

# Interaction of carbonation and chloride ions ingress in concrete

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## Abstract

Carbonation is one of the factors that can influence the chlorides transport. Despite the combined action of chlorides and carbonation being a reality, there is no consensus on the effect of this coupled environmental load on chloride transport. Accelerated tests were used to simulate this environment. Concrete specimens were cast using 0.60 water/cement ratio and CEM I 42.5R. After curing, half of the specimens were submitted to accelerated carbonation test (20°C, 55% RH and 4% CO<sub>2</sub>) for 1 and 7 months. The other half was protected with plastic film for the same period. Then, the specimens were submitted to the accelerated chlorides attack using: migration test and immersion test. Finished the immersion test, dust samples were extracted from surface to bulk and analysed to obtain free and total chloride profiles and pH profile. According to the obtained migration test results, the carbonation increases chloride transport regardless of the exposure time. According to the immersion test results, there is a decrease in chloride ions transport when the specimens are carbonated for 1 month and a noticeable increase when carbonation period increases to 7 months. The redistribution of pores in carbonated concrete and the reduction of chlorides binding capacity in carbonated concrete are related to these results.

**Keywords:** Chloride transport; Carbonation; Coupled deterioration; Accelerated test

## 1 Introduction

In terms of the durability of concrete structures and, consequently, its service life, the chloride ions transport into concrete has been the subject of several studies over the years [1–6]. As one of the most aggressive ions, chloride ion penetrates into the hardened concrete cover and reaches the surface of the steel reinforcement, causing the rebars corrosion [7]. Several factors can influence the chloride ions transport, including concrete carbonation. The chlorides transport can happen combined to carbonation, for example, in tunnels close to the sea, bridges subject to thaw salts, structures inserted in marine environments or specific industrial environments.

The study on the coupled deterioration by chlorides and carbonation is relatively recent and achieves greater expressiveness from the year 2000 [8–12]. Thus, despite the combined action of these mechanisms being a reality, there is no consensus on the interaction between chlorides and carbonation and its effects on chloride transport into concrete.

Chindaprasirt et al. [8], submitted Ordinary Portland Cement (OPC) mortars specimens to carbonation chamber (5% CO<sub>2</sub>, 23°C and 50% RH) for 28 days. After that the specimens were

tested through a rapid chloride penetration test, modified rapid migration test and chloride immersion test (3% NaCl solution). They concluded that the exposure to CO<sub>2</sub> environment has no influence in the chloride ingress into the samples studied. Chengfang et al. [10] subjected OPC concrete specimens to carbonation chamber (20% CO<sub>2</sub>, 20°C and 70% RH) for 14 and 28 days and after to immersion in 5% NaCl solution and drying in laboratory environment cycles to study the impact of CO<sub>2</sub> presence on chloride ingress. They concluded that the chloride diffusivity coefficient increases with the carbonation time. Malheiro [13] studied the behaviour of concrete and mortar specimens using wet and drying cycles, chloride migration test and chloride immersion test (15% NaCl), to combine carbonation (20°C, 55% RH and 4% CO<sub>2</sub>) and chloride transport, and observed contradictory results. She observed that sometimes the carbonation increases the chloride transport and sometimes the carbonation decreases it. She believes that this behaviour is related to carbonation depth reached. The lack of standardisation about the various parameters involved in the experimental work such as type of test, temperature, humidity and concentration of NaCl and CO<sub>2</sub> makes it difficult to compare these results [14].

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The combination of chlorides and carbonation action in the cementitious material is a complex chemical and physical phenomenon. On the one hand, the transformation of  $\text{Ca}(\text{OH})_2$  and others hydrates into carbonates, decreases the porosity of concrete [15–17] and, consequently, contributes to reduce the amount of penetrated chlorides. On the other hand, the pH decrease caused by carbonation [17] can contribute towards releasing chemically fixed chlorides and increasing free chlorides content, which may actively ingress into concrete. When these aggressive agents act at the same time, or in short intervals one after the other, these mechanisms and properties can be modified.

The present work contributes to this scientific discussion, carrying out an experimental investigation on the interaction between chlorides and concrete carbonation, combining these aggressive agents through accelerated tests in ordinary concrete.

## 2 Materials and specimens

Concrete specimens were made using OPC, CEM I 42.5R, which chemical composition is presented in Table 1. Fine aggregate with a fineness modulus of 2.96 and two coarse aggregates, one with particle sizes between 4 mm and 8 mm and the other with particle sizes between 6 mm and 12 mm were used. To accelerate the concrete carbonation and chloride transport, cylindrical concrete specimens ( $\phi 100 \times 200$  mm) with high water/cement ratio, 0.60, were cast. The concrete mixture is presented in Table 2.

**Table 1.** Chemical composition of CEM I 42.5R.

SiO <sub>2</sub>	%	20.33	K <sub>2</sub> O	%	0.76
Al <sub>2</sub> O <sub>3</sub>	%	4.59	Na <sub>2</sub> O	%	0.19
Fe <sub>2</sub> O <sub>3</sub>	%	3.06	Cl <sup>-</sup>	%	0.07
CaO	%	62.30	Loss on ignition	%	1.78
MgO	%	2.12	Insoluble residue	%	1.21
SO <sub>3</sub>	%	3.10	Remaining elements	%	0.49

**Table 2.** Concrete mixture design (1 m<sup>3</sup>) and its main properties.

Cement	kg	380.00	Compressive strength [MPa]	28 days	32.84
Water	l	228.00		90 days	35.09
Sand 0/4	kg	855.76		365 days	39.59
Coarse aggregate 4/8	kg	282.22	Capillary water absorption	kg/m <sup>2</sup> /min <sup>1/2</sup>	0.28
Coarse aggregate 6/12	kg	508.95	Open porosity	%	13.50
Slump	mm	20.50			

After casting, the specimens were protected from water evaporation with a plastic sheet and were stored in a humidity chamber (21 °C and 98% RH) for one day. Then, they were removed from the moulds and were cured immersed in tap water for 90 days. After, two types of specimens were prepared. The specimens type 1 were obtained by cutting the

original cylinders in three slices. The top and bottom of the cylinders were discarded (according to [11]), resulting in the following new specimens type 1:  $\phi 100 \times 50$  mm. The specimens type 2 were obtained by cutting the original cylinders in two slices. The top and bottom of the cylinders were discarded, resulting in the following new specimens type 2:  $\phi 100 \times 75$  mm.

## 3 Experimental programme

### 3.1 Preconditioning

The specimens were divided in two groups: reference and combined. Before accelerated concrete carbonation, the specimens of the combined group were subjected to preconditioning procedures to achieve the equilibrium with environmental humidity near 60% and accelerate the carbonation process. The preconditioning procedures included three stages. In the first stage, the specimens remained in a chamber with controlled temperature and humidity (20 °C and 60% RH) until to reach the constant mass. In the second stage, the specimens were prepared according to [14]: the lateral and bottom faces were painted with paraffin (type 1) or epoxy resin (type 2), the unique free face was used to allow a unidirectional flux during the exposure time. In the third stage, the specimens were wrapped with plastic film and put in an oven (40 °C), for 20 days, to distribute the internal humidity. The specimens of the reference group were wrapped with plastic film and stored in a laboratory environment during the preconditioning period.

### 3.2 Accelerated test – Carbonation chamber

After preconditioning procedures, the plastic film was removed from the combined group specimens and then they were subjected to accelerated carbonation in carbonation chamber (4% CO<sub>2</sub>, 20 °C and 55% RH), following the recommendations of the European Standard [19], for 1 and 7 months. The specimens of reference group remained wrapped with plastic film and stored in a laboratory environment during the carbonation periods.

### 3.3 Accelerated test – Non-Steady-State Migration Test (NMT)

Finished the carbonation periods (1 and 7 months), the paraffin was removed from the specimens type 1 and the NMT was performed on reference and combined groups, following LNEC specification [20]. These specimens were previously saturated with limewater under vacuum conditions and stored in the solution for  $18 \pm 2$  h. Afterwards, the specimens were placed in rubber sleeves. The catholyte was filled with a 10% NaCl solution and the anolyte with a 0.3M NaOH solution. The test duration and applied voltages were determined based on the initial current measured at 30 V. Finished the test, the specimens were split diametrically and perpendicular to the penetration direction of the aggressive agents. In the reference group, the chloride penetration depth was determined according to [20]. In the combined group, it was also necessary to determine the carbonation depth. In this case, halves were used in different ways: specimens were sprayed with a solution of 1% phenolphthalein in 70% ethyl alcohol [21] in order to

determine the carbonated depth; specimens were sprayed with 0.1 N  $\text{AgNO}_3$  to determine the chloride penetration depth (Figure 1).



**Figure 1.** Determination of the carbonation depth (left) and the chloride penetration depth (right) in combined group.

### 3.4 Accelerated test – Chloride immersion test

Finished the carbonation periods (1 and 7 months), the specimens type 2, reference and combined groups, were submitted to chloride immersion test following LNEC specification [22]. In this test, the specimens were immersed in limewater until reaching a constant mass. After, the specimens were immersed in a solution with 15% sodium chloride (related to the mass of water) for three months. The container used in the test remained closed to avoid water evaporation. Furthermore, the water temperature was monitored, and it remained around 20°C. Finished the exposure time, the specimens were continuously ground into powder in successive 5 mm thickness layers from the surface until a depth of 30 mm [23], which means six layers for each specimen. The extracted powder samples were used to determine the total chloride content by chemical analysis using the Volhard's method [24], as well as the free chloride content [25] and the pH profile [26].

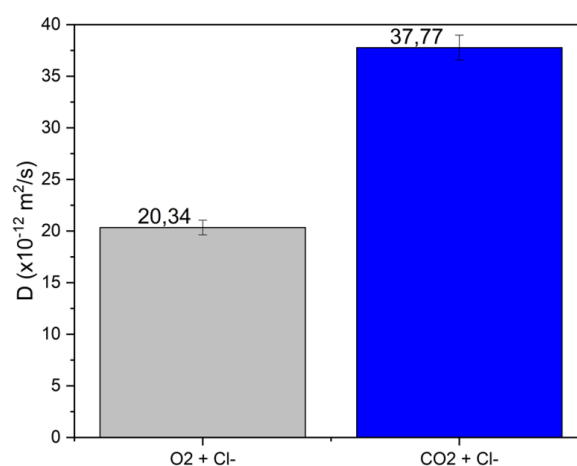
### 3.5 Mercury Intrusion Porosimetry (MIP)

The MIP was carried out in order to better study the carbonated region and its equivalent region in the reference group. The MIP tests were performed with an Auto Pore IV scanning mercury porosimeter, which had a pressure range from 0.10 to 33000 psi. The contact angle and the mercury surface tension considered were 130° and 0.485 N/m, respectively. Prior to the test, specimens were oven-dried at 60°C for 24 hours.

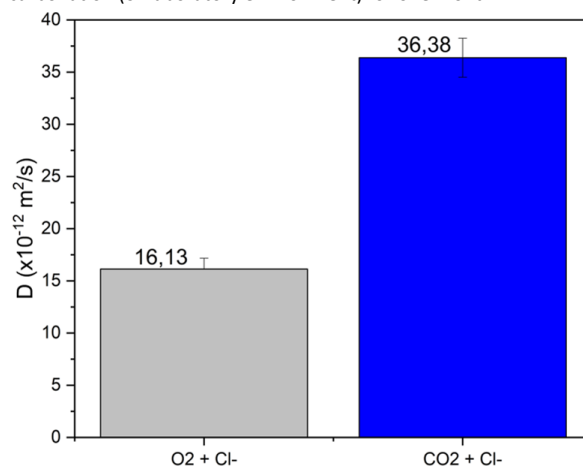
## 4 Results and discussion

### 4.1 Non-Steady-State Migration Test

Figures 2 and 3 show the non-stationary chloride diffusion coefficient ( $D$ ) obtained for reference and combined groups after one and seven months of carbonation, respectively.



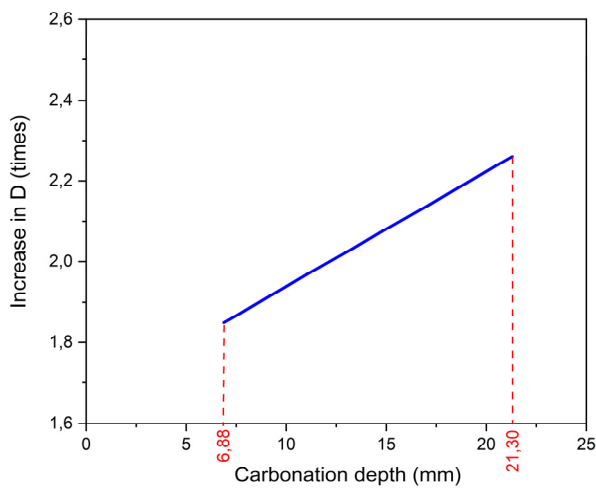
**Figure 2.** Chloride diffusion coefficient for specimens submitted to carbonation (or laboratory environment) for one month.



**Figure 3.** Chloride diffusion coefficient for specimens submitted to carbonation (or laboratory environment) for seven months.

According to the classification proposed by Gjorv [3], the values of  $D$  presented in Figures 2 and 3 represent concretes with reduced resistance to the penetration of chlorides. This is an expected result since concrete with a high water/binder ratio were cast to favour chloride penetration. Comparing Figures 2 and 3, it is observed a reduction in  $D$  value as the exposure time increases. In the reference group, this reduction is likely to be related to the concrete hydration process [27]. In the combined group, the carbonation degree must also be considered.

Concerning the chlorides transport in carbonated concretes, there is an increase in  $D$  for the combined group when compared to the reference group. This increase is more evident in specimens that were in the carbonation chamber for longer exposure time and, consequently, achieved more significant carbonation depths (Figure 4).



**Figure 4.** Relation between carbonation depth and increase in the chloride diffusion coefficient.

From Figure 4, it can be seen that the specimens carbonated for one month have an average carbonation depth equal to 6.88 mm and 1.85 times increase in the *D* achieved to the combined group when compared to the reference group. The specimens carbonated for seven months have an average carbonation depth equal to 21.30 mm and 2.26 times increase in the *D* achieved to the combined group when compared to the reference group.

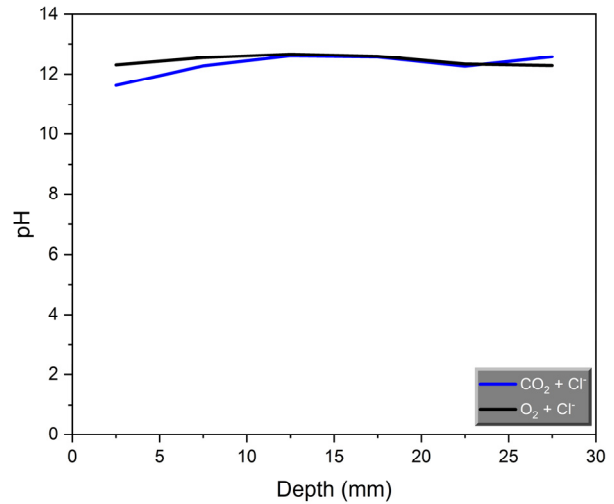
The increase in *D* may be related to the rise in the large capillary pores, caused by carbonation [15], that may provide a privileged pathway for the chlorides penetration. Furthermore, the carbonated concrete has its ability to bind chlorides reduced because there is a drop in pH values [28] and, consequently, there is an increase in the amount of free chlorides ions available to penetrate into the cementitious matrix. These hypotheses will be explored in the following section.

#### 4.2 Chloride immersion test

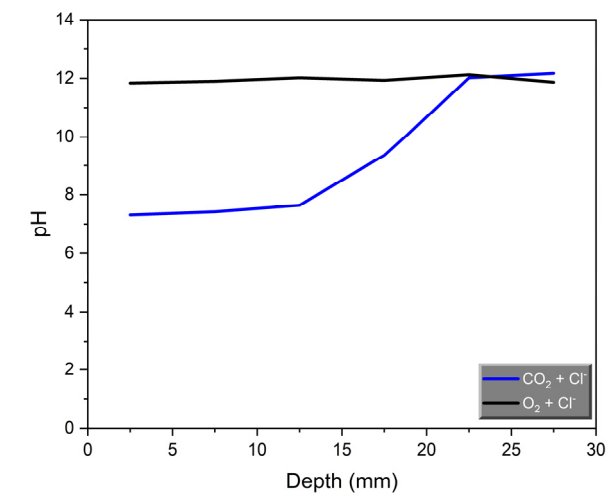
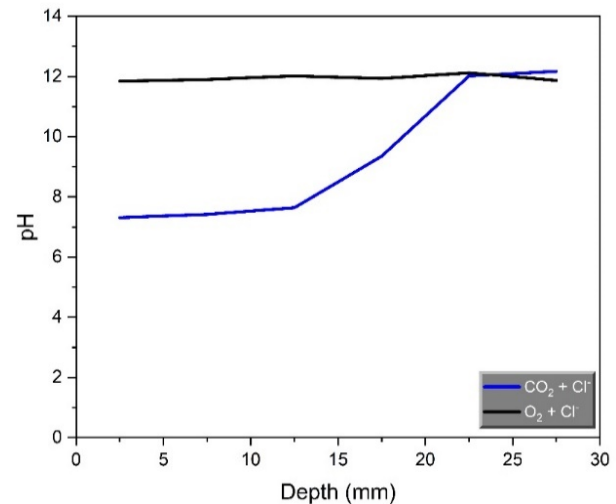
Figures 5 and 6 present the pH profiles to reference and combined groups. Figure 5 refers to specimens subjected to carbonation (or laboratory environment) for one month and to chlorides for three months. Figure 6 relates to specimens subjected to carbonation (or laboratory environment) for seven months and chlorides for three months.

From Figure 5, it can be seen two different profiles with the expected values for a non-carbonated concrete (~ 12.5 [29]). Despite that, the combined profile shows a slight decrease at the beginning of the profile, probably related to superficial carbonation. From Figure 6, it can be seen that the reference pH profile shows a stable behaviour, with the expected values for a non-carbonated concrete. However, carbonation is a mechanism that involves gradients and, therefore, no threshold is established between carbonated and non-carbonated zones. In this sense, for the combined profile, three distinct zones related to the different pH values were identified. The first one, until 15 mm, fully carbonated zone, shows a stable profile with values expected for carbonated concrete. The second one, between 15 and 25 mm, is a transition zone, partially carbonated due to the coexistence

of  $\text{Ca(OH)}_2$  and  $\text{CaCO}_3$ , where the pH value of the pore solution is higher than that in the fully carbonated zone. In the third one, between 25 and 30 mm, the profile is stable and presents values compatible with non-carbonated concrete.

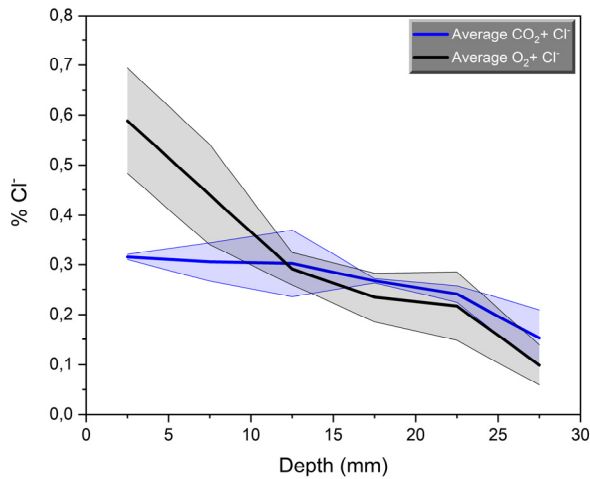


**Figure 5.** pH profiles for specimens submitted to carbonation (or laboratory environment) for one month.

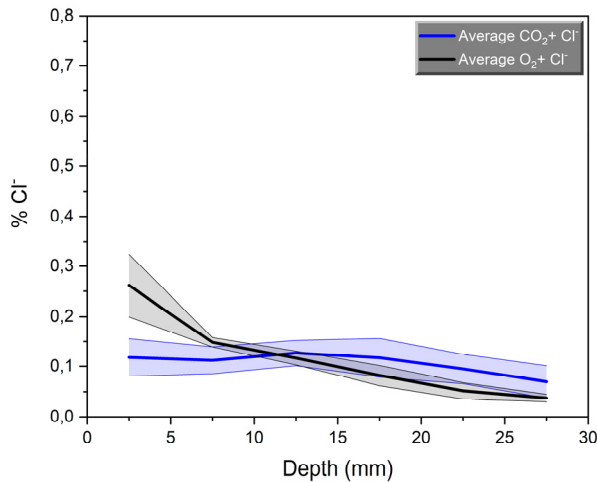


**Figure 6.** pH profiles for specimens submitted to carbonation (or laboratory environment) for seven months.

Figures 7 to 10 present the chloride profiles for the reference and combined groups. The values presented are the average of three samples analysed, as well as their standard deviation. In the Figures 7 and 8 it is possible to see the total and free chloride profiles, respectively, for specimens submitted to carbonation (or laboratory environment) during one month and to chlorides during three months.



**Figure 7.** Total chloride profiles for specimens submitted to carbonation (or laboratory environment) for one month.



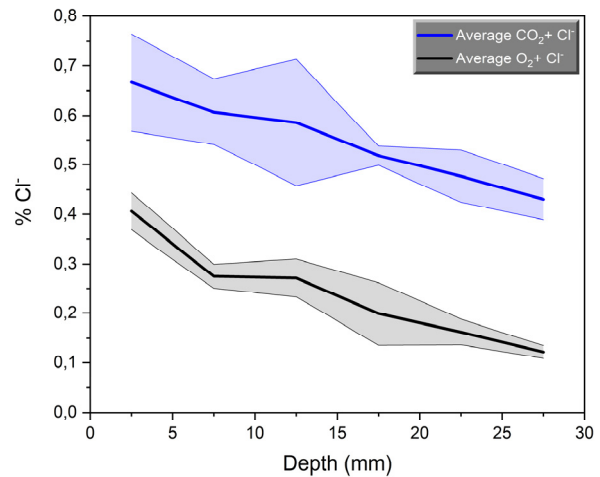
**Figure 8.** Free chloride profiles for specimens submitted to carbonation (or laboratory environment) for one month.

From Figure 7, it can be seen an expected behaviour for total chlorides profile in the reference group, that is, high chloride ions content in the superficial zone and a sharp drop in the bulk zone. Regarding the combined group, it can be seen a gradual reduction of chloride content across the profile. Furthermore, there is an expressive reduction in the chloride ions content of the initial zone (between 0 and 15 mm) when compared to the reference group. However, the bulk zone (between 15 and 30 mm) shows values close to the reference group, but slightly higher.

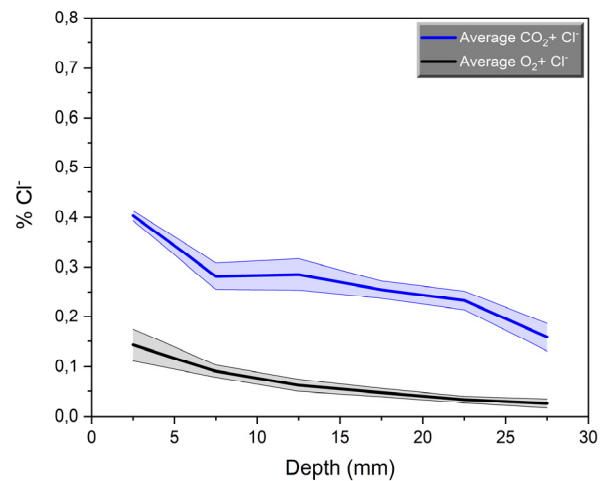
From Figure 8, it can be seen that free chlorides profile in reference group shows similar behaviour to total chlorides profile in the same situation. Regarding the combined group, there is a reduction in the chloride ions content of the initial zone (between 0 and 15 mm) when compared to the

reference group. However, it is important to observe a stable behaviour in the free chloride profile related to the combined group. The values are close to 0.1% Cl<sup>-</sup> across the profile.

In the Figures 9 and 10 it is possible to see the total and free chloride profiles, respectively, for specimens submitted to carbonation (or laboratory environment) during seven months and to chlorides during three months.



**Figure 9.** Total chloride profiles for specimens submitted to carbonation (or laboratory environment) for seven months.



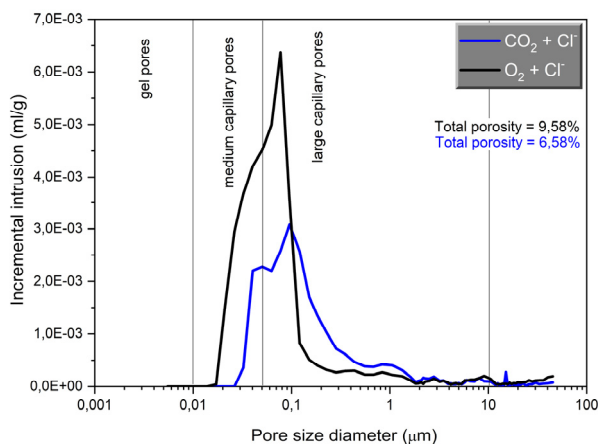
**Figure 10.** Free chloride profiles for specimens submitted to carbonation (or laboratory environment) for seven months.

When there is an increase in the exposure time (and a consequent increase in the carbonation zone), the chloride transport behaviour is modified. From Figures 9 and 10, it is clear that carbonation influences chlorides transport into concrete specimens. In this case, there is an important increase in total and free chlorides content throughout the entire combined profiles when being compared to the reference profiles. This increase happens regardless of the carbonation degree (total, partially or non-carbonated zone) and is accentuated with the increase in the depth. Regarding the total chlorides content, the increase varies between 1.6 (2.5 mm) and 3.5 times (27.5 mm). Regarding the free chlorides content, the increase is more noticeable and varies between 2.8 (2.5 mm) and seven times (22.5 mm).



Observing Figures 7 to 10, it is possible to say that carbonation influences chlorides transport in different ways. For superficial carbonation (Figures 7 and 8), there is a reduction in the chlorides content when compared to the reference group. To deeper carbonated zone (Figures 9 and 10), there is an increase in the chlorides content when compared to reference group.

The reduction in the chloride contents observed in Figures 7 and 8 can be caused by the probably decrease in the total porosity of superficial concrete, which is a consequence of the transformation of  $\text{Ca}(\text{OH})_2$  into carbonates [15]. On the other hand, the carbonation cause a redistribution of pore sizes, increasing the proportion of large capillary pores [15]. To understand the carbonated region and its equivalent region in the reference group, the MIP was carried out, and the results are by the statements above. From Figure 11, it is possible to see a reduction in the medium capillary pores and a slight increase in the large capillary pores in the combined group when compared to the reference group.



**Figure 11.** Incremental mercury intrusion versus pore size obtained for the carbonated zone (and equivalent), after seven months of carbonation (or laboratory environment).

The increase in the chloride contents observed in Figures 9 and 10 can be related to two main reasons: the increase in the proportion of large capillary pores (Figure 11); the reduction in the chloride binding capacity, indicated by the rise in the number of free chlorides in the combined profile when compared to the reference profile (Figure 10). This reduction in the binding capacity is related to the decrease in pH (Figure 6) caused by the carbonation process. The combination of chlorides and tricalcium aluminate, forming Friedel's salt ( $3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{CaCl}_2\cdot 10\text{H}_2\text{O}$ ), plays a significant role in this context due to their ability to binding chlorides [30]. However, according [30–32], this salt is not stable at low pH levels and thus previous combined chlorides can be released and be transported deeper into concrete.

## 5 Conclusions

The interaction between carbonation and chlorides in concrete was experimentally evaluated in this research. The effect of carbonation on chloride ions transport in concrete was investigated using different accelerated tests and different exposure times. It should be noted that the

accelerated tests create their own dynamic, different from the real scenario. Therefore, the results should be interpreted with caution.

On the one hand, the combination using migration test shows that carbonation increases chloride transport regardless of the exposure time. On the other hand, the combination using immersion test indicates that there is a decrease in chloride ions transport when the specimens are carbonated for one month and a noticeable increase when carbonation period increases to 7 months.

Based on the obtained results, it is possible to conclude that, under the studied conditions, the carbonation has an important influence on the chloride ions transport. Given the physical and chemical effects provided by carbonation in the cement matrix, it can be said that, in environments contaminated with chlorides and subject to carbonation, two effects occur simultaneously: redistribution on the pore structure and reduction in the chloride binding capacity. The chlorides transport behaviour will be a result of the combination of these two effects that are related to the degree of carbonation.

This behaviour can provide different impacts depending on the concrete characteristics, namely those produced with a cement different from CEM I or with incorporation/addition of supplementary cementitious materials.

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