



# Carbonation of recycled concrete fines as a treatment for promoting their recycling as supplementary cementitious material: influence of initial moisture on CO<sub>2</sub> uptake and reactivity<sup>☆</sup>

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## ABSTRACT

Construction and demolition waste (CDW) is a major global waste stream. Coarse aggregates from CDW are widely reused, but recycled concrete fines (RCf, <80 µm) remain underutilised, often ending up in landfills or used in low-value applications. Carbonation is a promising treatment to valorise RCf as a supplementary cementitious material (SCM), converting reactive calcium-bearing phases into calcium carbonate while permanently sequestering CO<sub>2</sub>. Hence, this study explores the semi-dry accelerated carbonation of RCf generated in a concrete recycling facility in the Netherlands, focusing on the influence of the initial moisture content, varied from 0 to 50 wt%. Pre-wetting the RCf prior to carbonation significantly influenced calcium carbonate formation, with higher moisture levels promoting calcite formation. Samples preconditioned with 10–50 wt% moisture demonstrated enhanced CO<sub>2</sub> uptake and a pronounced increase in specific surface area, from ~7 m<sup>2</sup>/g for raw RCf to up to ~20 m<sup>2</sup>/g for carbonated samples at intermediate moisture content. CO<sub>2</sub> uptake increased with increasing moisture, whereas reactivity did not follow the same trend. The 50 wt% condition achieved the highest CO<sub>2</sub> uptake but the lowest R3 reactivity among the carbonated samples, while intermediate moisture (~20 wt%) led to the highest specific surface area and favourable R3 reactivity and was selected for paste validation. Selected carbonated and uncarbonated RCf were subsequently used to replace 20% of the cement in paste mixtures and were compared against a limestone reference. RCf carbonated at 20 wt% moisture achieved a 13.4% increase in 28-day compressive strength compared with the limestone reference. These results demonstrate that RCf, often considered low-value waste, can be valorised as an SCM through moisture-controlled carbonation. Their use supports CO<sub>2</sub> utilisation in cementitious systems. It contributes to more circular construction practices, while offering a critical perspective on the application of mineral carbonation to materials produced in full-scale waste treatment plants.

## 1. Introduction

The reuse and recycling of concrete from buildings and infrastructure is recognised as one of the key strategies to reduce the demand for primary cement, thereby lowering the carbon emissions associated with the cement industry (IEA, 2018). In concrete recycling practices, the concrete waste from CDW is commonly processed to recover larger particles, which are often reused as substitutes for natural aggregates or as road-base materials (Agrela et al., 2012; Florea et al., 2014; Lacerda Gomes et al., 2023). However, the recycling process also generates a fine fraction known as RCf. This fine fraction consists of hydrated and

unhydrated cement paste, along with fine aggregate particles (Florea and Brouwers, 2013). Despite its potential as a supplementary cementitious material (SCM), RCf lacks large-scale applications and is often landfilled or downcycled, even in regions with advanced concrete recycling practices. RCf has attracted interest from both industry and academia, with research focusing on its recycling and on developing technologies to enhance its performance. These efforts aim to improve the properties of RCf, such as chemical reactivity and promote its incorporation into new cement-based materials (Doudi et al., 2024; Sousa et al., 2024; Teune and Schollbach, 2024; Wang et al., 2022; Zhang et al., 2024).

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Alternative valorisation approaches, such as mechanical activation, have been investigated to enhance reactivity through particle size reduction, increased surface area, and high-energy grinding (Rajczakowska et al., 2025; Sousa et al., 2024). However, the energy demand associated with mechanical activation can be considerably high (Rajczakowska et al., 2025). At the same time, RCf generated from concrete recycling can differ considerably from laboratory-produced cement-paste fines because it contains aggregate particles and may have undergone partial carbonation during the service life of the original concrete (Tabrizi et al., 2025). Therefore, it is important to study treatment routes such as mineral carbonation using RCf generated in full-scale CDW treatment plants rather than relying only on laboratory-produced materials.

The potential use of RCf as a supplementary cementitious material depends on the presence and accessibility of reactive phases, particularly residual C-S-H and amorphous silica- and alumina-containing phases. During carbonation, however, calcium-bearing hydrates are progressively decalcified. Carbonation of C-S-H involves decalcification and polymerisation, resulting in the formation of an amorphous silica-rich phase, while calcium is precipitated mainly as calcium carbonate (Morandau et al., 2014). The resulting silica-rich phase can contribute to pozzolanic reactivity, whereas calcite generally has a lower reactivity when used as SCM (Maia Neto et al., 2024; Zhao et al., 2024a). The extent of C-S-H decalcification and the nature of the resulting carbonation products depend on the carbonation conditions and the initial composition of the material. In particular, low-Ca/Si C-S-H is more resistant to decalcification and may result in a lower formation of reactive silica gel, which can limit the reactivity of conventionally carbonated RCf (Zhao et al., 2024a). Therefore, maximising CO<sub>2</sub> uptake does not necessarily maximise the reactivity of carbonated RCf as an SCM.

Accelerated carbonation presents a promising technology for the valorisation of RCf by combining CO<sub>2</sub> sequestration with the modification of its phase composition and microstructure. This process stores CO<sub>2</sub> by converting calcium-bearing phases, such as portlandite and C-S-H phases, into stable calcium carbonate polymorphs, while also forming reactive amorphous alumina-silica gel (De Weerd et al., 2019; Teune et al., 2023; Zajac et al., 2023a). One effective route is wet carbonation, which involves reacting the material in an aqueous system, typically as a slurry, under fully saturated conditions with excess liquid water (Snellings and Matschei, 2025). This method relies on the presence of liquid water to facilitate CO<sub>2</sub> dissolution and enhance reaction kinetics. Alternatively, semi-dry carbonation (also referred to as semi-wet carbonation) occurs under partially saturated conditions, where water is present as a thin liquid film or adsorbed moisture within the pore structure (Galan et al., 2015), enabling CO<sub>2</sub> dissolution and subsequent reactions with calcium-bearing phases. In practice, this process is commonly conducted in a climate chamber under controlled relative humidity (RH). Compared to wet carbonation, semi-dry carbonation offers lower water consumption, a simpler processing route, and efficient calcium extraction.

During semi-dry carbonation, environmental factors such as RH, temperature, and pressure significantly influence process efficiency and phase composition, and previous studies have explored the effect of these parameters (Gholizadeh-Vayghan and Snellings, 2022; Wu et al., 2022; Zajac et al., 2022). Relative humidity is particularly important for carbonation kinetics, as water is the medium in which CO<sub>2</sub> and calcium phases dissolve, facilitating their interaction through a dissolution-precipitation mechanism. In this gas-liquid-solid system, CO<sub>2</sub> dissolves in water to form carbonic acid (H<sub>2</sub>CO<sub>3</sub>), which lowers the pH and accelerates the dissolution of calcium-bearing phases, enriching the solution with calcium ions (Cizer et al., 2012; Daval et al., 2009). The calcium ions then react with carbonate ions to form calcium carbonate, predominantly as calcite, vaterite, aragonite, or amorphous calcium carbonate.

Research indicated that calcite, the most thermodynamically stable

phase, becomes dominant at very high RH (~100%), whereas intermediate RH levels lead to the formation of mixed polymorphs (Dubina et al., 2013). High RH facilitates rapid carbonation by promoting thick water films that enhance CO<sub>2</sub> dissolution and calcium carbonate nucleation at crystal-solution interfaces (Galan et al., 2015). Conversely, insufficient moisture limits carbonation at low RH (<30%) due to the absence of continuous water films necessary for CO<sub>2</sub> dissolution (Dubina et al., 2013; Galan et al., 2015), and moderate RH (40–75%) supports carbonation via thin water layers (Galan et al., 2015). However, the precipitation of carbonation products can reduce pore size and connectivity, while the formation of calcium carbonate layers may act as diffusion barriers, thereby limiting CO<sub>2</sub> access to unreacted calcium-bearing phases and reducing the overall progress of carbonation (Galan et al., 2015; Teune et al., 2023).

The carbonation of portlandite, ettringite, and C-S-H under different relative humidity (RH) conditions revealed that the predominant CaCO<sub>3</sub> polymorph depends on both RH and the Ca/Si ratio of C-S-H (Steiner et al., 2020). At 91% RH, calcite is the dominant phase forming from C-S-H with Ca/Si ratios of 1.5 and 1.2 after 28 days, whereas at 57% RH, vaterite is prevalent for a Ca/Si ratio of 1.5, and amorphous calcium carbonate forms for a Ca/Si ratio of 0.7 (Steiner et al., 2020). These moisture-dependent mechanisms are also reflected at the material scale, where carbonation of recycled concrete aggregates (RCA >3 mm) has been shown to improve physical and durability-related properties, including reductions in porosity and water absorption and enhanced freeze-thaw resistance, particularly under partially saturated conditions (e.g., intermediate relative humidity levels around 40–70%) (Gholizadeh-Vayghan et al., 2020). These findings highlight the influence of moisture on the carbonation pathway and the resulting carbonate phases.

Due to the complex and heterogeneous composition of RCf generated by CDW treatment, previous carbonation studies have used RCf produced by crushed cement paste under controlled laboratory conditions (Ho et al., 2020; Peng et al., 2023). In contrast, RCf sourced from recycling facilities contains additional variability arising from the presence of crushed aggregates and other impurities, which can affect its effective calcium availability and carbonation behaviour (Ho et al., 2021). Consequently, the carbonation behaviour of RCf generated in recycling facilities has received comparatively limited attention, particularly regarding its influence on carbonation efficiency and subsequent reactivity.

A recent study on the wet carbonation of industrial recycled concrete fines (RCf) showed that carbonation kinetics are strongly influenced by diffusion through the product layer and particle size (Tabrizi et al., 2025). Compared to laboratory-produced cement paste analogues, RCf generated in recycling facilities exhibited lower effective CaO availability, partial pre-carbonation inherited from service life, and pronounced particle-scale heterogeneity, which together influence carbonation kinetics and the relationship between CO<sub>2</sub> uptake and mechanical performance (Tabrizi et al., 2025). Kinetic analyses indicate that product-layer diffusion and particle size control reaction progress (Tabrizi et al., 2025). Moreover, the performance of carbonated RCf when used as an SCM is influenced by the replacement level and the nature of the carbonation products. Studies using 5–20% cement replacement have reported improvements in hydration and/or compressive strength, although the extent of these improvements varies with the carbonation route applied to the RCf and the resulting carbonate and silica phases (Tabrizi et al., 2025; Zhao et al., 2024b).

Previous studies have established that water availability influences the kinetics and phase assemblage of semi-dry carbonated recycled cement paste, while other work has demonstrated the potential use of carbonated recycled concrete fines in blended cement. However, less attention has been given to initial liquid-water addition as a process-control variable for producing an SCM from heterogeneous RCf generated in a full-scale recycling facility. In particular, is necessary to determine whether process conditions that favour CO<sub>2</sub> mineralisation

also favour the subsequent reactivity of the carbonated product. Therefore, this study evaluated five initial water additions (0–50 wt%) under fixed semi-dry carbonation conditions and related carbonation degree and carbonate phase assemblage to specific surface area, pore-volume distribution and R3 reactivity. Selected products were subsequently evaluated in cement paste at 20% cement replacement. The objective was to identify the moisture-dependent trade-off between CO<sub>2</sub> uptake and SCM-related functionality, rather than to optimise carbonation degree alone.

## 2. Materials and methods

### 2.1. Materials

The RCf used in this study were sourced from a recycling plant in the Netherlands and reported to consist of a mixture of different types of parent concrete. The RCf were generated through a patented crushing process specifically designed to recycle demolished concrete by crushing and sieving it to obtain clean aggregate. After being collected, the fines were dried at 40 °C (± 5 °C) for three days and then sieved to a size below 80 µm to remove gravel and obtain fines richer in cement paste. The sieved RCf had a median particle size (d<sub>50</sub>) of 28.20 µm, and the limestone used in the cement paste samples had a median particle size of 16.35 µm (see Appendix, Fig. A1). The RCf was further characterised in terms of chemical composition and particle size distribution. Quantitative chemical analyses were conducted using X-ray fluorescence spectrometry (XRF) with a PANalytical Epsilon 3-standardless OMNIAN method. The loss on ignition was performed at 1050 °C for 4 h, and the fines were prepared by fusing 1 g of the material with 10 g of lithium tetraborate and 300 µl of lithium iodide.

The chemical composition is presented in Table 1, and particle size distribution is shown in Appendix Fig. A1.

### 2.2. Methods

#### 2.2.1. Accelerated carbonation

The semi-dry carbonation of RCf samples was performed in a climate chamber under controlled CO<sub>2</sub>, RH, and temperature. To assess the impact of varying water content on the carbonation of RCf, the fines were pre-wetted with different proportions of liquid water, i.e., 0, 5, 10, 20, and 50 wt%. The selected moisture contents were chosen to cover a wide range relevant to semi-dry carbonation, from limited water availability to conditions where excess moisture may restrict CO<sub>2</sub> diffusion. Each sample contained 50 g of RCf and was thoroughly mixed with the designated amount of water before being evenly spread on metal plates (20 cm x 10 cm). The samples were then carbonated in a controlled environment at 35 °C for 3 h. This duration was selected based on literature indicating that carbonation reactions in cementitious materials occur rapidly during the initial hours of exposure, followed by a slower diffusion-controlled stage due to the formation of carbonate layers that limit CO<sub>2</sub> transport (Biasin et al., 2015; Wang et al., 2019). Therefore, a duration of 3 h was selected to allow the carbonation process to occur while avoiding an excessively long duration that could mask the influence of the initial moisture content. The RH and CO<sub>2</sub> concentrations in the climate chamber were maintained at 80% (± 5%) and 20%, respectively, with air as the balance gas. Residual moisture was removed immediately after carbonation through solvent exchange with isopropanol and diethyl ether, followed by filtration using a vacuum filter and drying at 40 °C in a ventilated oven for 1 h. The samples are referred to as RCf-NC for the raw fines without accelerated

carbonation, and “c-RCf-wX” for the carbonated samples (where X represents the water content added to the RCf, i.e., 0, 5, 10, 20, 50 wt%).

#### 2.2.2. Characterisation Techniques

Mineralogy was determined by powder X-ray diffraction (XRD) using a Bruker D2 X-ray diffractometer equipped with a Co tube, over a 2θ range of 7° to 80°, with a step size of 0.02°. The powders were milled in an XRD-Mill McCrone (RETSCH) for 8 min with silicon powder as an internal standard (10 wt%) and backloaded into the sample holders. The phases were identified and quantified with X'Pert HighScore Plus 2.2 PANalytical with the PDF2 database of ICDD and TOPAS Academic software v5.0, respectively.

The microstructural investigation was performed by scanning electron microscopy (JOEL JSM-5600) with a backscattered electron detector at an accelerating voltage of 15 kV. The heat flow and total heat released by the carbonated samples were evaluated using an isothermal calorimeter (Calmetrix, I-Cal Ultra 8-channel) at 40 °C for 7 days, in accordance with the R3 test procedure reported in ASTM C1897-20. Fourier-transform infrared spectroscopy (FTIR-ATR) spectra were obtained in the range of 400–4000 cm<sup>−1</sup> with a resolution of 2 cm<sup>−1</sup> by PerkinElmer Frontier FTIR–GladiATR equipment. Nitrogen sorption (TriStar II 3020, Micrometrics) was used to determine changes in specific surface area, and the pore size distribution was determined using the Brunauer-Emmett-Teller (BET) method.

Thermogravimetric analysis (TGA) was conducted for all samples using a Netzsch TG-209 Tarsus instrument. Before analysis, samples were thoroughly manually homogenised before taking the analytical portion to minimise sampling variability associated with the heterogeneous composition of the RCf. Approximately 40–45 mg powder samples were analysed under a nitrogen atmosphere with a heating rate of 10 °C/min from 40 to 1000 °C. The calcium carbonate content (CaCO<sub>3</sub>) was quantified based on the weight loss due to the loss of CO<sub>2</sub> from the CaCO<sub>3</sub>. The mass loss due to the decomposition of carbonates was determined within the temperature range for each sample based on the derivative curves. However, during accelerated carbonation in the climate chamber, the CO<sub>2</sub> uptake increased the total mass of the sample. Therefore, the residual mass at 1000 °C was used for normalisation to account for this mass increase and to enable comparison among samples with different inert contents (Teune and Schollbach, 2024). The normalised value (N) of the calcium carbonate content (CaCO<sub>3</sub>%wt) and the degree of carbonation were calculated according to:

$$N = \frac{WL_{1000^\circ C}^{RCf-Un}}{WL_{1000^\circ C}^{cRCf}} \quad (1)$$

$$m_{CaCO_3,N}(\%wt) = N \cdot WL_{CC} \cdot \frac{MmCaCO_3}{MmCO_2} \quad (2)$$

$$DoC(\%) = \frac{(m_{CC_{Norm,f}} - m_{CC_{Norm,i}}) \cdot \left( \frac{Mm_{CaO}}{Mm_{CaCO_3}} \right)}{M_{CaO_{RCf}}} \quad (3)$$

Where  $WL_{1000^\circ C}^{RCf-Un}$ ,  $WL_{1000^\circ C}^{cRCf}$  is the residual mass of the RCf samples at 1000 °C before and after carbonation, respectively.  $WL_{CC}$  is the weight loss at ~ 480–800 °C following the DTG curves for each sample,  $m_{CC_{Norm}}$  is the content of CaCO<sub>3</sub> before and after carbonation,  $M_{CaO_{RCf}}$  is the CaO content in the RCf, and Mm are the molar masses of CaCO<sub>3</sub> and CO<sub>2</sub>, respectively.

The compressive strength of cement pastes incorporating both

**Table 1**

Chemical composition expressed as oxide content determined by X-ray fluorescence (%wt) and loss on ignition (LOI).

| Oxide | SiO <sub>2</sub> | CaO   | Al <sub>2</sub> O <sub>3</sub> | Fe <sub>2</sub> O <sub>3</sub> | SO <sub>3</sub> | MgO  | K <sub>2</sub> O | MnO  | Others | LOI   |
|-------|------------------|-------|--------------------------------|--------------------------------|-----------------|------|------------------|------|--------|-------|
| RCf   | 56.98            | 16.65 | 5.40                           | 1.93                           | 1.48            | 1.68 | 0.82             | 0.09 | 0.68   | 14.29 |

carbonated and non-carbonated RCf was evaluated through compressive strength tests conducted at 1, 7, and 28 days. Fresh paste mixtures were cast into  $40 \times 40 \times 40$  mm cubic moulds, with three specimens tested per formulation at each curing age. The reported compressive strengths correspond to the mean of three measurements obtained using an Autotest mechanical testing machine at a loading rate of 2400 N/s. In all cases, 20% of the cement was replaced by RCf. The cement used was CEM I 42.5R, and a fixed water-to-binder (w/b) ratio of 0.33 was adopted based on preliminary tests. After casting, the specimens were demoulded after 24 h and then cured in water at  $20 \pm 1$  °C until the designated testing age. Each paste sample was designated using the prefix “P-” followed by the label of the RCf used. For example, P-cRCf-w20 refers to the cement paste containing carbonated RCf cRCf-w20.

### 3. Results and discussion

The XRD diffractograms of raw RCf and carbonated RCf (Fig. 1) showed that the raw material, RCf-NC, consisted of crystalline phases, including 55.1% of quartz ( $\text{SiO}_2$ ), 7.5% of feldspar (anorthoclase and albite), 6.5% of calcite ( $\text{CaCO}_3$ ), 3.1% of gypsum, 0.8% of portlandite ( $\text{Ca(OH)}_2$ ), 3.2% of ettringite, and an amorphous content calculated as approximately 20.9%. The results of the Rietveld quantitative phase analysis of initial RCf and carbonated samples after the mass normalisation are described in Table A-1. The presence of feldspar, gypsum, calcite, and quartz is likely related to the heterogeneous composition of the parent concrete, including contributions from natural aggregates and cement paste. Quartz and feldspar may originate from the aggregate fraction incorporated into the fines during crushing and sieving (Florea et al., 2014) while calcite and gypsum may be associated with the

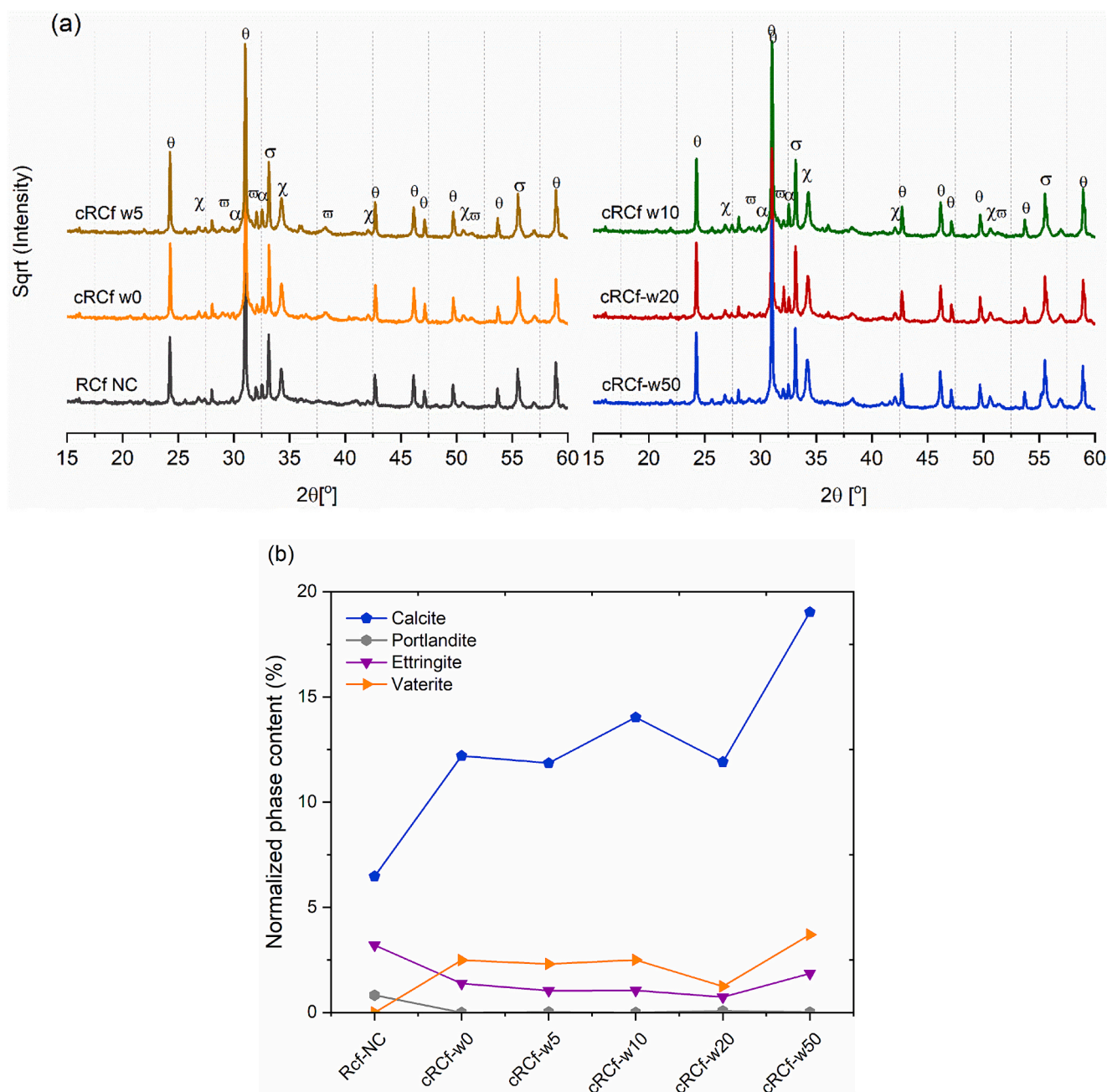


**Fig. 1.** XRD pattern (a) and QXRD content of normalized phases (b) of RCf before (RCf-NC) and after carbonation (cRCf-w0, cRCf-w5, cRCf-w10, cRCf-w20, cRCf-w50).  $\alpha$  = albite ( $\text{NaAlSi}_3\text{O}_8$ ),  $\theta$  = quartz ( $\text{SiO}_2$ ),  $\sigma$  = silicon ( $\text{Si}$ ),  $\chi$  = calcite ( $\text{CaCO}_3$ ),  $\omega$  = vaterite ( $\text{CaCO}_3$ ).

cement-paste fraction and the long-term exposure history of the parent concrete (Tokareva et al., 2023).

The contents of quartz and calcite are higher than those reported by other authors for RCf (Deng and Wu, 2024; Jiang et al., 2022) with reported values of around 30.9% and 5.8%, respectively. This discrepancy may be related to the more controlled lab crushing and sieving process used to generate RCf in those previous studies, which likely resulted in fewer aggregate particles in the fines. In contrast, similar quartz and calcite levels have been observed in previous studies involving RCf sourced directly from industrial recycling sites (Sousa et al., 2024; Teune and Schollbach, 2024). This composition indicates a relatively high fraction of non-reactive aggregate-derived phases and a lower proportion of reactive cement-paste phases, particularly C-S-H and portlandite, which may limit the use of these residues as SCMs.

After carbonation, calcium carbonate ( $\text{CaCO}_3$ ) polymorphs calcite and vaterite were identified in all samples (Fig. 1). The content of  $\text{CaCO}_3$

phases increased with moisture addition, reaching the highest content in the cRCf-w50 sample (Fig. 1). RCf samples carbonated with a moisture content above 5% showed a modest increase in calcite formation within three hours, as quantified by QXRD (Fig. 1b), indicating that water availability at the particle surface strongly influences carbonate precipitation (Galan et al., 2015; Gholizadeh-Vayghan and Snellings, 2022; Steiner et al., 2020).

Calcite was the predominant crystalline carbonate phase in all carbonated samples, while vaterite showed only minor variations, with a slight increase observed in cRCf-w50. The limited variation in vaterite and the absence of aragonite suggest that the carbonation conditions favoured the formation of more stable crystalline phases at higher moisture levels. Previous studies have demonstrated that the prevalence of specific  $\text{CaCO}_3$  polymorphs depends on factors such as the Ca/Si ratio of C-S-H and the relative humidity. Steiner et al. (2020) observed that under moderately humid conditions ( $\sim 57\%$  RH), a mixture of C-S-H

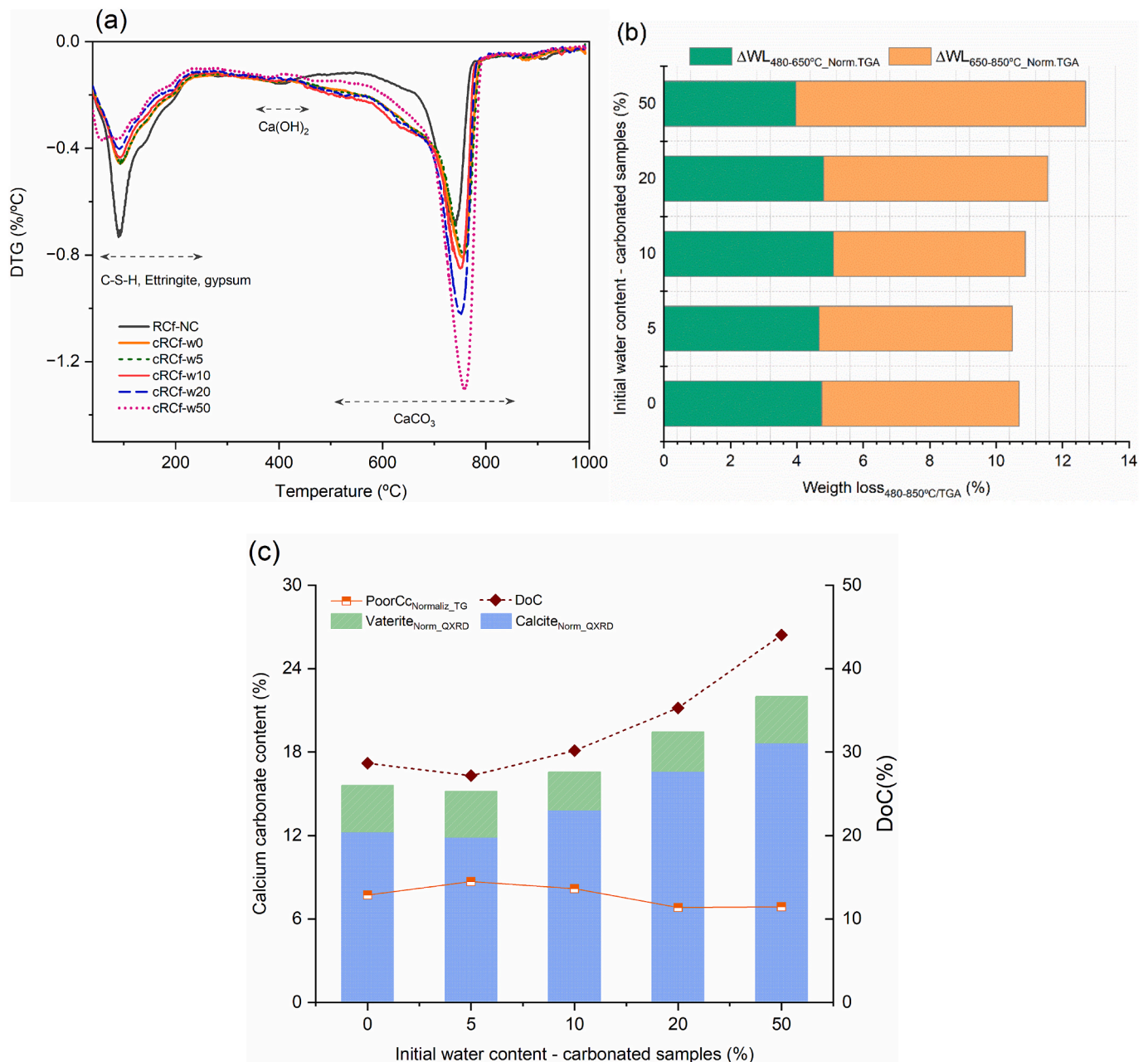


**Fig. 2.** TGA/DTG curves of (a) RCf before and after carbonation at different initial water contents, (b) calculated normalised weight loss for carbonated samples in the temperature range of calcium carbonate and (c) calcium carbonate content of RCf samples from QXRD and DTG/TGA and the degree of carbonation.

(Ca/Si  $\approx$  1.5) and ettringite produced vaterite as the dominant  $\text{CaCO}_3$  polymorph (with only minor presence of aragonite). Conversely, Sevelsted and Skibsted (2015) found that near-saturated  $\text{CO}_2$  exposure of C-S-H with a high Ca/Si ratio led to nearly complete decalcification of the hydrate and predominantly to calcite precipitation, whereas drier conditions or lower Ca/Si resulted in less extensive carbonation and a higher fraction of metastable carbonate phases. In our study, increasing the moisture content similarly shifted the carbonate assemblage towards a higher proportion of calcite (Fig. 1b). This is consistent with established carbonation mechanisms in cementitious systems and, in the context of RCf sourced from full-scale recycling facilities, is relevant for interpreting subsequent changes in material properties. The crystallinity of the carbonate phases can influence the accessibility of unreacted calcium-bearing phases to further carbonation, and the reactivity of the carbonated product toward cement hydration.

The presence of calcium carbonate polymorphs is also identified in the DTG curves of RCf samples (Fig. 2-a). The DTG curve of uncarbonated RCf (RCf-NC) exhibited a prominent peak within the 100–300 °C range, corresponding to the dehydration of ettringite, physically bound water, gypsum, and C-S-H (Rostami et al., 2012). This peak decreased with increasing initial moisture content during carbonation, indicating enhanced carbonation of cement hydration product, such as C-S-H, within 3 h.

In the cRCf-w50 sample, an additional split and shift towards lower temperatures (60–150 °C) were observed, attributed to the presence of loosely bound water and residual hydration products. The carbonation of C-S-H involves progressive decalcification through the removal of  $\text{Ca}^{2+}$  from interlayer and defect sites, leading to the formation of an amorphous silica-rich phase and the precipitation of calcium carbonate (Morandau et al., 2014; Wang et al., 2020). This process occurs alongside the carbonation of any portlandite present; however, in typical cement systems, CH carbonates more rapidly than C-S-H (Lagerblad, 2005). Yet, the peak at around 420 °C, associated with the decomposition of portlandite, is minimal in RCf (RCf-NC) and is consistent with QXRD results (Fig. 1), indicating the presence of less than 1% of calcium hydroxide in the uncarbonated RCf (RCf-NC). This suggests that carbonation in RCf is primarily governed by C-S-H and other calcium-bearing phases rather than CH (Morandau et al., 2014; Thierry et al., 2007).

The decomposition of calcium carbonate, one of the primary products of the carbonation of RCf, was observed (Fig. 2) in all carbonated samples at temperatures ranging from 460 °C to 850 °C (Ashraf and Olek, 2016; Scrivener et al., 2016). The corresponding normalised mass loss calculated for each sample is presented in Fig. 2b. In the DTG graph, a shoulder between 460 and 680 °C appears alongside the main carbonate peak at 700–750 °C (Rostami et al., 2012; Steiner et al., 2020). This shoulder is attributed to metastable carbonate phases, such as vaterite, amorphous  $\text{CaCO}_3$ , and poorly crystalline calcium carbonate. This poorly crystalline polymorph decomposes at a lower temperature than calcite, typically at 450–600 °C, while calcite, the well-crystalline form of  $\text{CaCO}_3$ , decomposes at 700–760 °C (Shao et al., 2025; Steiner et al., 2020).

Furthermore, the amount of calcium carbonate in the 650–850 °C range increased with the sample water content (Fig. 2-a), indicating enhanced product formation, particularly in samples with 10% (cRCf-w10), 20% (cRCf-w20), and 50 wt% (cRCf-w50) moisture content. Additionally, the main decomposition peaks around 740 °C (Fig. 2-a), shifted to  $\sim$  760 °C as the moisture content increased from 5 wt% (cRCf-w5) to 50 wt% (cRCf-w50), suggesting a slight increase in calcite crystallinity at higher moisture content. This shift can be linked to moisture-controlled carbonation pathways, where higher water availability promotes dissolution-precipitation mechanisms and favours the formation of more thermodynamically stable carbonate phases (Saeki et al., 2024; Steiner et al., 2020). Previous studies have shown that calcite predominates at high RH ( $\sim$ 75%), while vaterite and aragonite can coexist at intermediate RH ( $\sim$ 50%), and tend to gradually shift to calcite as

humidity increases (Dubina et al., 2013; Steiner et al., 2020).

Based on previous studies (Saeki et al., 2024; Xue et al., 2022) the presence of poorly crystalline calcium carbonate (PoCc) can be estimated from the difference between total  $\text{CaCO}_3$ , related to mass loss at 480–850 °C, and the crystalline carbonate content quantified by QXRD. The calculated PoCc and degree of carbonation were shown in Fig. 2, together with calcite and vaterite contents.

The results indicate that PoCc was present in all carbonated samples, with the highest level in the 10% water addition sample (cRCf-w10). A higher initial moisture content in the RCf led to a slight decrease in the poorly crystalline calcium carbonate fraction upon carbonation, specifically at 20% (cRCf-w20) and 50 wt% (cRCf-w50), while calcite and vaterite showed a significant increase (Fig. 2-c). Thus, the degree of carbonation increased with the amount of moisture added, reaching the highest value for the cRCf-w50 samples (Fig. 2). This transition suggests that increasing moisture not only enhances the extent of carbonation but also promotes the transformation of initially formed metastable carbonate phases into more stable and crystalline calcite (Cheng et al., 2025; Saeki et al., 2024).

The FT-IR spectra of raw and carbonated RCf are shown in Fig. 3. After carbonation, the samples exhibit a weak band at  $\sim$  3380  $\text{cm}^{-1}$  and at  $\sim$  1650  $\text{cm}^{-1}$ , corresponding to O-H stretching vibrations that can be associated with the presence of  $\text{H}_2\text{O}$  and C-S-H phases (Fernández-Carrasco et al., 2012). The band at around 3650  $\text{cm}^{-1}$ , related to portlandite (Saeki et al., 2024; Steiner et al., 2020) is not evident. The spectra shows the characteristic bands of the carbonate ion ( $\text{CO}_3^{2-}$ ), associated with both calcite and vaterite, including the  $\nu_2$  out-of-plane bending vibration near  $\sim$  875  $\text{cm}^{-1}$ , and the  $\nu_3$  asymmetric C-O stretching vibrations at approximately 1085  $\text{cm}^{-1}$  and 1430  $\text{cm}^{-1}$  (Chu et al., 2013; Cizer et al., 2012; Zhang et al., 2012). The absence of the aragonite band at  $\sim$  854  $\text{cm}^{-1}$  aligns with the XRD/QXRD results (Fig. 1). However, the band at 1085  $\text{cm}^{-1}$  may overlap with the quartz group band that can appear at around 1190  $\text{cm}^{-1}$  (Sáez del Bosque et al., 2020).

Additionally, a broad peak at  $\sim$  1420  $\text{cm}^{-1}$  ( $\nu_3$ ) and 1085  $\text{cm}^{-1}$ , attributed to carbonate groups, along with slight bands at  $\sim$  1645  $\text{cm}^{-1}$  and 2600–3700  $\text{cm}^{-1}$  associated with water, suggests the presence of amorphous or poorly crystalline calcium carbonate (Asta et al., 2020; Ihli et al., 2014). The broad peak at  $\sim$  1450  $\text{cm}^{-1}$ , indicative of the asymmetric stretching of  $\text{CO}_3^{2-}$  ions, arises from the disordered structure of ACC (Addadi et al., 2003; Lam et al., 2007). In contrast, the sample with higher water addition (cRCf-50) exhibited a noticeable shift from broader to narrower peaks, accompanied by a shift to higher wavenumbers (Fig. 3). This transition is likely related to increased

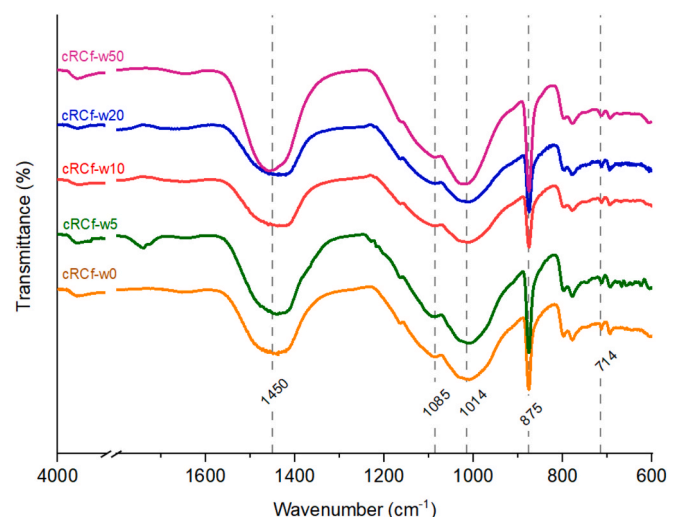


Fig. 3. FT-IR spectra of carbonated samples.

crystallization of carbonates, consistent with the formation of stable crystalline calcite, as observed in the QXRD results (Fig. 1).

A wide band between 890 and 1230  $\text{cm}^{-1}$  was also observed in the FTIR spectra (Fig. 3), associated with vibrations of Al-O and Si-O bonds, indicating the presence of both crystalline and amorphous phases, such as silica gel (Oh et al., 2024; Zajac et al., 2023b). The shoulder at 1160  $\text{cm}^{-1}$ , present in all samples, is linked to sulfate compounds such as gypsum (Yousuf et al., 1993). The Si-O stretching band, observed around 960–970  $\text{cm}^{-1}$ , is closely associated with the calcium silicate hydrate (C-S-H) phase. The narrowing and shifting of this peak signify the removal of calcium ions during carbonation, leading to the formation of Si-OH groups (Sáez del Bosque et al., 2020; Yu et al., 1999). This shift is particularly evident in the cRCf-50% sample. The shift of the silicate band to higher wavenumbers, along with changes in the relative intensities of the carbonate bands, suggests that increasing the liquid water content during carbonation in samples cRCf-5%, cRCf-10%, and cRCf-20% likely promotes the formation of poorly crystalline  $\text{CaCO}_3$ , as also supported by TGA mass loss analysis (Fig. 2). In contrast, the cRCf-50% sample exhibits sharper peaks related to crystalline calcite in the FTIR spectra (Fig. 3) and also a higher content of crystalline carbonate phases, as confirmed by QXRD analysis (Fig. 1).

The specific surface area (SSA) and cumulative pore volume results (Fig. 4-a) showed that as water content increased (0, 5, 10, 20, and 50 wt %), the SSA rose from 7.06  $\text{m}^2/\text{g}$  (RCf-NC) to 12.94 (cRCf-w0), 12.87 (cRCf-w5), 11.71 (cRCf-w10), 20.37 (cRCf-w20) and 18.38  $\text{m}^2/\text{g}$  (cRCf-w50). The pore volume curve of RCf-NC (Fig. 4-b) showed a lower overall pore volume, particularly in the mesopore range (2–50 nm), suggesting a more compact and less porous structure prior to carbonation. Upon carbonation, all the carbonated samples exhibited an increase in pore volume, especially around 3–5 nm, indicating the formation of mesopores, likely associated with carbonate precipitation. Notably, the cRCf-w20 also exhibited a significant rise in pore volume between 4–10 nm and an increase in the SSA (Fig. 4-a) of 115% compared to RCf-NC, indicating that 20% moisture provides favourable conditions for pore development and accessible surface area during carbonation.

The increase in specific surface area can be associated with the formation of finely distributed carbonate phases, including vaterite and poorly crystalline  $\text{CaCO}_3$ , which typically exhibit higher surface areas than well-crystallised calcite (Zhao et al., 2023). Consistently, the cRCf-w50 sample exhibited a rise in the pore volume peak within the 2–10 nm range, which can be attributed to the formation of both calcite and

vaterite, as also supported by QXRD and DTG analyses (Fig. 1 and Fig. 2, respectively). However, SSA decreased slightly at 50 wt% water addition (cRCf-w50), suggesting that this level of moisture reduces the effective pore network due to the formation of more crystalline carbonation products that partially block fine mesopores. Such pore blocking under near-saturated conditions is consistent with dissolution–precipitation mechanisms, where high moisture promotes crystal growth rather than the generation of new surface area (Addadi et al., 2003; Gholizadeh-Vayghan et al., 2020). In particular, the development of pores in the lower mesopore range ( $\approx 2$ –10 nm) is critical for reactivity, as these pores provide a favourable balance between accessible internal surface area and effective diffusion pathways for  $\text{CO}_2$  and dissolved ions. Very small gel pores ( $< 2$  nm) are often poorly accessible to  $\text{CO}_2$ , while larger capillary pores mainly facilitate transport but contribute less to reactive surface area, making mesopores especially relevant for carbonation efficiency (Scrivener et al., 2016; Steiner et al., 2020).

A similar moisture-dependent trend has been reported for recycled concrete aggregates, where carbonation under near-saturated conditions (e.g., water-saturated aggregates or very high RH) resulted in limited pore structure modification, while reduced moisture availability led to significant improvements in pore structure and surface densification (Gholizadeh-Vayghan et al., 2020; Pan et al., 2017). Fig. 5 shows SEM images of carbonated RCf samples with varying water contents: cRCf-w0, cRCf-w5, cRCf-w10, cRCf-w20, and cRCf-w50. Across all samples, finer particles were observed agglomerating onto larger aggregates. In the cRCf-w0 sample, only minor morphological changes were noted, with particles partially covered by carbonation products. As the initial water content increased in the carbonated samples, a visible progression in the formation and distribution of crystalline carbonate structures became apparent.

Rhombohedral  $\text{CaCO}_3$  crystals, characteristic of calcite, were observed in all samples (Fig. 5). Their appearance increased, becoming especially pronounced in cRCf-w10, cRCf-w20, and cRCf-w50. In cRCf-w10 and cRCf-w20, well-formed, tightly packed calcite crystals are visible, coating the surfaces of larger particles and filling voids. The cRCf-w20 and cRCf-w50 images show dense clusters of crystals, indicating more extensive carbonation at this moisture level. EDS point analysis of cRCf-w20 (Fig. 5) confirmed the presence of calcium (Ca), carbon (C), and oxygen (O), supporting the identification of calcium carbonate. Notably, cRCf-w20 and cRCf-w50 exhibited a higher concentration of calcite crystals compared to lower moisture samples (cRCf-w0 and cRCf-w5). No clear evidence of spherical vaterite morphology

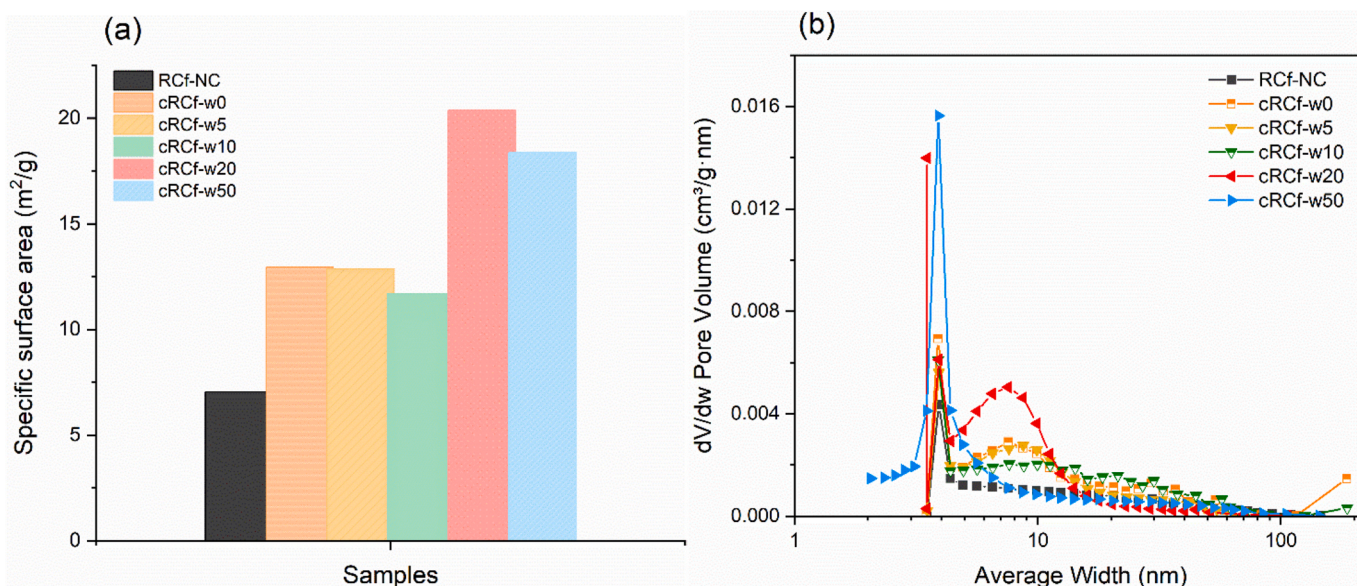


Fig. 4. (a) Specific surface area and (b) pore volume curves of RCf samples.

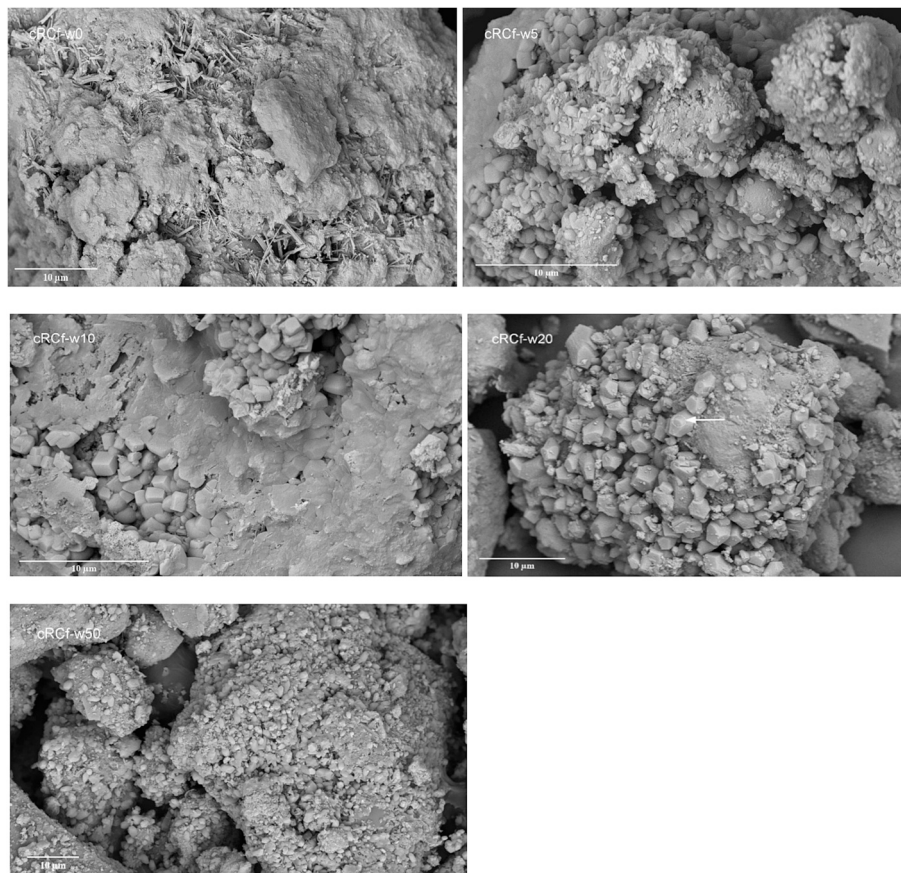


Fig. 5. Scanning electron microscopy images of carbonated RCf samples cRCf-w0, cRCf-w5, cRCf-w10, cRCf-w20 and cRCf-w50.

was observed, likely due to the formation of very fine vaterite crystals that fall below the resolution limit of the SEM used. Overall, the SEM analysis supported the conclusion that higher moisture content leads to more extensive and well-defined carbonation and an increase in SSA (Fig. 4).

The pozzolanic reactivity of raw and carbonated RCf was assessed using the  $R^3$  test. The resulting heat flow and total heat released curves are shown in Fig. 6, and detailed values for 1, 3 and 7 days of reaction are described in Table A.2. All samples exhibited an initial exothermic peak within the first hour (Fig. 6-a). As the initial moisture content increased in the carbonated samples, the intensity of the initial peak gradually decreased. No clear relationship was observed between the initial moisture content and the appearance of this first peak. The total heat released curve (Fig. 6) indicated that carbonation enhanced the pozzolanic reactivity of all samples compared to the uncarbonated RCf. cRCf-w50 exhibited the lowest total heat release, likely due to its content of crystalline calcium carbonate, particularly calcite, as identified by QXRD and DTG analysis (Fig. 1 and Fig. 2, respectively), which limits the availability of reactive phases. Additionally, the formation of a continuous calcium carbonate layer around the particles may have limited the accessibility of the reactive phases (Teune et al., 2023; Zajac et al., 2022). Although the sample was manually milled prior to testing to reduce this effect, it is still possible that some reactive phases remained partially encapsulated within this carbonate layer, thereby limiting their reactivity.

In the first 24 h, cRCf-w10 showed the highest early reactivity (89.71 J/g), followed by cRCf-w0 (87.65 J/g) and cRCf-w20 (85.12 J/g). cRCf-w20 achieved the highest total heat release after 3 and 7 days (Fig. 6), with 61.8% of its cumulative heat released within the first day and 30.9% between days 1 and 3, indicating more sustained reactivity beyond the initial reaction period. In contrast, cRCf-w10 released 74.2%

of its total heat within the first day and 15.1% between days 1 and 3, indicating a rapid early-age dissolution rate. No clear trend was observed among cRCf-w0, cRCf-w5, and cRCf-w10, as these samples exhibited similar crystalline and poorly crystalline calcium carbonate contents (Fig. 2), as well as comparable SSA values (Fig. 4). Despite this, cRCf-w10 released higher cumulative heat, particularly after 24 h of reaction. Samples with higher early reactivity, such as cRCf-w0, cRCf-w10, and cRCf-w20, were associated with the presence of poorly crystalline calcium carbonate and silica-rich phases, as also suggested by DTG analysis (Fig. 2) and SSA (Fig. 4). These components could readily dissolve, promoting C-S-H formation and accelerating reaction kinetics.

Conversely, cRCf-w50, despite having the second-highest SSA, exhibited lower reactivity, likely due to its higher crystalline calcium carbonate content. Hence, suggesting that the type and accessibility of the phases formed during carbonation influence reactivity more than carbonation extent alone, as also discussed by previous studies (Maia Neto et al., 2024; Teune et al., 2023). In addition, the initial RCf sample had an elevated quartz content, which might have hindered the development of pozzolanic reactivity. While calcite and quartz can provide nucleation sites for hydrates such as C-S-H, their direct contribution to pozzolanic reactions is limited. Although the reactivity of RCf has not been widely evaluated by the  $R^3$  test in the literature, previous studies have used the  $R^3$  test to assess the reactivity of different SCMs (Blotevogel et al., 2020; de Azevedo Basto et al., 2023; Londono-Zuluaga et al., 2022). The heat released after 7 days varies depending on the SCM tested, ranging from 50–300 J/g for natural pozzolans to 250–390 J/g for calcined clays (Londono-Zuluaga et al., 2022), and for inert materials, like quartz, this value is commonly below 50 J/g (Avet et al., 2016; Williams et al., 2023). Previous research (Sousa et al., 2024) found that RCf, sourced from full-scale recycling facilities, with high quartz content had a total heat release of 33.02 J/g<sub>scm</sub> after 7 days. During the  $R^3$  test,

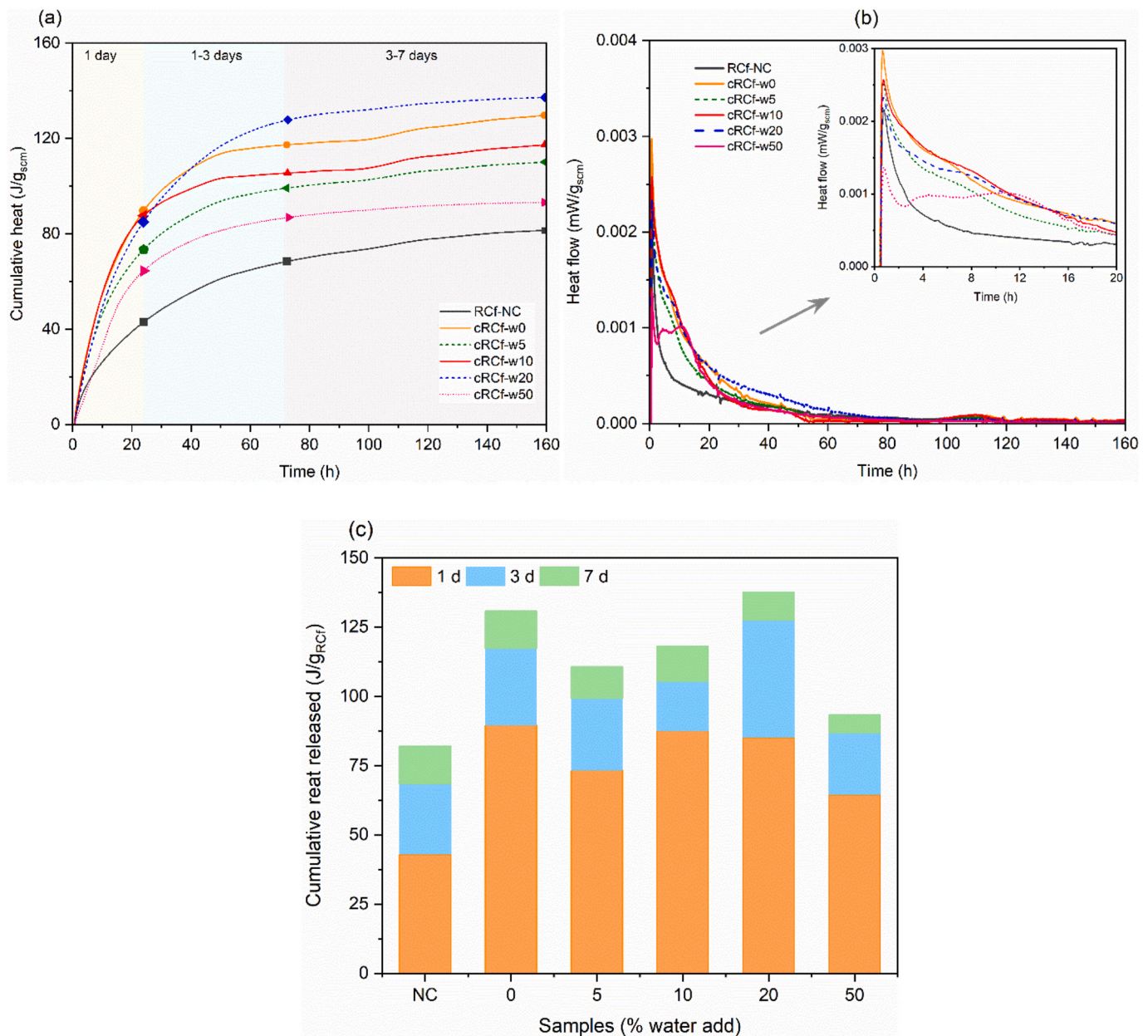


**Fig. 6.** Isothermal calorimetry results of (a) cumulative heat and heat flow curves for 7 days, (b) heat flow curves, and (c) cumulative heat released for each sample after 1, 3, and 7 days.

phases such as C-S-H and ettringite can be formed (Avet et al., 2016). Thus, poorly crystalline calcium-bearing phases within the SCM, in this case from the carbonated RCf samples, may dissolve in the alkaline environment, releasing calcium and other ions (e.g., Si, Al) that participate in pozzolanic and latent hydraulic reactions.

### 3.1. Properties of cement pastes incorporating carbonated RCf

To evaluate the effect of incorporating RCf into cement paste, both carbonated and non-carbonated fines were used to replace 20% of ordinary Portland cement, and the results are shown in Fig. 7. The reported compressive strengths correspond to the mean value of the three measurements, and standard deviations are provided in Appendix Table A3. The samples incorporated in the paste include RCf-NC, cRCf-w0, cRCf-w20, and limestone.

Fig. 7a shows clear differences between the formulations. At 1 day, the limestone and RCf-NC mixtures exhibited higher compressive

strength than the carbonated RCf mixtures, suggesting that the influence of carbonation on strength development became more evident at later curing ages. At later ages, P-RCf-NC showed only a slight reduction relative to the limestone reference (2.8%), whereas P-cRCf-w0 showed a reduction (9.9%), indicating limited benefit from carbonation without pre-wetting beyond the filler effect already provided by the uncarbonated fines. In particular, cRCf-w0 showed no improvement in strength compared to RCf-NC, whereas P-cRCf-w20 exhibited enhanced compressive strength after 28 days.

Although the incorporation of limestone in Portland cement formulations can enhance early hydration, higher substitution levels generally lead to reduced mechanical performance due to clinker dilution. This effect was also observed in the present study at a 20% replacement level. Moreover, the P-cRCf-w0 paste showed slightly lower 28-day strength than P-RCf-NC, indicating that carbonation without pre-wetting did not improve the effective contribution of the fines to strength development. This contrasts with the R3 calorimetry results (Fig. 6), where cRCf-w0

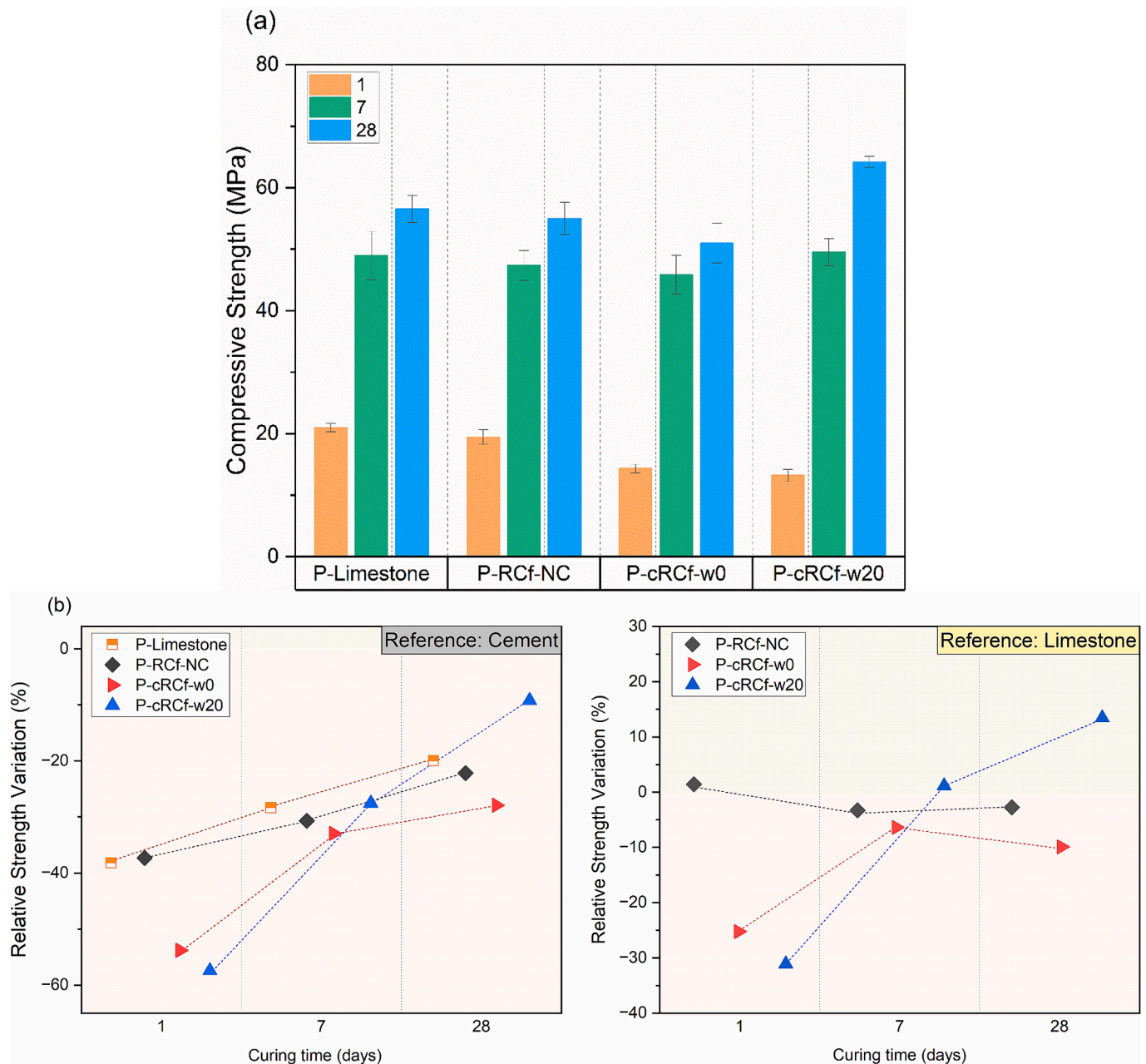


**Fig. 7.** (a) Mean compressive strength of paste mixtures containing 20% cement replacement at 1, 7, and 28 days; relative compressive strength change compared with (b) the cement reference and the limestone reference. Error bars in panel (a) represent one standard deviation based on three specimens.

exhibited the second-highest cumulative heat release among the carbonated samples. The difference indicates that the higher reactivity of cRCf-w0 measured under the R3 conditions was not directly translated into strength development in the cement paste. The R3 test evaluates SCM reactivity under controlled alkaline conditions, whereas strength development in cement paste also influenced by the interaction between the SCM and cement hydration (Suraneni et al., 2019).

In contrast, carbonation at intermediate moisture content (e.g., cRCf-w20) presented higher surface area and pore refinement (Fig. 4), which likely promoted nucleation of hydration products, resulting in higher compressive strength (Fig. 7). This may be related to the beneficial role of finely distributed calcite and filler-induced nucleation effects reported in the literature (Berodier and Scrivener, 2014; Lu et al., 2018). Previous studies have also shown that cement blends incorporating carbonated recycled concrete paste (RCP) can exhibit higher compressive strength than those using non-carbonated RCP or limestone, at a

similar replacement level, depending on the carbonation conditions (Lu et al., 2018; Zajac et al., 2023c).

It is known that limestone acts as a filler, enhancing particle packing density and providing nucleation sites for the cement hydrate phases (Berodier and Scrivener, 2014). In addition, limestone can chemically influence hydration through the formation of carboaluminate phases, contributing to ettringite stabilisation and modification of the hydrate assemblage (De Weerd et al., 2011; Sabtiwu et al., 2025). Furthermore, the effect of limestone is not governed solely by fineness or surface area, since interactions between particle chemistry and the cementitious system are also important in determining its effectiveness (Sabtiwu et al., 2025).

Similarly, fine calcium carbonate from carbonated RCf, as identified in the SEM analysis (Fig. 5) may also contribute to strength development through filler and nucleation effects, particularly at later ages. Quartz, present in the RCf, may also contribute to the filler effect and serve as a

nucleation site, although C-S-H nucleates preferably on limestone surfaces (Berodier and Scrivener, 2014).

Relative strength variation values were calculated to compare RCf mixes with limestone and cement references (Fig. 7-b). After 28 days of curing, the replacement with cRCf-w20 resulted in a 13.4% increase compared to the P-Limestone. In comparison, cRCf-w0 and RCf-NC showed reductions of 9.9% and 2.8%, respectively. Previous studies reported (Lu et al., 2018; Mehdizadeh et al., 2022; Zhang et al., 2024) that the addition of 10–20% carbonated RCf led to an improvement in compressive strength when compared to OPC cement samples. Among the RCf, cRCf-w20 showed the highest SSA and cumulative pore volume (Fig. 4), which likely promoted nucleation of hydration products.

Carbonation under low-moisture conditions may be limited by transport processes, influencing the distribution of carbonate phases within the particles (Gholizadeh-Vayghan and Snellings, 2022; Saeki et al., 2024). This can affect the accessibility of residual reactive phases when the carbonated fines are later used in cement paste, thereby reducing their effective contribution to hydration and strength development (Yuan et al., 2024). Calcite offers favourable interfacial properties and lower nucleation energy barriers for hydrated phases due to its stable crystal structure (Zhao et al., 2023). In contrast, the P-cRCf-w0 paste showed lower compressive strength than P-Limestone at all curing ages, with reductions of approximately 25.2%, 6.4%, and 9.9% after 1, 7, and 28 days, respectively. The limited increase in specific surface area observed for cRCf-w0 suggests that the reactive phases formed during carbonation were not sufficiently accessible to effectively participate in hydration reactions. By comparison, the performance of P-RCf-NC may be associated with the presence of residual hydrated phases (such as C-S-H) and calcite, which can contribute to strength development by providing nucleation sites for the growth of hydration products (Lu et al., 2018).

P-cRCf-w20 exhibited 37.1% lower strength than P-Limestone at 1 day, followed by slight improvement at 7 (1.1%) and an increase at 28 days (13.4%). This trend indicates a transition from slower early-age kinetics to enhanced later-age reactivity, as reflected in the evolution of relative strength (Fig. 7b).

Overall, RCf-NC and cRCf-w0 showed lower or comparable strength than limestone reference, whereas cRCf-w20 showed a clear strength enhancement at 28 days. Despite lower early-age strength, likely due to delayed hydration, the improved performance of cRCf-w20 is associated with the higher specific surface area and mesoporosity observed after carbonation, together with the presence of more accessible reactive phases. These features may have supported further hydration at later ages, resulting in greater strength development.

The influence of moisture is further influenced by the initial characteristics of the material. Variations in cement paste content, inert phases, and initial porosity affect the optimal moisture content for carbonation. Bustamante et al. (2024) showed that fines derived from higher w/c ratio pastes (~0.7) required around 20% moisture to reach maximum densification, whereas those from lower w/c ratio pastes (~0.5) peaked at approximately 10%. This suggests that more porous materials can accommodate higher moisture levels before diffusion limitations occur, while denser materials reach this limit at lower moisture contents.

Carbonation effectiveness was therefore controlled by the accessibility and distribution of reaction products within the microstructure. At intermediate moisture (~20 wt%), carbonation resulted in increased surface area and mesoporosity, enhancing the availability of reactive phases, whereas higher moisture (50 wt%) promoted the formation of more crystalline and less accessible carbonate phases despite greater CO<sub>2</sub> uptake, consistent with observations under high moisture conditions (Bao et al., 2025; Cheng et al., 2025).

Unlike earlier studies focused primarily on laboratory-produced recycled concrete paste, wet carbonation, or carbonation degree alone, this study evaluates RCf generated in a full-scale recycling facility across five initial moisture contents under a fixed semi-dry carbonation

treatment and links phase evolution, surface area and pore structure with R3 reactivity and performance at 20% cement replacement.

#### 4. Conclusion

Recycled concrete fines (RCf) generated during concrete recycling remain largely underutilised or are downcycled despite containing calcium-bearing phases that can be carbonated, which could support their use as supplementary cementitious materials (SCMs). Developing valorisation strategies for RCf requires treatment methods that can both improve reactivity and, ideally, sequester CO<sub>2</sub>. This study investigated the influence of semi-dry carbonation of RCf generated in a concrete recycling facility on the phase composition, microstructure, reactivity, and subsequent performance. Moreover, the use of industrially produced RCf highlights the need to evaluate mineral carbonation beyond laboratory-generated materials. Therefore, under the semi-dry carbonation conditions investigated, increasing the initial water addition increased the degree of carbonation and favoured the formation of crystalline calcium carbonate. However, this trend did not produce a corresponding increase in R3 reactivity. The cRCf-w20 sample exhibited the highest measured specific surface area and the highest cumulative R3 heat release after three and seven days, whereas cRCf-w50 showed the highest carbonation degree but the lowest R3 response among the carbonated samples. Paste validation of cRCf-w20 also demonstrated favourable 28-day performance at 20% cement replacement relative to limestone.

#### CRedit authorship contribution statement

**L.N. Sousa:** Writing – original draft, Visualization, Methodology, Investigation, Data curation, Conceptualization. **K. Schollbach:** Writing – review & editing, Supervision, Resources, Conceptualization. **H.J.H. Brouwers:** Writing – review & editing, Supervision, Project administration, Funding acquisition, Conceptualization.

#### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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#### Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.wasman.2026.115866>.

#### Data availability

Data will be made available on request.

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