

On the influence of mix design and pre-conditioning on the CO₂-uptake of cementitious materials

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Abstract. The cement and concrete sectors are known for their significant contribution to CO₂ emissions. Carbonation curing of concrete precast elements in a CO₂-rich atmosphere is a sustainable approach to reducing the carbon footprint of these industries while simultaneously enhancing the mechanical properties of cement-based materials. This study systematically investigates the influence of mix design and pre-conditioning on CO₂ uptake efficiency and the mechanical performance of mortars. The findings highlight that both factors play a crucial role in optimizing carbonation efficiency. Notably, the study demonstrates that maximizing CO₂ sequestration of 3 wt.% of the mortar can be achieved through controlled pre-curing while mechanical properties are preserved or even improved. This study confirms that carbonation curing can be seamlessly integrated into the precast concrete manufacturing process without requiring additional processing steps. This breakthrough paves the way for practical implementation in industrial settings, offering the dual benefit of carbon reduction and material performance enhancement.

1 Introduction

The cement industry stands as a significant contributor to global CO₂ emissions, accounting for approximately 7-8% of the world's total anthropogenic CO₂ emissions [1]. These emissions result from the calcination of calcium carbonate, the combustion of fossil fuels in high-temperature processing units, and electricity usage for facility operations. Therefore, cement producers constantly seek solutions to reduce the environmental impact of their production, and cement plants must achieve carbon neutrality by 2050, in line with the 13th United Nations Sustainable Development Goal [1,2].

Carbonation curing of precast concrete under a CO₂-rich atmosphere is an approach to decreasing carbon footprint that has attracted the attention of the cement and concrete industries. The approach of CO₂ curing of precast concrete is reported as a promising method for permanently storing CO₂ in concrete [3,4]. To date, carbonation curing of concrete under a CO₂-rich atmosphere is recommended for non-reinforced precast concrete, as CO₂-curing lowers the pH from ~13 to below 10, which weakens the passivation layer on steel bars, making them susceptible to corrosion [5]. In this approach, after mixing and casting, CO₂ is introduced to the cement-based material to accelerate the curing phase. It allows for a rapid strength gain and has been shown to improve product density and durability [6,7]. The fast strength gain also permits the precast industries to ensure a fast production turnover [8]. The concrete mix design,

e.g., water-to-binder ratio (w/b) and aggregate particle size, are crucial factors to the success of CO₂ curing [9]. The carbonation curing process for precast concrete typically involves three stages: pre-conditioning, carbonation, and post-conditioning [10].

The pre-conditioning stage aims to reduce pore water content to optimize CO₂ diffusion through the concrete matrix during the carbonation [11]. This step is usually performed at 20–25 °C and 40–60% relative humidity (RH) [11]. Additionally, pre-conditioning time is also reported as an important factor for the CO₂ curing of cement-based materials. As such, a wide range of curing conditions (wet, in mold, off mold, open air, and steam curing) and various curing durations from 3 to 72 h have been reported in the literature [5,12]. To the best of the authors' knowledge, the effect of pre-conditioning on the CO₂ uptake of pre-cast concrete has rarely been reported in the literature. RILEM TC 281-CCC [13] critically reviews 17 standards for carbonation resistance testing, emphasizing how differences in pre-conditioning (e.g., curing, humidity, sample drying) alter carbonation depths and CO₂ absorption rates. Based on this review, a shorter pre-conditioning time (e.g., 14 days) may underestimate carbonation resistance for blended cements, as internal moisture redistribution continues beyond this timeframe [13]. Another RILEM round-robin study involving 22 labs revealed large discrepancies in carbonation rate for CEM I, II/B-V, and III/B due to inconsistent pre-conditioning (e.g., drying conditions) [14]. Based on this report, the variation of preconditioning between

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carbonation tests as an effective parameter makes it difficult to attribute the change in carbonation depth to the applied duration of CO₂-curing alone [14]. Therefore, the preconditioning step needs to be adjusted based on specific mix design and pre-cast production conditions to maximize the CO₂ uptake of pre-cast concrete. In the carbonation stage, with the objective of hydration acceleration and early-age strength development, precast concrete is exposed to CO₂ under controlled conditions [10]. CO₂ concentration, curing time and temperature, RH, and pressure are crucial parameters that affect the CO₂ uptake of cement-based materials [10,15]. Literature reported on 25-100% CO₂ concentration, 4-96 h of curing time, 23-95 °C of curing temperature, 30-90% RH, and 0.1-13.9 bar of CO₂ pressure [16,17] to maximize the CO₂ uptake by concrete. It is worth mentioning that, based on the literature, 50-60% RH and room temperature are optimum conditions, which maximize carbonation efficiency [10,18]. In the post-conditioning stage, concrete is stored under controlled environment conditions at 20-25 °C and RH of 40-95%, which allows for further hydration of unreacted cement particles by consuming the remaining free water of concrete, resulting in the stabilization of concrete properties [62]. Nevertheless, most experimental studies kept the carbonated samples in a humidity-controlled chamber at 25 °C and 95% RH for 27 days [62–64].

This work aims to contribute to the decarbonization of the precast concrete industry in Portugal by studying the possibility of integrating a CO₂-curing step into the production process. The primary goal is to maximize CO₂ uptake in cementitious materials while minimizing additional CO₂ emissions, particularly by avoiding high-temperature or high-pressure carbonation. To do so, cement pastes and mortars were formulated using two cement types—CEM I 52.5 R and CEM II/B-L 32.5N. Unlike most existing studies, this research focuses on the impact of mix design, w/b ratio, and pre-conditioning steps (such as drying and humidity homogenization) on CO₂ uptake and mechanical properties of mortar. Two sets of cement pastes and six sets of mortars were prepared to study the mix design, w/b ratio, and pre-conditioning parameters. The carbonation progress in cement pastes and mortar was evaluated using a phenolphthalein indicator. Thermogravimetric analysis (TGA) was used to identify and quantify the CO₂ uptake during the carbonation process. The mechanical properties of mortars were assessed to study the effect of pre-conditioning and CO₂-curing on the flexural and compressive strength. The findings provide new insights into the potential of integration of the CO₂-curing step into the production process in the precast concrete industry, emphasizing mix design over carbonation conditions.

2 Materials and Methods

2.1 Materials

Silica sand, a combination of calibrated siliceous sands from Areipor - Areias Portuguesas, composed of over

98% silica was used as a natural aggregate. CEM I 52.5 R (CEM I) with clinker $\geq 95\%$, minor constituents $\leq 5\%$, and calcium sulfate as a setting regulator, and CEM II/B-L 32.5 N (CEM II) with $65\% \leq \text{clinker} \leq 79\%$, $21\% \leq \text{limestone} \leq 35\%$, minor constituents $\leq 5\%$ and calcium sulfate as setting regulator, both produced by CIMPOR, Portugal, were used as binders. CO₂ gas from Air Liquide with 99.9% purity was used for the carbonation of samples.

2.2 Cement Paste and Mortar Preparation

Two cement pastes were prepared using CEM I and CEM II with the w/b = 0.35. The samples were de-moulded after 48 hours of curing in a closed plastic bag at room temperature. Further, samples were subjected to CO₂ curing at 60% RH, 23 °C, and 25% CO₂ concentration for 72 h in a ARALAB FitoClima 300 EP carbonation chamber.

Mortar samples were prepared with CEM I and CEM II with various w/b ratios, cement-to-aggregate (c/ag) ratios, and different pre-conditioning, including 48 h of mold curing, 48 h mold curing followed by 24 h drying at 60 °C, and introducing humidity homogenization steps (wrapping in plastic bags and left for 3 days) after curing and drying.

The methodology used for mixing the ingredients was based on European Standard EN 1015-2 [19]. Prismatic specimens of 40 × 40 × 160 mm were produced based on European Standard EN 1015-11 [19]. The specimens were cured in moulds for 48 h at 20 ± 2°C and an RH of 90 ± 5%.

Further, mortar samples were subjected to CO₂ curing at 60% RH, 23 °C, and 25% CO₂ concentration for 24 h in the same carbonation chamber mentioned previously.

2.3 Techniques

The phenolphthalein indicator (a solution of 1 wt.% phenolphthalein in ethanol) was used on a freshly broken surface of cement paste or mortar to assess the carbonation depth. After applying the indicator, carbonation depth was measured using a calliper, and an average of four measurements was considered. Samples for mechanical properties evaluation, namely flexural and compressive strength, were subsequently cured in controlled environment conditions (room temperature and 60% RH) until being tested at 7 and 28 days.

The flexural and compressive strength was measured in triplicate for each set of mortar samples, and an average value was considered.

TGA was performed to assess the carbonation potential of cement pastes and mortars. The mass loss of the samples was recorded while samples were heated continuously from 25 to 1000 °C at a uniform rate of 10 °C.min⁻¹ in an N₂ atmosphere (3 L/h). The temperature range from 400–500 °C, which corresponds to the dehydroxylation of Ca(OH)₂ [20] allows the calculation of the free Ca(OH)₂ content of the sample (Eq.1), which can be used to estimate the carbonation potential of a cementitious material. The temperature range from 500–

900 °C corresponds to the decarbonisation of CaCO_3 [20], allowing the calculation of CaCO_3 content (Eq.2) and CO_2 uptake of the samples (Eq.3). The quantity of CaCO_3 formed during the carbonation and the remaining Ca(OH)_2 was calculated using Eq.1 and 2.

$$M_{CH} = \Delta M_{400-500^\circ\text{C}} \times \frac{M_{CH}}{M_{H_2O} \times (M_{\text{initial}})} \quad \text{Eq.1}$$

$$M_{CaCO_3} = \Delta M_{500-900^\circ\text{C}} \times \frac{M_{CaCO_3}}{M_{CO_2} \times (M_{\text{initial}})} \quad \text{Eq.2}$$

Table 1 Composition and pre-conditioning states of mortars prepared in this work.

Cement type	c/agg	w/b	Pre-conditioning
CEM I	1:4	1	48 h mould curing
CEM II	1:4	1	48 h mould curing
CEM II	1:4	0.5	48 h mould curing
CEM II	1:2	0.5	48 h mould curing
CEM II	1:4	0.5	48 h mould curing + 24 h drying at 60°C
CEM II	1:4	0.5	48 h mould curing + 24 h drying at 60°C + 72 h homogenization

Additionally, based on Eq.3, the maximum CO_2 uptake can be determined by the difference between the CaCO_3 content before and after carbonation.

$$\text{CO}_2 \text{ Uptake (\%)} = \Delta \text{CaCO}_3 \times \frac{M_{CO_2}}{M_{CaCO_3}} \quad \text{Eq.3}$$

Where ΔCaCO_3 is the difference between CaCO_3 before and after carbonation, M_{CO_2} and M_{CaCO_3} are the molar masses of CO_2 and CaCO_3 , respectively.

3 Results and Discussions

3.1 Carbonation Progress Using Phenolphthalein Indicator

Initially, cement paste was used to study the carbonation progress and CO_2 uptake during the accelerated carbonation process. To do so, two sets of samples were prepared, using CEM I and CEM II with w/b = 0.35, as discussed in more detail previously. Carbonation curing was performed for 72 h at 60% RH, 23 °C, and 25% CO_2 concentration. To evaluate the carbonation depth, samples were tested by phenolphthalein indicator at 0, 5, 8, 12, 24, 48, and 72 h of carbonation curing. Figure 1 shows the results of the carbonation depth for cement paste prepared using CEM I (PC I) and CEM II (PC II). At 0 h, samples were tested by phenolphthalein indicator to ensure no significant carbonation had occurred during the preparation process, and for both sets of samples, carbonation was not detected before CO_2 curing (Figure 1a and b). For PC I, no carbonation was detectable until 48 h of carbonation (Figure 1a). Solely a carbonation depth of 2.4 mm was observed at the end of curing time (72 h). In contrast, the carbonation depth in PC II shows an increasing trend during the carbonation, being 3, 6, and 9.1 mm at 12, 24, and 72 h of carbonation, respectively (Figure 1b).

Where $\Delta M_{400-500^\circ\text{C}}$ and $\Delta M_{500-900^\circ\text{C}}$ are the weight lost at the indicated temperature range regarding the Ca(OH)_2 and CaCO_3 , respectively. $M_{\text{Ca(OH)}_2} = 74.093$, $M_{H_2O} = 18.015$, $M_{CaCO_3} = 100.086$, and $M_{CO_2} = 44.01$, and M_{initial} is the initial weight of the sample.

The higher carbonation depth of PC II compared to PC I can be attributed to the low Ca/Si ratio of C–S–H, whose carbonation leads to shrinkage [21]. As a result, coarser pores are formed in the carbonated zone of PC II compared to PC I, which facilitates the further diffusion of CO_2 , i.e., further carbonation [21].

Additionally, the presence of SCM in CEM II results in a different phase assemblage of PC II, e.g., fewer calcium-bearing phases form and are available for carbonation. As a result, less pore refinement takes place during the carbonation, facilitating CO_2 diffusion [21]. However, the low level of porosity and compact structure of both cement pastes (PC I and PC II) hinder the diffusion of CO_2 and the respective carbonation degrees.

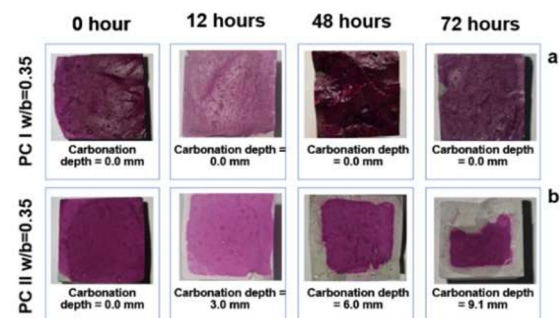


Figure 1 - Carbonation progress in a) PC I and b) PC II during the 72 h of carbonation in 23 °C, 60% RH, and 25% CO_2 concentration.

Therefore, to follow the objective of this work, mortar samples with a specific mix design, which offer more porous structures, were considered more appropriate to be tested.

Therefore, the study of CO_2 curing was performed on the four sets of mortar samples with different mix designs, varying in cement type (CEM I and CEM II), w/b ratio (1 and 0.5), and cement to sand volumetric ratio (volumetric ratio) (1:4 and 1:2) as shown in Table 1. The same

carbonation conditions used for the cement pastes were applied, but the CO₂ curing time was reduced to 24 h.

First, mortars with a volumetric ratio of 1:4 and w/b = 1.0 were prepared using CEM I and CEM II (Figure 2a and b) to correlate with previous results (PC I and PC II) and evaluate the effect of cement type on the carbonation progress of mortars. Table 2 shows the carbonation depth of mortars tested by phenolphthalein indicator at 0, 5, 12, and 24 h of carbonation curing. By the end of the curing time (24 h), CEM I mortar reached a maximum carbonation depth of 12.9, while CEM II mortar achieved a total carbonation (T.C.). Comparing these results with the results obtained for cement pastes (Figure 1a and b), the mortar samples exhibited higher carbonation depth, as the incorporation of natural aggregates in mortars resulted in a more porous structure, facilitating CO₂ diffusion and enhancing the carbonation reaction [22]. As discussed previously, the higher carbonation depth in CEM II mortars can be attributed to the presence of SCMs, which results in less pore refinement during carbonation, creating a structure that further facilitates CO₂ diffusion compared to CEM I mortars [23,24]. Further, to study the effect of the w/b ratio on the carbon uptake of the mortar, a set of mortars was prepared using CEM II with a volumetric ratio of 1:4 and a w/b = 0.5 (Table 1).

It is worth mentioning that as samples prepared with CEM II showed higher carbonation depth, CEM II was used to study the effect of other parameters in this work. Figure 2c and Table 2 show the results of the carbonation depth for these mortars.

Comparing these results with previous carbonation depth measured for CEM II with a volumetric ratio of 1:4 and a w/b = 1 (Figure 2b), it can be concluded that a 50% reduction of the w/b ratio resulted in a reduction of carbonation depth of the same order of magnitude ($\approx 50\%$) by the end of the carbonation time (24 h).

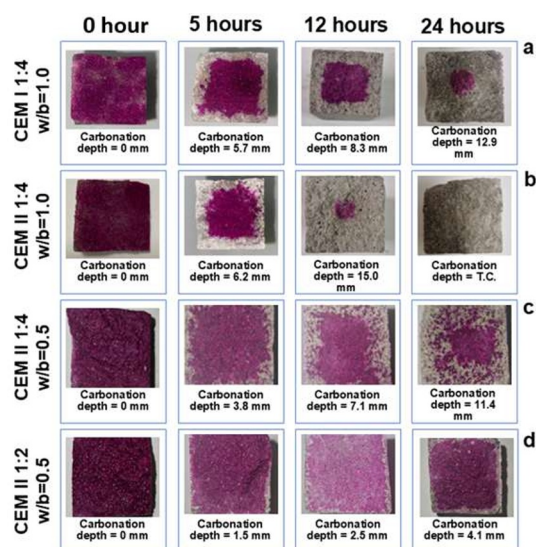


Figure 2 - Carbonation progress during the 24 h carbonation at 60% RH, 23 °C and 25% CO₂ concentration of mortar samples prepared with a) CEM I at w/b = 1 and volumetric ratio = 1:4, b) CEM II at w/b = 1 and volumetric ratio = 1:4, c) CEM II at w/b = 0.5 and volumetric ratio = 1:4, and d) CEM II at w/b = 0.5 and volumetric = 1:2.

In fact, the reduction of the mortar's w/b ratio reduces its porosity, which attenuates the CO₂ diffusion, promoting a less efficient CO₂ uptake.

To study the effect of volumetric ratio on CO₂ uptake of mortar, a set of mortars was prepared using CEM II with a w/b = 0.5 and a volumetric ratio of 1:2. Figure 2d and Table 2 show the carbonation depth of these samples. In fact, this mix design yielded a low-porosity mortar, which restricted CO₂ diffusion into the mortar matrix [25]. Comparing with previous results (Figure 2b and c), it can be concluded that the combined effect of lowering the w/b and reducing the volumetric ratio results in reducing the CO₂ diffusion due to the reduced porosity, resulting in significant attenuation in carbonation progress.

The study demonstrates that mortar mix design critically influences CO₂ penetration during curing. Based on these results, a CEM II 1:4 mortar with w/b = 0.5 was selected for further process optimization, as the partial carbonation observed in this mortar enables the possibility to explore and implement changes in the curing process to enhance CO₂ uptake efficiency.

To study the effect of drying on the CO₂ uptake of mortar, a set of mortars was prepared with CEM II 1:4 w/b=0.5. After 2 days of curing in plastic bags, samples were dried for 24 h at 60 °C. Afterwards, samples were subjected to carbonation at 60% RH, 23 °C, and 25% CO₂ concentration. Figure 3b illustrates the effects of the drying process on the carbonation depth of mortar samples. To facilitate the comparison, the previous results obtained without drying were added to Figure 3 (Figure 3a). Table 3 shows the results of the carbonation depth for these mortars. For dried samples, the carbonation depth was substantially lower than that of samples without drying (Figure 3a and Table 3). From this time onward, it was not possible to measure the carbonation depth due to an irregular carbonation progress of the mortars; the carbonated zone (no color by phenolphthalein) was detected at the internal section of the sample while the outer layer was still pink (Figure 3b). This irregular carbonation may be attributed to fast drying of the outer layer resulting in a non-homogeneity in the humidity distribution along the sample cross section. Therefore, a new process was tested by incorporating a homogenization step to redistribute the humidity after drying and prior to the CO₂ curing. Figure 3c and Table 3 show the carbonation depth of these mortar samples. At 5 h, the average carbonation depth was 1.2 mm, showing a slight increase in comparison to previous samples (Figure 3b). However, the irregular carbonation was still detected at 12 h, making the carbonation depth measurement impossible. Interestingly, by the end of carbonation (24 h), the sample was totally carbonated. Comparing the three processes, CO₂ curing (Figure 3a) led to the most rapid initial carbonation. The incorporation of a drying step appeared to disrupt carbonation, while the addition of a homogenization step resulted in the complete carbonation of samples after 24 h of CO₂-curing. This suggests that both humidity level and homogeneous distribution of humidity are crucial for optimal carbonation and CO₂ sequestration. It is worth noting that the humidity content of samples studied in this work was

measured to be 7, 2.3, and 0.5% before drying and after 8 and 24 h of drying at 60 °C, respectively, confirming the extremely low humidity of the sample after 24 h of drying. Further investigation was carried out using TGA to understand the chemical changes during the carbonation for these three campaigns.

Table 2 – Carbonation progress during the 24 h CO₂ curing of mortar samples with different mix designs.

Mortar mix	Carbonation depth (mm)			
	0 h	5 h	12 h	24 h
CEM I 1:4 w/b=1.0	0.0	5.7	8.3	12.5
CEM II 1:4 w/b=1.0	0.0	6.2	15.0	T.C.
CEM II 1:2 w/b=0.5	0.0	1.5	2.5	4.1
CEM II 1:4 w/b=0.5	0.0	3.8	7.1	11.4

3.2 TGA of Carbonation Progress

TGA was performed to assess the carbonation degree and CO₂ uptake of PC II (the sample showed a higher carbonation depth by phenolphthalein indicator) before and after CO₂ curing. To do so, TGA was carried out for PC II samples before and after 12 and 72 h of CO₂ curing. The quantity of Ca(OH)₂ and CaCO₃ in PC II before and after carbonation was calculated using Eq.1 and 2. Figure 4a shows the TGA and DTG curves of PC II before and after 12 and 72h carbonation. Before carbonation, the quantity of CaCO₃ and Ca(OH)₂ were calculated to be 30 and 11.1 wt%, respectively (Figure 4b). Upon the carbonation, the quantity of CaCO₃ shows an increasing trend, attaining the value of 34.1 and 46.3 wt% at 12h and 72h of carbonation, respectively. This increasing trend was accompanied by a decrease in the quantity of Ca(OH)₂, as was expected. The Ca(OH)₂ quantity was reduced to 10.1 after 12 h of carbonation and attained a value of 6 wt% by the end of 72 h (Figure 4b).

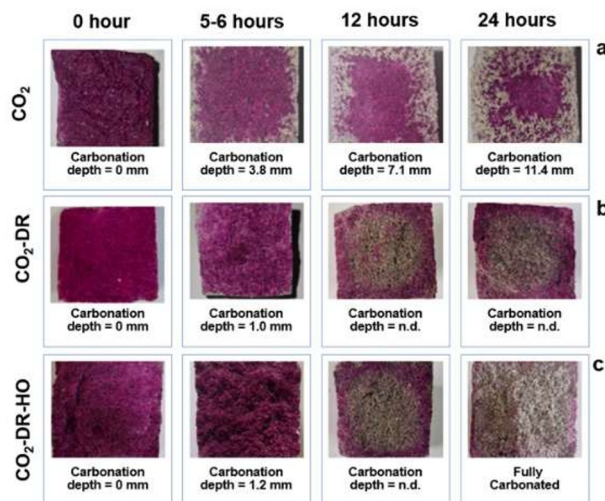


Figure 3 – Carbonation progress in mortar samples prepared with CEM II at w/b = 0.5 and volumetric ratio = 1:4, using different CO₂ curing process a) conventional CO₂ curing, b) CO₂ curing with drying (CO₂-DR), c) CO₂ curing with drying and homogenization (CO₂-DR-HO).

Table 3 - Carbonation progress during the 24 h carbonation

Pre-conditioning	Carbonation depth (mm)			
	0 h	5 h	12 h	24 h
CO ₂	0.0	3.8	7.1	11.4
CO ₂ -DR	0.0	1.0	n.d.	n.d.
CO ₂ -DR-HO	0.0	1.2	n.d.	T.C.

The CO₂ uptake of PC II was calculated based on Eq.3. As a result, CO₂ uptake was calculated to be 1.8 wt% at 12 h of carbonation. Further CO₂ curing until 72 h results in a CO₂ uptake of 7.2 wt% by the PC II sample. The results obtained confirmed that a longer CO₂ curing leads to higher CO₂ uptake of cement paste as CO₂ can diffuse further into the cement paste matrix, dissolve in pore solution, and form calcium carbonate in contact with Ca²⁺ ions present in the pore solution [26]. The TGA results also reveal that by the end of the carbonation process (72 h), there was still some residual Ca(OH)₂ (6 wt%) in the carbonated PC II (Figure 4b). This indicates that PC II still has substantial capacity for further CO₂ uptake. This also is in line with the result obtained by testing carbonated samples with the phenolphthalein indicator (Figure 1b), which showed, at the end of 72 h of carbonation, CO₂ diffused only 9 mm (≈ half of the full depth). This phenomenon is justified by the formation of a thin layer of CaCO₃ around the Ca(OH)₂ particles, which decelerate both CO₂ diffusion and Ca(OH)₂ dissolution, two essential parameters for carbonation progress [27]. These results also reconfirmed that cement paste with low porosity is not the best solution to estimate the CO₂ uptake of cementitious materials.

TGA was also performed for mortars prepared without a drying process and with drying and homogenization steps being performed before enforced CO₂ carbonation. Therefore, TGA was performed to assess the impact of the drying and homogenization process in the carbonation degree and CO₂ uptake of CEM II 1:4 w/b = 0.5 mortars before and after CO₂ curing. Figure 6a1 shows the TGA and DTG curves of mortars, which were conventionally cured for 48 h without any drying process involved.

Before CO₂ curing, the quantity of CaCO₃ and Ca(OH)₂ were calculated to be 22.9 and 7.1 wt%, respectively (Figure 5a1). After 24 h of carbonation, the quantity of CaCO₃ increased to 29.7 wt%, which was accompanied by a decrease of Ca(OH)₂ content to the value of 2 wt% (Figure 5a2). Correlating these results with the carbonation depth, Figure 3a indicates that the mortar still has some carbonation potential due to the presence of residual Ca(OH)₂ by the end of CO₂-curing, mainly in the centre of the sample.

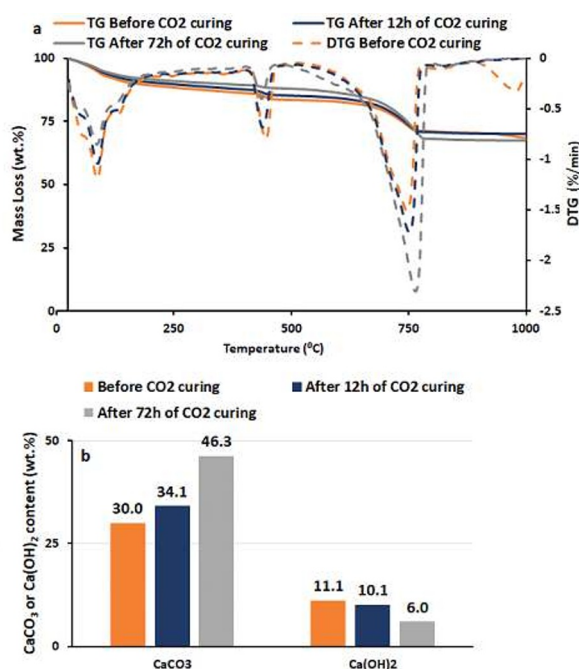


Figure 4- a) TGA and DTG curves of PC II before and after 12 and 72h carbonation, b) Evolution of CaCO₃ and Ca(OH)₂ content during the carbonation compared with a sample before carbonation.

Nevertheless, total carbonation did not happen due to the formation of CaCO₃ that densified the matrix, clogged the pores, and reduced further CO₂ diffusion [20,28,29]. Based on TGA, a CO₂ uptake of 3 wt% was calculated for these mortars after 24 h of CO₂ curing.

Later, a drying step was included in the curing process. Figure 5b1 shows the TGA and DTG curves of these mortars before and after CO₂-curing. Before CO₂ curing, CaCO₃ and Ca(OH)₂ contents were calculated to be 9.0 wt% and 1.9 wt%, respectively (Figure 5b2). These values were substantially lower than the same values for mortars without the drying step. Probably, over-drying caused a water starvation which inhibits the hydration process [6]. Therefore, even before carbonation, the quantity of Ca(OH)₂ was ≈73 wt% lower than in mortars prepared without the drying step. After carbonation, the quantity of CaCO₃ increased to a value of 13.0 wt%. This increase was accompanied by a reduction in Ca(OH)₂ content,

which decreased to 1.0 wt% by the end of carbonation (Figure 5b2).

The incorporation of the drying step led to a maximum CO₂ uptake of 2.4 wt% achieved by the end of carbonation. Thus, the drying step negatively affected the formation of hydration products and CO₂ uptake of mortar samples. As mentioned previously, 24 h of drying led to a global humidity level of 0.5%, which is too low for the hydration and carbonation of prepared mortars [30,31].

A homogenization step was added to the previous campaign to reduce the effect of the humidity gradient along the sample cross section. Figure 5c1 shows the TGA and DTG curves of mortar samples before and after carbonation, while both drying and homogenization steps were included in the curing process. Before carbonation, the quantities of CaCO₃ and Ca(OH)₂ were calculated to be 9.9 wt% and 3.0 wt%, respectively. The quantity of Ca(OH)₂ shows approximately a 58 wt% increase in comparison with the previous campaign, where only the drying step was included. Probably homogenization or extra conventional curing days (samples wrapped in plastic bags and left for 3 days) results in slight progress in the hydration process, i.e., formation of more Ca(OH)₂. After 24 h of carbonation, the quantity of CaCO₃ increased, reaching the value of 16.2%. This increase was mainly due to the carbonation of Ca(OH)₂ that reduced from 3.0 to 1.2 wt% over 24 h of curing (Figure 5c2). The incorporation of both drying and homogenization steps resulted in a maximum CO₂ uptake of 2.8 wt%, which was slightly higher than the value calculated for the process with drying (2.4 wt%). However, despite this improvement, the incorporation of drying still led to lower overall CO₂ sequestration values compared to processes without drying. This reduction in CO₂ uptake can be attributed to the negative impact of drying on hydration product formation. The lower content of hydrated products in dried mortars before curing indicates that the drying process interferes with the hydration reactions [6]. In fact, while the addition of homogenization slightly improved the carbonation progress (measured by phenolphthalein indicator (Figure 3c), it still resulted in lower CO₂ uptake than processes without drying. Furthermore, the initial process without drying is not only more efficient in terms of CO₂ capture but also simpler to implement on an industrial scale.

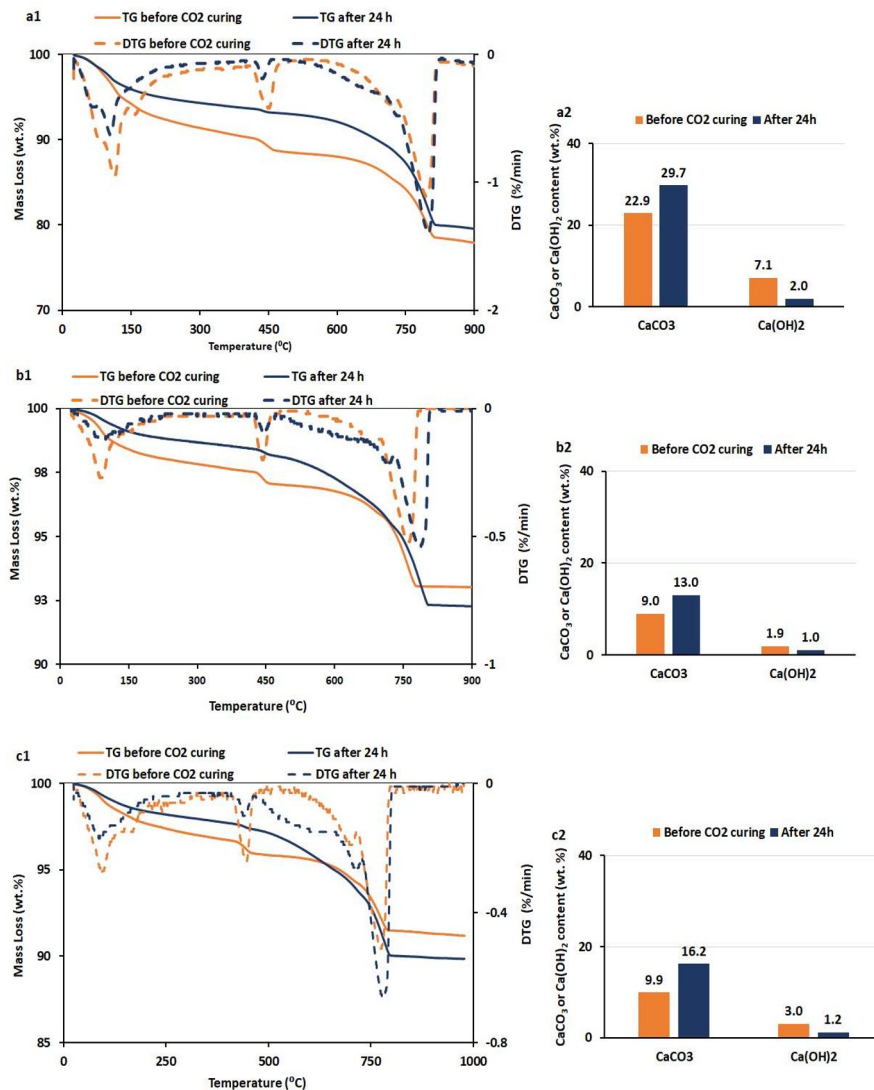


Figure 5 - TGA and DTG curves before and after carbonation and evolution of CaCO₃ and Ca(OH)₂ quantity during the carbonation for mortars prepared with CEM II 1:4 w/b = 0.5: a1 and a2) only CO₂ curing performed, b1 and b2) drying performed before CO₂ curing, c1 and c2) both drying and humidity homogenization were performed before CO₂ curing.

3.3 Effect of Mix Design and Carbonation on the Mechanical Properties

The effect of CO₂ curing on the mechanical properties of mortars developed in this work was explored. All samples were examined in triplicate, and the average value was considered. Figure 6a and Table 4 show the 7-day and 28-day flexural and compressive strength of mortars prepared using CEM I with a volumetric ratio of 1:4 and w/b = 1 after conventional curing (without enforced carbonation) and after 24 h of carbonation curing at 23 °C, 60% RH, and 25% CO₂ concentration. At 7 days, the CO₂-cured mortar outperformed the conventionally cured mortar, and the flexural strength was 21.6% higher than of the conventionally cured samples. This initial difference indicates that early carbonation facilitates rapid strength development, likely due to the fast formation of CaCO₃, which densifies the microstructure and enhances early-age mechanical properties [32,33]. At 28 days, the flexural strength of conventionally cured mortar shows an increase in comparison to the same value at 7 days. On the

contrary, the 28-day flexural strength of CO₂-cured mortar shows a slight decrease in comparison to its 7-day strength. The reduction in flexural strength for the CO₂-cured sample could be attributed to competition between the carbonation and hydration processes. While carbonation forms CaCO₃, which densifies the matrix, prolonged CO₂-curing can disrupt hydration processes and amplify pore structure changes, leading to a positive net porosity, which leads to a decrease in mechanical properties [22]. It is worth mentioning that in blended cement (e.g., CEM II), due to the presence of low Ca/Si of C-S-H, such a reduction in mechanical properties is more pronounced [21]. The compressive strength of these mortars (CEM I with a volumetric ratio of 1:4 and w/b = 1), which is depicted in Figure 6a2 and shown in Table 4, shows a slightly different trend. The compressive strength of both CO₂-cured and conventionally cured samples at 7 days has the same value, with both showing an increasing trend at 28 days. The lower compressive strength of CO₂-cured samples can be explained by competition between carbonation-induced densification and increased porosity,

as discussed previously. Nevertheless, the effect of the presence of possible defects and microcracks (formed during the enforced carbonation) is less pronounced in compressive strength than in flexural strength. In fact, compressive loading primarily closes existing microcracks rather than propagating them [34], and this justifies the slightly higher compressive strength of mortar at 28 days after CO₂-curing.

Figure 6b1 and Table 4 show the flexural strength of mortar prepared using CEM II with a volumetric ratio of 1:4 and w/b = 1. The flexural strength shows the same trend as previous samples (Figure 6a1), but overall flexural strength values are lower for mortar prepared with CEM II. The compressive strength of these samples is shown in Figure 6b2 and Table 4, following the same trend as the flexural strength of these samples. Comparing CEM I mortar mechanical properties (Figures 6a1 and a2) with CEM II mortars (Figures 6b1 and b2) reveals that the mechanical properties of CEM II mortars are significantly lower than those of CEM I mortars due to the reduced quantity of clinker. As mentioned previously, in blended cement (e.g., CEM II), due to the presence of low Ca/Si of C-S-H, a reduction in mechanical properties is more pronounced when compared with CEM I [7].

To investigate the effect of the reduction of the w/b ratio, a set of mortars was prepared using CEM II with a volumetric ratio of 1:4 and w/b = 0.5. The same conventional curing and CO₂-curing were applied. Figures 6c1, c2, and Table 4 show the flexural and compressive strength of these mortar samples, respectively. The flexural strength of conventionally cured samples at 7 days was slightly higher than that of the CO₂-cured samples (Figure 6c1 and Table 4). However, as curing time elapses (28 days), both conventionally and CO₂-cured samples attain almost the same values of flexural strength. Additionally, the overall flexural strength values are substantially higher than those of mortars prepared with w/b = 1 (Figure 6b1 and b2). Probably, reduced porosity due to the reduction of the w/b

ratio results in a more efficient pore refinement during the carbonation. Figure 6c2 shows the compressive strength of mortars with w/b = 0.5 after conventional and CO₂-curing at 7 days and 28 days. At 7 days, CO₂-cured samples show substantially higher compressive strength than conventionally cured ones. After 28 days of curing, the conventionally cured samples showed an increasing trend and outperformed the CO₂-cured samples. In contrast, the CO₂-cured samples show a slight reduction at 28 days. Comparing compressive strength at 28 days with the same values of the previous campaign (w/b = 1), a substantial increase of 323% and 347% was observed for conventional and CO₂-cured samples, respectively. The study reveals that even though higher w/b is in favor of higher carbonation depth (Figures 1b and c), it negatively affects the mechanical properties of mortar. Nevertheless, mortar samples with w/b = 0.5 still have a capacity of CO₂ uptake as the carbonation progressed only to half the depth of samples after 24 h.

Further, the effect of changing the volumetric ratio was studied by preparing a set of mortars using CEM II with a volumetric ratio of 1:2 and w/b = 0.5. The flexural strength of CO₂-cured samples was higher than that of conventionally cured samples at 7 days and 28 days (Figure 6c1, c2 and Table 4). The compressive strength of these mortars shows identical values for both curing methods after 7 days and 28 days. This can be justified by the negligible carbonation depth of these samples after 24 h of carbonation (Figure 1d).

In fact, the low porosity of the designed mortar, on the one hand, and the higher quantity of clinker available to react with CO₂ at the surface (pore refinement due to the formation of CaCO₃), on the other hand, hinders the further diffusion of CO₂ to the samples. Comparing these results with the results obtained in the previous campaign (volumetric ratio of 1:4 and w/b = 0.5), a substantial increase in mechanical properties was observed due to the higher quantity of CEM II available in the formulation [35]

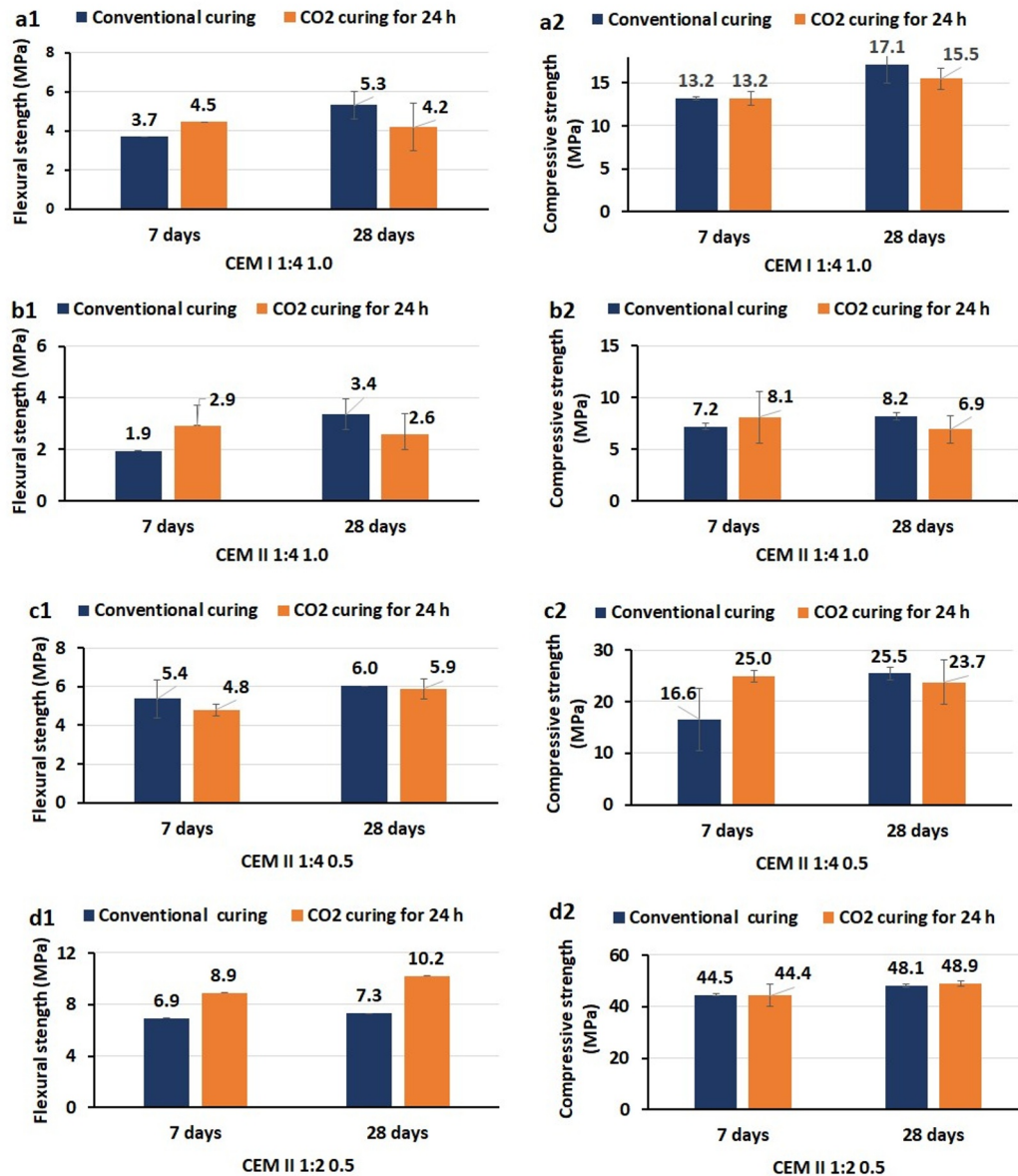


Figure 5- Flexural and compressive strength of mortar samples after conventional curing (orange data) and CO₂-curing-curing (blue data) for a1 and a2) CEM I with a volumetric ratio of 1:4 and w/b = 1, b1 and b2) CEM II with a volumetric ratio of 1:4 and w/b = 1, c1 and c2) CEM II with a volumetric ratio of 1:4 and w/b = 0.5, and d1 and d2) CEM II with a volumetric ratio of 1:2 and w/b = 0.5.

3.4 Effect of Pre-conditioning and Carbonation on the Mechanical Properties

As mentioned previously, the effect of drying and homogenization on the CO₂ uptake and mechanical properties of mortar was studied. Figures 7a, b, and Table 5 show the flexural and compressive strength of mortar samples prepared using CEM II with a volumetric ratio of 1:4 and w/b = 0.5 and subjected to three different curing conditions. For samples cured in conventional curing without any drying. The compressive strength of these samples (without any

drying at 28 days also were identical. Upon drying at 60 °C for 24 h, both the flexural and compressive strength of conventional-cured and CO₂-cured samples decreased substantially. The same samples showed a 51% and 63% reduction in compressive strength in 7 days and 28 days, respectively. For CO₂-cured samples, the drying process also provoked pronounced decreases in mechanical properties. The reduction of 52% and 54% in flexural strength was observed at 7 and 28 days, respectively

▪

Table 4 - Flexural and compression strengths of mortar samples tested in this study after conventional(conv.) curing and CO₂ curing for 24 h.

Mix design	Flexural strength (MPa)				Compressive strength (MPa)			
	7 days		28 days		7 days	28 days	7 days	28 days
	Conv. curing	CO ₂ curing	Conv. curing	CO ₂ curing	Conv. curing	CO ₂ curing	Conv. curing	CO ₂ curing
CEM I 1:4 w/b=1.0	3.7	4.5	5.3	4.2	13.2	13.2	17.1	15.5
CEM II 1:4 w/b=1.0	1.9	2.9	3.4	2.6	7.2	8.1	8.2	6.9
CEM II 1:2 w/b=0.5	5.4	4.8	6.0	5.9	16.6	25.0	25.5	23.7
CEM II 1:4 w/b=0.5	6.9	8.9	7.3	10.2	44.5	44.4	48.1	48.9

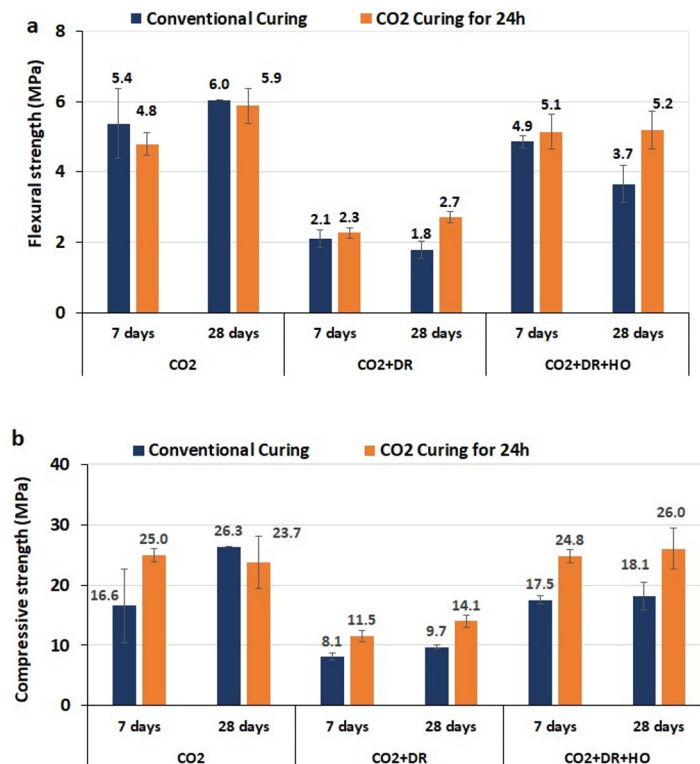


Figure 6 - a) Flexural and b) compressive strength of mortar subjected to CO₂ curing, drying and CO₂-curing (CO₂+DR) and drying, humidity homogenization and CO₂-curing (CO₂+DR+HO) at 7 and 28 days.

The compressive strength of CO₂-cured samples shows a reduction of 54% and 40% at 7 and 28 days, upon drying. The slight lower reduction in mechanical properties of CO₂-cured samples compared to the conventionally cured ones can be attributed to the formation of a higher quantity of CaCO₃, which resulted in pore refinement and slightly higher mechanical strength [20]. A homogeneous process (plastic warping mortar samples for 3 days) was performed to eliminate the un-homogeneity in the humidity profile of samples as surface evaporation results in lower humidity at the surface and higher in the center of samples. Surprisingly, both flexural and compressive strength show a substantial improvement compared with previous campaigns (24 h drying). In fact, for both conventional and CO₂-cured samples, the measured values of flexural and compressive were very close to those without drying. For conventional cured samples, the flexural strength shows a decreasing trend after 28

days, while the flexural strength of CO₂-cured samples was almost identical after 28 days. The compressive strength of conventionally cured samples and CO₂-cured samples shows a slight increase after 28 days, and the values were very similar to those of mortar samples without any drying. Therefore, the homogenization process efficiently increased the carbonation depth (Figure 3) without negatively affecting the mechanical properties of the mortar samples. As discussed previously, homogenization, on the one hand, improves the humidity distribution and, on the other hand, provokes extra curing (samples wrapped in plastic bags and left for 3 days), resulting in slight progress in the hydration process, i.e., formation of more Ca(OH)₂ (confirmed by TGA). This extra Ca(OH)₂, during the enforced carbonation, forms a slightly higher quantity of CaCO₃, and this can justify the higher mechanical properties of CO₂-cured samples after homogenization in comparison to conventionally cured ones.

Table 5 Flexural and compression strengths of mortar samples prepared using CEM II with a volumetric ratio of 1:4 and w/b = 0.5 with different pre-conditioning.

Pre-condition	Flexural strength (MPa)				Compressive strength (MPa)			
	7 days		28 days		7 days	28 days	7 days	28 days
	Conv. curing	CO ₂ curing	Conv. curing	CO ₂ curing	Conv. curing	CO ₂ curing	Conv. curing	CO ₂ curing
CO ₂	5.4	4.8	6.0	5.9	16.6	25.0	26.3	23.7
CO ₂ -DR	2.1	2.3	1.8	2.7	8.1	11.5	9.7	14.1
CO ₂ -DR-HO	4.9	5.1	3.7	5.2	17.5	24.8	18.1	26.0

4 Conclusions

This study aimed to evaluate the feasibility of integrating CO₂ curing into the production process of pre-cast cementitious materials (only for unreinforced concrete). Initial experiments focused on the CO₂ curing of prismatic specimens composed solely of cement paste using two types of cement (CEM I and CEM II). The results indicate that the low porosity of the cement paste (PC I and PC II) limits CO₂ diffusion, resulting in a constrained carbonation depth. TGA confirmed a CO₂ uptake of 7.2 wt% for PC II after 72 h of carbonation. Despite such a level of CO₂ uptake, 6 wt% of Ca(OH)₂ remained uncarbonated, suggesting the further potential of CO₂ uptake, which is restricted due to limited accessibility of CO₂ to unreacted phases.

Both the cement pastes and mortars prepared with CEM II exhibited higher carbonation depth compared to those made with CEM I. After prolonged carbonation (72 h), the carbonation depth of PC II was 3.8 times higher than PC I. For mortar prepared with CEM II after 12 h of carbonation, the carbonation depth was 1.8 times higher than for CEM I made mortar.

The reduction of both the w/b ratio and the volumetric ratio reduces the carbonation depth of mortar made with CEM II. Therefore, the mix design substantially affects the carbonation depth as the porosity level changes effectively. The pre-conditioning (curing, drying, and humidity homogenization) affects

carbonation depth, CO₂-uptake, and the mechanical properties of mortar. Conventional 48h of CO₂ curing created the most favourable condition for effective CO₂-uptake of mortar samples with a negligible negative effect on mechanical properties. The CO₂-uptake was 3% after 24 h of carbonation. While an additional drying step by extreme reduction of humidity negatively affects the CO₂-uptake and mechanical properties of mortars, further humidity homogenization by protection with a plastic film, attenuates the negative effect on mechanical properties. Despite the higher carbonation depth for samples subjected to extra drying and homogenization steps, TGA confirmed the lower CO₂ uptake. Therefore, the integration of the CO₂-curing step in pre-cast concrete manufacturing may be possible without considering any extra steps. Based on the obtained results and taking into consideration the vast variety of precast concrete products, CO₂ curing can be suggested for a selective type of precast products, namely non-structural with optimum porosity to ensure CO₂ diffusion through the pores. The humidity level of products at the carbonation step and the porosity of products are crucial factors for successful CO₂ uptake and will be considered in future works. The techno-economic assessment of CO₂-curing integration also needs to be studied in the future.

CRedit authorship contribution statement

David Bastos: Writing – original draft, Investigation, Conceptualization, Methodology. **Mahboobeh Attaei:** Writing – original draft, Conceptualization, Methodology, Investigation. **Cinthia Maia Pederneiras:** Writing – original draft, Conceptualization, Methodology, Investigation. **Rosario Veiga:** Writing – Methodology, Conceptualization, Supervision. **António Santos Silva:** Methodology, Conceptualization, Supervision.
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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.

Data availability

No 3rd party data was used for the research described in the article.

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