

Impact of CO₂ exposure on cement hydration mechanism

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Abstract

In the construction area, cementitious materials (especially concrete and mortar) continue to be the most demanded worldwide. However, the cement industry is responsible for the emission of around 5% of global carbon dioxide (CO₂) emissions. The area of accelerated carbonation has shown promising results, namely through a new approach that consists of the incorporation of CO₂ in cementitious mixtures. This work aims to analyze the influence of carbonation during the hydration mechanism of cement pastes, to evaluate the phenomenon of CO₂ incorporation during production and its impact on pastes composed of cement and calcium hydroxide addition. The experimental campaign involved the production of pastes with three different compositions, depending on the constitution of the binder mixture and different levels of exposure to CO₂. Subsequently, the pastes were characterized in the fresh state (consistency, density, setting time and calorimetry), in the hardened state (mechanical resistance), and subjected to chemical (XDR and TGA) and microstructural (SEM and MIP) characterization tests. Exposure to CO₂ affects the development of the microstructure, which appears more disconnected and presents the arising of pores in larger size ranges, a situation that was observed regardless of the presence of CH. This situation corroborates the results obtained in the mechanical tests since all the carbonated pastes reduced their compressive strength as well as a lower hydration degree and less formation of hydration products (C-S-H). The CO₂ absorption is benefited by the introduction of CH and leads to a greater formation of amorphous CC and CH.

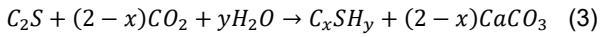
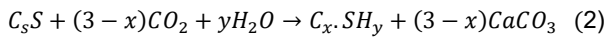
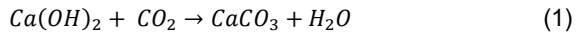
Keywords: Accelerated carbonation; CO₂ absorption; Mechanical strength; Hydration products.

1 Introduction

Cement is the main element in the construction sector since it is present in the constitution of most building materials. It is estimated that cement industry is responsible for about 5% of global carbon dioxide emissions, outcome of calcium carbonate calcination process [1]. To achieve the new environmental goals set at international level, several studies have appeared in literature based on the use of CO₂ during the production and curing

of cementitious mixtures, taking advantage of the favorable effects of the carbonation reactions when it occurs simultaneously with cement hydration [2]. In the carbonation curing technique, the porosity of cementitious mixtures allows the diffusion of CO₂ and subsequently, through reactions between CO₂ and cement minerals (equation 1 [3]) the formation of CaCO₃ crystals occurs. In the presence of water, calcium silicates

also react with CO₂ according to equations (2,3) [4], producing a hybrid of calcium-silicate-hydrate and calcium carbonate [5].



Several authors have been investigating a new approach consisting in the introduction of CO₂ during the production of cement mixtures. This introduction can be materialized through different ways: injection of gaseous CO₂ into the mix [6], use of carbonated water in the mix [7], or performing the mix in a carbonation chamber [8].

Although there is no consensus on the reported results, most authors suggest an acceleration of the hydration reactions [9] associated with a reduction in the setting time [6,10], variations in microstructure and reaction products depending on the amount of CO₂ [5]. Regarding the mechanical behavior, opinions differ among many authors and the contribution of CO₂ can be positive or negative depending mainly on the amount of CO₂ absorbed by the mixture [2].

In CarbonCure case study, mechanical results found out that the injection of 0.05% CO₂ (% to the mass of cement) allows a reduction in cement content without affecting the mechanical strength development over time [5]. However, it remains open what is the optimal CO₂ content to be absorbed by the concrete, which does not affect the mechanical and microstructure properties.

According to majority of authors, the increase in strength and acceleration of hydration is associated with the formation of CaCO₃ nanoparticles that act as nucleation points in the interstitial solution improving the precipitation of hydration products. [11]

2 Dissertation objectives

This work aims to study the hydration and carbonation mechanisms of cement pastes in the

presence of CO₂, to understand how the carbonation can affect the setting and hardening during pastes hydration. Also, the work investigates if the mechanical and microstructural can be enhanced through the incorporation of calcium hydroxide in pastes composition.

3 Materials and Methods

3.1 Materials

The Portland cement CEM I 42,5R provided by SECIL has a mineralogical composition of C₃S=58.3%, C₂S=15.4%, C₃A=5.3%, C₄AF=10.1% and a specific surface (Blaine) of 3640 cm²/g. The chemical composition of cement is CaO=63.18%, SiO₂=19.43%, Al₂O₃=5.39%, Fe₂O₃=3.24%, MgO=2.03%, TiO₂=0.31%, P₂O₅=0.02%, SrO=0.05%. The calcium hydroxide used contains more than 92% of CH and was manufactured by Calcidrata. It was also used a superplasticizer (SP), Master Ease 5025, which has a density of 1.058 (g/cm³) and pH of 5.3 at 20 °C. The mixing water was provided by the main water supply. The CO₂ gas supply was provided by AirLiquide, at 99.95% purity.

3.2 Definition of pastes composition

For each composition (Table 1), mixtures of 1.8 liters were performed and a water/binder ratio (w/b) of 0.44 was adopted to ensure a predefined spread of 100 mm. The parameter associated with the amount of CO₂ was evaluated by the different mixing times; then, the addition of CH was analyzed through two contents, 3% and 6% (% to mass of binder). For ensuring a good CH dispersion it was used a SP (0.3% to mass of binder). The mixtures produced in an atmospheric environment with normal carbon dioxide concentration followed the general indications of the standard NP EN 196-3 (2006).

Table 1 Mix proportions for cement pastes produced with/without the carbonation method

Designation of pastes	Cement (g)	CH (g)	Binder(g)	Water (g)	SP (g)	Mixture Time (min)	Presence of CO ₂ in atmosphere
REF	2500	-	2500	1092,5	7,5	3	N
CIM 5						5	Y
CIM 10						10	Y
CIM 15						15	Y
CIM 45						45	Y
CIM 90						90	Y
CIMC	2425	75	2500	1092,5	7,5	3	N
CIMC 45						45	Y
CIMC 90						90	Y
CIMC 2	2350	150	2500	1092,5	7,5	3	N
CIMC2 45						45	Y
CIMC2 90						90	Y
CIM control	2500	-	2500	1092,5	7,5	45	N
CIMC control	2425	75	2500	1100	-	3	N

3.3 Production of carbonated pastes

3.3.1 Carbonation Chamber

The system used to produce carbonated pastes, Figure 3.2, achieves an atmosphere with a controlled concentration (80%) of CO₂, monitored through a sensor (Datalogger). Cement, water and SP were set in container that is placed inside the carbonation chamber. CO₂ was pumped at a constant flow until the percentage of 80% was reached, and then, the mixing process begins.

The addition of CH is dispersed in the water by magnetic stirring for 10 minutes, then the cement is added, and the container is placed inside the carbonation chamber. In the production of the pastes with 45min and 90min of time production, a mixing cycle of 10min was performed (5min mixing and 5min pause) to avoid mixer machine overheating. Pastes' temperature was measured throughout production at 5min intervals as a check parameter.



Figure 3.1 Production system for carbonated pastes

3.4 Characterization of cement pastes

In the fresh state, the workability and apparent bulk density of the pastes were evaluated through the slump test and bulk density according to NP EN 445:2008. The pastes CIMC2 45 and CIMC2 90 showed low fluidity and therefore it was necessary to use the standard NP EN 1015-3 (2006). The setting time was evaluated by the Vicat method according to EN 196-3 (2005) without the determination of the normal consistency, which was not possible due to the mixing process of the carbonated pastes.

To evaluate the development of hydration heat of the produced pastes, the temperature of the pastes was monitored from the end of production until 30 hours, carried out by using 80g specimens of pastes inside a thermally sealed case. Specimens from the different pastes were cast in prismatic molds with $4 \times 4 \times 16 \text{ cm}^3$, demolded after 1 day, and cured in a moist chamber until testing age. The mechanical characterization involved compressive strength tests performed according to standard NP EN 1015-11 (2006). Tests were carried out at 1, 3, 7, 14, 28 and 90 days of curing age.

Hydration and carbonation products formed in cement paste samples were examined by quantitative x-ray diffraction (XRD) and thermogravimetric analysis (TGA) at different ages. Microstructural analysis was carried out by scanning electron images (SEM) and mercury intrusion porosimetry (MIP) at age 1 and 3 days.

The hydration stoppage method was the same for all the samples, but it was applied at the different testing ages according to several tests, based on [12]. Firstly, the sample was dried in an oven at 80°C for 1 hour, then it was put in 50ml of isopropanol for 50 min. Then, isopropanol was replaced by diethyl ether for more 30min, finally, the ether was removed, and the sample was dried in an incubator for 40min at 40°C . All samples were stored inside the desiccator in vacuum until testing.

Regarding the TGA analysis, it was also necessary to sieve the material through ASTM sieves with 0.150mm and 0.075 mesh opening. In the same way, samples for DRX analysis were ring milled to a fine powder and spiked with 10% zinc oxide to allow the quantification of cement phases.

4 Results and discussion

4.1 Influence of CO_2

4.1.1 Fresh density and workability of cement paste

The carbonation process during mixing causes a reduction of the spread diameter meaning a

reduction of the fluidity of cement pastes (figure 2), which was already observed in previous studies [13]. The fresh density showed a clear tendency to increase with the increase of the carbonation time.

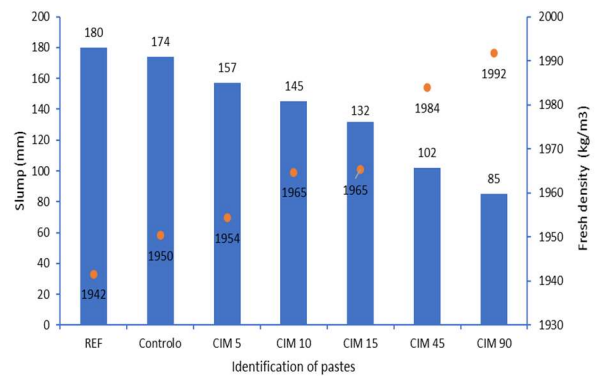


Figure 4.1 Slump and Fresh density

The increase from 45 min to the maximum time in the carbonation chamber implies a 17% (17 mm) reduction in the spreading diameter, substantially lower than the difference between the REF and the CIM45 (27%, 48 mm). This result suggests that the impact of CO_2 is higher at the beginning of the mixing process and becomes less significant when it is longer than 45 min (Figure 4.2).

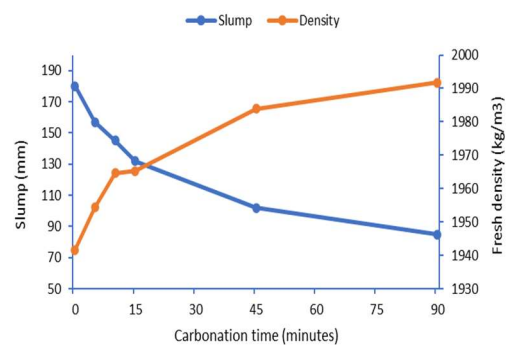


Figure 4.2 Influence of carbonation time on fresh state

4.1.2 Evolution of hydration reactions

The initial and final setting times for the control paste produced without carbonation method were 135 and 201 min, respectively. These two times were reduced by 65 and 45 min, respectively, for the paste produced by using the carbonation method (Table 2). This situation suggests that the presence of CO_2 during the mixing process works as a setting

accelerator, a result that follows the trend reported by other authors [5].

Table 2 Set time of cement pastes.

Paste	Set time (minutes)	
	Initial set	Final set
REF	255	345
CIM 90	190	300

Figure 4.2 shows the temperature evolution of the pastes REF and CIM 90 overtime, where are also marked the beginning (i) and end of setting (f) for both pastes. Initially, there was a higher reactivity in the CIM 90 paste, however, there was a reduction of the peak temperature (hydration of C_3S), which happens slightly earlier compared to the REF paste. The setting accelerator effect of CO_2 observed in these tests is related to the decrease in fluidity observed in carbonated pastes. This accelerator effect will have corresponded to an acceleration of chemical reactions resulting in a faster rate of compound formation that should have an impact on the increase in density.

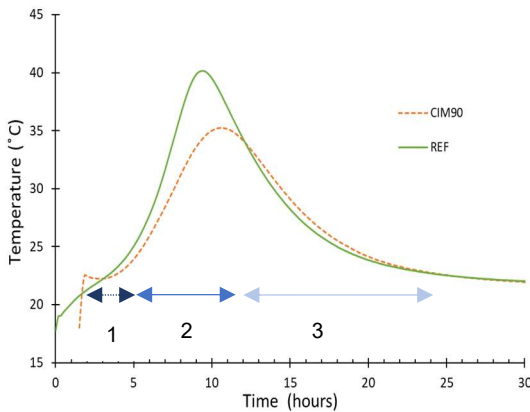


Figure 4.2 Temperature evolution during hydration. 1- Induction period; 2-Acceleration period; 3-Low activity period; (CIM 90)

4.1.3 Mechanical behaviour

The flexural and compressive strength tests yielded average results at 1, 3, 7, and 28 days of the hardened specimens (Figure 4.3). Regardless of the mixing time in the carbonation chamber, the incorporation of CO_2 is always detrimental to the compressive strength. Contrary to expectations, it was not possible to find an optimum content that

would allow an acceleration of the reactions in the first phase, with a consequent impact on the strength suggested in [6]. Despite the variability of results, a generally reduction in strength is observed with increasing CO_2 content.

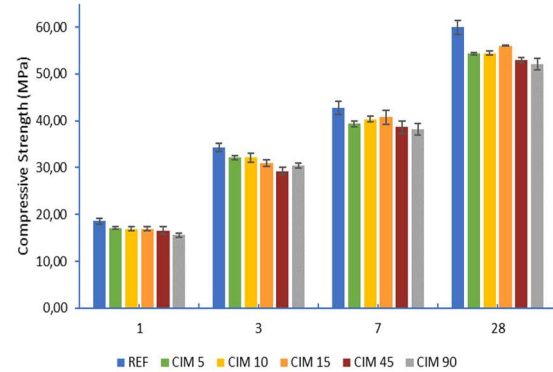


Figure 4.3 Compressive Strength for different pastes

The pastes subjected to longer carbonation times show greater reductions in strength, both to REF and to the other pastes (CIM 5, CIM 10, and CIM 15) at younger ages (89% and 85% at 1 day and 85% and 88% at 3 days), with some recovery, albeit slight, of this resistance after 7 days of age (90% and 89% at 7 days and 88% and 87% at 28 days)

The differences between pastes subjected to maximum and minimum carbonation times suggest that CO_2 effect is not linear with the increase of carbonation time. Thus, and despite some uncertainty in the results due to their apparent variability, they suggest a reduction in strength that increases with exposure to CO_2 ; however, the rate of this reduction decreases with the age of the pastes, probably related to some capacity of compensation by later hydration reactions.

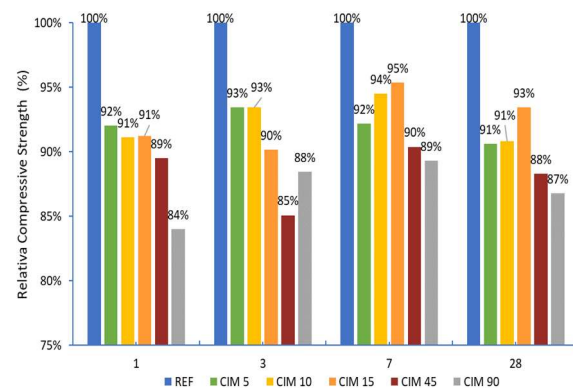


Figure 4.3 Relative compressive strength

4.1.4 Impact on hydration and reaction products

On the one hand, the XRD and TG analysis of the REF paste identified the initial formation of amorphous CH that progressively transforms into crystalline material, figure 4.6. On the other hand, CIM 90 paste showed a higher amount of amorphous CH at 3 days and less formation of crystalline CH throughout the hardening reactions.

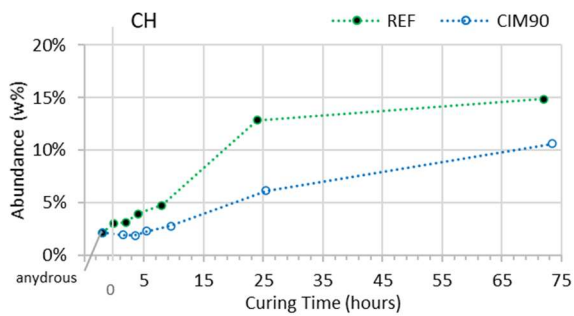


Figure 4.4 Development of CH quantity over curing time.

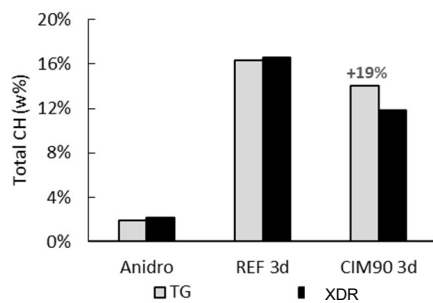


Figure 4.5 Crystalline CH (DRX) versus all CH identified (crystalline and amorphous).

Thus, the presence of CO₂ should cause a higher initial formation of amorphous CH (with the presence of agglomerates of these same particles) associated with a reduction of the hydration rate throughout the hydration period, as well as a lower degree of hydration at 3 days identified in the TG analysis.

The exposure to CO₂ during production did not allow reaching significant values of CO₂ absorption, which is related to the similar CC content between the pastes REF and CIM 90. The reactions between CO₂ and calcium ions do not trigger a consumption effect of these ions. The higher solubility and the consequent increase of Ca²⁺ content in the solution

delay the precipitation of hydration products over time, which justifies the lower values of α and the lower amount of formed CSH. This phenomenon also allows explanation of partial recovery of resistance in the long term, after the progressive restoration of pH and CSH formation conditions.

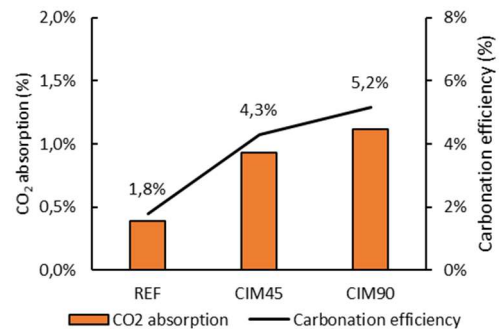


Figure 4.6 CO₂ absorption and carbonation efficiency for different productions conditions.

4.1.5 Influence of CO₂ on microstructure

Figure 4.7 shows the images from SEM analysis performed for the REF (a) and CIM 90 (b) pastes at 1day. The presence of CO₂ lead to a more disconnected structure and smaller particles in the CIM 90. This situation is related to the results obtained during the mechanical characterization tests, reinforcing the fact that CO₂ negatively affects the formation and growth of hydration products causing a looser and less resistant structure.

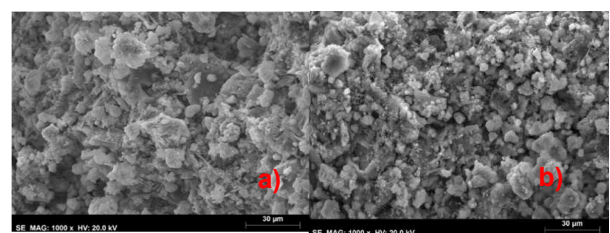


Figure 4.7 SEM images from REF and CIM 90 pastes

The presence of CO₂ caused a slight increase in total pore volume compared to non-carbonated paste, corresponding to a variation from 30.1% to 31.4%. REF paste exhibits a porosity based on smaller pore sizes, with a critical peak at 0.1 µm. In contrast, the paste CIM 90 is distinguished by a coarser porosity and a curving peak on size of 1 µm, and distribution with greater amplitude in the size of

the pores (Figure 4.8). The increase in total porosity and the presence of larger pores agree with the observation of a more disconnected microstructure (less dense) verified in SEM analysis and justify the lower mechanical resistance achieved by carbonated pastes.

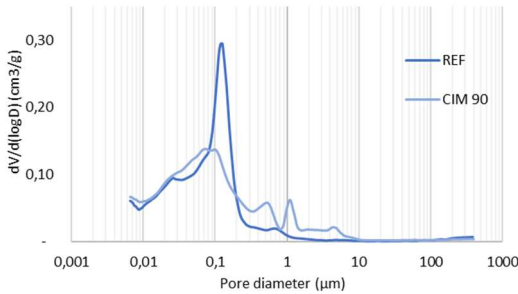


Figure 4.8 Porosimetry of different pastes.

4.2 Influence of CH

4.2.1 Fresh density and workability of cement/lime paste

A similar trend was observed on workability for the pastes with CH, i.e., the reduction of the spreading diameter with the introduction of the carbonation process, according to Figure 4.9. On the other hand, the introduction of CH made the pastes slightly less fluid and caused an immediate reduction of the density mass compared to the pastes produced without CH, regardless mixing conditions.

Similar behavior was observed with increasing CH content (6%), which required the use of an alternative slump test. Still, a reduction of 7% in the spread of the paste CIMC2 90 compared to CIMC2 45 can be noted.

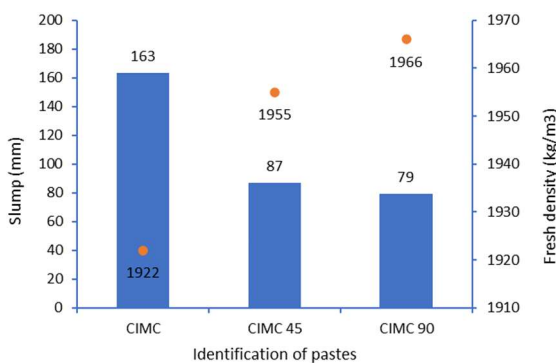


Figure 4.9 Spread and fresh density for cement/lime pastes

4.2.2 Evolution of hydration reactions

Table 3 shows the setting time for cement/lime pastes produced with or without using the carbonation method. The introduction of 3% CH reduced the initial and final setting times compared to REF, suggesting an acceleration of the setting reported by [14], attributed to the larger specific area of CH contributing to higher precipitation. The CIMC paste showed intermediate values between REF and CH pastes, indicating the existence of a threshold value of CH, above which the beneficial effect of CH on the acceleration of setting is blocked by the lower amount of clinker present in the paste.

In the situation of carbonation during mixing, the effect of acceleration of setting due to CO_2 is intensified by the presence of CH, proven by the reduction of the setting time of paste CIMC 90 compared to paste CIM90. CIMC2 90 showed the longest setting time among the carbonated pastes, confirming the hypothesis described above.

Table 3 Set times of cement/lime pastes.

Paste	Set time (min)	
	Initial set	Final set
REF	265	350
CIM 90	190	280
CIMC	230	320
CH90	175	245
CIMC2	260	345
CIMC2 90	250	305

Figure 4.10 represents the evolution of paste temperature over time influenced by carbonation (CIM 90 vs CIM), by the presence of CH (CIM vs CIMC), and by both simultaneously (CIM vs CIMC 90). The introduction of CH in 3% corresponded not only to a higher peak of hydration compared to the REF paste but also to a greater rate in the initial development of the reactions, since it presents a higher initial temperature, in agreement with the results obtained in the setting time. The increase in the amount of CH penalized hydration, the paste

CIMC2 reached the hydration peak later and associated with a much lower temperature compared to the other pastes.

In pastes with applied carbonation, the introduction of CH caused greater initial heat release, higher in paste CIMC2, and shorter intervals until reaching the temperature peak associated with the hydration of C3S. However, the introduction of CH did not reduce the CO₂ penalizing effect on hydration since there is a sharp reduction of the hydration peak in carbonated pastes.

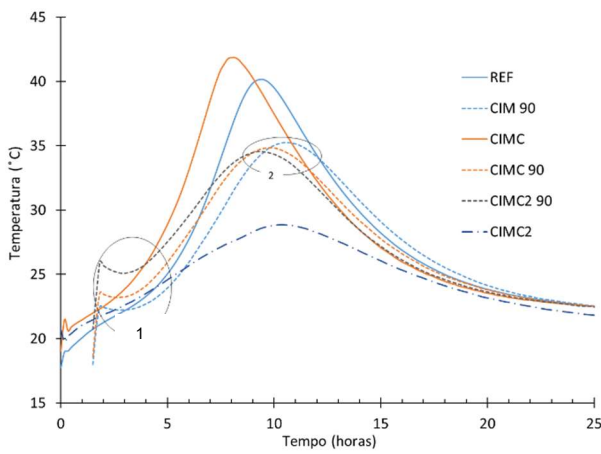


Figure 4.10 Temperature evolution for all pastes

4.2.3 Mechanical behaviour

Figure 4.11 represents the evolution of compressive strength at young ages and the simultaneous impact of the introduction of CH and CO₂. It was found that the introduction of CH did not overcome the negative effect of CO₂, the pastes with CH showed similar results to those without CH when subjected to the carbonation method.

CIMC paste showed similar resistance to REF paste, which may have been achieved by the introduction of CH. Without the presence of CO₂, there is no formation of CC, so there must have been an acceleration caused by the presence of CH, which has the potential to act as a nucleation point, a situation also suggested in other investigations [14].

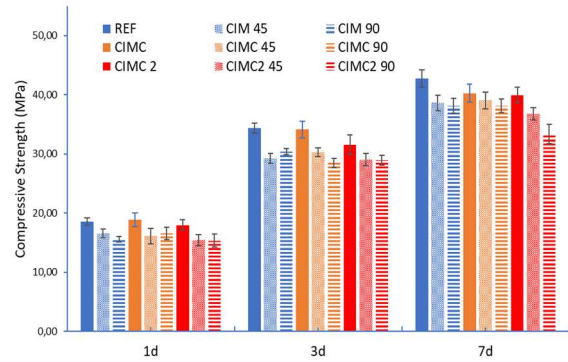


Figure 4.11 Compressive strength for cement/lime pastes at young ages.

At higher ages, especially at 28 days, the paste with 3% of CH showed relative losses of 8% and 7% due to carbonation at 45 and 90 minutes, while the losses of the CIM pastes were 12% and 13%, respectively (Figure 4.12). Despite the negative effect of CO₂ affecting the strength of pastes, the incorporation of CH is beneficial because it allows partial recovery of mechanical strength over time, clearly at higher ages (28d and 90d).

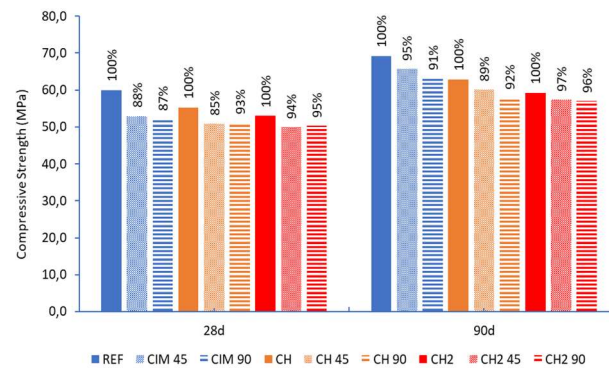


Figure 4.12 Compressive Strength and relative loss due to mixture compositions.

The pastes with 6% of CH showed lower relative losses at 28d compared to the incorporation of CO₂ at 45 and 90 minutes, 3% and 4% respectively. In general, it was found out that the impact of CO₂ is slightly reduced by increasing CH, however, this behaviour did not show a regular pattern over time.

4.2.4 Impact on hydration and reaction products

The CIMC paste showed higher initial dissolution together with a higher formation of amorphous material compared to the REF paste, which was expected given the behaviour observed in the setting time and calorimetry tests. Through TG analysis, it was found that the incorporation of CH causes an increase in the amount of amorphous CH. In the CIMC paste, 44% of the CH formed during hydration is composed of amorphous material, while in the REF paste, almost all the quantified CH is in the crystalline phase.

It was verified that the presence of CH is favourable to the permanence of new CH compounds in the amorphous state. It should be noted that in the chapter on the influence of CO₂ (4.1), the same conclusion was obtained regarding the presence of CO₂. Figure 4.1e3 supports the hypotheses that, except paste REF, whose hydration is not influenced by the presence of additions, the other pastes presented significant initial amounts of amorphous CH that may evolve later to crystalline material at a slower rate.

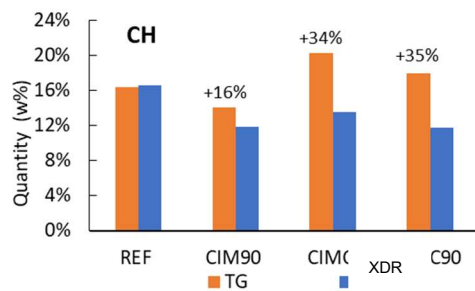


Figure 4.13 Crystalline and amorphous CH quantification at 3 days.

Although the variations between the pastes are relatively low, the CIMC 90 paste showed higher CC values at 3 days, supported by both TG and XRD. Thus, this result confirms that the presence of CH benefits the carbonation reactions, allowing a greater formation of CC (amorphous and crystalline), which can be seen at 3 days.

The results showed a very similar evolution of the amorphous, C₃S, and CH phases between the

CIM 90 and CIMC 90 pastes. In the calorimetry test, the induction period was identified in both pastes subjected to CO₂; however, the presence of CH should be responsible for the higher reactivity observed in CIMC 90 paste after the end of the induction phase, due to a typical filler setting accelerator effect, as previously mentioned.

Regarding the absorption of CO₂ by the pastes, although the pastes with CH reached absorbed CO₂ values lower than 2%, it was found that CH slightly benefits carbonation reactions, due to the higher formation of CC and higher absorbed CO₂ at 3 days.

4.2.5 Influence of CO₂ on microstructure

Despite the introduction of CH in the binder mixture, the same adverse effect on the development of hydration products was observed, probably, caused by the presence of CO₂ during pastes production. The paste CIMC90 presents a looser microstructure and less bonding between the particles compared to the paste CIMC according to the images obtained by SEM at 1 day with a scale of 1000x (Figure 4.14), which supports the lower values of compressive strength obtained in CH carbonated pastes. The presence of ettringite and portlandite in both samples is noteworthy, which was expected due to the higher amount of CH in the binder mixture, it was also to distinguish in the paste CIMC90 crystals that may be agglomerates of calcite particles.

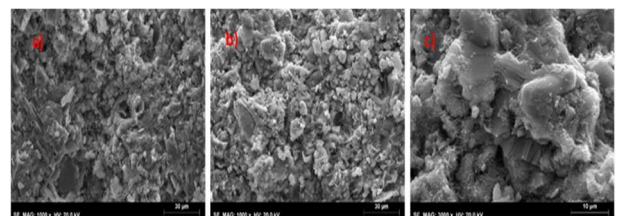


Figure 4.14 SEM images captured at 1 and 3 days for cement/lime pastes.

Despite slight differences observed in the MIP results (Table 4), from samples at 3 days, there is not only an increase in total porosity due to the introduction of CH but also, as previously observed,

higher porosity in the pastes subjected to carbonation method during production.

Table 4 Total porosity for cement pastes

Paste	REF	CIM90	CIMC	CIMC 90
Porosity (%)	30,1	31,4	32,9	34,1

CIMC paste differs from REF by showing higher amount of small pores, with greater emphasis in the range 0.01 μm to 0.1 μm . This increase may be associated with the larger specific surface area that benefits the precipitation of compounds, a situation that is also associated with the higher initial reactivity of paste CH compared to REF, observed in the evolution of reactions during hydration.

The paste CIMC 90 not only shows higher total porosity than the paste CIM90 but also more pore size content in higher ranges (0.5-10 μm). The carbonation mechanism continues to cause a coarser porosity and the presence of larger pores, which is related to less dense microstructures (figure 4.15)

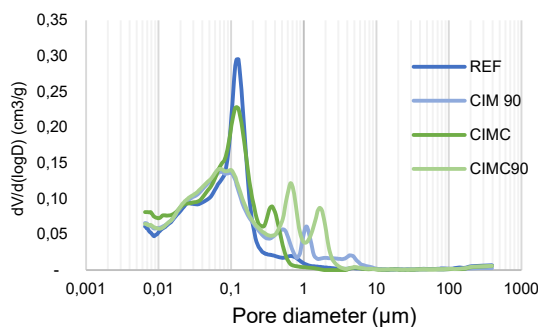


Figure 4.15 Porosimetry for the different pastes.

5 Conclusions

5.1 Final conclusions

The presence of CO_2 during paste production caused an acceleration in the pre-nucleation period (dissolution phase) but delayed the acceleration period. The incorporation of CH in the binder mixture composition has a filler-type phenomenon, i.e., it increases nucleation rate which helps the growth of hydration products such as C-S-H throughout the acceleration period. When both additions act simultaneously, CIMC 90 paste, the

effects are overlapped, with a faster setting time and longer time needed to reach the acceleration period when compared with the situation without CO_2 .

In summary, it was verified that a penalization of the mechanical strength due to CO_2 for the various exposure times adopted, whose impact on the hydration of the pastes is not linear with the increase of the exposure time to CO_2 . On the other hand, the higher solubility, and the consequent increase in Ca^{2+} content delay the precipitation of hydration products over time, which can support the lower hydration degree and the lower amount of C-S-H formed. This also could explain the partial recovery of strength in the long term, after the progressive restoration of pH and CSH formation conditions.

. Although the introduction of CH did not overcome the detrimental effect of CO_2 on hydration, there was a lower loss of strength in the pastes with CH and a higher amount of CO_2 absorbed.

5.2 Suggestions for future studies

Throughout the work, the high variability and uncertainty about the effects of carbonation were referred, which demonstrates the need for further research on this subject. Given the diversity of possible study strategies, some hypotheses that may be relevant are suggested:

To evaluate the impact of the greater permanence of amorphous CH in pastes subjected to additions and its possible relationship with the reduction of compressive strength. To explore the effect of exposure to CO_2 of pastes with reduced mixing times and the influence of pressure in the carbonation chamber. In a slightly distinct concept, to analyse the possibility of carbonation of cement waste for the formation of calcium carbonate.

6 References

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