

Upcycling recycled concrete fines and wheat straw fibres into carbonation-cured circular cementitious composites

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ABSTRACT

This study investigates the combined use of recycled concrete fines (CF) and wheat straw fibres to develop cementitious composites under accelerated carbonation curing. Mixtures containing 8 wt % wheat straw fibres and 0–20 wt% CF as partial cement replacement were cured at 20 °C and 50 °C under carbonated and non-carbonated conditions. Carbonation reduced porosity and increased bulk density, resulting in a 33–43% increase in thermal conductivity and improved mechanical performance. The presence of CF appeared to enhance carbonation through a combination of residual reactive phases and physical effects related to particle packing and transport, contributing to higher strength development. Thermogravimetric analysis confirmed increased CaCO₃ formation at elevated temperature. Total CO₂ storage reached up to 0.207 kg CO₂/kg material, combining mineral and biogenic contributions. These results show that combining CF and wheat straw fibres under accelerated carbonation curing can improve material performance while increasing CO₂ storage.

1. Introduction

Efforts to reduce the environmental impact of cement production have focused on strategies, including the development of more energy-efficient processes, the use of alternative binders, and technologies that enable CO₂ uptake [1–3]. Among these, the partial replacement of clinker with supplementary cementitious materials is one of the main strategies to reduce CO₂ emissions associated with cement production [4–6].

In parallel, the recycling of construction and demolition waste (CDW) has gained increasing attention as a way to reduce the consumption of natural resources and the environmental impact of construction materials. This approach promotes material reuse and reduces the demand for virgin resources in construction [1,7]. Recycled concrete fines (CF) are generated during the crushing and classification of end-of-life concrete, producing a fine fraction that can represent around 20–30% of the original concrete mass, depending on the recycling process. These fines are mainly composed of hydrated cement paste and fine particles, but remain less utilised than coarse recycled aggregates and are often downcycled.

In addition to mineral recycling strategies, lignocellulosic agricultural residues have been incorporated into cementitious composites to modify pore structure, reduce density, and improve thermal insulation [8,9]. Due to its cellular and porous structure, wheat straw shows low thermal conductivity and high moisture sensitivity, which makes it attractive for lightweight insulating materials,

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although it also raises durability concerns under unfavourable conditions [10].

The transition towards circular bio-based materials relies on valorising residual biomass and integrating waste streams into new value chains, reducing dependence on virgin resources and fossil-based materials [11,12]. However, the incorporation of wheat straw fibres introduces challenges, as degradation under alkaline conditions and dimensional instability can increase porosity and reduce mechanical performance [13]. Previous studies have mainly focused on wheat straw ash as a supplementary cementitious material, with reported optimal replacement levels around 10% and associated improvements in mechanical performance due to pozzolanic reactions [14].

Carbonation curing has been recognised as a method that can simultaneously enhance material performance and enable CO₂ uptake [8,15]. During carbonation, CO₂ reacts with hydration products such as portlandite and C-S-H to form CaCO₃, leading to microstructural densification and improved mechanical performance [15,16]. The efficiency of carbonation depends on CO₂ transport, moisture availability, and pore structure, as carbonate precipitation both densifies the matrix and progressively limits further CO₂ ingress [17]. Recent studies have also shown that carbonation treatment of recycled concrete fines can improve material properties while reducing environmental impact [18].

In addition, recycled concrete powder has been used to produce dense cementitious materials through combined compaction and carbonation [19]. Carbonation depends on transport conditions within the material, particularly CO₂ diffusion and moisture distribution. As carbonation progresses, diffusion becomes limiting, and CO₂ uptake typically reaches 20–50% of the theoretical potential [15].

In fibre-reinforced composites, the additional porosity introduced by natural fibres may influence carbonation by affecting moisture distribution and CO₂ transport. Carbonation can partly compensate for strength reductions in porous systems through microstructural changes [20,21], although the combined effect of recycled concrete fines, natural fibres, and carbonation curing remains insufficiently explored.

Related studies also show that internal moisture and transport pathways influence carbonation behaviour, for example, biochar has been reported to enhance carbonation and strength under CO₂ curing in MgO-based binders [22]. Although the chemistry differs from OPC systems, these findings indicate that porous additions can affect carbonation through moisture distribution and CO₂ transport.

While previous studies have investigated carbonation of recycled fines or fibre-reinforced systems separately, their combined interaction has not been widely studied. In particular, how recycled concrete fines and lignocellulosic fibres jointly influence carbonation efficiency, microstructural evolution, and CO₂ storage under controlled curing conditions remains insufficiently explored. Therefore, this study investigates the combined effects of CF incorporation and wheat straw fibres under accelerated carbonation curing, focusing on the assessment of microstructure, thermal properties, mechanical performance, and CO₂ storage.

2. Materials and methods

2.1. Materials

Concrete fines (CF) supplied by Circulair Mineraal (the Netherlands) were used as partial cement replacement in the composite. The material presented a particle density of approximately 2.70 g/cm³ and was sieved to <250 µm prior to mixing to ensure a consistent particle size distribution. The particle size distribution is shown in Figure A.1 (Appendix), indicating a predominantly fine particle fraction, with d₅₀ and d₉₀ values of 44.74 µm and 130.53 µm, respectively.

Wheat straw fibres were supplied by Strobouwer (the Netherlands). The fibres originated from the same batch previously characterised and studied by Ref. [10]. Therefore, the physical properties reported in that study were adopted here. These include a density of approximately 1.01 g/cm³, fibre lengths ranging from 0.5 to 20 mm, and an open porosity of about 92% [10].

The cement used to prepare the mixes was CEM I 52.5R (3.15 g/cm³), and clean, potable water was used. Particle size distribution was determined by laser diffraction (Malvern Mastersizer 2000) using isopropanol as a dispersant. The chemical composition of CF and cement was obtained by X-ray fluorescence (XRF) analysis (PANalytical Epsilon 3, standardless OMNIAN method) and is presented in Table A.1 (Appendix).

The recycled concrete fines (CF) are characterised by a high SiO₂ content (62.38 wt%) and relatively low CaO content (12.63 wt%), reflecting the presence of quartz phases and residual cementitious material. In contrast, the cement is dominated by CaO (61.36 wt%), consistent with its clinker-based composition. The higher loss on ignition (LOI) of CF (15.92 wt%), compared to cement (4.65 wt%), indicates the presence of pre-existing carbonates and hydrated phases.

XRD analysis of the CF (Figure A.2, Appendix), supported by quantitative phase analysis (Table A.2, Appendix), identified 56.6% quartz (SiO₂), 8.1% feldspar (albite), 9.6% calcite (CaCO₃), 2.5% gypsum, and 2.5% ettringite, along with an estimated 16.6% amorphous content (Table A.2, Appendix). The portlandite content was below 1%, which is consistent with the thermogravimetric analysis (Figure A.3, Appendix). Previous findings report similarly low Ca(OH)₂ content in CF generated from the concrete recycling industry [23]. The minerals quartz, albite, gypsum, and calcite likely originate from the residual natural aggregates in the parent concrete and cement carbonation.

2.2. Mix design

Four mix designs were developed to assess the influence of concrete fines (CF) content and wheat straw fibres on the properties of cementitious composites. To evaluate the influence of CF incorporation while avoiding excessive dilution effects and reduced mixture stability, replacement levels of 5–20 wt% were studied.

The wheat straw dosage was selected based on a combination of previous literature and preliminary mixture optimisation. Previous studies have shown that natural fibre content affects workability, fibre dispersion, and mechanical performance in cement-based composites, requiring optimisation depending on the specific matrix and fibre characteristics [9,24]. Preliminary trials were carried out to determine the fibre dosage, leading to a fixed content of 8 wt% relative to cement mass. Higher fibre dosages reduced mixture homogeneity and fibre dispersion during preliminary mixing trials, while lower dosages resulted in less pronounced effects on density reduction and composite behaviour. Hence, the selected 8 wt% fibre content provided a balance between workability and binder demand. The flowchart of the experimental procedure is shown in Fig. 1.

As the wheat straw fibres are highly water absorbent, they were pre-hydrated prior to mixing. Pre-hydration was done using a 1:1 water-to-straw ratio to prevent additional water absorption during mixing. The fibres are highly hygroscopic [10], and the selected ratio was intended to reduce rapid moisture uptake during mixing and improve fibre dispersion, as commonly reported for natural fibre-reinforced cementitious composites [9], while avoiding excessive local water accumulation that could hinder CO₂ transport during carbonation [21]. The same procedure was applied for all mixtures to ensure experimental consistency. However, the water absorption behaviour of the fibres was not directly quantified in the present study. A more detailed characterisation of the wheat straw used in this study, including its hygrothermal behaviour and related properties, can be found in previous studies [10,25].

The fibres were first blended with water for 2 min, after which cement was added and mixed for another 2 min. CF was then introduced and mixed for 1 min, followed by the addition of the remaining water. Finally, the mixture was homogenised at high speed for 2 min. The water-to-binder (w/b) ratio was fixed at 0.3 for all mixtures.

Fresh paste samples were cast in standard prism moulds (40 × 40 × 160 mm), compacted to eliminate air voids, and covered with plastic sheets to prevent moisture loss. The specimens were initially cured under controlled conditions (25 °C, 65% RH) for 3 days before being transferred to their respective curing environments.

2.3. Curing conditions

After casting, all samples were sealed with plastic foil and kept in the moulds for 3 days in a controlled room (≈20–25 °C, 65% RH). After demoulding, the specimens were transferred to the respective curing environments. The carbonation conditions were selected to represent accelerated curing environments commonly used in carbonation studies. A CO₂ concentration of 20% was chosen as a representative concentrated industrial CO₂ stream relevant to carbonation applications [26], while 80% RH was maintained to provide sufficient moisture for carbonation reactions. The curing temperatures of 20 °C and 50 °C were selected to evaluate carbonation behaviour under ambient and moderately elevated temperature conditions, as temperature is known to influence carbonation kinetics and CO₂ transport within cementitious materials [27].

Carbonation was carried out in a climate chamber at either 20 °C or 50 °C with 20% CO₂ and 80% RH for 4 days. Specimens were subsequently stored under sealed conditions until the respective testing age. For comparison, non-carbonated samples were kept sealed in plastic foil and cured at the same temperatures in a laboratory oven for the same duration, but without CO₂ exposure. In this study, mixes are denoted NF8 for the control (0% CF), and CFX-T-C/NC, where X indicates the CF replacement level (5, 10, or 20%), T the

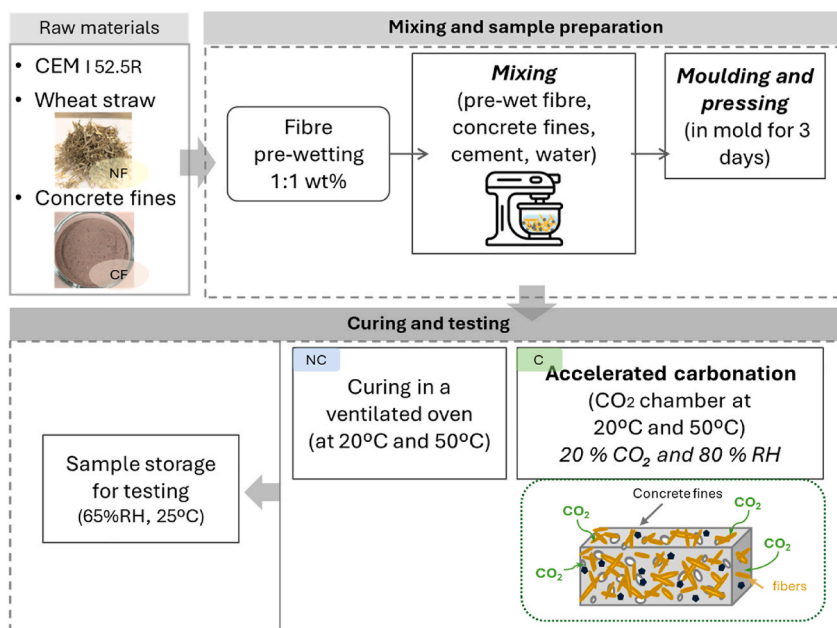


Fig. 1. Experimental workflow illustrating raw materials, mix preparation, and curing procedures for carbonated (C) and non-carbonated samples (NC).

curing temperature (20 °C or 50 °C), and C/NC specifies carbonation or oven curing. For instance, CF10-20-C corresponds to a mix with 10% CF and carbonated at 20 °C.

2.4. Methods

2.4.1. Sample characterisation

2.4.1.1. Thermal conductivity, porosity, and bulk density. Prior to characterisation, specimens were dried in an oven at 40 °C for 3 days to minimise differences in internal moisture content. Specimens allocated to later-age testing were kept sealed in plastic foil until the drying step. Given that thermal transport and pore structure strongly influence the performance of composite cement materials, both properties were evaluated. The thermal conductivity of each mix was assessed using two samples placed side by side on the ISOMET 2104 device, with a precision of ± 0.001 W/(m·K). To improve accuracy, each pair of side-by-side specimens was measured three times at different positions (upper, middle, and bottom) with a 3-min pause between measurements. This approach aimed to minimise potential deviations caused by environmental fluctuations or instrument drift. Furthermore, total porosity was calculated from bulk and particle density as follows:

$$\text{porosity (\%)} = 1 - \frac{\rho_{\text{bulk}}}{\rho_{\text{particle}}} \quad (1)$$

where ρ_{bulk} represents the bulk density, and ρ_{particle} corresponds to the particle density measured using a helium pycnometer (Micromeritics AccuPyc II 1340). The bulk density was calculated as the ratio between the sample mass and the total specimen volume (256 cm³).

2.4.1.2. Compressive and flexural strength. Mechanical performance was evaluated through flexural and compressive strength tests. Flexural strength was evaluated in accordance with EN 196-1 at 7 and 28 days, with a three-point bending test (INSTRON). The test setup featured a support span of 100 mm, and loading was applied at a constant rate of 1 mm/min. The compressive strength (AUTOMAX5) was measured at a constant load rate of 2400 N/s at 91 days. The later testing age was selected to ensure microstructural stabilisation, considering the relatively high content of natural fibres in the mixtures.

After flexural testing, phenolphthalein solution was used as a pH indicator. This indicator is widely used to estimate carbonation depth. It turns colourless in carbonated zones where pH drops below 9.0 and magenta in uncarbonated areas with pH above 9.5, providing a clear visualisation of the carbonation front.

2.4.1.3. Thermogravimetric analysis and CO₂ storage. To evaluate the overall CO₂ storage capacity of the developed composites, both biogenic and mineral CO₂ contributions were considered. Biogenic CO₂ associated with wheat straw fibres was calculated following EN 16449:2014, which converts biomass carbon content into equivalent atmospheric CO₂. This carbon corresponds to CO₂ absorbed during plant growth and stored within the lignocellulosic structure. As all mixtures contained the same fibre dosage (8 wt%), the biogenic CO₂ contribution was considered constant across all formulations.

Total CO₂ storage was defined as the sum of biogenic CO₂ and mineral CO₂ contributions:

$$\text{CO}_{2,\text{total}} = \text{CO}_{2,\text{bio}} + \text{CO}_{2,\text{mineral}} \quad (2)$$

This approach allows comparison of the overall CO₂ storage capacity of carbonated and non-carbonated specimens as a function of curing conditions and concrete fines content. The biogenic CO₂ contribution from wheat straw fibres was estimated from the initial biomass carbon content according to EN 16449:2014 and treated as constant across all mixtures due to the fixed fibre dosage. Although the effects of carbonation curing on the lignocellulosic structure were not directly evaluated, previous studies have shown that alkaline cementitious environments may promote degradation of lignin and hemicellulose in natural fibres, whereas carbonation may reduce matrix alkalinity and create a less aggressive environment for fibre stability [28,29]. However, long-term changes in fibre composition and biogenic carbon stability were not investigated in the present study.

Thermogravimetric analysis (TGA) was performed to assess the degree of carbonation and quantify mineral CO₂ storage within the binder phase. Samples were crushed and sieved (<90 µm) to remove straw fibres and isolate the mineral fraction. The resulting powders were dried at 40 °C for 24 h prior to testing. TGA was performed under a nitrogen atmosphere at a heating rate of 10 °C/min from 40 to 1000 °C using a NETZSCH Tarsus instrument.

Mineral CO₂ content was calculated from the mass loss observed between 600 and 800 °C, corresponding to the decarbonation of calcium carbonate ($\text{CaCO}_3 \rightarrow \text{CaO} + \text{CO}_2$), according to:

$$\text{CO}_2(\%) = \frac{\Delta m_{600-800}}{m_i} \times 100 \quad (3)$$

where $\Delta m_{600-800}$ is the mass loss within the decarbonation interval and m_i is the initial sample mass. The equivalent calcium carbonate content was calculated as:

$$\text{CaCO}_3(\%) = \text{CO}_2^m(\%) \times \frac{100}{44} \quad (4)$$

where 44 g mol^{-1} and 100 g mol^{-1} are the molar masses of CO_2 and CaCO_3 , respectively. The mineral CO_2 storage was expressed as:

$$CO_2^m \left(\frac{\text{kg}}{\text{kg}} \right) = \frac{CO_2^m (\%) }{100} \quad (5)$$

where $CO_2^{\text{mineral}} (\text{kg} / \text{kg})$ denotes the amount of CO_2 stored through mineral carbonation per unit mass of material. As the CF was sourced from an industrial recycling stream, the CO_2 storage calculation accounted for the mineral CO_2 content determined by TGA, including both pre-existing carbonates and carbonates formed during carbonation curing. Therefore, the effect of carbonation was evaluated by comparison with the corresponding non-carbonated reference samples.

3. Results and discussion

3.1. Thermal and microstructural properties

The influence of carbonation and CF replacement on thermal and microstructural properties is presented in Fig. 2a and b, including thermal conductivity, porosity, and optical microscopy images. Overall, thermal conductivity increased with increasing bulk density and decreasing porosity, reflecting the more compact structure of the material. Since all specimens were subjected to the same conditioning procedure prior to characterisation, the observed differences are therefore interpreted primarily in terms of changes in

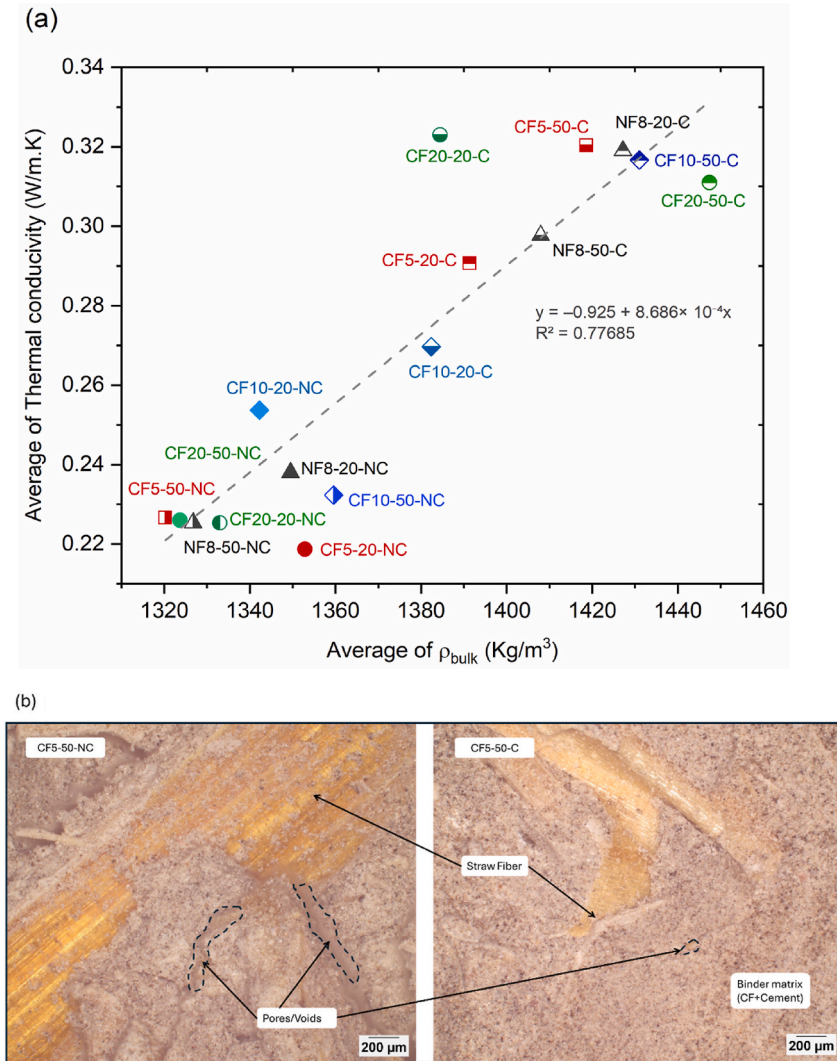


Fig. 2. (a) Correlation between average thermal conductivity and bulk density for all samples at 20 °C and 50 °C based on data provided in Table A.4 in Appendix A. Each data point represents the average value for one mixture and curing condition. (b) Optical microscope images of CF5-50-NC (left) and CF5-50-C (right).

microstructure rather than differences in moisture content.

Carbonation increased thermal conductivity at both curing temperatures (20 °C and 50 °C). Compared with the non-carbonated samples, conductivity increased by approximately 33–43%, depending on mixture composition and curing condition. The largest increase was observed for CF20-20-C (0.323 W/m·K), corresponding to a ~43% increase relative to CF20-20-NC (0.226 W/m·K). Similar improvements were measured for CF5-50-C and CF20-50-C. Since wheat straw fibres have inherently low thermal conductivity [10], the observed changes can be mainly attributed to modifications of the cementitious matrix rather than the fibre phase. The thermal conductivity values obtained in this study (0.22–0.32 W/mK) were comparable to those reported for other bio-based and lightweight construction materials, including cement-based lightweight blocks incorporating textile waste (0.149–0.294 W/mK) [30], gypsum composites incorporating rice straw fibres (0.233 W/mK) [31], and straw-based cementitious composites (0.240–0.259 W/mK) [32].

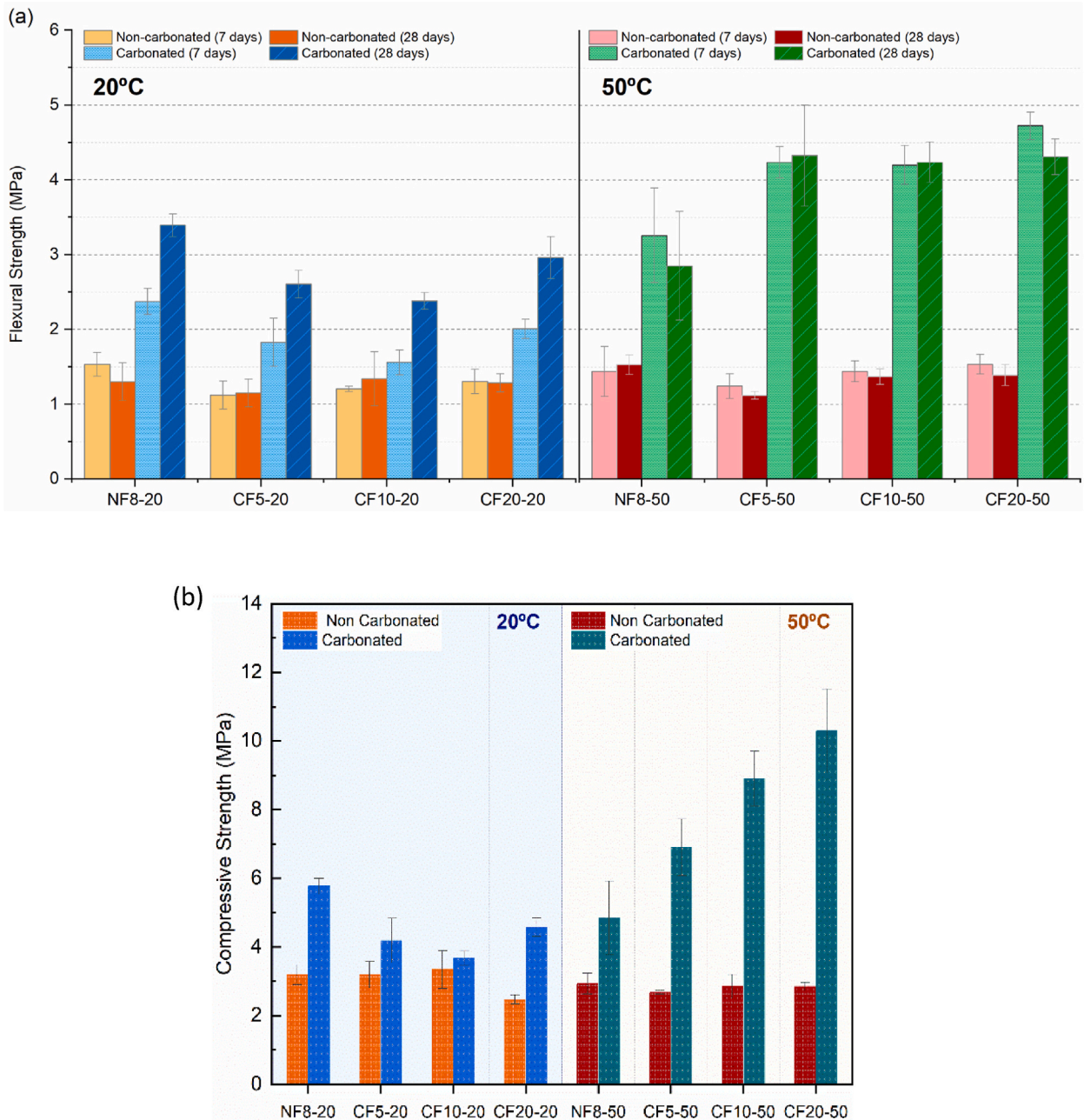


Fig. 3. (a) Flexural strength at curing ages of 7 and 28 days, (b) compressive strength at 91 days of carbonated and non-carbonated samples cured at 20 °C and 50 °C.

In the non-carbonated samples, bulk density showed only limited variation with increasing CF content. At 50 °C, values remained in a narrow range from 1320 to 1354 kg/m³, while at 20 °C they ranged from 1324 to 1353 kg/m³. After carbonation, most mixtures showed reduced porosity and increased density. At 20 °C, the reduction in porosity ranged from about 7.0% to 14.7%, whereas at 50 °C the largest reduction reached 10.8% for CF20-50-C.

These changes are consistent with the increase in thermal conductivity and indicate that carbonation leads to a denser matrix. The effect was more evident at 50 °C, in line with higher carbonation under elevated temperature. Carbonation involves both pore filling through CaCO₃ formation and local changes associated with decalcification of hydration products [33,34].

CF10-20-C deviated from the general trend, showing lower density than its non-carbonated counterpart at 20 °C. This behaviour may be related to local heterogeneity in particle packing or moisture distribution. Recycled concrete fines typically contain micro-cracks and porous adhered mortar, which contribute to a heterogeneous structure. Carbonation further modifies this structure by converting cement hydration products (mainly portlandite and C-S-H) into carbonate phases and silica-rich gel, while reducing pore volume and average pore size, thereby affecting density and transport behaviour [35].

Fig. 2b shows optical microscopy images of CF5-50 in the non-carbonated (CF5-50-NC) and carbonated (CF5-50-C) states. In the non-carbonated sample, visible voids are present around the straw fibres and within the matrix. In contrast, the carbonated sample shows a more continuous matrix with fewer visible voids around the fibres.

3.2. Mechanical properties and carbonation depth

Mechanical performance was evaluated through flexural and compressive strength tests (Fig. 3). A small number of individual measurements (7 out of 96) were excluded from the plotted results following a simple outlier screen applied within each test type and curing condition. The dataset is provided in Appendix A (Table A.5, Appendix). Carbonation enhanced mechanical performance across all mixtures, increasing both compressive and flexural strength.

At 50 °C, all carbonated samples (NF8, CF5, CF10, and CF20) exhibited higher strength compared with the corresponding non-carbonated mixes, with the magnitude of improvement generally becoming more pronounced at higher concrete fines content. Among these, CF20 showed the largest increase in strength. This behaviour is consistent with previous studies on recycled concrete powder, where carbonation promotes strength development through the formation of CaCO₃ bridges between particles, improving interparticle bonding [19].

CF20-50 showed an increase in compressive strength from 2.84 MPa to 10.29 MPa after carbonation. NF8-50 showed the lowest compressive and flexural strength among the carbonated samples. The increase in compressive strength generally aligned the mineral CO₂ uptake trend, indicating a close relationship between carbonation extent and strength development. In addition, the strength enhancement observed after carbonation is consistent with previous studies showing that carbonate precipitation fills pores and densifies the microstructure, leading to improved mechanical performance [36,37].

Among the carbonated samples, those cured at 50 °C showed the highest strength values (Fig. 3), suggesting that higher curing temperature improved carbonation and contributed to strength development under the investigated conditions. Similar trends have been reported for recycled concrete fines, where increasing carbonation temperature raised the carbonation degree (from ~4% at 20 °C to ~30% at 100 °C) and CO₂ uptake (up to ~21%), reflecting a higher extent of reaction [35]. Lu et al. [36] also observed that higher CO₂-curing temperatures increased both carbonation depth and compressive strength, with specimens cured at 20 °C and 50 °C showing higher strength than those cured at 5 °C, reaching increases of up to ~35–60% at early ages.

Carbonate precipitation contributes to pore filling and the formation of interparticle crystal bridges, improving load transfer within the matrix [19]. In compacted recycled concrete powder, carbonation enabled compressive strengths above 40 MPa by shifting particle bonding from physical contact under pressure to chemical CaCO₃ bridge bonding [19]. Similar benefits were reported for carbonated recycled concrete fines in UHPC, where carbonation improved compressive strength by 1.73–3.69% relative to the corresponding uncarbonated fines, while also reducing cumulative pore volume by about 25% [18].

The higher strength gains observed in mixtures containing CF (CF5-50, CF10-50, and CF20-50) suggest that recycled concrete fines further influence strength development, likely related to improved particle packing and microstructural refinement. Previous studies have reported that CF incorporation can enhance mechanical performance through improved packing density and crack-control mechanisms within the matrix [35,38–40]. However, recycled powders generally have lower intrinsic reactivity than Portland cement, which can limit strength development at higher replacement levels unless activation mechanisms such as carbonation are involved.

In contrast, NF8 samples, which contain no CF, showed smaller strength gains under carbonation. NF8-50 increased in compressive strength from 2.93 MPa (NF8-50-NC) to 4.85 MPa (NF8-50-C), while flexural strength increased from 1.53 MPa to 2.85 MPa. Although carbonation improved strength even without CF, the increase was lower than in mixtures containing CF. This difference is likely related to the absence of concrete fines, which reduces the availability of reactive surfaces for carbonation. CF particles typically contain adhered mortar and micro-porous regions that may facilitate CO₂ ingress and provide accessible CH and C-S-H for carbonation reactions, leading to more effective microstructural modification. Consequently, NF8 mixtures likely undergo a lower extent of carbonation and show less pronounced densification compared with CF-containing mixes. Similar trends have been reported in cement-bonded fibreboards, where carbonation curing enhanced both CO₂ uptake and mechanical performance [21].

Although elevated temperature can influence carbonation kinetics, moisture availability, and transport processes within cementitious materials [27], the non-carbonated samples showed only limited differences between 20 °C and 50 °C. In contrast, substantially larger improvements were observed in the carbonated series, particularly for mixtures containing CF, suggesting that carbonation was the dominant factor contributing to the observed strength enhancement under the investigated conditions. Carbonated samples cured

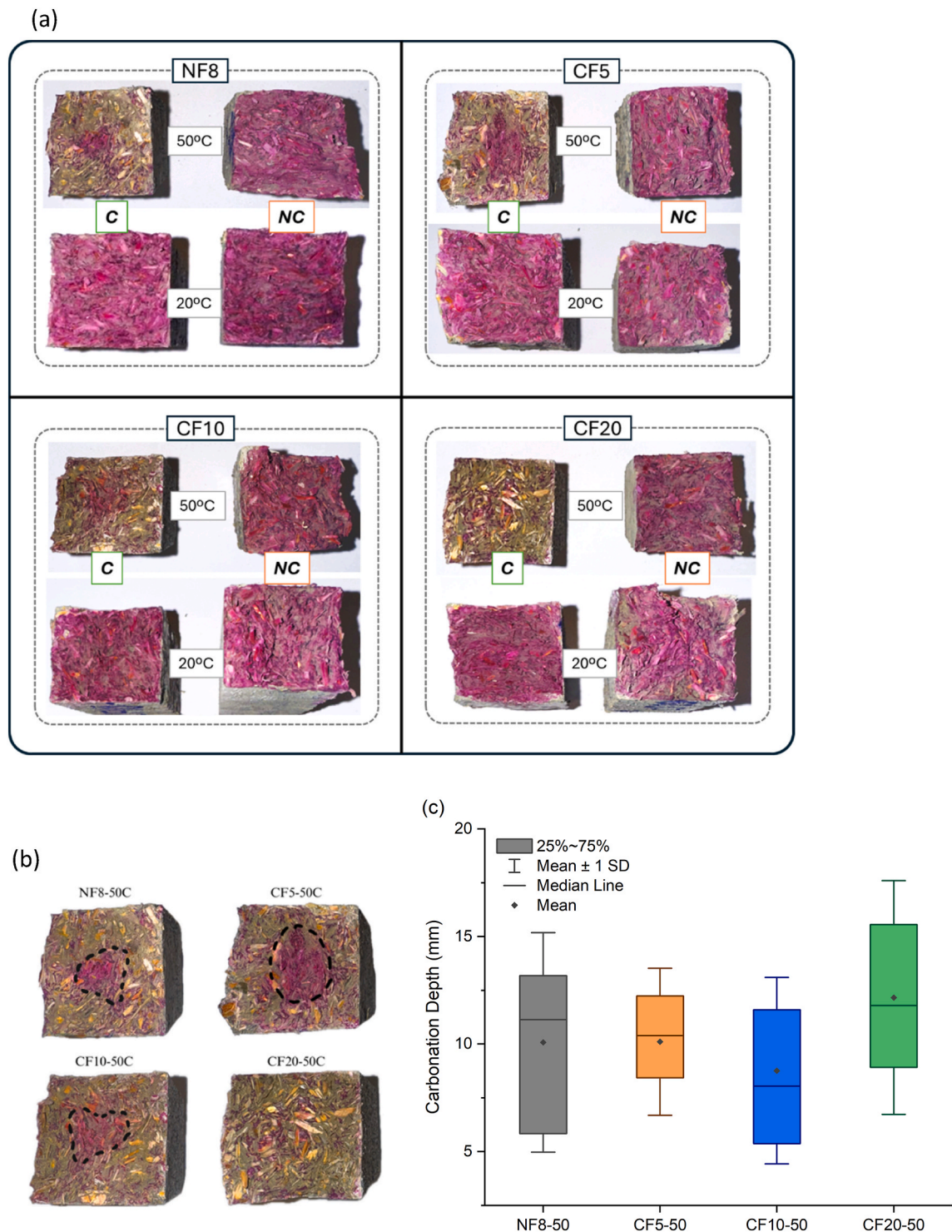


Fig. 4. (a) Phenolphthalein test images of non-carbonated (NC) and carbonated (C) samples for all mixtures (NF8, CF5, CF10, and CF20), cured at 20 °C and 50 °C. (b) Representative cross-sections of samples carbonated at 50 °C showing carbonation fronts. (c) Carbonation depth distributions for samples carbonated at 50 °C, presented as boxplots based on 50 measurements per sample, showing the median, mean, interquartile range, and standard deviation.

at 50 °C consistently showed higher strength than those cured at 20 °C, indicating that the effect is mainly associated with enhanced carbonation rather than temperature alone. These observations are consistent with the densification associated with carbonation, which aligns with the strength increases shown in Fig. 3, particularly for CF10 and CF20.

To evaluate the extent of carbonation, the pH indicator test was conducted. Fig. 4a illustrates the results, showing no visible carbonation penetration in the non-carbonated samples (NF8-NC, CF5-NC, CF10-NC, and CF20-NC). As expected, no clear differences were observed between the two curing temperatures for the non-carbonated series.

As shown in Fig. 4b, carbonated samples cured at 50 °C exhibit notably greater carbonation depths compared to those cured at 20 °C, indicating that elevated temperature accelerated the carbonation process. This behaviour is likely related to enhanced CO₂ diffusion and faster reaction kinetics at higher temperature, which promote deeper carbonation fronts and increased reaction extent [36]. Local transport conditions within the fibre-containing matrix may also influence carbonation penetration. Higher temperatures also facilitate interactions between CO₂ and cementitious phases, thereby increasing overall carbonation efficiency.

In contrast, at 20 °C (Fig. 4a), carbonation was limited and not clearly detected by the pH indicator test, likely due to limited carbonation depth and reduced CO₂ transport within the matrix [41]. The absence of a clearly visible phenolphthalein carbonation front at 20 °C highlights the limitation of pH indicator methods for detecting partial or low-degree carbonation. Carbonation at 20 °C may therefore still have occurred, but below the visual detection limit of the indicator [29].

Image analysis was performed using ImageJ software to quantify carbonation depth from phenolphthalein sections. For each sample, 50 measurements were obtained by tracing line segments from the exposed surface to the carbonation front. The resulting carbonation depth distributions are presented in Fig. 4c as boxplots, allowing visualization of the median, mean, and standard deviation, highlighting the variability of the measurements. The boxplots indicate differences in both carbonation depth and measurement variability among the mixtures, highlighting the heterogeneous nature of carbonation penetration within the composites.

The effect of CF incorporation is therefore not directly related to bulk porosity alone. Although CF can facilitate carbonation through additional reactive phases and porous microstructural regions, the relationship between CF content and carbonation behaviour did not follow a linear trend under the investigated conditions. In particular, CF10-50 exhibited lower carbonation depth than CF5-50 and CF20-50, suggesting competing effects between pore refinement, moisture distribution, and CO₂ transport within the heterogeneous fibre-containing matrix. Intermediate CF contents may locally reduce pore connectivity, thereby limiting effective carbonation penetration in specific regions of the samples.

In contrast, lower CF contents, such as CF5-50, may limit carbonation penetration even at elevated temperature, as reflected by the larger uncarbonated core area observed in Fig. 4. This suggests that carbonation depth was primarily governed by temperature and, within a given temperature, influenced by CF content through a combination of reactive phase availability and local transport conditions.

Although the visual pH-indicator method has limitations in detecting partial carbonation, the carbonated samples exhibited a denser microstructure with fewer visible voids, consistent with the porosity reductions measured in Fig. 2a.

3.3. Thermogravimetric analysis and CO₂ storage

The TGA-DTG curves of samples cured at 20 °C and 50 °C are presented in Fig. 5. The first peak, located between 50 and 220 °C, corresponds to dehydration and decomposition of hydration products such as C–S–H, ettringite, and physically bound water [42]. The mass loss in this region tends to be higher for non-carbonated samples, particularly at 20 °C, while carbonated samples show reduced intensity due to partial consumption and decalcification of hydration products during carbonation. A minor peak around 300 °C may be associated with residual straw fibres that could not be removed. The peak near 460 °C corresponds to the decomposition of

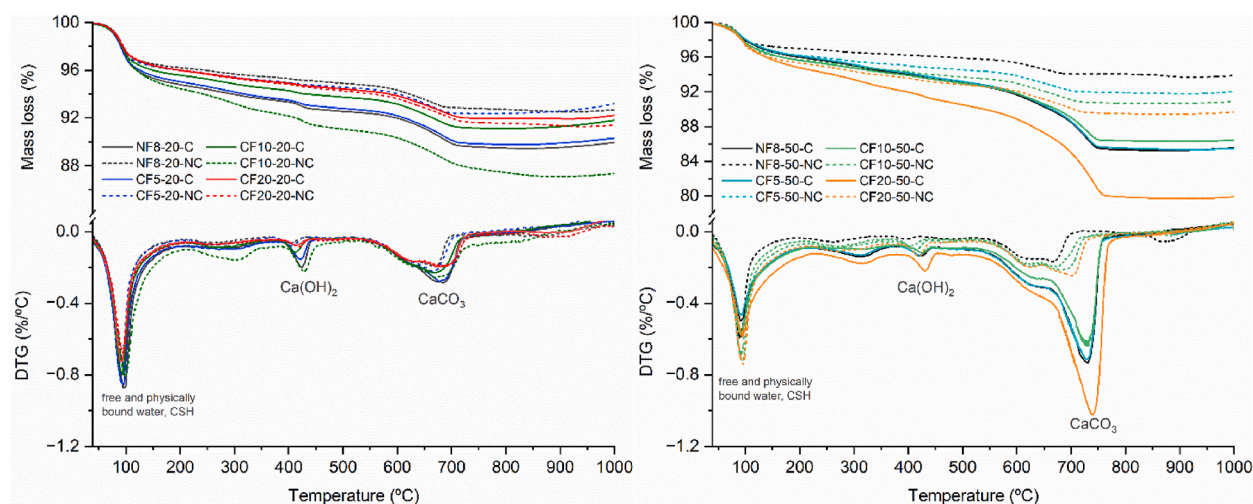


Fig. 5. DTG curves of cured samples at (a) 20 °C and (b) 50 °C.

portlandite ($\text{Ca}(\text{OH})_2$), one of the main phases reacting with CO_2 during carbonation. Lastly, the region around 550–900 °C is associated with the decomposition of calcium carbonate (CaCO_3), which is the main product of carbonation and forms through carbonation of calcium-bearing hydration products and residual reactive phases [42].

As expected, carbonation was more extensive in samples cured at 50 °C, as indicated by the stronger carbonate decomposition region in the DTG curves compared with specimens carbonated at 20 °C. At the same time, the reduced peaks associated with portlandite and hydration products suggest progressive conversion of calcium-bearing phases into carbonate products, primarily calcite.

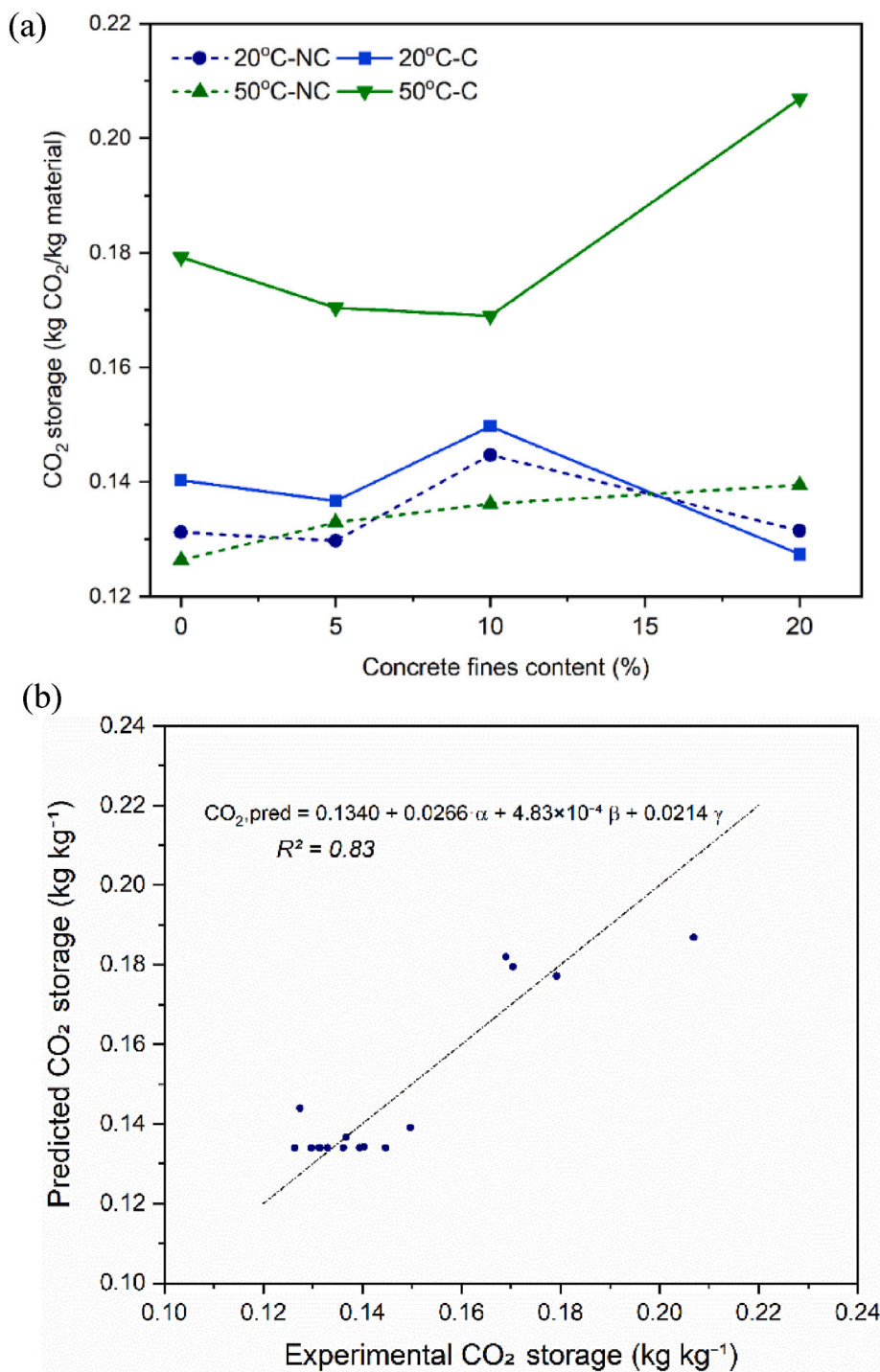


Fig. 6. (a) Total CO_2 storage of all mixtures as a function of concrete fines (CF) content, calculated as the sum of mineral CO_2 from TGA and biogenic CO_2 from wheat-straw fibres, (b) experimental and regression-predicted values used to evaluate model accuracy.

Although elevated temperature reduces CO₂ solubility in pore solution [27,36], increased CO₂ diffusion and faster reaction kinetics under partially saturated conditions appear to dominate, resulting in more extensive carbonation under the investigated curing conditions [36,43]. Elevated temperature may facilitate CO₂ transport by increasing permeability and diffusivity while reducing pore water content, thereby promoting carbonation reactions [27].

The stronger CaCO₃ peaks observed in samples cured at 50 °C indicate a higher degree of carbonation and are consistent with the higher mechanical strengths reported in previous sections. The influence of concrete fines became more evident under accelerated carbonation conditions. While differences between mixtures were relatively small at 20 °C, carbonated samples cured at 50 °C showed an overall increase in carbonate peak intensity, with CF20-50-C exhibiting the strongest response.

It is worth noting that the compatibility between the wheat straw fibres and the cementitious matrix may also be influenced by the incorporation of recycled concrete fines, particularly through changes in matrix alkalinity, porosity, and moisture distribution. Recycled concrete fines likely modify the pore structure due to their residual cement paste and higher porosity, while carbonation can promote CaCO₃ precipitation and matrix densification. Under carbonation curing, the alkalinity of the cement matrix tends to decrease, which may also reduce the exposure of fibres to highly alkaline conditions commonly associated with the degradation of lignocellulosic components [9,29]. However, fibre degradation was not directly evaluated in the present study, and further investigation of fibre–matrix interactions and long-term fibre stability under carbonation curing conditions is therefore recommended for future work.

Quantitative analysis of the mass loss in the region 600–800 °C, which is associated with CaCO₃ decomposition, confirmed the carbonation trends identified in the DTG profiles. The corresponding CaCO₃ contents and mineral CO₂ uptake calculated from TGA are summarised in Table A.3 (Appendix). The results show a clear influence of curing temperature on the extent of carbonation. Samples carbonated at 20 °C exhibited mineral CO₂ storage values between 0.017 and 0.040 kg CO₂/kg material, whereas carbonation at 50 °C substantially increased uptake to values between 0.059 and 0.097 kg CO₂/kg. It is worth noting that carbonation at 20 °C remained relatively limited, resulting in small differences in carbonate-related mass loss between carbonated and non-carbonated samples. Under these conditions, local variations in moisture distribution and accessibility of reactive phases within the heterogeneous fibre-containing matrix may have a greater influence on the calculated mineral CO₂ uptake. However, the experimental trends remain consistent, particularly the clear increase in carbonation extent observed at 50 °C. The highest mineral CO₂ storage was observed for CF20-50-C (0.097 kg CO₂/kg), highlighting the combined effect of elevated temperature and concrete fines on carbonation efficiency.

Based on the total CaO content measured by XRF (Table A.1, Appendix), the theoretical maximum mineral CO₂ uptake was estimated at approximately 0.43, 0.42, 0.41, and 0.39 kg CO₂ per kg binder for NF8, CF5, CF10, and CF20, respectively. This theoretical estimate represents an upper bound, as it assumes that all CaO present in the binder is reactive and can fully carbonate. In practice, this value is difficult to achieve because part of the calcium may already be present in stable phases such as calcite, as evidenced by the 9.6% calcite identified in the CF by QXRD (Table A.2), reducing the amount of Ca available for further carbonation.

The difference between theoretical and measured uptake is also influenced by internal transport limitations. Porous additions can enhance carbonation by improving moisture distribution and CO₂ diffusion; for example, biochar has been shown to increase carbonate formation and strength in CO₂-cured reactive MgO composites [22]. In parallel, carbonation-induced CaCO₃ precipitation contributes to pore filling and microstructural refinement in recycled cementitious materials [44], with similar effects reported in other carbonated systems such as slag-based binders [45].

In contrast, non-carbonated mixtures exhibited lower CO₂ contents, reflecting only pre-existing carbonates and the absence of carbonation-induced reactions. A minor deviation was observed for CF20-20-C, where the carbonated specimen showed slightly lower carbonate-related mass loss than the corresponding non-carbonated sample. This behaviour is likely associated with local microstructural heterogeneity introduced by fibre–matrix interactions, which may affect moisture distribution and accessibility of reactive phases during carbonation.

To assess the total CO₂ storage potential, mineral carbonation was combined with the biogenic CO₂ contribution from wheat-straw fibres. Biogenic CO₂ was calculated following EN 16449:2014 using:

$$P_{CO_2, bio} = m_{bio} \cdot cf \cdot \frac{44}{12} \left(1 + \frac{w}{100} \right) \quad (6)$$

where m_{bio} is the mass of biogenic material, is cf the carbon fraction, and w is the moisture content. Owing to the fixed straw dosage (8 wt%) used in all mixtures, the biogenic contribution remained constant at approximately 0.110 kg CO₂/kg material. The total CO₂ storage for each sample was then obtained as the sum of biogenic and mineral contributions ($CO_{2, storage} = CO_{2, bio} + CO_{2, TGA}$), thereby enabling direct comparison of overall CO₂ storage across mixtures, curing temperatures, and carbonation conditions, as shown in Fig. 6a.

Hence, Fig. 6a shows that total CO₂ storage increases primarily with curing temperature, while CF content influences the magnitude of the response, with the highest storage observed for CF20 under 50 °C carbonation. Carbonated samples at 20 °C also store more CO₂ than the non-carbonated samples, although the increase is more modest due to slower reaction kinetics. These chemical trends are consistent with the microstructural densification inferred from porosity and thermal conductivity results (Fig. 2). This behaviour also aligns with the deeper carbonation fronts observed in the phenolphthalein test (Fig. 4), indicating consistent chemical and visual evidence of enhanced carbonation.

At 20 °C, CF10 showed the highest mineral CO₂ storage (0.040 kg CO₂/kg), exceeding both NF8 (0.030 kg CO₂/kg) and CF20 (0.017 kg CO₂/kg), indicating that moderate CF replacement may provide a more favourable balance between reactive surface availability and pore accessibility than higher CF contents under limited carbonation conditions. Increasing the curing temperature

from 20 °C to 50 °C produced a substantial rise in mineral CO₂ storage across all mixtures; for example, NF8 increased from 0.030 to 0.069 kg CO₂/kg, while CF20 increased from 0.017 to 0.097 kg CO₂/kg, indicating that curing temperature strongly influences carbonation extent within the investigated conditions. Under 50 °C carbonation, the effect of CF content became more evident, with mineral CO₂ storage increasing from 0.069 kg CO₂/kg (NF8) to 0.097 kg CO₂/kg (CF20), suggesting that higher concrete fines content enhances carbonation efficiency when carbonation reactions are sufficiently activated at elevated temperature.

The highest mineral CO₂ value measured (0.097 kg CO₂/kg material for CF20-50-C) corresponds to approximately 97 kg CO₂ per ton of material. When combined with the biogenic contribution from wheat straw, total CO₂ storage reached up to approximately 0.207 kg CO₂/kg material, indicating the potential of combining bio-based constituents with carbonation processes to enhance carbon storage in cementitious composites. The higher total CO₂ storage observed for CF20-50-C coincides with the largest mechanical improvements (Fig. 3), indicating that enhanced carbonation not only increases carbon uptake but also contributes to matrix densification and strength development.

These CO₂-uptake levels are consistent with the wider accelerated-carbonation literature, where achieved uptake depends strongly on curing route, pore connectivity, and the progressive self-limiting effect of CaCO₃ precipitation on further CO₂ ingress [17]. The stronger response at 50 °C observed here is also consistent with the general effect of temperature on carbonation kinetics and transport. In addition, studies on carbonation-active binders indicate that porous constituents can support internal carbonation by improving moisture regulation and transport pathways [22]. Although the present system differs chemically, the results indicate that internal moisture distribution and transport influence the extent of carbonation.

To quantify the combined influence of curing conditions and mixture composition on CO₂ storage, a multiple linear regression model was developed using the experimental dataset presented in Fig. 6a. This regression is an empirical fit to the present dataset and is therefore intended for interpolation within the investigated parameter space (CF of 0–20 wt%, 8 wt% wheat straw fibres, 20/50 °C, 20% CO₂, 80% RH, curing age of 7 days), rather than for prediction outside these conditions.

The experimental total CO₂ storage values were obtained as the sum of mineral CO₂ quantified by TGA (Table A.3, Appendix) and the constant biogenic contribution from wheat-straw fibres. The model considers three independent variables: carbonation condition (α , coded as 0 for non-carbonated and 1 for carbonated samples), concrete fines content (β , CF replacement level, %), and curing temperature (γ , coded as –1 for 20 °C and +1 for 50 °C). The resulting empirical equation is expressed as:

$$CO_{2,pred} = 0.1340 + 0.0266 \alpha + 4.83 \times 10^{-4} \beta + 0.0214 \gamma \quad (7)$$

where $CO_{2,pred}$ represents the predicted total CO₂ storage (kg·kg^{–1}). Although carbonation conditions and curing temperature define the overall reaction regime, the regression results indicate that CF content modulates the effectiveness of carbonation, particularly under accelerated curing conditions. The contribution of CF to CO₂ storage is not attributed solely to carbonation reactivity. The investigated CF contained significant amounts of quartz and pre-existing calcite (Table A.2), together with limited reactive hydrated phases. Therefore, the observed influence of CF on CO₂ uptake likely reflects residual carbonation potential within adhered mortar combined with indirect physical effects related to particle packing and CO₂ transport within the composite, rather than CF acting primarily as a reactive calcium source.

Fig. 6b compares experimental and predicted values, showing reasonable agreement ($R^2 = 0.83$), indicating that the model captures the main trends of CO₂ storage within the investigated experimental domain. However, despite the relatively high correlation coefficient, the proposed relationship should be interpreted as a descriptive model within the investigated experimental range rather than a general predictive formulation. Additional datasets covering broader material compositions and curing conditions would be necessary for further validation and generalisation.

Bio-based construction materials can contribute to climate mitigation not only through emission reduction but also via biogenic carbon storage in long-lived applications [11]. This combined contribution from mineral carbonation and biogenic carbon storage highlights the dual carbon-capture potential of the developed composites, where both the cementitious matrix and the bio-based reinforcement contribute to overall CO₂ uptake.

4. Conclusion

This study shows that the combined use of wheat straw fibres, recycled concrete fines, and accelerated carbonation curing can produce cementitious composites with improved performance and CO₂ storage potential. Mixtures incorporating 8 wt% wheat straw and 0–20 wt% concrete fines replacing cement showed that, although the effect of concrete fines was not strictly linear, higher replacement levels generally led to greater microstructural densification, mechanical performance, and CO₂ uptake after carbonation.

Accelerated carbonation curing was the dominant factor governing microstructural development and overall performance. Carbonation, particularly at 50 °C, reduced porosity and increased bulk density, resulting in higher thermal conductivity (≈33–43%) and improved mechanical behaviour. The largest improvement was observed for CF20-50-C, where compressive strength increased from 2.84 MPa to 10.29 MPa after carbonation. In contrast, non-carbonated samples showed only minor differences between curing temperatures, indicating that temperature alone had a limited influence in the absence of CO₂. While carbonation increased thermal conductivity, the measured values remained within the range reported for comparable bio-based lightweight construction materials, indicating that matrix densification enhanced mechanical performance while maintaining thermal conductivity values comparable to those of similar bio-based construction systems.

Wheat straw fibres contributed to reduced density and improved thermal behaviour while also providing biogenic carbon storage and may have influenced internal moisture distribution and CO₂ transport during carbonation. Thermogravimetric analysis confirmed

progressive conversion of hydration products into carbonate phases, with mineral CO₂ uptake increasing with both curing temperature and concrete fines content. When combined with the biogenic contribution from wheat straw fibres, total CO₂ storage reached up to approximately 0.207 kg CO₂/kg material.

Overall, the results indicate that recycled concrete fines, bio-based fibres, and accelerated carbonation curing act together to improve microstructural, thermal, mechanical, and environmental performance. The empirical regression model relating total CO₂ storage to carbonation condition, curing temperature, and concrete fines content captured the main experimental trends ($R^2 = 0.83$), and can be used to describe the CO₂ storage trends observed within the investigated system.

Future work may focus on higher replacement levels of concrete fines, alternative locally available natural fibres, and the long-term behaviour of these composites, particularly the stability of fibre–matrix interactions and carbon storage under varying moisture and temperature conditions. Further investigation of carbonation behaviour over extended curing periods would improve understanding of transport limitations and carbonation progression in fibre-containing systems. Additional validation across a broader range of material compositions and curing conditions would also help establish the wider applicability of the proposed CO₂ storage relationship.

CRediT authorship contribution statement

Leila Nobrega Sousa: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Validation, Visualization, Writing – original draft. **Oliwia Korecka:** Data curation, Investigation, Writing – original draft. **Koh Chuen Hon:** Conceptualization, Data curation, Methodology, Writing – original draft. **Katrin Schollbach:** Supervision, Validation, Writing – review & editing. **H.J.H. Brouwers:** Funding acquisition, Supervision, Writing – review & editing.

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.jobbe.2026.116535>.

Data availability

Data will be made available on request.

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