

Resistance to chloride-ion penetration of different concrete mixtures subjected to long-term immersion tests

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Abstract

This paper aims to characterize and evaluate the chloride penetration in concretes produced with different types of aggregates and binders, as well as to analyze the adequacy of using the chloride diffusion coefficient, determined from laboratory accelerated tests, to characterize the durability of these concretes when exposed in a saline submerged environment.

To do this, a vast experimental campaign was carried out, involving the production of different types of concrete and their characterization in terms of resistance to chloride penetration, based on accelerated migration tests and semi long-term immersion tests. Three types of aggregate of different density (limestone, Leca and Stalite) and three types of addition (silica fume, fly ash and limestone filler) at varying replacement percentages, were used in the concrete production.

In general, it can be confirmed that the main factor ruling the diffusion and penetration of chlorides was the quality of the cement paste (water/binder ratio). The incorporation of LWA did not significantly influence the diffusion coefficient, but in the concretes with more porous LWA a decrease in chloride penetration resistance occurred. The immersion test was able to characterize the potential contribution of the pozzolanic additions in the chloride penetration resistance and is therefore the more appropriate test to simulate reality. Furthermore, it can be concluded that the use of the RCMT test for the characterization of the resistance to chloride penetration into concrete must be done separately, depending on the type of binder and, eventually, the type of aggregate.

1. Introduction

Among the various existing degradation mechanisms, chloride-induced reinforcement corrosion is considered one of the main causes of degradation of reinforced concrete, causing a reduction in its service life. The attack of the reinforcements by this type of ion promotes an increase of its corrosion rate, leading to important losses in structural elements resistance.

Concrete durability is evaluated by the performance of the constructions or from laboratory tests that simulate the degradation of the concrete. In the case of resistance to chloride attack, the concretes have been analyzed based on laboratory tests that simulate the development of their actions, or indirectly from tests that aim to characterize the microstructure of the concrete ([1], [2]). Several methods have been developed and can be classified according to the driving force responsible for transporting chlorides in the concrete (diffusion or migration) and depending on the variation of the chloride concentration in the concrete during the test (steady state or non-steady state). In recent years, the rapid chloride migration test, developed by Luping [3], has been adopted in several concrete characterization studies to evaluate its resistance to chloride penetration ([4] [5] [3] [6] [7]). The normative documents fib 34 [8] and specification LNEC E465 [9] adopt this test in their suggested methodology for the prediction of concrete's service life in real environment. According to Chlortest [10], Luping's test is considered the best accelerated laboratory test to qualify the durability of concrete in relation to chloride attack.

However, laboratory tests (generally accelerated) have limitations when it comes to evaluating the concrete's performance if inserted in real structures, as laboratory tests cannot cover all phenomena that govern its degradation. Particularly, the Rapid Chloride Migration Test (RCMT) is based solely on the expeditious determination of the diffusion coefficient, which is not the only

parameter responsible for the penetration of chlorides in the concrete. Therefore, it's important to assess the capability of the laboratory tests in the characterization of the concrete durability, under non-accelerated exposure conditions, namely considering different exposure environments, different concrete compositions and types of binders.

2. Experimental program

2.1. Materials and methods

The experimental campaign involved the production of numerous NWC and LWAC with several water/binder (w/b) ratios (0.35, 0.45 and 0.55) and additions. To produce the mixtures, two types of LWA were used: Leca (expanded clay aggregate) and Stalite (expanded slate aggregate). Normal weight aggregates (NWA) consisted of crushed limestone composed by coarse gravel 1 (CG), coarse gravel 2 (CG2) and fine gravel (FG), and natural silica sand, composed of fine sand (FS) and coarse sand (CS). The aggregates physical properties are summarized in Table 1. In addition, cement type CEM I 42,5R and superplasticizer, when needed, were also used to produce the mixtures. The additions used to improve concrete properties were fly ash (FA), silica fume (SF) and limestone filler (LF).

Table 1 - Aggregate properties

Property	Normal weight aggregates					Lightweight aggregates	
	Fine sand	Coarse sand	Fine Gravel	Coarse gravel 1	Coarse gravel 2	Leca	Stalite
24h water absorption, $w_{abs,24h}$ (%)	0,19	0,26	0,73	0,35	1,05	16,28	3,57
Particle dry density, ρ_p (kg/m ³)	2605	2617	2646	2683	2618	969	1483
Loose bulk density, ρ_b (kg/m ³)	1569	1708	1309	1346	1325	632	760
Shape index	-	-	34(SI ₄₀)	20(SI ₂₀)	15(SI ₁₅)	1(SI ₁₅)	10(SI ₁₅)

2.2. Mix proportions and concrete mixing

In this study, the concrete compositions were chosen in order to cover a wide range in terms of density and compressive strength. As resumed in Table 2, several mixtures were produced from the different w/b ratios, aggregates and binders considered. The mixtures formulation was done considering two methods: Faury and the methodology proposed by Bogas [11], adapted to the specific case of LWAC.

The concrete was produced using a vertical axis mixer with bottom discharge. Firstly, the aggregates were introduced in the mixer and moistened for 3 minutes with 2/5 of the mixing water. Secondly, cement and additions, the remaining water and, when needed, the superplasticizer (for mixtures with 0,35 and 0,45 w/b ratio), were added to the mixture. Final mixture time was about 7 minutes long. Due to the high porosity of LWA, these are subjected to pre-saturation prior to the concrete production. This way, the water absorbed by the LWA during the concrete production was reduced and it was possible to better control the workability and effective water/cement ratio.

For the physical and mechanical characterization of concrete the following specimens were produced for each mix: three 150mm cubic specimens for compressive strength tests at 28 days, according to EN 12390-3 [12] and two 100mm cubic specimens for concrete dry density at 28

days. For the durability tests, the following specimens were also produced for each mix: three sawed $\phi 100 \times 50$ cylindrical specimens for the rapid migration test, according to LNEC E-463 [3] and one 150mm cubic specimen for the bulk diffusion test, according to LNEC E-390 [13].

Table 2 - Mix proportions

w/b ratio	Mixes	Aggregate	Type of binder	Addition (wt % of binder)	Cement content (kg/m ³)	Coarse aggregate (l/m ³)	Natural sand (l/m ³)		Effective water (l/m ³)	
							Fine	Coarse		
0,35	B35I	Natural	CEM I	0%	450	436	80	154	157,5	
	B35IIAD		CEM II/A-D	6%SF		433	80	153		
	B35IIAV		CEM II/A-V	15%FA		431	80	152		
	B35IIBV		CEM II/B-V	30%FA		419	72	164		
	B35IIAL		CEM II/A-L	15%LF		427	87	153		
	B35IIBL		CEM II/B-L	30%LF		424	80	159		
	S35I	Stalite	CEM I	0%	355	100	214			
	L35I	Leca	CEM I	0%	355	114	201			
	0,45	B45I	Natural	CEM I	0%	400	412	106	146	180
		B45IIAD		CEM II/A-D	6%SF		411	106	146	
B45IIAV		CEM II/A-V		15%FA	408		105	145		
B45IIBV		CEM II/B-V		30%FA	406		104	144		
B45IIAL		CEM II/A-L		15%LF	411		104	146		
B45IIBL		CEM II/B-L		30%LF	409		106	145		
S45I		Stalite	CEM I	0%	355	100	213			
L45I		Leca	CEM I	0%	355	126	186			
0,55		B55I	Natural	CEM I	0%	350	401	114	154	192,5
		B55IIAD		CEM II/A-D	6%SF		400	113	153	
	B55IIAV	CEM II/A-V		15%FA	399		106	159		
	B55IIBV	CEM II/B-V		30%FA	395		105	158		
	B55IIAL	CEM II/A-L		15%LF	400		107	160		
	B55IIBL	CEM II/B-L		30%LF	399		106	159		
	S55I	Stalite	CEM I	0%	355	114	201			
	L55I	Leca	CEM I	0%	355	134	181			

2.2.1. Curing process

The curing conditions were defined in accordance to the main normative followed for each studied property. The demoulding process occurred 24 hours after the concrete production. Subsequently, the specimens were placed under different curing conditions, depending on the test to which they would be subjected. The specimens for the compressive strength test were water cured until the age of testing (28 and 90 days). The specimens for the rapid migration test and immersion test were water cured during the first 7 days and then placed in a controlled chamber at a temperature of 22 ± 2 °C and relative humidity of $60 \pm 5\%$ until the age of testing.

2.3. Test methods

2.3.1. Rapid Chloride Migration Test

The non-steady state rapid chloride migration test was performed according to specification LNEC E-463 [3]. The test consisted of applying an external electrical potential during 24h, forcing the chloride ions to migrate into the specimen. In order to determine the specimen chloride penetration, a colorimetric method was used. This method consisted in half-splitting the specimen and spray both halves with a silver nitrate solution, which changed the concrete color in the presence of chloride ions. The specimen's chloride penetration resistance was characterized by the diffusion coefficient, $D_{Cl,RCMT}$, calculated according to Eq. (1), where T represents the average value of the initial and final temperature of the anodic solution (°C), L is the specimen's thickness

(mm), U is the absolute value of the applied voltage (V), t is the test duration (hours) and x_d is the average value of the measured chloride penetration depth (mm). Before the test, all specimens were saturated in a $\text{Ca}(\text{OH})_2$ solution.

$$D_{cl,RCMT} = \frac{0,0239 \cdot (273 + T) \cdot L}{(U - 2) \cdot t} \times \left(x_d - 0,0238 \cdot \sqrt{\frac{(273 + t) \cdot L \cdot x_d}{U - 2}} \right) \quad [x10^{-12} \frac{m^2}{s}] \quad (1)$$

2.3.2. Immersion Test in NaCl solution

The non-steady state immersion test was performed according to the specification LNEC E-390 [13]. The test basically consisted in submerging the specimens in a saturated solution of $\text{Ca}(\text{OH})_2$ and 15% NaCl, during 90 days. After the defined period, concrete samples were collected at various depths (0–2.5 mm, 2.5–7.5 mm, 7.5–12.5 mm, 12.5–17.5 mm, 17.5–22.5 mm and 22.5–27.5 mm) and chloride profiles were built. The concrete content at every depth was determined applying the potentiometric method. This method consisted of the following steps: attack of the collected concrete sample with a nitric acid solution; addition of a silver nitrate and iron ammonium sulfate solution and titration with potassium thiocyanate until a white precipitate was formed; lastly, the chloride content was quantified as a function of the volume of the titrated solution.

After this, the obtained chloride profiles were adjusted, by non-linear regression, to Fick's 2nd law (Eq.(2)) in order to obtain an estimate of D_{Cl} and c_s . In Eq.(2), $c(x,t)$ is the chloride content at depth x and after exposure time t (years), c_s is the surface chloride content(), D_{Cl} is the diffusion coefficient (m^2/s), $erfc$ is the complementary error function.

$$c(x, t) = c_s \cdot \left[erfc \left(\frac{x}{2 \cdot \sqrt{D_{cl} \cdot t}} \right) \right] \quad (2)$$

As the diffusion coefficient, D_{Cl} , may not be enough to properly characterize chloride penetration into concrete, it's relevant to calculate the penetration parameter K_c [$mm/year^{0.5}$]. This parameter is calculated through Eq. (3) and simultaneously combines the effect of D_{Cl} and c_s into one value, allowing a direct comparison of concrete compositions performances to chloride attack. In Eq.(3), c_r is the critical chloride content and was assumed to be 0,05% of binder weight (as suggested in NTbuild443) and c_i [% of binder weight] is the initial chloride content of the concrete.

$$K_{Cr} = 2 \cdot \sqrt{D_{cl} \cdot t} \cdot erf^{-1} \left(\frac{C_s - C_r}{C_s - C_i} \right) \quad (3)$$

3. Results and discussion

Slump, fresh density, ρ_f , dry density, ρ_d , compressive strength, f_{cm} (28 and 90 days), structural efficiency, f_{cm}/ρ_d , RCMT diffusion coefficient, $D_{Cl,RCMT}$, and immersion test diffusion coefficient, $D_{Cl,Im}$, surface chloride content, c_s , and penetration parameter, K_{Cr} , values are summarized on table 3, for each tested mixture.

Table 3 - Slump, fresh density, dry density, compressive strength, structural efficiency, RCMT diffusion coefficient and the immersion test diffusion coefficient, surface chloride content and penetration parameter measurements.

Mixes	Slump cm	ρ_f kg/m ³	ρ_d kg/m ³	$f_{c,28d}$ MPa	$f_{c,28d}/\rho_d$ x10 ³ .m	$f_{c,90d}$ MPa	$f_{c,90d}/\rho_d$ x10 ³ .m	$D_{Cl,RCMT}$ x10 ⁻¹² m ² /s	$D_{Cl,Im}$ x10 ⁻¹² m ² /s	c_s %wt binder	K_{Cr} mm/year ^{0.5}
B35I	13,2	2450	2334	76,99	3,3	79,78	3,4	9,97	2,32	2,3	15,9
B35IIAD	10,5	2390	2292	78,04	3,4	82,68	3,6	5,95	1,32	1,6	10,1
B35IIAV	14,5	2380	2300	63,18	2,7	78,32	3,4	9,73	1,29	3,1	13,3
B35IIBV	13	2330	2274	55,98	2,5	71,64	3,2	9,74	1,17	4,3	13,7
B35IIAL	10,5	2410	2308	61,98	2,7	69,82	3,0	12,03	3,67	2,3	19,9
B35IIBL	15	2400	2282	53,21	2,3	57,46	2,5	15,12	15,43	1,7	34,4
S35I	12	1940	1921	66,13	3,4	71,54	3,7	10,61	2,72	2,6	17,8
L35I	10	1910	1659	35,93	2,2	38,45	2,3	12,01	3,81	2,4	20,6
B45I	13,5	2390	2268	58,05	2,6	62,27	2,7	14,32	4,23	2,4	21,9
B45IIAD	12,5	2360	2220	51,48	2,3	59,68	2,7	9,02	2,68	3,3	19,7
B45IIAV	14,6	2380	2250	49,64	2,2	63,46	2,8	16,71	2,82	4,0	22,0
B45IIBV	14,4	2370	2249	44,58	2,0	62,88	2,8	18,51	1,25	4,4	14,6
B45IIAL	14,1	2370	2258	52,31	2,3	53,32	2,4	18,69	5,47	2,4	25,5
B45IIBL	14,2	2360	2236	40,76	1,8	43,60	2,9	-	23,34	2,4	55,2
S45I	13	1940	1825	51,14	2,8	55,44	3,0	11,82	4,16	2,2	20,7
L45I	12	1870	1622	30,27	1,9	34,61	2,1	13,65	6,98	3,4	31,8
B55I	15,8	2340	2227	47,67	2,1	53,08	2,4	16,96	4,74	3,4	27,8
B55IIAD	13,4	2330	2199	44,53	2,0	50,93	2,3	14,20	5,22	3,8	29,9
B45IIAV	18,8	2350	2232	38,63	1,7	52,14	2,3	23,34	5,78	4,4	32,4
B55IIBV	19,2	2360	2205	30,69	1,4	46,06	2,1	25,22	4,47	4,4	27,6
B55IIAL	16	2340	2232	40,76	1,8	42,39	1,9	-	14,29	3,5	48,0
B55IIBL	14	2330	2228	29,95	1,3	30,94	1,4	-	41,29	4,2	86,5
S55I	13,8	1930	1799	41,13	2,3	46,04	2,6	16,26	4,87	3,6	27,2
L55I	14	1830	1599	26,09	1,6	29,64	1,9	19,47	11,21	4,9	45,1

3.1. Compressive strength

The results suggest that the compressive strength in NWAC is strongly conditioned by the cement paste quality (i.e., by the w/b ratio of the composition). In LWAC, the compressive strength is no longer so influenced by this factor, since it is also conditioned by the resistant capacity of the LWA. NWAC obtained higher compressive strength results compared to LWAC. Even though Stalite specimens reported lower results compared to NWAC with same the composition, it should be noted that this kind of concrete obtained the highest structural efficiency of the test. This way, the results suggest that the LWA of Stalite are oriented to produce high strength concrete, while Leca are better suited to produce medium strength concrete. In terms of NWAC produced with additions, it's interesting to note the evolution of the compressive strength reported in the fly ash concretes. The evolution observed between the compressive strength tests, suggests that the pozzolanic activity in this kind of concrete only starts to be significant during that period.

3.2. Rapid chloride migration test

The diffusion coefficients obtained from the rapid chloride migration test varied between 5,95 and 25,22 x10⁻¹² m²/s. In the present work, after spraying silver nitrate solution in the sectioned halves (as the colorimetric method suggests), it was found that in three specimens the resulting whitish precipitate filled the whole sprayed area. This indicates that the chloride penetration front has completely passed through the test specimen and the diffusion coefficient is not possible to calculate. In Figure 1 the RCMT test results are resumed.

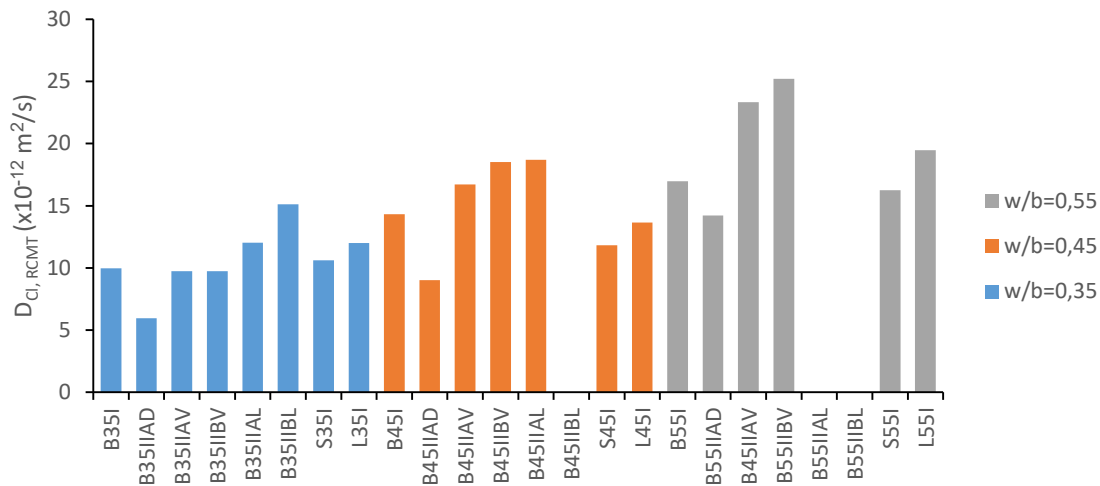


Figure 1 – Chloride diffusion coefficients obtained for all mixtures in RCMT test

The results show that the w/b ratio was the main factor conditioning the diffusion coefficients determined in the RCMT test. In another hand, it's also possible to verify that, in general, the aggregate type has a small influence in the results. Bogas [11] and Liu et al. [14] reported similar tendencies. However, on a more detailed analysis, Leca LWAC showed, on average, a slightly lower resistance to chlorides penetration when compared to the NWC of the same composition. Stalite LWAC reported similar diffusion properties similar as NWC.

Regarding NWC with additions, analyzing Figure 1 It's possible to conclude that silica fume was the only type of addition that produced an effective $D_{Cl, RCMT}$ reduction. It's also possible to note that this reduction is more relevant for lower w/b ratios, losing its efficiency for higher w/b ratios. As stated, this test is performed at 28 days. This way, pozzolanic reactions don't develop properly and fly ash particles tend to only have a physical effect, acting mainly as filler, failing to counteract the decrease in cement content and the consequent reduction of hydration products. This dilution effect of the amount of hydration products was also verified in NWC in which the cement was replaced by limestone filler. Therefore, the diffusion coefficient in fly ash and limestone filler concretes increased with the increase of the w/b ratio and percentage of substitution.

3.3. Immersion Test in NaCl Solution

The diffusion coefficients obtained from the immersion test (90 days) varied between 1,17 and 41,29 $\times 10^{-12}$ m²/s, the surface chloride content between 1,6 and 4,9 % of binder weight and the penetration parameter varied between 10,1 and 86,5 mm/year^{0,5}. In Figure 3 Figure 4 and Figure 5 these parameters are summarized.

A good fit of the obtained chloride profiles to the curves deduced by Fick's second law (Eq. (2)) was verified.

The fact that in all chloride profiles there is a progressive decrease of chloride concentration, shows that diffusion was the main mechanism of transport during the test.

Just like observed in the RCMT test, paste quality appeared to be the factor with the highest influence on chloride penetration.

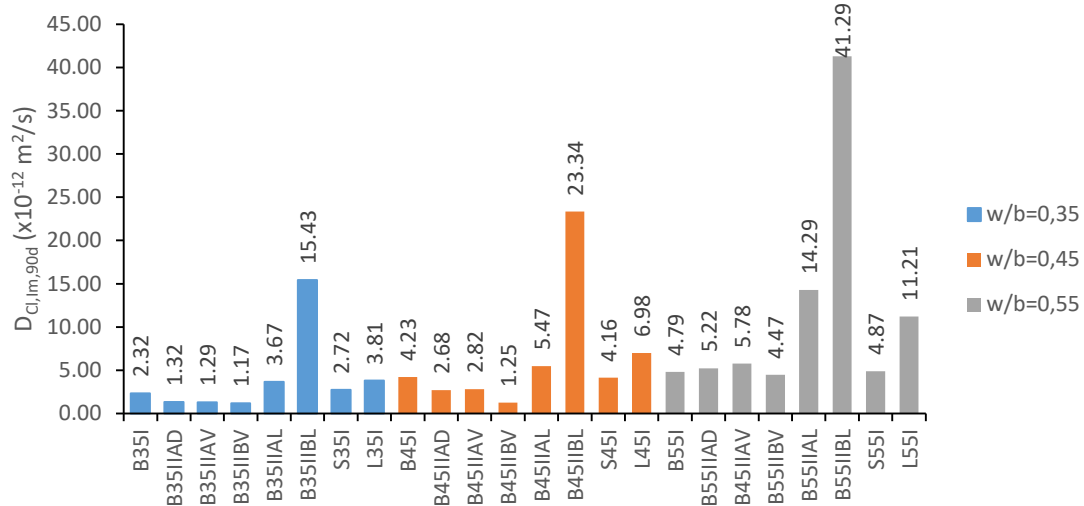


Figure 2 – Chloride diffusion coefficients obtained for all mixtures in the immersion test

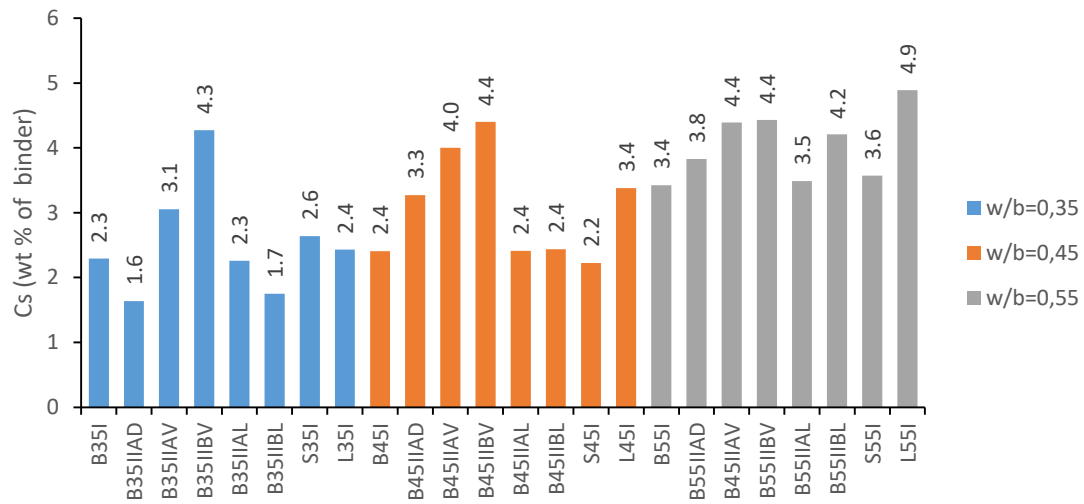


Figure 3 – Surface chloride content obtained for all mixtures in the immersion test

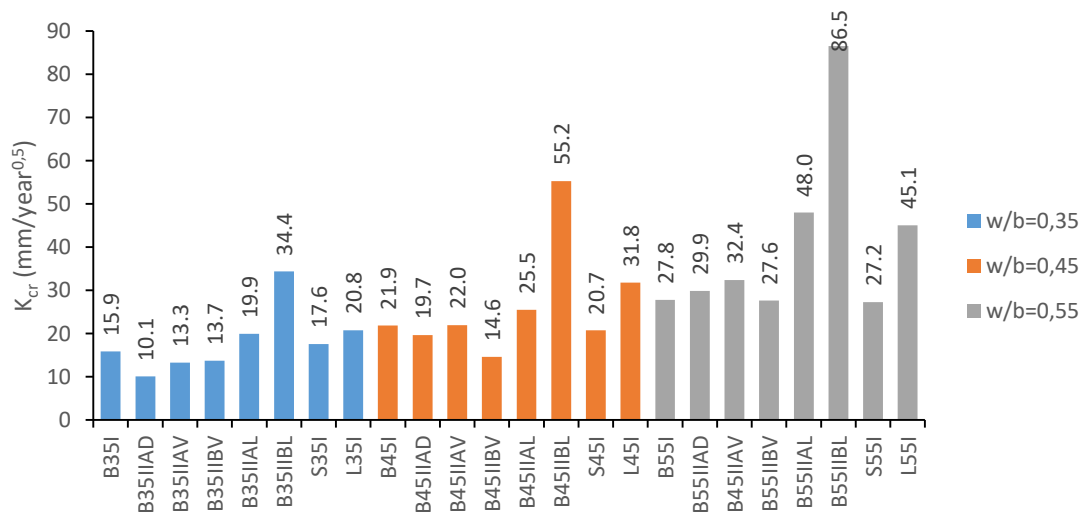


Figure 4 – Penetration parameters obtained for all mixtures in the immersion test

In relation to the type of aggregate, the mixtures with Leca showed lower resistance to penetration of chlorides, resulting in higher diffusion coefficients and surface chloride concentration. Contrary to what other authors reported ([15], [16]) participation of LWA in chloride diffusion was not negligible. To such, the fact that the immersion tests were performed under conditions in which the LWA were saturated, must have contributed. However, as verified by other authors in accelerated tests [17, 18], LWAC with Stalite, showed similar behavior to the NWC of the same composition. The higher porosity and diffusivity of LWA compared to NWA must have been compensated by the better quality of the transition zone, typically associated to LWAC. The surface chloride concentration increased with the increase of the aggregates porosity, especially in the concrete with higher w/b ratio. This tendency is attributed to the fact that porous aggregates function as sites for the accumulation of chlorides, increasing their concentration in the concrete surface areas, where they're more accessible.

Contrary to what was observed in the RCMT test, cement substitution by fly ash resulted in an increased chloride penetration resistance. The introduction of fly ash in the mixtures implied a significant decrease in the chloride diffusion coefficient, that allowed to compensate the increase of the surface chlorides concentration. Thus, comparing to NWC without additions, there was a reduction in the penetration parameter. It can also be observed that this decrease grew as the substitution percentage increased. The more advanced age at which the immersion tests were conducted and the more favorable conditions for chloride ions binding justify the different observed trends, compared to the migration tests.

Despite the difficulty of dispersing silica fume, the mixtures with 6% of these additions got the lowest diffusion coefficients (as the mixtures with fly ash) and the lowest chloride penetration parameters. These results justify the option of some Nordic countries to consider the incorporation of a minimum amount of silica fume in concrete subjected to severe marine environments [19]. As observed in the migration tests, the use of silica fume was less efficient in the concrete with higher w/b ratio. The surface concentration of chlorides was little affected by the incorporation of this type of addition.

The immersion tests confirmed the poor performance of the mixtures with limestone filler. As observed in the migration test, there was a significant increase in the chloride diffusion coefficient, regardless of the w/b ratio and substitution percentage.

3.4. Correlation between tests

Both migration and immersion tests were able to clearly distinguish the quality of the different types of concrete, allowing their classification in terms of performance. For this, contributed the fact that both tests were similarly affected by the variation of the w/b ratio.

Since there's a significant maturity disparity between the specimens, when the migration test and immersion test were carried out, implying therefore different chloride binding capacity conditions, there was a poor correlation (Figure 5) between the diffusion coefficients obtained from these tests, especially in mixtures with fly ash, where the pozzolanic reactivity is lower. The migration test, in the case of mixtures with fly ash, should be performed for ages greater than 28 days.

There was also a greater penalization of the chloride diffusion coefficient of concretes with more porous lightweight aggregates (Leca), compared to the results obtained in the migration tests. This should be due to the different saturation conditions of the aggregates in both tests. However, there was a high correlation between tests when some types of binder (CEM II/A-V, CEM II/B-V, CEM II/B-L and CEM I (Leca)) were not considered (Figure 6).

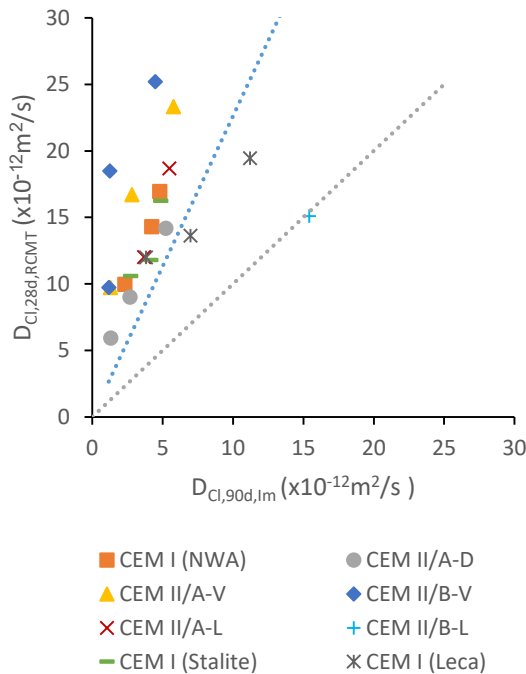


Figure 5 - Correlation between $D_{Cl,RCMT,28d}$ and $D_{Cl,Im,90d}$ (considering all mixtures tested)

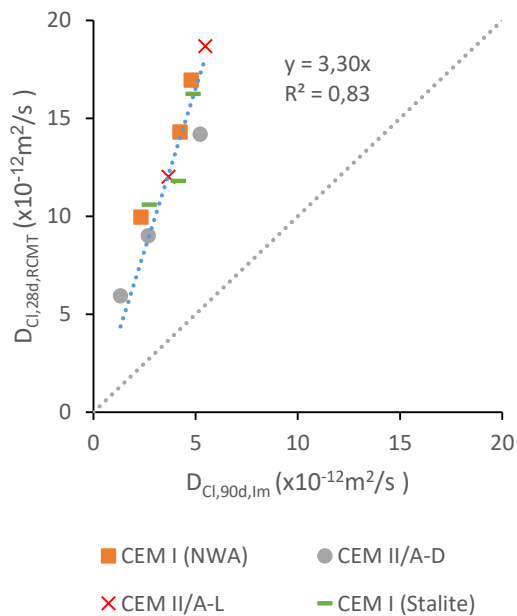


Figure 6 – Correlation between $D_{Cl,RCMT,28d}$ and $D_{Cl,Im,90d}$ (not considering CEM II/A-V, CEM II/B-V, CEM II/B-L and CEM I (Leca))

It can be concluded that in these cases, the RCMT test can be considered to classify the concrete diffusion properties, when subjected to a XS2 environmental exposure class. However, the penetration of chlorides does not depend only on the chloride diffusion coefficient.

Comparing the chloride diffusion coefficients obtained in both tests (Figure 7 and Figure 8), with the penetration parameter obtained in the immersion test, it's possible to conclude that the variation of the chloride surface content between different concrete types can have a significant influence on their durability.

This way, the establishment of a chloride penetration resistance classification based only on the RCMT test may be inadequate and should be done separately for each type of binder and, possibly, for each type of aggregate.

Nevertheless, it's possible to obtain a high correlation between the immersion test chloride diffusion coefficient and the penetration parameter (Figure 8), due to the fact that both parameters are strongly affected by the w/b ratio, and D_{Cl} and C_s vary the same way as a function of this factor.

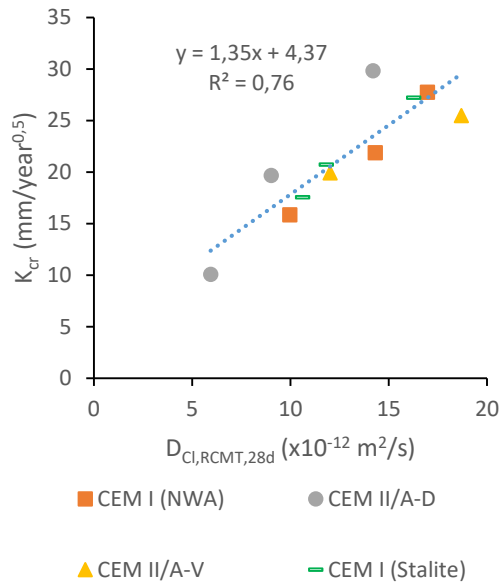


Figure 7 – Correlation between K_{cr} and $D_{Cl,RCMT}$ (CEM I (NWA), CEM II/A-D and CEM II/A-L; LWAC with Stalite)

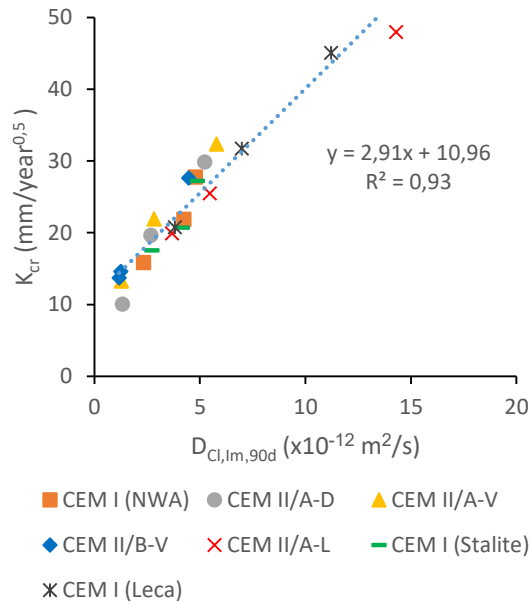


Figure 8 - Correlation between K_{cr} and $D_{Cl,Im}$ (CEM I (NWA), CEM II/A-D, CEM II/A-V, CEM II/B-V and CEM II/A-L; LWAC with Leca and Stalite)

4. Conclusions

In the present study, the following main conclusions have been drawn:

- Stalite LWA are oriented to produce high strength concrete, while Leca are better suited to produce medium strength concrete;
- RCMT and immersion tests results are mainly influenced by the paste quality (w/b ratio) and type of binder, while the type of aggregate appeared to be less relevant;
- As the penetration of chlorides does not depend only on the chloride diffusion coefficient, the establishment of a chloride penetration resistance classification based only on the RCMT test may be inadequate and should be done separately for each type of binder and, possibly, for each type of aggregate.

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