy-loss spectroscopy (EELS), and Raman spectroscopy also indicated the formation of an
amorphous phase containing Mg, O, H, and C under both ambient and concentrated CO₂ curing [22, 28].

With the compressive strength ranging from 10 to 70 MPa [17, 18, 29], RMC-based concrete is applicable in various areas where PC is currently used. Yet, the integrity of the composites under fire or high-temperature exposure has not been explored. Such understanding is critical and would contribute to possible design recommendations as the material gains traction in the construction industry. Indeed, every building material is vulnerable to fire hazards in a real-time environment. Therefore, its stability under high temperatures is vital not only to the overall structural integrity but also to the safety of the occupants of the structures. In this context, the fire resistance of PC-based concrete has been extensively studied [30-32]. Due to the lower thermal conductivity of concrete [33] and its ability to retain a considerable proportion of strength at high temperatures (200-400 °C) [34, 35], international building codes have traditionally set up guidelines for fire protection of structural components by specifying minimum concrete covers to protect the embedded reinforcement [36, 37]. On the other hand, the thermal decomposition of hydration and carbonation products such as nesquehonite, hydromagnesite, and brucite inherent in RMC-based concrete, were individually studied previously [38-41]. The loss of physically bound water from the HMCs occurs in the temperature range of 50-300 °C, whereas the dehydroxylation of brucite and HMCs takes place at nearly 300-500 °C, while the decarbonation starts at about 440 °C. The high thermal decomposition temperature, non-toxicity, and high specific heat make Mg(OH)₂ an attractive fire retardant additive for both polymers and natural fibers composites [42-44]. However, the thermal decomposition of brucite with and without the presence of HMCs as a composite has not been thoroughly investigated, not to mention its thermo-mechanical property evolution, relevance, and implications for building applications.

This study aims to assess the thermal stability of RMC-based concrete at elevated temperatures (50-400 °C) as specified in building code requirements [45, 46]. A comprehensive investigation was performed on RMC-based composites to provide new insights into the influence of high-temperature exposure on their mechanical and physicochemical properties. A focus was made on the evolution of the micro-mechanical, compressive strength, porosity, morphological and crystal properties of the hydration and carbonation phases. Concrete samples prepared with a hydration agent were cured under ambient and accelerated carbonation conditions. The thermal stability of the concrete was first assessed by its residual compressive strength and
microstructural changes after exposure to the elevated temperatures followed by cooling to room temperature. Porosity as an indicator of matrix densification and strength gain was then quantified with mercury intrusion porosimetry (MIP). The morphology evolution of the samples was analyzed by both scanning electron microscopy (SEM) and environmental scanning electron microscopy (ESEM). Meanwhile, the phase transformation was investigated with Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), thermogravimetric analysis (TGA), and confocal Raman spectroscopy [47-49]. Finally, nanoindentation, a technique generally used for characterizing the micro-mechanical properties of cement-based materials and their binding phases [50-55], was used to estimate the elastic modulus of the carbonated samples. The indentation results provided signatory elastic modulus data that could be assigned to phases in the composites.

2 Materials and Methods

2.1 Materials

RMC (commercial name “calcined magnesite 92/200”) used in this study was obtained from Richard Baker Harrison (UK). The physical and chemical properties of RMC are listed in Table 1. The reactivity of RMC (i.e., measured by the time required to neutralize 0.25 M of acetic acid by 5 grams of RMC [8]) was recorded as 520 sec. Magnesium acetate ((CH₃COO)₂Mg), obtained from VWR (Singapore), was used as the hydration agent (HA) at a concentration of 0.1 M (i.e., based on previous research [17]) to promote the hydration process. Saturated surface dry (SSD) gravel with a particle size of 4.7–9.5 mm was used as the aggregate for preparing the concrete samples.

2.2 Sample preparation and methodology

Table 2 shows the composition of the prepared concrete samples for this study. The mixture proportion was designed based on the preliminary results and previous studies [5, 18, 21]. (CH₃COO)₂Mg 0.1 M solution was used to improve the hydration and carbonation of RMC concrete samples [17, 56]. The ratio of (CH₃COO)₂Mg 0.1 M solution to binder was as high as 0.65 to achieve a workable mix due to the high water consumption of RMC as a result of its fine particle size distribution and porous microstructure. Fine aggregates were not included in order to enable the extraction of the carbonated paste from the matrix without any
contamination and to ensure accurate quantification of the hydration and carbonation phases via XRD and TGA. After mixing, the concrete was cast into 50 × 50 × 50 mm³ cubic molds, consolidated by a vibration table, and leveled by a trowel. After casting, one set of the samples was cured under accelerated carbonation with a CO₂ concentration of 20% (labeled as C20), while the second set was cured under the ambient condition with an average atmospheric CO₂ concentration of ~ 0.04% (labeled as Amb). For both sets, the relative humidity and temperature were maintained at 80 ± 5% and 30 ± 1.5 °C, respectively. All samples were demolded after 24 hours of casting, and, for compression strength measurement, they were exposed to the two curing conditions for up to 28 days to investigate the strength development.

The effect of high-temperature exposure was evaluated by heating the samples at 14 days to 50, 100, 200, 300, and 400 °C in an oven. A heating rate of 5 °C/min was employed to reach the desired temperature. The target temperature was held for 2 hours as per standard fire safety testing procedures for buildings [45, 46, 57]. Once the heating cycle was completed, the samples were cooled down to room temperature at 5 °C/min. The samples were annotated by C followed by the CO₂ concentration used for curing and T followed by the temperature at which they were heated for the adopted duration. Thus, the C20-T50 represents a sample cured under 20% CO₂ concentration and heated to 50 °C for 2 hours. All sample sets were prepared in triplicates, and the compression testing was performed. Paste fragments were extracted from the cubes crushed during the strength testing and were stored in isopropanol for at least seven days to stop hydration. The samples were then dried under a vacuum for another three days before use for all analyses that followed.

2.2.1 Compressive strength

The compressive strength of the concrete sample was measured by uniaxial loading in triplicates at 1, 3, 7, 14, and 28 days. The equipment used for this purpose was a Toni Technik Baustoffprüfsysteme machine, operated at a loading rate of 55 kN/min. The samples exposed to the high-temperature regimes were also tested at 14 days for residual compressive strengths within 24 hours after the heating to prevent further carbonation and carbonation.

2.2.2 Porosity
The average pore size distribution of the samples was determined using AutoPore IV 9500 high-pressure mercury intrusion porosimetry (MIP) system (Micromeritics) with a pressure capacity of up to 413 MPa (60,000 psi). The paste fragments approximately 10 mm in size from selected cubic samples were used to carry out the MIP analysis. The obtained MIP data was significantly scattered, and therefore the ‘locally weighted scatterplot smoothing (Lowess)’ was applied to get a better insight into the average pore size distribution of the samples.

2.2.3 Microstructural analysis

Scanning electron microscopy (SEM) was carried out with a Zeiss Evo 50 electron microscope to investigate the morphology evolution of the carbonation products within the prepared samples. Before the SEM analyses, the vacuum dried samples were mounted onto aluminum stubs using double-sided adhesive carbon tape. The whole assembly was subsequently coated with gold to prevent charging. To characterize the morphology, microstructure, and damage progression of the hydration and carbonation products, environmental SEM (ESEM) was performed on the 28-day sample with Quanta FEG 450 equipped with a heating stage. The sample was heated from 25 °C in a stepwise manner to 50, 100, 200, 300, and 400 °C at the rate of 20 °C/min. Before the image acquisition, the sample was held at these target temperatures for 30 minutes to reach equilibrium.

2.2.4 FTIR

The Fourier transformed infrared (FTIR) spectra of samples, used to identify functional groups of carbonation phases, were recorded using an FTIR spectrometer (Cary 670, Agilent) fitted with a diamond attenuated total reflectance (ATR) accessory. The samples were crushed into fine powders and deposited directly on to the diamond lens of the spectrometer. The spectra were then collected in the transmission mode with a wavenumber resolution of 1 cm\(^{-1}\). For each sample, 32 scans were recorded and averaged in the range of 800 cm\(^{-1}\) to 1900 cm\(^{-1}\), where the main vibration bands of HMCs were observed.

2.2.5 XRD and TGA for phase quantification
Powder X-ray diffraction (PXRD) patterns were recorded via Philips PW 1800 spectrometer using Cu Kα radiation ($\lambda = 1.54060$ Å) with a scanning rate of 0.017° per step in a 2θ range of 5° to 70°. The internal standard used for quantification purposes was calcium fluorite (CaF₂) mixed at a concentration of 2 wt.%. The quantitative phase analysis of the samples was obtained by the reference intensity ratio (RIR) technique [58-60], as described in the existing literature [18, 25].

Thermogravimetric analysis (TGA) was conducted on Perkin Elmer TGA 4000 equipment in a temperature range from 30 to 950 °C with a heating rate of 10 °C/min under nitrogen flow. To quantify the amount of different phases in each sample, derivative thermogravimetry (DTG) curves were deconvoluted according to the Gaussian area deconvolution method to reveal the area corresponding to the decomposition of each phase.

2.2.6 Confocal Raman microscopy

Structural characterization of the exterior surfaces of the respective paste samples was performed with Raman spectroscopy. The technique has been successfully used to identify chemical composition and atomic configuration of both anhydrous and hydrated phases of cement [48, 49, 61] and was shown to be sensitive to the carbon-oxygen (C-O) vibration bond at local atomic environments [62]. The features made it a good candidate to evaluate the prevalence of the carbonation phase in the RMC composites. The Raman spectra were recorded by raster scanning under a confocal Raman microscope, Alpha300 RA from WITec GmbH (Ulm, Germany), equipped with a Zeiss EC Epiplan 20x objective, a 488 nm laser and an SP2300i detector from Princeton Instruments (Acton, MA). The detector had a graze of 600 l/mm for scanning in low resolution from 120 to 4000 cm⁻¹. Calibration was performed on a silicon wafer using the 520 cm⁻¹ emission band prior to sample analysis. The area scanned in confocal Raman mode was 170 by 170 μm with 170 points per line and 170 lines per image under an integration time of 0.2 sec. Data analysis was performed by using the Project 5.0 software from Witec. The spectra were first recalibrated by zeroing the position of the Rayleigh band. An average spectrum of the whole scanned area was then obtained, on which the functions of cosmic ray removal, smoothing, and background subtraction were used.

2.2.7 Nanoindentation for micro-elastic property characterization
Sample fragments whose size ranged between 10-15 mm were embedded in epoxy using 30 × 30 mm cylindrical silicone molds. The pellet samples were ground with sandpaper prior to polishing by oil-suspended diamond bits sequentially with sizes of 9, 6, 1, and 0.05 µm. The finished surfaces achieved a root-mean-square (RMS) roughness of approximately 100 nm, measured by atomic force microscopy (AFM). Nano-indentation was then performed on the polished surface using a Keysight G-200 Nanoindenter equipped with a diamond Berkovich tip. A programmable technique where the hardness and modulus properties of the material were measured at a specified target penetration depth was adopted [50, 63]. A target penetration depth of 500 nm, about 5 times the surface roughness, was set to achieve an optimum interaction volume larger than the nanogranular globules [64] but smaller than the homogeneous isotropic region of a typical phase [65], thus to achieve repeatable and accurate test data. A surface detection stiffness of 200 N/m and a dwell period of 10 s at the maximum load (to account for thermal drift and creep) were implemented [64, 65]. A total of 64 indentations were made in an 8 × 8 matrix at a spacing of 50 µm per region per sample. In a typical indentation experiment, both elastic and plastic deformation occurs during loading, and as a result, the initial unloading branch of the load-displacement curve was used to quantify the elastic modulus. The elastic modulus (E_p) of the indented material was estimated from Eq. 1.

\[
\frac{1}{E_r} = (1-\nu^2)/E_p + (1-\nu_i^2)/E_i
\]

(1)

where E_r is the resultant or effective indentation modulus, \(\nu\) is the Poisson’s ratio of the test material, E_i and \(\nu_i\) are the elastic modulus and Poisson’s ratio, respectively, of the indenter. For a diamond tip, E_i = 1141 GPa and \(\nu_i = 0.07\) [63]. The deconvolution was based on the probability density functions (PDFs) of the tested results plotted with a bin size of 5 GPa. Lorentz distribution was chosen with manually selected initial peak locations. Iterations were subsequently performed to achieve a respective coefficient of determination R^2 greater than 0.99.
3 Results and Discussion

3.1 Compressive strength and thermal stability

Fig. 1 (a) shows the compressive strength of RMC samples until 28 days of curing. The samples under the accelerated carbonation condition (C20) exhibited a significant strength development, while the samples under ambient curing (Amb) produced relatively low compressive strength. The accelerated carbonation improved the compressive strength by 5 times after 3 days of curing. This proved the important role of carbonation in the strength development of RMC-based concretes. The initial rapid rate of carbonation enabled the C20 samples to achieve 51 MPa at 7 days. Afterward, pore reduction with the resulting diffusion control of CO$_2$ and the precipitation of carbonate phases on uncarbonated Mg(OH)$_2$ prevented the progress of the carbonation reaction and reduced the rate of strength gain [26, 70]. As a result, C20 samples exhibited a marginal strength gain to 58 MPa at 14 days and 60 MPa at 28 days. It is noted that due to diffusion control arising from the matrix densification, the carbonation level was postulated to be non-uniform throughout the sample depth. This was proven in a previous study with X-ray computed tomography based on a correlation between grayscale and sample density [27, 70]. Quantification of the sequestered CO$_2$ with TGA also confirmed the uneven carbonation where more extensive carbonation was observed for the exterior regions of RMC samples even after prolonged CO$_2$ exposure [26].

Fig. 1 (b) presents the residual compressive strength of the RMC samples after being subjected to the high-temperature regimes. The compressive strength of the Amb samples increased from 10 MPa to 15 MPa and 30 MPa when subjected to 100 and 200 °C, respectively. This significant increase in the compressive strength could be attributed to dehydration, which improved the cohesion and binding action of brucite, an increase in brucite content under exposure to high temperatures [56], and an improved morphology of the formed brucite. The second mechanism could be a result of the additional conversion of unhydrated MgO to brucite induced by an internal autoclaving condition under the high temperature and the evaporation of water [71, 72]. However, the compressive strength of the Amb samples slightly decreased to 25 MPa and then dropped sharply to 5 MPa when subjected to 300 and 400 °C, respectively. The decrease in the compressive strength within this temperature range could be attributed to the
dehydroxylation of brucite and decomposition of HMCs. The water loss from the dehydration and dehydroxylation at this high-temperature range can generate high vapor pressure inside the pores, leading to sample spalling [73]. On the other hand, the compressive strength of the C20 samples increased to 65 MPa when subjected to 50 °C, then slightly dropped and remained stable at nearly 56 MPa when subjected to temperatures ranging between 100 and 300 °C. However, like the Amb samples, the compressive strength of the C20 samples dropped drastically to 8 MPa when heated further to 400 °C.

3.2 MIP analysis

Fig. 2 (a) and (b) show the MIP pore size vs. pore volume distribution of the samples cured under ambient and accelerated carbonation conditions, respectively, followed by exposure to the high temperatures regimes. In the case of the Amb samples, the pore size distribution remained relatively stable with main peaks centered at 0.6 μm up until 100 °C. The large-range pore size for the Amb sample became smaller at 200 °C, attributable to the induced hydration of the remaining MgO. Diffused distribution peaks in size range of 0.01-0.1 μm were observed in all the Amb samples, confirming that smaller pores were also present in minor proportions. When heated further to 400 °C, the Amb-T400 sample showed a significant increase in the number of smaller pores, as indicated by another high-intensity distribution peak at 0.02 μm, attributable to the thermal decomposition of brucite that occurs at nearly 300 °C.

As expected, the accelerated carbonation caused an extensive formation of HMCs, which led to significant densification of the microstructure and a reduction in pore sizes. The main peaks occurred at 0.02-0.03 μm for the sample cured under 30 °C. A diffused peak, however, was seen at nearly 0.8 μm, representing a small proportion of larger pores. As compared to the Amb samples, the pore size distribution of the C20 samples was found to be much more sensitive to heat treatment. When heated at 100 °C, the primary distribution peak splits into multiple smaller peaks centered at about 0.02, 0.01, and 0.008 μm. The change was attributed to sample dehydration and likely desorption of water from HMCs, leading to a small decrease in the compressive strength. Heating further to 200 °C and 400 °C induced further formation of smaller pores below 0.005 μm, and larger pores with diffused peaks centered in the range of 0.3-0.7 μm were also noted. This resulted in microstructural heterogeneity and a sharp decline in the compressive strength. The large variation in pore size distribution at high temperatures
of the RMC samples cured under the accelerated carbonation condition could be closely
attributed to the decomposition onset of different HMCs and the loss of physically adsorbed
water. Detailed thermodynamic investigations of HMCs [74] revealed that under the ambient
pressure, nesquehonite began to dehydrate at temperatures as low as 46 °C and a reversible
transformation between the nesquehonite and hydromagnesite could occur at approximately 68
°C. On the other hand, the ambient RMC samples displayed greater temperature stability and
pore size distribution since they are primarily composed of brucite whose thermal
decomposition is initiated at nearly 270 °C.

3.3 SEM and ESEM

Fig. 3 and

Fig. 4 reveal the microstructures of the Amb and the C20 samples subjected to high
temperatures after 14 days of curing, respectively. The Amb samples with low carbonate
content showed a porous microstructure of tiny platelet brucite with size < 1 µm (Fig. 3 (a)), explaining its low compressive strength. The accelerated hydration, when subjected
to 50 °C, resulted in the additional formation of brucite and denser microstructure in the Amb-
T50 samples (Fig. 3 (b)). The improvement to the morphology of the hydration products was also achieved
when the Amb samples were subjected to 100 °C and 200 °C (Fig. 3 (c) and (d)) with disk-like morphology of brucite observed in the respective samples. The disk-like brucite agglomerated to form a rose-like structure with a diameter of ~1 µm in
the Amb-T100 sample and up to ~2 µm in the Amb-T200 sample. This development in brucite
morphology would contribute to the strength development when the Amb samples were heated
up to 200 °C. Agglomerated brucite of an irregular shape with a width of up to ~1 µm was also
seen in the Amb-T300 sample (Fig. 3 (c)).
Platelet-like brucite started to disappear due to dehydroxylation that occurred at 300 °C. Extensive dehydroxylation at 400 °C caused decomposition of brucite flakes, leading to a porous microstructure (Fig. 3 (f)) and the associated strength decline in the Amb-T400 sample relative to the Amb-T200 sample.

The accelerated carbonation facilitated the formation of HMCs and led to the dense microstructure of the C20 sample after 14 days of carbonation (Fig. 4 (a)). Rosette-like hydromagnesite (~1 μm diameter) formed on top of a solid layer of nesquehonite (~0.5 μm diameter) and provided the filler effect and binding properties in the samples. Cracking was observed on nesquehonite when the C20 samples were subjected to 50 °C (Fig. 4 (b)), attributed to its structural instability induced by the loss of bound water at 50 °C [40]. Despite the cracking, there was an increase in the size of nesquehonite as its diameter rose up to 1.2 μm. The improvement in the crystal size, along with the additional formation of brucite as noted in the Amb samples, explained the increase in the compressive strength of the C20-T50 sample when compared with the C20-T30 sample. In the C20-T100 sample, nesquehonite needles were observed to remain interlinked as a dense carbonate phase despite extensive damage to hydromagnesite (Fig. 4 (c)). When the C20 samples were further subjected to 200 °C and 300 °C, nesquehonite seemed to disappear, while hydromagnesite was reduced in size (Fig. 4 (d) and (e)) as also noted in [40]. As the heating proceeded to 400 °C, the dehydroxylation coupled with the decarbonation led to the decomposition of most of the HMCs and brucite, which resulted in the porous microstructure and a sharp strength decline in the C20-T400 sample.

ESEM images for the C20-T30 sample subjected to different heating temperatures are presented in Fig. 5. A focus was made on an area consisting of unreacted MgO (circled) and
HMCs, mainly of nesquehonite. Consistent with the SEM observation for the samples under the prolonged heating regimes, cracks in nesquehonite occurred at the temperature of 50 °C due to dehydration, as indicated by an arrow in Fig. 5 (b). However, as the temperature increased, the extent of cracking was noticeably less as compared with the same samples under the prolonged heating. The longer heating duration was postulated to provide more time for dehydration and chemical decomposition of the reaction products, thus resulting in a higher shrinkage and subsequent cracking. The decomposition of HMCs and a corresponding reduction in their unit volumes were observed when the temperature increased from 300 °C to 400 °C, as marked by dashed rectangles in Fig. 5 (e) and (f).

3.4 FTIR

Fig. 6 (a) and (b) show the FTIR spectra of the samples cured under the ambient and the accelerated carbonation conditions, respectively, and heated to temperatures ranging from 30 °C to 400 °C. In the case of the Amb samples heated to 30 °C, 100 °C, and 200 °C, the broad peak centered at 1420 cm\(^{-1}\) and an adjacent diffused shoulder at 1476 cm\(^{-1}\) corresponded to the hydromagnesite and dypingite phases [75]. The presence of nesquehonite could hardly be predicted through a diffuse band at 1648 cm\(^{-1}\), which collectively represented the OH\(^{-}\) bending vibration in water present in both the hydromagnesite and nesquehonite phases [76]. However, when the samples were heated to 400 °C, which was above the decomposition temperature (350 °C) of HMCs [77], the peak center shifted to 1458 cm\(^{-1}\) with a diffused shoulder at 1402 cm\(^{-1}\), representing the disintegration of HMCs. In the case of the C20 samples heated to 30 °C and 100 °C, the trend was similar except for a new diffused shoulder centered at 1517 cm\(^{-1}\), which was exclusive to the antisymmetric stretching vibration of CO\(_3^{2-}\) in the nesquehonite phase with a minor contribution from antisymmetric stretching vibration of CO\(_3^{2-}\) in the hydromagnesite phase, as reported in several studies [76, 78]. In addition, the low-intensity bands at 850 and 880 cm\(^{-1}\), corresponding to the bending vibration of CO\(_3^{2-}\), collectively represented the hydromagnesite and dypingite phases with a possible existence of the huntite phase [76]. This confirmed that the formation of a variety of HMCs was promoted under the accelerated carbonation, leading to microstructural densification and the associated significant gain strength. At 200 °C, the diffused shoulder at 1517 cm\(^{-1}\) disappeared, showing the instability of the nesquehonite phase. Near the decomposition temperature of HMCs (i.e. at 400 °C), the characteristic and broad HMCs peak centered at 1420 cm\(^{-1}\) disappeared and flattened out, which was consistent with a previous high-temperature FTIR study on nesquehonite [78].
This thermal decomposition of nesquehonite and other HMCs led to increased porosity and a sharp decline of mechanical properties, as evident in Fig. 1.

### 3.5 XRD

The XRD patterns of samples subjected to the high temperatures after curing under the ambient and the accelerated carbonation are shown in Fig. 7 (a) and (b), respectively. Periclase (MgO; PDF #00-043-1022) and brucite (Mg(OH)$_2$; PDF #00-007-0239) were observed with the main peaks at 42.9° and 38.1° 2θ, respectively. Dypingite (4MgCO$_3$·Mg(OH)$_2$·5H$_2$O; PDF #00-029-0857), nesquehonite (MgCO$_3$·3H$_2$O PDF #00-020-0669), hydromagnesite (4MgCO$_3$·Mg(OH)$_2$·4H$_2$O; PDF #00-025-0513), and artinite (Mg$_2$CO$_3$(OH)$_2$·3H$_2$O; PDF #00-006-0484) were the main carbonation products observed with the main peaks at 8.1°, 13.7°, 15.2°, and 32.8° 2θ, respectively. A comparison of the HMC phases at 14 days before heating (T30) based on the intensity of fluorite (main peak at 28.2° 2θ) as the internal standard revealed higher intensities of HMCs within the samples under the accelerated carbonation curing than the samples under the ambient curing. This higher HMCs content of the carbonated samples was responsible for their higher strength.

As the samples were heated to 50 °C (T50), the brucite content increased in both the samples under the ambient and accelerated carbonation conditions. This increase in brucite content proved the accelerated hydration of MgO under 50 °C. In addition to brucite, the increase in the formation of artinite within the C20 sample was obvious. The formation of artinite could be from the reaction of uncarbonated brucite with ambient CO$_2$ or dissolved CO$_2$ in the pore structure. After heating to 50 °C, the Amb samples retained their compressive strength despite the additional formation of brucite, while the C20 sample with the additional formation of brucite and artinite achieved an increase in the compressive strength by 7 MPa. This highlighted the important role of HMC formation in contributing to the strength development of RMC-based concretes.

As the heating proceeded further, the brucite and artinite contents of the Amb samples increased and were associated with a further increase in the compressive strength until 200 °C. Despite an increase in artinite, the C20 samples exposed to heating experienced an overall loss in the HMCs content and the compressive strength. Their reduction in the HMCs content could be due to the loss of the bound water at around 100 °C to result in a partial collapse of the
nesquehonite structure [40, 41]. The brucite content was gradually reduced when the samples were subjected to 300 °C due to its dehydroxylation. This was also associated with the small strength loss in both the Amb and the C20 samples. As the samples were heated to 400 °C, the artinite content decreased in the Amb samples due to its dehydroxylation, yet significantly rose in the C20 sample. The incline of artinite within the C20 sample could be attributed to its transient phase from the decomposition of other HMCs such as nesquehonite and hydromagnesite as governed by Eq. 2 and 3, respectively. The dehydroxylation and decarbonation after 400 °C led to an increase in the periclase content and the significant strength drop, as previously shown in Fig. 1.

2MgCO₃·3H₂O → Mg₂CO₃(OH)₂·3H₂O + CO₂ + 2H₂O  \quad (2)

4MgCO₃·Mg(OH)₂·4H₂O → Mg₂CO₃(OH)₂·3H₂O + 3MgO + 3CO₂ + H₂O  \quad (3)

The quantification of MgO within each sample is provided in Table 3. It is confirmed that the content of MgO in both the Amb and the C20 samples was reduced up to 200 °C due to its conversion to brucite. At higher temperatures, the MgO content increased as a result of dehydroxylation and decomposition of the HMCs. Also, a reduction in the pore solution pH was found to induce the hydration of MgO [17, 79]. In this study, it is postulated that the dissolution of CO₂ under the accelerated carbonation condition lowered the pore solution pH and consequently improved the hydration of MgO within the C20 samples. This mechanism led to lower unhydrated MgO contents within the samples cured under the accelerated carbonation compared to those cured under the ambient condition.

3.6 TGA

The TGA results of the samples subjected to the ambient and accelerated carbonation conditions and followed by exposure to the high temperatures are presented in Fig. 8 (a) and (b), respectively. Without heating, the carbonated samples at 14 days showed a higher mass loss than the corresponding ambient-cured samples. The higher mass loss within the C20 samples could be due to the additional decomposition of HMCs. An endothermic peak responsible for the dehydration of water bonded to HMCs was observed at ~100 °C. A strong endothermic peak corresponding to the decomposition of brucite, accompanied by the
dehydroxylation (e.g., artinite) and decarbonation (e.g., nesquehonite) of HMCs was observed at around ~360-380 °C. A shoulder peak related to the decarbonation of HMCs was observed at ~440 °C within the C20 samples. Furthermore, two endothermic peaks corresponding to the decarbonation of HMCs were observed at ~540 °C and 600 °C. The samples subjected to higher temperatures (e.g., T300 and T400) had both experienced partial dehydration and dehydroxylation, thereby giving lower mass losses than the samples exposed to lower temperatures (e.g., T30 and T50). The three main decomposition steps of all samples were consistent with the patterns presented in previous studies [38, 40, 80-84] and are summarized below:

Dehydration of water bonded to HMCs (e.g., artinite, nesquehonite, and dypingite) from 30 °C to 200 °C, as shown in Eq. 4 [40, 80-84]:

\[
x_{\text{MgCO}_3} \cdot y_{\text{Mg(OH)_2}} \cdot z_{\text{H}_2\text{O}} \rightarrow x_{\text{MgCO}_3} \cdot y_{\text{Mg(OH)_2}} + z_{\text{H}_2\text{O}}
\]  

Decomposition of uncarbonated brucite (Eq. 5), dehydroxylation of HMCs such as artinite and dypingite (Eq. 6) from 200 °C to 450 °C [40, 81-84]:

\[
\text{Mg(OH)_2} \rightarrow \text{MgO} + \text{H}_2\text{O}
\]  

\[
x_{\text{MgCO}_3} \cdot y_{\text{Mg(OH)_2}} \rightarrow x_{\text{MgCO}_3} + y_{\text{MgO}} + y_{\text{H}_2\text{O}}
\]  

And decarbonation of residual magnesium carbonate (Eq. 7) from 450 °C to 950 °C [40, 84]:

\[
\text{MgCO}_3 \rightarrow \text{MgO} + \text{CO}_2
\]  

The mass loss values associated with each of the hydration and carbonation phases obtained by calculating the area of their respective deconvoluted DTG curves are summarized in Table 4. With no high temperatures, all the mass losses would be associated with the dehydroxylation of brucite and HMCs formed during the hydration and carbonation period. The data in Table 4 thus represent the decomposition remaining after the high-temperature regimes. It is observed that the mass losses due to the dehydration in the C20 samples were more significant in comparison to the values in the Amb samples. This was attributed to the higher content of
HMCs formed during the accelerated carbonation of the C20 samples. According to Table 4, dehydration and dehydroxylation obtained from TGA exhibited a decreasing trend when the samples were heated beyond 200 °C, attributable to partial decomposition of the reaction products under the heat treatment. The decarbonation contents in the Amb sample remained stable until 300 °C, while the decarbonation in the C20 samples noticeably decreased when they were subjected to temperatures greater than 50 °C. In comparison to the Amb samples, the C20 samples subjected to 200-300 °C attained higher dehydroxylation. The finding suggested their higher hydration during the carbonation period and a decreased dehydroxylation rate of brucite due to a physical barrier effect induced by the formation of HMCs.

An increase in the mass loss due to dehydroxylation was noted in the Amb samples when they were heated to 100 °C (21.5 wt.% at 30 °C vs. 22.6 wt.% at 50 °C and 23.2 wt.% at 100 °C). The result confirmed that the enhanced hydration of the unhydrated MgO to form brucite under the heating regimes. Though, heating at 200 °C reduced the mass loss corresponding to dehydration and dehydroxylation of the Amb sample to 1.4 wt.% and 21.7 wt.%, respectively.

Interestingly, the compressive strength of the Amb sample under 200 °C significantly increased when compared to the samples under 100°C (as seen in section 3.1). One possible explanation for the phenomenon is the significant change in morphology of hydration and carbonation products (as shown by SEM), which improved the microstructure and consequently the compressive strength. It is also observed that the mass losses due to dehydration slightly decreased when the heating temperature rose from 200 to 300 °C (1.4 wt.% vs. 1.2 wt.%), while other products were left almost unchanged. This reduction in the dehydration was attributed to the loss of 5 MPa in the compressive strength of the Amb-T300 sample when compared to the Amb-T200 sample. When the heating temperature increased to 400 °C, significant decomposition corresponding to the dehydroxylation and decarbonation occurred. This resulted in the decrease in the total mass loss (27.4 wt.% vs. 19.8 wt.%) and was responsible for a 20 MPa reduction in the compressive strength within the Amb-T400 sample relative to the Amb-T300 samples (25 MPa vs. 5 MPa).

Similar to the Amb samples, the mass loss corresponding to the dehydroxylation within the C20 samples increased when they were heated up to 100 °C (22.8 wt.% vs. 25.6 wt.%), attributable to the additionally accelerated hydration of the unhydrated MgO. However, the decomposition of HMCs within the C20 samples occurred during the heating, as indicated by the corresponding decrease in the remaining dehydration. For heating from 30 °C to 50 °C, the
dehydration decreased from 9.6 wt.% to 9.2 wt.% while the decarbonation was lowered from 6.4 wt.% vs. 5.7 wt.%. Despite the decomposition of HMCs and the reduction of total mass loss (38.8 wt.% vs. 37.9 wt.%), the compressive strength of the C20-T50 sample was higher than that of the C20-T30 sample. At 100 °C, the decomposition due to the dehydration and decarbonation was offset by the additional hydration of unhydrated MgO, leading to a similar total mass loss in the C20-T50 sample (37.9 wt.) and the C20-T100 sample (38 wt.%). However, the loss of 9 MPa in the compressive strength of the C20-T100 indicated the dominant role of the HMCs in dictating the mechanical properties of the composite. At higher temperatures, the C20 samples experienced further decomposition of the hydration and carbonation products. This was reflected by their reduced total mass losses of 33.3 wt.% at 200 °C and 31.4 wt.% at 200 °C vs. 38.8 wt.% 100 °C. Though their compressive strengths were of a similar magnitude, and this reinforced the fact that in addition to the content of hydration and carbonation, the morphology played a critical role in strength retention. For the heating of 400 °C, the C20-T400 sample retained dehydroxylation of only 13.3 wt.% vs. 23.9 wt.% for the C20-T300. Aggravated by the remaining small dehydration (2.2 wt.) and decarbonation (4.3 wt.%), the sample underwent a drop of 45 MPa in the compressive strength, as indicated in Fig. 1 (b).

3.7 Confocal Raman microscopy

Fig. 9 presents Raman spectra obtained for the external surfaces of the samples under both the ambient and accelerated carbonation conditions and subjected to heating at 30 °C, 50 °C, 200 °C, and 400 °C for 2 hours. Normalization was performed with respect to the maximum intensity between Raman shifts of 50 cm⁻¹ and 4000 cm⁻¹. According to Raman spectra obtained from RRUFF database for brucite (R040077), nesquehonite (R050639), hydromagnesite (R060011), dypingite (R070086), and artinite (R060166), the peaks at 277 cm⁻¹, 442 cm⁻¹, 726 cm⁻¹, and 1086 cm⁻¹ are attributed to OH⁻ vibration modes in brucite. An additional peak for brucite at 3652 cm⁻¹ was also reported by other researchers [85]. Meanwhile, the peaks in the vicinity of 1100 cm⁻¹ are associated with that of C=O vibration in CO₃²⁻ within carbonation products with slight shifting to the right as compared with the corresponding frequency peak of OH⁻ in brucite. The peak positions from the database are consistent with the Raman spectra presented previously for nesquehonite [80], hydromagnesite [81], and dypingite and artinite [86].
The Amb samples exposed to heating at 30 °C and 50 °C exhibited peaks at 277 cm\(^{-1}\), 444 cm\(^{-1}\), and 3638 cm\(^{-1}\), associated with OH\(^{-}\) vibration due to the presence of Mg(OH)\(_2\) in the matrix system. Heating the Amb samples up to 200 °C did not have a significant effect on the spectrum profile, and this further reinforced the argument that the morphology evolution contributed to strength enhancement of the samples. The decomposition of brucite at temperatures between 360 °C and 380 °C led to less intense Raman peaks for the Amb-T400 sample. It is important to note that the application of the background subtraction adopted in data processing resulted in a high noise level and apparent high peaks in the vicinity of 1093 cm\(^{-1}\) and 3646 cm\(^{-1}\), as shown in Fig. 9 (a) for the Amb-T400 sample. Conversely, Fig. 9 (b) revealed that the accelerated carbonation induced the formation of carbonation products, which was evident by peak centers at 1181.1 cm\(^{-1}\) for the C20-T30 and 1118.3 cm\(^{-1}\) for the C20-T50 sample. As compared with the Amb samples, the carbonated samples showed greatly diminished relative intensities in the region of 3638 cm\(^{-1}\) at heating temperatures of 30 °C and 50 °C, indicating a reduced presence of brucite due to its conversion to the carbonation phases. Peaks at low frequency match those of artinite, which was expected to be present as part of the carbonation products. On the other extreme, the spectrum bands in the vicinity of 3416 cm\(^{-1}\) could be assigned to moisture in the system [80]. For the C20-T200 sample, the peak band for moisture became significantly reduced due to dehydration. Also, the peak band in the vicinity of 3638 cm\(^{-1}\) for OH\(^{-}\) vibration of brucite became more apparent while the peak at 1183 cm\(^{-1}\) became relatively less intense and shifted to 1098 cm\(^{-1}\), attributable to an increase in Mg(OH)\(_2\) content and partial decarbonation of HMCs. Increasing the target heating temperature from 200 °C to 400 °C led to greatly reduced peak intensities when compared with those of the C20-T200 sample as a result of further dehydroxylation and decarbonation. Nevertheless, the peak at 1098 cm\(^{-1}\) assigned to OH\(^{-}\) in Mg(OH)\(_2\) persisted. This could be attributed to the presence of HMCs forming layers around brucite and acting as a physical barrier to delay its dehydroxylation.

### 3.8 Nanoindentation

Relative frequency plots of elastic modulus data from the nanoindentation experiment are given in Fig. 10 and Fig. 11 for samples cured under the ambient and accelerated carbonation conditions, respectively. The average elastic modulus values for individual nanoindentation tests are also given in Fig. 12. In line with the compressive strength results, it is evident that heating up to 300 °C enhanced the modulus of the Amb samples as marked by a shift of the relative frequency profiles to the right and higher average modulus values as compared with
that of the Amb-T30 sample. Interestingly, there is an insignificant difference in the relative frequency plots and the resulting average modulus values for Amb-T100 and Amb-T200 (Fig. 10 (c) and (d)). This is consistent with the TGA data, where individual mass losses corresponding to dehydration, dehydroxylation, and decarbonation were very similar among the samples. The onset of decomposition of brucite at a heating temperature of 300 °C led to a slight increase in low modulus phases, as translated into a corresponding reduction in the compressive strength. At 400 °C, the decomposition of brucite led to a more porous microstructure and a significant increase in the low-modulus peak (Fig. 10 (f)).

When compared with the ambient condition, the accelerated carbonation greatly enhanced micro-mechanical properties of the composite as reflected by a significant increase in the high-modulus phase in the C20-T30 sample (Fig. 11 (a)). Using Lorentz non-linear fit for the sample, the average modulus for HMCs was estimated to be 17.6 GPa. A similar elastic modulus distribution was observed for the C20-T50 and C20-T100 samples (Fig. 11 (b), and (c)), despite a higher compressive strength in the C20-T50 sample. It is anticipated that a possible modulus enhancement, due to improvement in the crystal size of nesquehonite and additional formation of brucite at 50 °C heating, was offset by the nano and micro-crack formation, which was not translated to the overall macro response of the concrete under the compressive testing. Crack propagation in concrete usually initiates from micro defects at the interfacial zones [87], thus obscuring the influence of smaller-scale cracks in the matrix on the overall compressive response. Otherwise, the nano and macro-scale cracks could influence the nanoindentation data due to an interaction micro-volume underneath the indenter tip, whose size was estimated to be approximately 10 times the indentation depth [88, 89] or 5 µm for the case of this study. Consistent with the TGA results where a high level of dehydration of HMCs started to occur at 200 °C, a corresponding reduction in medium modulus (in the range of 17.6 GPa) phases and the associated increase in low modulus phases were observed in the C20-T200, C20-T300, and C20-T400 samples (Fig. 11 (d), (e), and (f)). Despite retaining a higher compressive strength than the Amb-T400 sample, the C20-T400 sample exhibited a higher relative frequency for the low-modulus phases, as also reflected by the lower average modulus value (Fig. 12). The incongruence was attributed to more formation of nano and macro-scale cracking in the C20-T400 sample due to its more intense decomposition at the heating temperature of 400 °C.
With the goal of proving and expanding the potential application areas of reactive MgO cement (RMC) mixes for buildings, this study investigated the strength, composition, and microstructure of RMC-based concrete mixes cured under ambient and accelerated carbonation conditions, followed by exposure to high temperatures ranging from 30 °C to 400 °C. For the ambient-cured samples, the hydration of the remaining MgO was accelerated under heating up to 200 °C. This additional hydration densified the microstructure, which was composed of brucite with an improved morphology. This contributed to significantly higher compressive strengths in the samples (i.e., 10 MPa vs. 30 MPa). At the heating temperatures of 300 °C and 400 °C, the dehydroxylation of brucite led to a significant loss of its binding ability. This reduction in brucite was accompanied by an increase in the pore size (i.e., mostly in the range of 0.5-0.7 μm), resulting in a strength decrease to 5 MPa at 400 °C. Consistently, the ambient-cured samples exhibited enhanced micromechanical properties at up to 100 °C. Further exposure to higher temperatures at up to 300 °C did not have a significant influence on their nanoindentation modulus values due to a combined action of crack formation and morphology change.

For the samples cured under the accelerated carbonation condition, the formation of HMCs not only enhanced the binding ability of the matrix, but also provided a filler effect. This improvement led to a reduction in the pore size, resulting in a significant improvement in their compressive strengths compared with the ambient-cured samples (58 vs. 10 MPa). Exposure to a temperature of 50 °C stimulated the hydration of the remaining MgO to provide an increased amount of brucite. The reaction between dissolved CO$_2$ and Mg(OH)$_2$(aq,s) improved the morphology of HMCs, which resulted in a further increase in the compressive strength from 58 MPa to 65 MPa. Despite the additional formation of brucite at 100-300 °C, the dehydroxylation and decarbonation of HMCs and the associated cracking led to a slight reduction in the compressive strength of the heated samples from 58 to 56 MPa. The dehydroxylation and decarbonation of HMCs were associated with the transformation of nesquehonite and hydromagnesite to artinite at temperatures of 100-400 °C. The presence of HMCs, forming protective layers around brucite, acted as a physical barrier and decreased its dehydroxylation rate. The strong decarbonation of the carbonated samples at 400 °C led to the loss of HMCs and an increase in the pore size from 0.02-0.03 μm to 0.3-0.7 μm, which resulted in a significant
reduction in compressive strength from 58 MPa to 8 MPa as well as the loss of nanoindentation modulus from 17.6 GPa to 2.6 GPa based on frequency plot deconvolution.

The work contributes to unraveling the mechanical and microstructural performance of composites produced with reactive magnesium oxide cement (RMC) under high temperatures or simulated fire scenarios. Based on the obtained results, it follows that despite the compositional and morphological changes observed at high temperatures, RMC-based composites cured under ambient and accelerated conditions remain mechanically stable without a significant reduction in performance at temperatures as high as 300 °C (i.e. when heated for up to 2 hours at 10 °C/min). These findings highlight the stability of RMC-based concrete under extreme operating conditions. Also, improved fire resistance of the composites may be achieved by including nucleation materials or carbonation agents to preferentially form more stable HMCs. The incorporation of microfibres is also postulated to enhance the structural stability by delaying the cracking initiation within the carbonation products. These topics have been completed [25, 90] or are being investigated by the co-authors.

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Table 1 Chemical composition and physical properties of RMC.

<table>
<thead>
<tr>
<th>Chemical composition (%)</th>
<th>Physical properties</th>
<th>Specific gravity (g/cm$^3$)</th>
<th>Specific surface area (m$^2$/g)</th>
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Table 2 Compositions of RMC-based concrete used in this study.

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Table 3 MgO content within each sample, obtained by XRD.

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Table 4 Mass losses of all samples, obtained by TGA.

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Fig. 1 Compressive strength of concrete samples: (a) cured for up to 28 days and (b) heated for 2 hours after 14 days of curing
Fig. 2 MIP pore size distribution of samples cured under (a) ambient and (b) accelerated carbonation conditions and heated at different temperatures
Fig. 3 SEM images of samples after 14 days of ambient curing, followed by an exposure to: (a) 30 °C, (b) 50 °C, (c) 100 °C, (d) 200 °C, (e) 300 °C and (f) 400 °C
Fig. 4 SEM images of sample after 14 days of 20% CO₂ curing, followed by an exposure to: (a) 30 °C, (b) 50 °C, (c) 100 °C, (d) 200 °C, (e) 300 °C and (f) 400 °C
Fig. 5 ESEM images of 14-day sample cured under 20% CO$_2$ at different scanning temperatures: (a) 30 °C, (b) 50 °C, (c) 100 °C, (d) 200 °C, (e) 300 °C and (f) 400 °C
Fig. 6 FTIR spectra of samples cured under (a) ambient and (b) accelerated carbonation conditions heated at different temperatures
Fig. 7 XRD patterns of concrete samples after heating, cured under: (a) ambient and (b) 20% CO₂ conditions
Fig. 8 TGA and DTG of samples after heating, cured under: (a) ambient and (b) 20% CO₂ conditions
Fig. 9 Raman spectra with 600 g/mm detector of samples cured under (a) ambient and (b) 20% CO₂ conditions and subjected to heating at 30 °C, 50 °C, 200 °C, and 400 °C for 2 hours.
Fig. 10 Nanoindentation results of samples after ambient condition curing, followed by exposure to: (a) 30 °C, (b) 50 °C, (c) 100 °C, (d) 200 °C, (e) 300 °C, and (f) 400 °C
Fig. 11 Nanoindentation results of samples after 14 days of 20% CO₂ curing, followed by exposure to (a) 30 °C, (b) 50 °C, (c) 100 °C, (d) 200 °C, (e) 300 °C, and (f) 400 °C
Fig. 12 Average elastic modulus based on the nanoindentation of heated samples after 14 days of curing under ambient and 20% CO$_2$ conditions.