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## EXPERIMENTAL STUDY ON CARBONATION AND CHLORIDE MIGRATION COEFFICIENTS OF SELF-COMPACTING CONCRETE (SCC) WITH BINARY AND TERNARY MIXTURES OF FLY ASH (FA) AND LIMESTONE FILLER (LF)

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### Abstract

This paper's goal is to present the results of an experimental study on the behaviour of SCC with binary and ternary mixtures incorporating high quantities of FA and LF regarding degradation mechanisms, namely carbonation and chloride migration.

Despite the requirements in terms of hardened state properties for SCC being the same as for conventional concrete (CC), the mix quantities used are different and that can substantially affect the SCC's durability, namely regarding in terms of carbonation and chloride penetration. It is therefore relevant to expand the knowledge on the way the aggressive agents mentioned diffuse or penetrate SCC.

For that purpose, a total of 11 SCC mixes were produced: 1 with cement only (C); 3 with C+FA in 30%, 60% and 70% substitution; 3 with C+LF in 30%, 60% and 70% substitution; 4 with C+FA+LF in combinations of 10-20%, 20-10%, 20-40% and 40-20% substitution respectively.

The resistance against CO<sub>2</sub> penetration into the SCC was tested by an accelerated laboratory test procedure described in specification LNEC-E391 and in recommendation RILEM CPC-18. The resistance to chloride ions migration was tested resorting to non-steady-state migration tests, according to specification LNEC-E463 based on NT-BUILD-492.

The results obtained indicate that the properties studied are strongly conditioned by the type and quantity of additions used. The use of ternary mixtures also proves to be extremely favourable, confirming a beneficial effect of the synergy between these additions.

**Keywords:** Self-compacting concrete; Durability; Carbonation; Chloride migration; Fly ash; Limestone filler.

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## 1 INTRODUCTION

According to report ACI 201.2R [1], the durability of concrete is defined by its capacity to resist the different types of degradation mechanisms, both of concrete itself and the reinforcements embedded in it. Reinforced concrete should maintain all the project requirements, namely its functionality, safety and aesthetics, without maintenance costs higher than initially predicted.

There are several factors that may alter the service time of a reinforced concrete structure and they do not depend exclusively on its intrinsic properties. There are numerous conditions, both of environmental exposure and in service, which may lead to the deterioration of reinforced concrete. In the reinforced concrete's deterioration process mentioned, it is possible to identify a set of degradation mechanisms that condition the behaviour of concrete and the set that causes reinforcement's corrosion. The specific concrete's deterioration processes can be subdivided in mechanical, physical, chemical and biological processes.

The mentioned concrete's deterioration processes, despite not acting directly on the reinforcement, have an indirect action on them because, as they deteriorate concrete, they allow an easier and faster penetration of the reinforcement's degradation agents. As for the specific corrosion processes of the reinforcement, they occur essentially by carbonation and by chloride penetration that, despite not attacking directly the cover concrete, act on the reinforcement's passive protection layer by decreasing its alkalinity.

Regarding SCC's carbonation, it is possible to find, in the publications consulted, different results. While some authors mention, for example, not having found significant differences in the carbonation depth between SCC and conventional concrete (CC) with the same mechanical resistance and for the same exposure period, other authors present penetration values, for SCC, up to 40% below those of equivalent CC [2]. Like the remaining durability properties of SCC, the results available about chloride penetration are scarce and sometimes somehow contradictory in what concerns essentially the factors (in terms of SCC composition) that influence the referred chloride penetration [2; 3].

Therefore, this paper intends to evaluate the influence of the use of FA and LF on the degradation mechanisms carbonation and chlorides diffusion coefficient in binary and ternary SCC mixes at three ages (28, 91 and 182 days) resorting to specification LNEC-E391 and recommendation RILEM CPC-18 for the carbonation test and according to NT Build 492 and specification LNEC-E463 for the rapid chloride migration test.

## 2 EXPERIMENTAL PROGRAMME

### 2.1 Materials and mix proportions

The following materials were used: one type of cement complying with NP EN 197-1 (cement type I-42.5 R with specific gravity of 3.14; two mineral additions: fly ash (FA) complying with NP EN 450-1 and NP EN 450-2 with specific gravity of 2.30 and limestone filler (LF) complying with specification LNEC-E466 with specific gravity of 2.72; two limestone coarse aggregates complying with NP EN 12620, gravel 1 with specific gravity of 2.59,  $D_{max}$  of 11 mm and water absorption of 1.46% and gravel 2 with specific gravity of 2.64,  $D_{max}$  of 20 mm and water absorption of 0.78%; two siliceous sands complying with NP

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EN 12620, one coarse (0/4) with specific gravity of 2.55, fineness modulus of 3.70 and water absorption of 1.10% and one fine (0/1) with specific gravity of 2.58, fineness modulus of 2.03 and water absorption of 0.70%; a third-generation high-range water-reducing admixture ( $S_p$ ) complying with NP EN 934-1 and NP EN 934-2 (a modified polycarboxylic liquid admixture with a density of 1.07) and tap water complying with NP EN 1008.

With the goal of scoping all variants of contents used in the mixes and the corresponding analysis of the binary and ternary mixes of FA and LF, 11 SCC mixes were produced according to the NP EN 206-9. These data is shown in Table. 1.

Table 1: Mix proportions and basic properties of SCC

Mixes		SCC1.100C	SCC2.30LF	SCC2.60LF	SCC2.70LF	SCC3.30FA	SCC3.60FA	SCC3.70FA	SCC4.10FA20LF	SCC4.20FA10LF	SCC5.20FA40LF	SCC5.40FA20LF
CEM I 42.5 R	[kg/m <sup>3</sup> ]	707	512	297	222	503	290	218	506	506	297	293
Fly ash	[kg/m <sup>3</sup> ]	---	---	---	---	158	318	373	53	106	109	215
Limestone filler	[kg/m <sup>3</sup> ]	---	190	386	449	---	---	---	125	63	257	127
Superplasticizer	[kg/m <sup>3</sup> ]	7	5	3	3	5	4	3	5	5	3	3
Water	[l/m <sup>3</sup> ]	189	175	168	170	183	180	178	180	180	168	175
Fine aggregate 0/1	[kg/m <sup>3</sup> ]	436	450	457	456	443	447	448	446	446	457	451
Fine aggregate 0/4	[kg/m <sup>3</sup> ]	287	297	301	300	292	294	295	294	294	301	297
Coarse aggregate 1	[kg/m <sup>3</sup> ]	417	417	417	417	417	417	417	417	417	417	417
Coarse aggregate 2	[kg/m <sup>3</sup> ]	283	283	283	283	283	283	283	283	283	283	283
W/C	[-]	0.27	0.34	0.57	0.76	0.36	0.62	0.82	0.36	0.36	0.57	0.60
W/CM	[-]	0.27	0.34	0.57	0.76	0.28	0.30	0.30	0.32	0.29	0.41	0.35
W/FM	[-]	0.27	0.25	0.25	0.25	0.28	0.30	0.30	0.26	0.27	0.25	0.28
Basic properties												
Slump flow	[mm]	770	710	710	680	680	670	660	780	740	690	650
V-funnel	[s]	9.3	10.3	9.1	9.9	7.3	8.4	8.6	9.3	10.8	9.1	10.0
L-box	[-]	0.91	0.89	0.85	0.82	0.84	0.81	0.79	0.91	0.90	0.89	0.83
$f_{cm,28d}$	[MPa]	83.6	70.1	42.3	30.5	68.4	54.0	35.3	63.4	70.9	47.8	49.1
$f_{cm,91d}$	[MPa]	85.5	70.0	42.8	32.6	71.7	62.5	48.9	70.4	75.8	57.9	56.9
$f_{cm,182d}$	[MPa]	88.2	74.1	49.2	35.5	69.5	59.9	49.6	71.1	74.7	59.9	55.9

In order to evaluate only the change in the unit substitution ratios of cement by mineral additions ( $f_{ad}$  by volume), the following conditions were taken into account: the volumetric ratio between mortar and coarse aggregates' content ( $V_m/V_g=2.625$ ), as well as the absolute volumes of coarse aggregate ( $V_g=0.268 \text{ m}^3/\text{m}^3$ ) and mortar ( $V_m=0.702 \text{ m}^3/\text{m}^3$ ), were kept constant; the volumetric ratio between the total powder content, cement and mineral additions, and fine aggregates in the mix ( $V_p/V_s=0.80$ ) was kept constant; the volumetric ratio between water and fine material content in the mix ( $V_w/V_p$ ), as well as the percentile ratio in mass between the high-range water reducing admixture ( $S_p$ ) and the fine material content ( $S_p/p\%$ ), varied depending on the need for water and  $S_p$  of each mix in order to obtain the self-compacity parameters according to the works of Nepomuceno and Oliveira [4] and Silva et al. [5].

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## 2.2 Test methods and sample preparation

The specification LNEC-E391 and the recommendation RILEM CPC-18 were used to determine the resistance to carbonation. For each SCC reference, a 100 mm diameter and approximately 50 mm thickness sample was tested, in each of the four ages (7, 28, 91 and 182 days of exposure in the CO<sub>2</sub> chamber). All the moulds tested were subjected to a process of humid cure by immersion in water at 20 ± 2 °C until 14 days prior to the start of the test, i.e., the entry into the carbonation chamber. At that time (14 days), the moulds were sectioned and its tops were protected with an insulating coating. Then, the moulds were conditioned at 20 ± 2 °C and 50% of RH, until entering the carbonation chamber (28 days). At that time, the moulds were placed in a chamber with 5±1% of CO<sub>2</sub>, 60±5% of RH and a temperature of 23±3 °C. With 7, 28, 91 and 182 days of exposure to the conditions mentioned, the moulds were removed from the chamber, sectioned and, using a colorimetric method (with phenolphthalein at 0.1%), a measure of the carbonation depth was made.

The test procedure used to determine the chloride diffusion coefficient was the one described in NT Build 492 and specification LNEC-E463. For that purpose, three cylindrical moulds with 100 mm diameter and 50 mm height were used, for each mix and age studied. After the adequate time of wet curing (climate room at 20 °C ± 2 °C and more than 90 % RH), the moulds were pre-conditioned in vacuum and immersed in a saturated calcium hydroxide (Ca(OH)<sub>2</sub>) solution in distilled water. Afterwards, the moulds were connected to a rubber sleeve in order to contain the anodic solution of sodium hydroxide in distilled water (NaOH 0.3 N), without chlorides. The rubber sleeve, with the mould properly sealed according to NT Build 492 and specification LNEC-E463, was inserted in the sodium chloride catholyte solution (at 10% in NaCl mass in tap water). Then, through the stainless steel plaques on the mould's tops, an electric potential (according to the specification in point 6.4.4 of NT Build 492 or LNEC-E463) was applied, which forced the transportation by migration of the chloride ions through the mould. After a given test period, the mould was broken by being axially split and the rectangular sections obtained were sprayed with a silver nitrate solution (AgNO<sub>3</sub> 0.1 N). From the visible white silver chloride precipitation, the penetration depth was measured. Based on the penetration depth measurement performed as well as in other parameters, it was possible to calculate the chloride (D<sub>nssm</sub>) diffusion coefficient in non-stationary regime from the simplified equation (1):

$$D_{nssm} = \frac{0.0239 (273 + T)L}{(U - 2)t} \left( x_d - 0.0238 \sqrt{\frac{(273 + T)Lx_d}{U - 2}} \right) \quad (1)$$

D<sub>nssm</sub> = non-steady-state migration coefficient (x 10<sup>-12</sup> m<sup>2</sup>/s); U = absolute value of the applied voltage (V); T = average value of the initial and final temperatures in the anolyte solution (°C); L = thickness of the specimen (mm); x<sub>d</sub> = average value of the penetration depths (mm); t = test duration (hours).

## 3 TEST RESULTS AND DISCUSSION

### 3.1 Carbonation test

In a first analysis of the results on carbonation depth showed in Table 2, one can conclude that, for the SCC mixes with values of f<sub>ad</sub> up to 30%, no carbonation effect is observed during 6 months of exposure in the accelerated carbonation chamber. This happens independently of

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the type of additions used, that is, in the binary mixes with LF (SCC2), FA (SCC3) and  $f_{ad}$  of 30%, in the ternary mixes with global  $f_{ad}$  also of 30% and obviously in the SCC1 mixes (with cement only), being practically impenetrable to carbonation at the ages studied. For those mixes, no measurable carbonation was detected, at any of the ages studied and consequently the coefficient were set at a minimum value of 1 mm/year<sup>0.5</sup>.

Table 2: Depth of carbonation and the corresponding coefficients for all the mixes

Mix	$d_{km,7d}$ [mm]	S	$d_{km,28d}$ [mm]	S	$d_{km,91d}$ [mm]	S	$d_{km,182d}$ [mm]	S	$K_c$ [mm/year <sup>0.5</sup> ]	$R^2$
SCC1.100C	<1	-	<1	-	<1	-	<1	-	1.00	-
SCC2.30LF	<1	-	<1	-	<1	-	<1	-	1.00	-
SCC2.60LF	5	0.58	9	0.50	13	0.58	16	0.96	22.57	0.97
SCC2.70LF	10	0.50	19	0.50	28	1.00	48	0.50	64.23	0.97
SCC3.30FA	<1	-	<1	-	<1	-	<1	-	1.00	-
SCC3.60FA	5	0.58	10	0.50	11	1.15	16	1.26	20.78	0.91
SCC3.70FA	9	0.58	14	0.82	18	0.50	32	2.63	40.89	0.94
SCC4.10FA20LF	<1	-	<1	-	<1	-	<1	-	1.00	-
SCC4.20FA10LF	<1	-	<1	-	<1	-	<1	-	1.00	-
SCC5.20FA40LF	6	0.50	13	0.96	23	1.50	26	2.71	37.96	0.96
SCC5.40FA20LF	5	0.50	12	1.15	21	2.22	24	0.82	35.53	0.97

The remaining mixes show a significantly distinct behaviour, with carbonation right at 7 days of exposure in the accelerated carbonation chamber. Analysing the values shown in Table 2, it is possible to see that the binary mixes with  $f_{ad}$  of 60%, independently of the addition used, are those that show greater resistance to carbonation, with average depths, after 6 months of exposure, between 16 mm and 20 mm. As for the binary mixes with  $f_{ad}$  of 70%, one can see greater penetrations than those with  $f_{ad}$  of 60% and with a greater increase between 3 and 6 months of exposure in the accelerated carbonation chamber. Nevertheless, these mixes show a clear difference in the resistance to carbonation between the SCC2 (with LF) and the SCC3 (with FA). In the case of the first, with LF (SCC2), the depth varies between 47 and 48 mm and in the seconds, with FA (SCC3), varies between 31 and 32 mm, all at 6 months of exposure.

The ternary mixes with global  $f_{ad}$  of 60% show a resistance to carbonation slightly lower comparing to the binary mixes with the same  $f_{ad}$ . Those mixes (SCC5) obtain a carbonation depth, after 6 months of exposure, between 24 mm and 26 mm, that is, 6 mm to 7 mm greater than the corresponding binary mixes.

The carbonation of concrete can also be quantified by the corresponding coefficient, by analysing the graphs of the depth of carbonation *versus* time, by the expression  $x=k\sqrt{t}$  ( $x=kt^{(1/n)}$  with  $n=2$ ). By analysing the graphs shown in Figure 1, one can find that the variation of the carbonation depth as time elapses is adequately characterised by the expression mentioned, showing reasonable correlation coefficients, always greater than 0.9.

As found by other authors, e.g. Neville [6], Bertolini et al. [7] or Siddique [8], it is possible to conclude, in our work, for the mixes with W/C ratio lower than approximately 0.36 (namely, SCC1, W/C=0.27; SCC2.30LF, W/C=0.34; SCC3.30FA and SCC4 W/C=0.27), that the carbonation was negligible, considering an exposure of up to 6 months.

From the analysis of the results obtained, it is possible to state that, in the case of the SCC



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produced with a lower W/C ratio, in general terms, its greater compacity creates an almost impenetrable barrier to CO<sub>2</sub>, significantly delaying its penetration. To this high compacity mixes is usually associated a low porosity accessible to water (essentially capillary pores), which represents the main route for CO<sub>2</sub> penetration. As confirmed in Silva and de Brito [9; 10] with the results of water absorption by immersion and capillary absorption, these are the mixes (SCC1, SCC2.30LF, SCC3.30FA and SCC4) with the lowest accessible porosity values and also the lowest capillary coefficients.

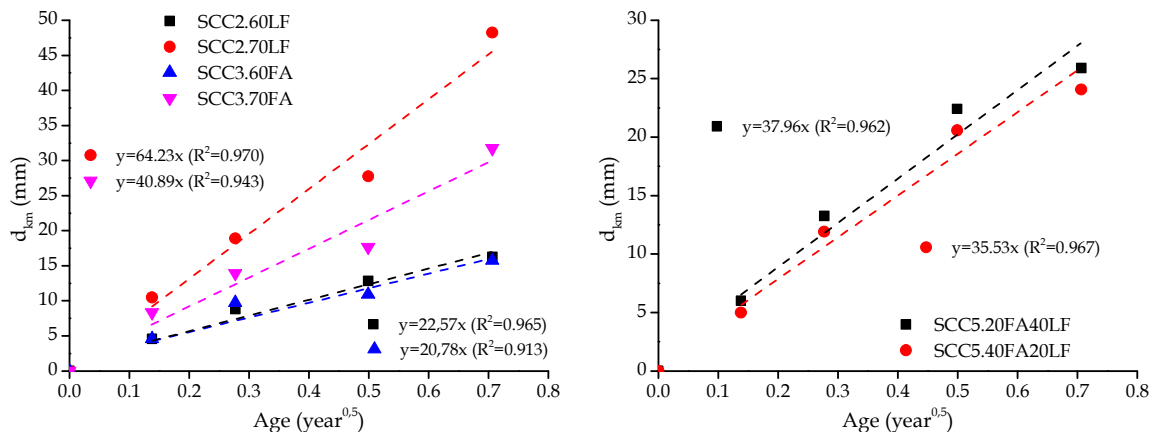


Figure 1: Depths of carbonation as function of the  $\sqrt{t}$  for all the mixes

## 3.2 Chloride migration test

In a first analysis of the results obtained, chlorides penetration resistance is strongly conditioned by the use of mineral additions in the SCC production and by their type. This can be observed in the values for SCC3 with FA, which are significantly lower when compared to those for SCC2 with LF. These results confirm the ones obtained, for example, by Zhu and Bartos [11] and Audenaert et al. [12]. Comparing our results with those of the authors mentioned, one should highlight that the use of FA may result in a higher resistance to chlorides penetration when compared to the concrete produced only with C or to concrete produced with C + LF. Zhu and Bartos [11] state that the increase of resistance to chloride penetration due to the incorporation of FA observed may be the result of the chloride ions passage by diffusion being more difficult or even blocked, since the FA particles, which are rounder, significantly contribute to a higher compacity of the particles, both of the SCC paste matrix and of the ITZ itself around the coarser aggregates.

Regarding the  $D_{nssm}$  variation with age and the  $f_{ad}$  value, it is observed that those differences are minimal and decrease significantly with age, i.e. for all the values of  $f_{ad}$  the variation from 91 days to 182 days is negligible.

The same is not true for the SCC3 with FA. For these mixes, a small decrease is observed in the value of  $D_{nssm}$  with the increase of  $f_{ad}$  to 60% at 91 days. That value tends to stabilize, mainly for values of  $f_{ad}$  of 60% at 182 days. For  $f_{ad}$  of 70%, the value of  $D_{nssm}$  tends to increase despite being still significantly below the one obtained for the SCC2 with LF.

Interpreting the data in Figure 2, there is a consistent coherence when compared to the variation of the  $D_{nssm}$  with the tests age as well as with the  $f_{ad}$  used. One should also mention the coherence with the results obtained by the other authors mentioned. The best behaviour is

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that of the mix with 30% of FA (SCC3.30FA) at all ages when compared to all the mixes produced (except for SCC2.60FA which at 91 days shows a slightly lower  $D_{nssm}$ ).

From the analysis of Figure 2, it is observed that the ternary mixes show a very satisfactory behaviour when compared to the remaining mixes. One should highlight the behaviour shown at the first ages by SCC5.40FA20LF with a  $D_{nssm}$  lower by 27.6% than SCC3.60FA (both with total  $f_{ad}$  of 60%). That difference fades as age increases, nevertheless maintaining small values of the migration coefficients for both SCC. The ternary mix SCC4.20FA10LF (with total  $f_{ad}$  of 30%) shows, at 28 days, a value for  $D_{nssm}$  very close to that of SCC3.30FA, with a difference between them of approximately 3.3%. At 182 days, the difference mentioned increases to approximately 50%, nevertheless still remaining the lowest value of the ternary mixes at that age.

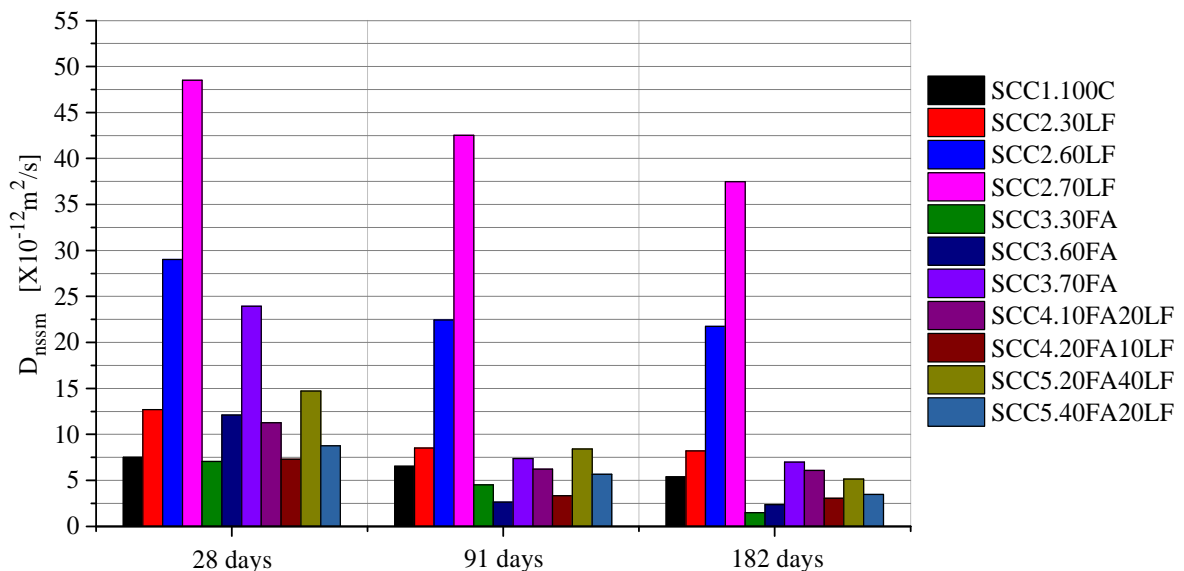


Figure 2: Results of the non-steady-state migration coefficient

## 4 CONCLUSIONS

This study showed that for SCC mixes with  $f_{ad}$  values of up to 30%, one cannot observe any carbonation effect, for 6 months of exposure in the accelerated carbonation chamber. This occurs independently of the type of additions used, i.e. in the binary mixes with LF (SCC2), FA (SCC3) and  $f_{ad}$  of 30%, in the ternary mixes with the same global  $f_{ad}$  of and obviously in the SCC1 mixes (cement only), revealing to be practically impenetrable to carbonation at the ages studied.

The variations in carbonation depth with the value of the W/C ratio observed in this work agree with what was found by other authors, namely that, for the mixes with W/C ratios lower than approximately 0.36, carbonation was negligible, for an exposure period of up to 6 months.

The chloride migration coefficient is strongly influenced by the amount and type of additions used, as well as by the test age of concrete. The use of FA in partial substitution of C reduces the chloride migration coefficient essentially due to the improvement of the concrete's permeability. If, on the one hand, the use of FA causes an increase in the concrete's porosity in the hardened state at younger ages, it also causes a reduction in the average pores

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diameter with consequences such as a decrease in the concrete's permeability and a denser ITZ, making it less susceptible to the penetration of chlorides.

The ternary mixes show very satisfactory results when compared to the remaining mixes. The behaviour of the mix with  $f_{ad}$  of 20% FA and 10% of LF should be highlighted. The incorporation of FA in the ternary mixes turned to be very beneficial to the behaviour of those mixes concerning the penetration of chlorides.

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