CHLORIDE PENETRATION IN CONCRETE UNDER COMPRESSION OR SPLITTING TENSILE LOAD REPRESENTING 60 - 65 PER CENT OF THE ULTIMATE LOAD

Hugo Egüez Alava [1,2], Nele De Belie [1], Geert De Schutter [1]

(1) Magnel Laboratory for Concrete Research, Ghent University, Belgium
(2) Escuela Superior Politécnica del Litoral, ESPOL, Guayaquil, Ecuador

Abstract

A critical loading ratio is reached at 50% of concrete ultimate load. Beyond this point the presence of micro cracks exerts a big influence on the chloride transport. For compression the strain distribution in the sample developed micro-cracking system that is more or less homogeneously distributed in the sample. Therefore it is expected the same chloride profile regardless of the position. When combined splitting tensile load and chloride attack are acting on concrete, the main micro-crack system is located on the loading plane. This contributes to regular and alike shaped chloride profiles if they are taken from planes that are kept parallel to the splitting one. Prismatic samples (100 x 100 x 400 mm) made of OPC binder were exposed to a 3% by weight sodium chloride solution while maintaining a loading ratio equal to 60 % of the maximum capacity. Cubic samples (side = 150 mm) made of OPC and 50% BFS had similar chemical exposure while applying a permanent splitting tensile load corresponding to 65 % of their breaking capacity. After the exposure, ground layers were obtained from the samples to determine the total chloride ingress into the concrete by means of potentiometric titration and the effect of the load on the chloride ingress was assessed.

1. Introduction

One distinguishing feature presented by hardened concrete is its porosity. The nature of openings in cement paste depend on different conditions. For instance, the type of cement and binder along with their physical and chemical characteristics shape the porous nature of this material. Furthermore other conditions such as water/binder ratio, hydration time, curing temperature, and aggregate/paste ratio also outline the nature of this more or less changeable assembly. Micro-structure can be modified in time due to external factors of chemical and physical nature. Some of them can be considered aggressive and affect the durability of concrete. In contrast others help to improve the pore system. Concretes at field sites commonly have their voids partly occupied by solutions. Water containing salt can enter the

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concrete pores by capillary absorption. This process is also called convection. The penetration magnitude of convection will be governed by other material features for example the size of the pores, existence of cracks and initial saturation degree. Once harmful salts have gone into the pore system they can travel further inside through diffusion, the degree of salt ingress is captured in the so-called diffusion coefficient. It depends not only on voids volume and size and amount of saturation, but also on the physicochemical nature of the cement or binder gel and its interaction with the aggressive ions in solution. Movement of ions into concrete is also influenced by cycles of wetting and drying, pressure and temperature. These factors contribute to the complexity of ionic transportation into the concrete.

The volume of pores in concrete is always changing, sometimes due to an on-going paste hydration and another time it is modified by its deterioration. It also can be considerably modified by an applied load to the concrete. Consequently it is probable that the movement of deleterious ions into concrete by convection and diffusion may be altered by the type and magnitude of the load applied to it. Chloride movement produced by convection and diffusion is depressed when a slight compressive load is applied to concrete. Contrarily an opposite effect is observed when the applied load exceeds a certain limit. Usually when 30 or 50 % of the concrete's full compressive capability is surpassed, some micro cracks are created. These fresh pathways give an increase to the chloride intrusion. Under direct tensile load the rate of chloride infiltration surges gradually as the load is increased. While in splitting tensile loading, a critical stress has to be surpassed in order to increase the chloride transport into concrete.

The proposition of this research is not only to analyse the chloride ingress into loaded concrete from a physical point of view. Conversely the high chemical interaction that chloride ions have with cementitious binders it is well known. In order to compare these influences, concretes made of ordinary Portland cement (OPC), high sulfate-resistant cement (HSR) and OPC with 2 replacement levels of blast furnace slag (BFS) were exposed to a 3% sodium chloride solution while maintaining concrete specimens under permanent compressive and splitting tensile loads.

2. Experimental investigation

2.1 Materials and preparation of specimens

Five types of concrete mixes –S0, S50, S70, HSR, and S0-b– were prepared for the experiment. The mix composition are presented in table 1.

Aggregates consist of silica gravel and natural sand with a nominal maximum size of 16 mm. Three different binders were used. A Portland type I 52.5 cement with no special property for durability. Another type I 52.5 N cement but with high sulphate resistance (HSR) property. Blast furnace slag addition was utilized to replace part of the OPC in amounts of 50 and 70 % by weight. Characteristics of binders are shown in table 2.

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Table 1: Mix proportions of concrete.

Designation	w/b ratio	Water (kg/m^3)	CEM I (kg/m^3)	CEM I (HSR) (kg/m^3)	BFS (kg/m^3)	Sand (kg/m^3)	Gravel (kg/m^3)
S0	0.451	156	345	0	0	771	1085
S50	0.451	156	173	0	173	771	1084
S70	0.453	154	102	0	238	759	1067
HSR	0.451	157	0	347	0	774	1090
S0-b	0.450	166	369	0	0	840	1025

A high range water reducer (Polycarboxylate based) was employed in amounts between 0.2 – 0.4 % by weight of binder in order to plasticize the fresh mix to obtain a slump between 160 – 210 mm before casting the samples.

Cubical samples of 150 mm side were moulded for the splitting test setups with mix designation S0, S50, S70, and HSR. The specimens were stored after casting for 7 days at 20 ± 2 ℃ and RH>95%, the cubes were then demoulded and stored for an additional 21 days at 20 ± 2℃ and 60 ± 5% RH.

Table 2: Binder properties

Ingredient	CEM I 52.5 N	CEM I 52.5 N HSR	BFS
	Mineral phase (%)		
C_3S	57.49	57.71	-
C_2S	10.81	18.45	-
C_4AF	13.12	12.32	-
C_3A	7.90	2.50	-
Ingredient	Chemical composition (%)		
CaO	63.37	63.90	41.24
SiO_2	18.90	21.62	36.37
Al_2O_3	5.74	3.53	9.83
Fe_2O_3	4.31	4.05	0.26
SO_3	3.34	2.40	1.62
MgO	0.89	1.82	7.41

Concrete prisms of square section (100 x 100 mm^2) and a height of 400 mm were cast for the compression test setup. They were stored 28 days after demoulding in a moist chamber at 20 $\pm$ 2 ℃ and RH>95%.

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2.2 Experimental setup

For the splitting load setup 3 cubes representing each mix of S0, S50, S70, and HSR were tested at the age of 28 days. The strength properties of the materials as well as the corresponding load utilized for the experiment at 65% of their maximum capacity are shown in table 3.

Table 3: Average splitting loads for 3 cubes (150 mm side) and permanent applied load

Sample #	28 days Ave. max. Load kN	Var. Coeff. %	28 days Splitt. Strength MPa	Permanent load 65% of max. kN
S0	145.5	1.9	4.1	94.6
S50	142.5	4.9	4.0	92.6
S70	124.0	1.8	3.5	80.6
HSR	168.7	1.8	4.8	109.6

Before the salt plus load exposure 2 cubes representing each mix were vacuum saturated. Afterward the cube's faces were covered with black silicon plus aluminium foil leaving only two parallel opposite faces without this application. One of the faces will serve to attach one flexible reservoir of 640 cm^3 capacity which will contain a circulating 3% sodium chloride solution. On the other face, a similar capacity reservoir will be placed, but only with saturated $Ca(OH)_2$ solution whose function is to keep the sample saturated, provide calcium ions in case of leaching and to check for any unexpected chloride filtration.

The reservoir with the attacking salt solution is connected to an 8 litres stock solution tank provided with an aquarium pump with capacity to pump the solution at a flow rate of 23 l/h.

The cubes are subjected to a permanent splitting stress by 2 opposed and coaxially aligned plywood strips 160 mm in length, 25 mm wide and 3 mm of thickness and are placed in the middle axis of the loading face. The wooden strips rest between the metal plates of a mechanical rig and the loading faces of the cubes as shown in figure 1. The load is transferred by an hydraulic jack which is connected to a pressurized cylinder which contains a nitrogen/oil mixture.

For the compressive loading setup 2 prism were tested for uniaxial compression at the age of 28 days. The obtained average strength result is shown in table 4. Afterward 2 prism were loaded coaxially until reaching 60 % of the reference maximum load as shown in figure 2. The same type of loading rig was used as the one utilized for the splitting test setup. After the prisms were loaded they were exposed to a 3% sodium chloride solution. The salt solution was recirculated trough a rectangular section reservoir of 80 mm width by 160 mm length and 50 mm depth which was attached to the middle part of the exposed face. The solution was pumped from an eight liter capacity stock solution using an small aquarium pump.

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After certain time of the combined attack exposure the samples were taken out from their respective rigs. Subsequently they were subjected to profile grinding starting from the chloride exposed face.

The powder for chloride determination was obtained by a diamond drilling bit of 10 mm in diameter. The drilling head was fixed on a rotating vertical shaft. The sample was placed on an horizontal moving table capable of providing 2 controlled perpendicular displacements.

The sampling ground surface consist in a groove whose long axis is oriented paralell to the splitting plane or to the prism's axis This consist on an indentation 25 mm long, 10 mm wide and 1 – 2 – 3 or 4 mm depth. Usually the first layers close to the exposed surface were ground at 1 mm depth interval. Subsequently the depth's intervals were increased. After reaching the final depth, the obtained groove was cleaned with pressurized air and filled with epoxy, thus it can be used for further chloride exposure.

The sampling for the profiles exposed to compression were taken with the groove axis always oriented paralell to the prism axis at any location of the exposed surface. This was done considering the homogeneous strain distribution in that surface. On the contrary, the sampling for the splitting tensile exposure were taken at paralell planes located at 0 - 20 – 40 mm away from the splitting plane within the exposed surface.

The obtained powdered layers were treated with nitric acid to extract the total chloride content. The utilized procedure is described in EN 14629 "Determination of chloride content in hardened concrete". This was done by potentiometric titration with a 0.01 M silver nitrate solution.
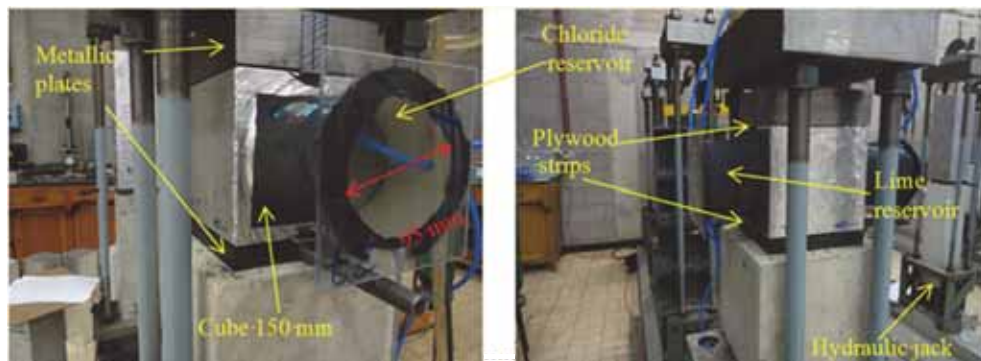

Figure 1.Setup for combined chloride attack plus splitting tensile loading.

Afterward the obtained chloride penetration profiles were analysed and an error function solution was applied to Fick's second law in order to obtain the apparent diffusion coefficient and the modelled chloride surface concentration.

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Table 3: Compressive loads for 2 prisms (100x100 mm, h = 400 mm) and permanent load

Sample #	Ave. Load (100%) kN	Var. Coeff. %	Compr. Strength MPa	60% of Max. kN
S0-b	568.9	1.1	56.9	341.4

Figure 2.Setup for combined chloride attack (2 prism), plus compressive loading.

2.3 Results and discussion

2.3.1 Micro crack observation for concretes under splitting tensile loading

In order to investigate the nature of micro cracking in concrete exposed to the combined attack, samples were taken from all concrete specimens for thin section microscopy. Special care was taken in order to obtain a rectangular section centered around the splitting plane and perpendicular to it as shown in figure 3 left.
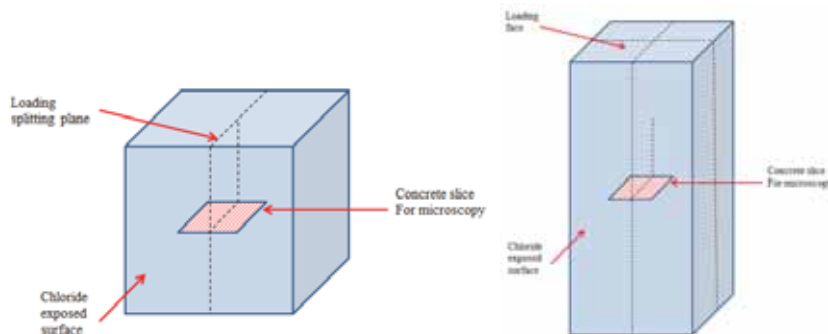

Figure 3. Schematic view of concrete slice extraction for microscopy (left splitting test, right compressive test).

The concrete slices of rectangular shape (width = 30 mm, depth = 50 mm) were then impregnated with fluorescent epoxy under vacuum and finally the sample was polished to obtain a 25 μm thickness of concrete for microscopic observation.

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Under the microscope a main crack oriented parallel to the splitting plane was observed as shown in figure 4. The crack width is variable and can reach an opening close to 100 µm at the surface but it narrows with concrete depth. The crack is typically seen to run along some smooth texture aggregates, it jumps from aggregate to aggegate traversing the paste and also fracturing some irregular shaped stones. This main crack may tend to subdivide into smaller branches as the depth increases. A very fine network of microcracks (crack width between 1 – 4 µm) was also noticed connected perpendiculary to this main crack shaft.
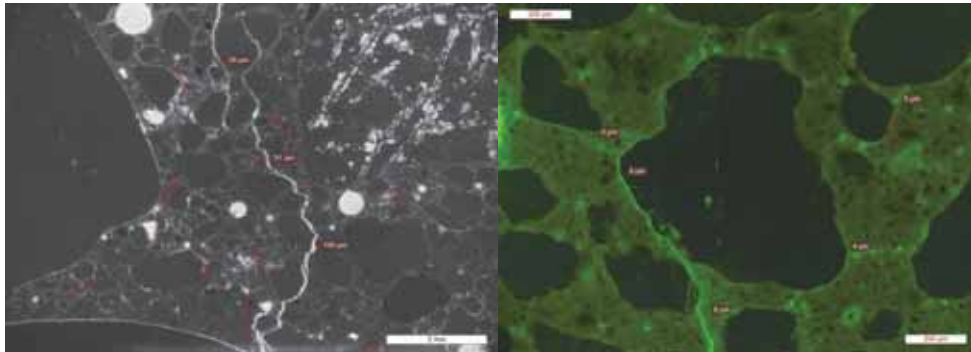

Figure 4. Micro cracking details under fluorescent light (grey tones left), showing fine crack network (red arrows) perpendicular to the main crack. Detail of micro cracking pattern (right). For S70 after 30 weeks chloride exposure.

The intensity of cracking seems to be different according to the binder type. For the pure Portland cements S0 and HSR the same cracking pattern was found, but in their case it seems that the developed micro-cracks are filled again by a self-healing product sometime after their formation. That is why in these 2 types of concretes, the crack interior appears somehow blurred. On the contrary the S50 and S70 types of concrete shown a very clean micro-cracked pattern where it is easy to verify even 1 µm sized microcracks.

2.3.2 Micro-crack observation for concrete under compressive load
A slice of concrete of similar dimension as the samples representing the splitting test was taken for microscopy observations. The sample was taken perpendicular to the exposed face as shown in figure 3.

Figure 5 shows the typical micro crack network and its orientation. Some of the cracks develop with a sub-parallel orientation forming a small angle (25 degrees approximately) with the exposed surface. Another set of micro-cracks intersect the latter forming an internal angle of 108° approximately. This pattern is not always constant and some irregular orientation has also been observed. The width of the micro-cracks is finer than the one observed in the splitting test. The widest showed an average width of 8 – 10 µm and they are reduced to reach 1 - 3 µm openings.

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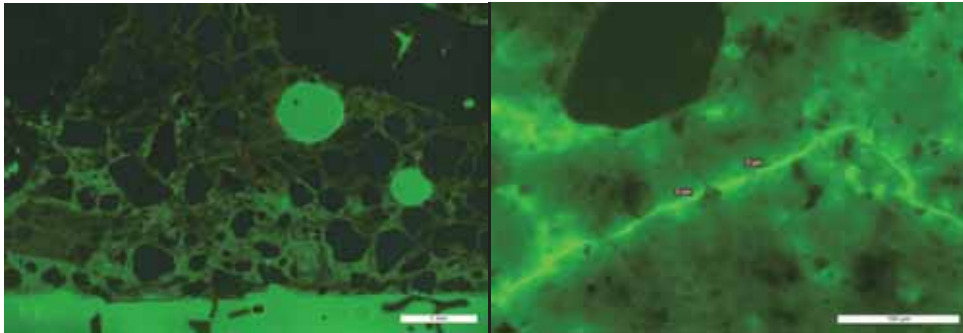22-24 August 2016, Technical University of Denmark, Lyngby, Denmark

Figure 5. Micro cracking details under fluorescent light, showing a fine crack network (red arrows). Detail of micro cracking pattern (right). For S0 under 60% compressive ratio and 34 weeks chloride exposure.

2.3.3 Obtained chloride profiles for concrete under compressive load

Chloride transport in concrete under compressive loading remains more or less homogeneous as long as a critical loading ratio is not surpassed. This critical loading is reached when micro cracking starts to be the main factor in chloride transport within the concrete. It is mostly agreed [1] that the critical loading ratio for concrete under compression is reached at nearly 50% of the ultimate or highest load. Beyond this point it is assumed that the micro cracking pattern starts to enhance the chloride transport within the concrete.

There has been some agreement about the lower limit for the micro crack size beyond which the chloride diffusion coefficient is increased. According to several researchers (Ismail 2004, Francois 2005, Djerbi 2008) this lower limit would be 30 µm, while others (Yoom et al 2014) (Maes M. 2015), place this critical limit at 12 and 10 µm respectively. Afterwards the diffusion coefficient is increased until an upper limit is reached and beyond that, the chloride diffusion in the crack is just like dispersion of chloride in plain water. The upper size limit has been set by (Ismail, Francois, Kato and Djerbi) in a range between 75 and 125 µm.

It would be very simple to point out that the penetration of chloride ions through a cement matrix can be explained only with the known model of Fick's second law. Chloride ions are chemically active, as is the hydrated cement paste. At the beginning chlorides penetrate through the outermost layers of concrete. These layers are usually enriched with cement paste. This zone is more porous and the dominant transport here is by convection. After the convection zone and in non cracked paste, chlorides leave the aggressive solution reacting chemically with hydrated cement products. They are also physically adsorbed on the huge specific surface of CSH that acts as filter for these ions, decelerating the chloride ingress.

The presence of microcracks alters the delicate balance and therefore the ability of healthy cement paste to delay chloride entrance. It has been observed in this study that the effect promoted by microcracking has similar characteristics as if the convection zone is gradually increased over time.

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Figure 6 shows a typical chloride penetration profile when concrete has been subjected to a combination of compressive stress and chloride solution exposure.
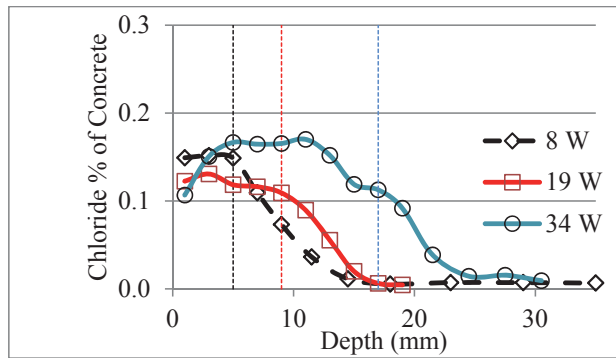

Figure 6. Acid soluble chloride content penetration after 8, 19 and 34 weeks, for S0-b exposed to 60% permanent compressive stress ratio and 3% chloride solution. Dashed vertical lines show the expansion of the convection zone (Δx) with time.

As seen in Figure 6, the layers obtained within the convection zone show a deviation towards lower values in the chloride content. Those points deviate from the normal fitting obtained at higher depths. The convection zone is a region with micro-cracks presence and where an aggressive solution is interacting with the paste. That provokes a reduction in the chloride adsorption capacity of CSH. Therefore unexpected low chloride points are seen in this region. Considering the movement of the convection zone the expression shown in equation (1) is proposed:

$$C(x,t) = C_i + (C_{s\Delta x} - C_i) * (1 - \text{erf}\left[\frac{x - \Delta x}{\sqrt{4Dt}}\right]) \tag{1}$$

Where $C(x,t)$ is the total chloride concentration at depth x ($x > \Delta x$) and exposure time t (s), Δx is depth of the convection zone (m), $C_{s\Delta x}$ is the surface chloride concentration (%) but measured at Δx, C_i is the initial chloride concentration (%) in the concrete and D is the apparent chloride diffusion coefficient (m^2/s) which is obtained by modelling the chloride profile in a given period.

Table 4: Depth of convection zone, modelled surface concentration (% by weight of concrete) and diffusion coefficient applying equation (1) to profiles from figure 6

Time	Δx	Surf. Conc.	D x 10^{12}	
weeks	mm	at Δx (%)	m^2/s	R^2
8	5	0.151	2.779	0.998
19	9	0.116	0.958	0.977
34	17	0.117	0.583	0.974

To characterize the chloride penetration in micro-cracked concrete, it is important to consider both parameters being the depth of the convection zone which is changing with time (Δx),

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and the difussion coefficient obtained at $x > \Delta x$. Table 4 shows the calculated parameters obtained from the chloride profiles of figure 6.

2.3.4 Obtained chloride profiles for concrete under splitting tensile loading

It is perceived from the petrographic analysis that the microcracking pattern provoked by the 65% splitting strenght ratio is more intense than that produced by the compressive test. This statement relates to the size and frequency of cracks found, which are larger and abundant in the indirect tensile test.

The shape of the chloride profile remains more or less alike as long as the ground profile is taken parallel to the splitting plane. Figure 7 shows the chloride profile obtained for S0 mix.
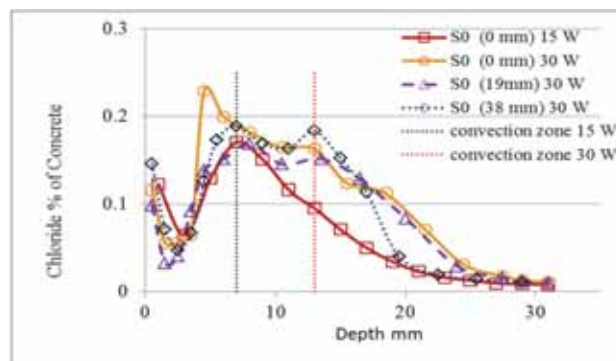

Figure 7. Total chloride content penetration profiles ground parallel to the loading axis (deviation distance from axis in parentheses), for S0 exposed to 65% permanent splitting tensile stress ratio and 3% chloride solution at 15 and 30 weeks. Dashed vertical line shows the location of the convection zone (Δx) with time.

Several researchers have noticed an inner higher peak in the chloride profile of concrete subjected to carbonation [2,3]; some also found this behaviour in concretes exposed to drying-wetting cycles [4]. Others have also noticed this conduct in cracked concrete [5]. In our case it is believed that microcrack formation promotes a higher ingress of chloride ions into this zone. Afterwards chlorides encourage calcium leaching from CSH sites and reduces the binding capacity (physical or chemical) of this gel. Probably it also provokes a lowering of the pore solution pH, a condition very similar to the effect of paste carbonation. In such condition the chloride content is lowered from the concrete surface towards inner zones until a lowest value is reached. The lowest value of the chloride content in our test is reached between 2-5 mm from the exposed surface. Subsequently, there is an increase in the chloride content until a higher peak is reached at inner depths. In some type of binders like S0 shown in Figure 7, two peaks are formed at 30 weeks of exposure and the chloride content forms a plateau between them. The second peak is located where the bulk condition is reached and normal difussion takes place afterwards. The more intense micro-cracking system found in splitting tensile exposure provoke more damage and lower binding capacity for chlorides. Consequently their obtained profiles were lower than those observed in compression and their difussivities were larger.

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To calculate the chloride penetration parameters for the splitting tensile tests, equation (1) is also employed. Table 5 presents the obtained parameters for the chloride profiles from S0 shown in Figure 7.

Table 5: Depth of convection zone, modelled surface concentration (% by weight of concrete) and diffusion coefficient applying equation (1) to profiles from figure 7

Time weeks	Axis dev. mm	Δx mm	Surf. Conc. at Δx (%)	$D \times 10^{12}$ m^2/s	R^2
15	0	7	0.176	4.009	0.995
30	0	13	0.166	2.154	0.969
30	19	13	0.142	2.569	0.925
30	38	13	0.193	0.868	0.976

3. Conclusions

When the considered concretes are subjected to sustained compressive and splitting tensile load levels of 60 and 65 % respectively of the maximum load capacity micro-cracks are formed. It was observed that the micro-crack width in compression ranged from 1 to 10 µm. In the other hand splitting tensile tests provoked a wider microcrack system located on the splitting plane and it can reach up to 100 µm in width. A thinner microcrack pattern of 1 to 10 µm width is connected perpendicularly to the main splitting micro-crack system.

Chloride ions ingress into the microcrack system and alter the properties of the gel and the pore solution. The penetration profile seems to be very similar to that found after chloride intrusion in carbonated concrete or under the effect introduced by repetitive cycles of wetting and drying in concrete. The principal observed features on the chloride profile consist firstly in a zone of low chloride binding capacity located close to the exposed surface. Next there is the presence of a chloride content peak which usually progresses in time to deeper zones. Finally there is the normal diffusion profile which develops afterward in areas where the physical-chemical damage has not yet altered the concrete.

Acknowledgements

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