

1. Introduction

Keywords:

-Chloride-ion penetration resistance;
-Chloride profiles;
-Diffusion coefficient;
-Surface chloride concentration;
-Penetration parameter;
-Structural lightweight aggregate concrete

The durability of lightweight concrete has been increasingly investigated, namely chloride attack, which is one of the main mechanisms of concrete deterioration, leading to the corrosion of reinforcement, causing the loss of section and consequent loss of structural strength.

However, the study of structural lightweight aggregate concrete (SLWAC) still has certain knowledge limitations regarding its exposure to a real environment.

Currently, laboratory and real environment tests have been used to analyse the effect of chloride action on SLWAC. Since the tests in real environment are more time consuming, it is usual to focus the studies of the durability of concrete on the accelerated/semi-accelerated exposure to chlorides in laboratory. However, it is necessary to perform tests in a natural environment, which reflect reality, allow model calibration and the establishment of relations with laboratory tests.

As the tests in a real environment better reflect the real behaviour of the evolution of chloride penetration in concrete, it is precisely in this context that this study was developed.

In this sense, a large experimental campaign was carried out in a real environment, and the influence of the main concrete composition parameters, as well as external factors, was assessed namely the diffusion coefficient, the chloride surface concentration and the penetration parameter.

2. Experimental programme

2.1. Materials

For the production of SLWAC slabs, 4 types of lightweight coarse aggregate with very different porosities were used: two expanded clay aggregates obtained in Portugal, with the commercial names Leca and Argex, and Argex had two different grain size classes (Argex 2-4: 70% and Argex 3-8F: 30%); one aggregate originating from the United Kingdom, with the commercial name Lytag, which consists of sintered fly ash and finally an expanded slate aggregate from the USA with the commercial name Stalite.

For the production of the reference slabs (NWC), 2 normal weight aggregates (NA) of crushed limestone with different grain sizes, one gravel 1 (66%) and another rice berry (34%), were used.

For both types of concrete, fine aggregates were used, corresponding to coarse sand (70%) and fine sand (30%). Three types of additions were also used: silica fume (SF), fly ash (FA) and lime filler (LF). A polycarboxylate-based superplasticizer was used in mixtures with higher compactness.

2.2. Mixture composition

For the composition of the concrete mixture, 3 water/binder ratios (w/b) were established (0.35, 0.45, 0.55) and several types of binder: CEM I 42.5R; CEM II/A-D (6% FA); CEM II/A-V (15% FA); CEM II/B-V (30% FA) and CEM II/A-L (15% LF) (Table 1). All this in accordance with the portuguese standard NP EN 197 (2012) and the LNEC specification E 464 (2007).

The mixtures were produced in a vertical shaft mixer and bottom discharge. In general, the lightweight aggregates pre-soaked for 24 hours, then dried superficially with absorbent towels and placed in the mixer with sand and 50% of the mixing water. After two minutes of mixing, the mixture was left to rest for 1 minute, the cement and part of the remaining water were added. Then, 1 minute later, the superplasticizer was added with 10% water. The mixture lasted 7 minutes. In the case of Argex, the aggregates were not pre-soaked for 24 hours, but were placed directly dry in the mixer with additional water.

Table 1 - Composition of mixtures of SLWAC and NWC for each type w/b ratio

Type of binder	Mineral addition (wt % of binder)	V _{coarse aggregate} (L/m ³)	w/b ratio					
			0,35		0,45		0,55	
			M _{binder} (kg/m ³)	V _{sand} (L/m ³)	M _{binder} (kg/m ³)	V _{sand} (L/m ³)	M _{binder} (kg/m ³)	V _{sand} (L/m ³)
CEM I	-	350	450	314	400	310	350	315
CEM II/A-D	6% SF	350	-	-	400	307	-	-
CEM II/A-V	15% FA	350	-	-	400	304	-	-
CEM II/B-V	30% FA	350	-	-	400	297	-	-
CEM II/A-L	15% LF	350	-	-	400	306	-	-

2.3. Location and exposure conditions of the slabs

SLWAC and NWC slabs were produced with dimensions of 0.40 x 0.30 x 0.10 m with different types of aggregate, binder and w/b, with a reinforcement of 12 mm diameter bars and 3 cm of concrete cover. These were placed in advance in the Cascais bay, belonging to the District of Lisbon, on 19 and 21 March 2014 at low tide hours. They were placed in three different exposure environments, according to the Portuguese standard NP EN 206 (2013) and the LNEC specification E 464 (2007) - XS1, XS2 and XS3. However, the slabs placed in XS1 were not addressed in this study (Figure 1). A total of 34 slabs were studied.



Figure 1 - Slabs in different environmental exposure conditions XS2 (left) and XS3 (right)

For the characterization of the environment, meteorological and sea water data were used, provided by Maretec - Marine, Environment and Technology Center, obtained from the MOHID modelling system for the Bay of Cascais. The collected data from March 2014 to March 2019, demonstrated that there were no significant changes in the environmental conditions during the exposure period.

The average temperature was 16 °C, the average relative humidity was 81% and the annual average precipitation was 785 mm. The mean sea water temperature was 16 °C and the average chloride concentration was 19.5‰. A chemical test of chloride concentration in seawater was also performed and 21 g/L was obtained [1].

After 5 years of exposure, samples were taken from each slab according to the method suggested by Rilem TC 178-TMC (2013). For slabs in XS2 and XS3 environments, 12 samples were taken from 2 adjacent holes. The samples were collected with spacing of approximately 5 mm until reaching 60 mm depth. In total, more than 400 powder samples were collected.

The AASHTO T260 was used to determine the chloride concentration. A 1.5 g of sample powder was weighted, 50 ml of distilled water were added and finally 1 ml of 65% nitric acid (HNO₃) was added. After this process, the beaker containing the mixture was placed on the magnetic stirrer for 10 minutes in order to homogenize the mixture of the sample salts extraction. After this process, 5 ml of buffer solution were added, and the mixture was filtered in order to eliminate the solid particles. Finally, the potential of the solutions was measured through the electrode inserted in the pH/ISE meter. The obtained value was inserted in the calibration curve in order to obtain the chloride content of each sample in % of sample mass.

Through the values obtained from the chloride concentrations and the depths at which they were obtained, it was possible to trace the points of the chloride profile. Based on equation (1), parameters C_s (chloride surface concentration) and D_{cl} (diffusion coefficient) were obtained through non-linear regression, using the method of the least squares, through the use of software Rstudio 1.2.1335.

$$C(x, t) = C_i + (C_s - C_i) \left[1 - \operatorname{erf} \left(\frac{x}{2\sqrt{D_{cl} \times t}} \right) \right] \quad (1)$$

- C_s: surface chloride concentration (%binder mass)
- C_i: initial chloride concentration (%binder mass)
- x: chloride penetration depth (mm)
- D_{cl}: chloride diffusion coefficient (mm²/year)
- t: exposure period (years)
- erf: error function

3. Analysis and discussion of results

3.1. Chloride Profile

In general, there was a decrease in chloride concentration with depth. However, the opposite trend was observed near the surface, and the maximum chloride content was not observed at the surface. In the literature, some authors disregard the first chloride measurements, corresponding to the first 10 mm depth in the adjustment of the profile to Fick's 2nd law curve [1], [3].

As was generally predicted, chloride profiles showed a non-linear decrease in chloride concentration according to the depth of the slab and were well adjusted to equation 3.3 with correlation coefficients, in general, higher than 0.95 (Table 2), thus highlighting that chloride transport occurred essentially through diffusion.

At a depth of 30 mm, corresponding to the reinforcement covering zone, the maximum chloride concentration reached approximately 1.65% and 1.20% of the cement mass, for the environment XS2 and XS3, respectively.

In LNEC specification E465 (2007), a value between 0.3-0.6% of the binder mass for the critical chloride content is defined. Some slabs reached values above this one, meaning that reinforcement corrosion may

have been reached. On the other hand, Frederiksen [4] argues that this only occurs for values higher than 2.7% of the binder mass. In the slabs where the perforation exposed the reinforcement, no signs of corrosion were detected, which indicates that the critical chloride content was not reached.

In general, after 5 years of exposure, the various chloride profiles presented higher chloride concentration values compared to the ones observed after 1 and 3 years of exposure to the same environmental conditions. However, this was not observed for all the studied concretes. Although, in depth, the chloride concentration was higher at the surface, this trend was not always observed, regardless of the type of aggregate and the exposure class. As mentioned, some authors also obtained chloride profiles in which the chloride concentration near the surface decreased over time [1], [5] which can be explained by the fact that they are subject to varying conditions over time or by the position from which the sample was taken due to the fact that the concrete is not homogeneous.

In a general case, the highest concentrations of chlorides occurred in concrete that was subject to the environment XS2, regardless of the depth. It should be noted that, in the reference concrete, for the three different water/cement ratios, (w/c), in two situations, the chloride concentration is superficially higher in XS3. The SLWAC, in general, had higher chloride concentrations in XS2 environment, which can be justified by the fact that the concrete in this environment is saturated, and as such the aggregates are filled with water and are able to participate in the chloride diffusion.

Table 2 - Diffusion coefficient, D_{cl} , chloride surface concentration, C_s , and correlation coefficient of the results in the adequacy to the Fick's curve, R^2 , of the various types of concrete for the two exposure classes studied

w/b ratio	Type of binder	Type of aggregate	XS2			XS3		
			D_{cl} ($\times 10^{-12}$ m^2/s)	C_s (% wt binder)	Fick's R^2	D_{cl} ($\times 10^{-12}$ m^2/s)	C_s (% wt binder)	Fick's R^2
0,35	CEM I	Leca	2,21	3,24	0,99	1,54	3,98	0,99
		Stalite	0,92	3,31	1,00	1,02	2,54	0,98
		NA	1,30	2,05	0,99	1,08	2,69	0,98
		Argex	1,64	2,69	0,99	1,55	2,94	1,00
		Lytag	-	-	-	1,55	4,50	0,97
	CEM I	Leca	3,23	2,43	1,00	3,67	2,07	0,99
		Stalite	2,82	1,89	0,97	3,25	1,40	0,99
		NA	3,87	2,48	0,99	2,98	2,04	0,99
		Argex	3,36	2,80	1,00	2,93	2,08	0,99
		Lytag	2,98	2,03	1,00	-	-	-
0,45	CEM II/A-D	Leca	-	-	-	3,00	3,14	1,00
		NA	-	-	-	1,82	3,40	0,99
	CEM II/A-V	Leca	-	-	-	1,24	3,44	0,96
		NA	-	-	-	1,40	2,89	0,96
	CEM II/B-V	Leca	-	-	-	0,68	4,47	0,97
		NA	-	-	-	0,64	4,12	1,00
	CEM II/A-L	Leca	-	-	-	7,48	1,65	0,98
		NA	-	-	-	7,05	1,76	0,98
0,55	CEM I	Leca	7,96	2,13	0,98	6,93	2,12	0,99
		Stalite	6,94	1,78	0,97	7,39	1,54	0,99
		NA	5,82	1,45	0,98	6,63	1,63	0,98
		Argex	-	2,74	1,00	-	1,77	0,87
		Lytag	-	-	-	-	-	-

In general, the concrete produced with Leca and Argex showed higher chloride penetration for the two exposure environments compared to NWC and SLWAC with less porous aggregates, for different w/c ratios. On the other hand, in general, NWC and SLWAC with Stalite showed the lowest chloride concentration values (Figure 2).

For the XS3 exposure environment and w/c ratio of 0.35, SLWAC with Lytag presented the highest values of Cl⁻ concentrations (Figure 2). This trend was also verified by Real [1].

Several authors state that concrete with more porous aggregates represent higher concentrations of chlorides [6], [7]. Bogas [6] also concluded that these concretes, by presenting a more open structure and a superficial film of the particles with less compactness, contribute, in general, to a higher chloride absorption.

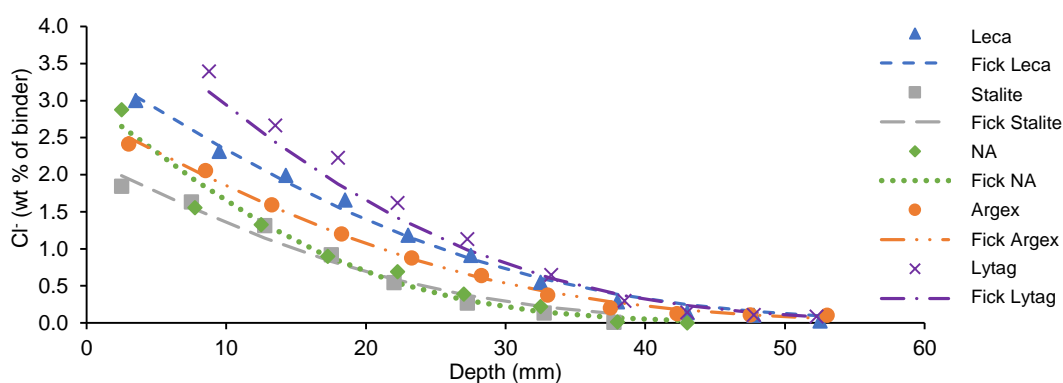


Figure 2 - Chloride profiles of SLWAC and NWC with w/b ratio of 0,35, after 5 years of exposure to class XS3

It was generally found that the higher the w/c ratio, the higher was the concentration of chloride ions along the depth of the slab, for w/c ratios between 0.35 and 0.55. This behaviour was, in general, similar for the various types of SLWAC. Besides, in the NWC no significant differences were noted, neither in XS2 nor XS3 environment.

3.2. Chloride diffusion coefficient

For concrete subject to environmental exposure class XS2, irrespective of its composition, the D_{cl} values ranged from 0.92×10^{-12} to 7.96×10^{-12} m²/s, while for concrete exposed to the environment XS3, the D_{cl} values ranged from 1.02×10^{-12} to 7.48×10^{-12} m²/s (Table 2).

The chloride diffusion coefficient increased with the increment of the w/c ratio for all types of aggregate and different exposure classes (Figures 3 and 4). The increase in the w/c ratio is directly related to the increase in the porosity of the cement paste and to a higher interconnectivity, associated with a decrease in the chloride binding capacity [1].

In general, the diffusion coefficient presented the same order of magnitude of values for the various concretes, not being significantly influenced by the type of aggregate. Several authors reached similar findings [1], [6].

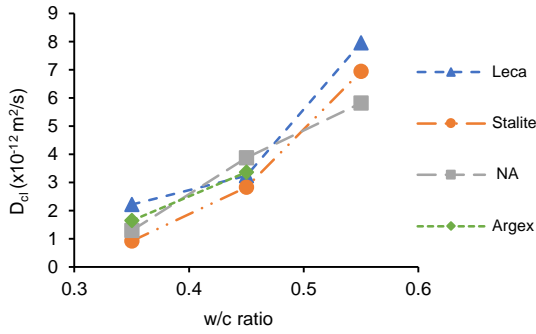


Figure 3 - Chloride diffusion coefficients, D_{cl} , of concrete with different types of aggregate and w/b ratios, after 5 years of exposure to XS2

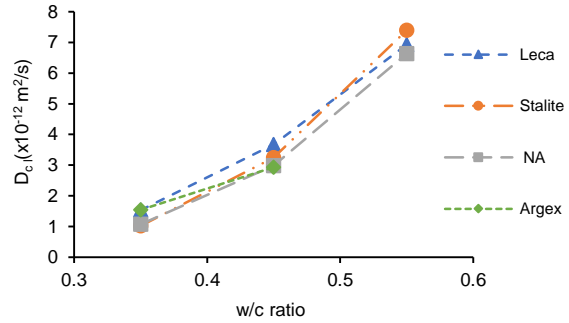


Figure 4 - Chloride diffusion coefficients, D_{cl} , of concrete with different types of aggregate and w/b ratios, after 5 years of exposure to XS3

Over the exposure period to which the slabs were subjected, in a general way, the diffusion coefficient decreased from 1 to 5 years, regardless of the type of mixture or exposure (Figure 5 and 6), which would be expected according to the literature [1], [2], [8].

It was not possible to find a clear relation between the chloride diffusion coefficient and the environmental exposure class to which the concrete was subjected, and the results were very similar for both XS2 and XS3 exposure environments.

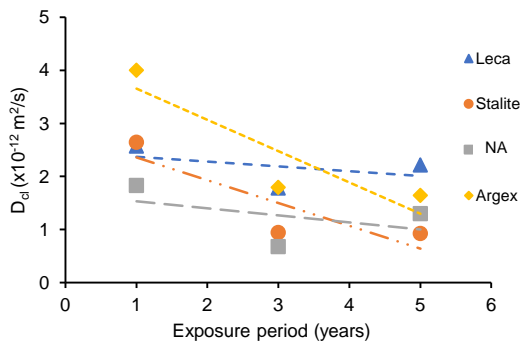


Figure 5 - Chloride diffusion coefficient, D_{cl} , of SLWAC and NWC, with w/b ratio of 0,35 and different types of aggregate, after 5 years of exposure to class XS2

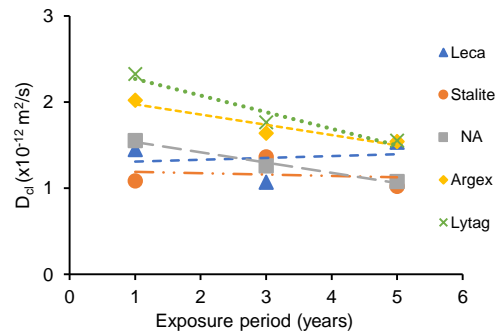


Figure 6 - Chloride diffusion coefficient, D_{cl} , of SLWAC and NWC, with w/b ratio of 0,35 and different types of aggregate, after 5 years of exposure to class XS3

3.3. Surface chloride concentration

For the present study, the surface chloride concentration of concrete exposed to XS2 for 5 years varied between 1.45% and 3.31% of the binder mass and for concrete exposed to XS3 varied between 1.40% and 4.50% of the binder mass.

In general, the surface chloride concentration had a slight tendency to decrease with the w/c ratio, for both XS2 and XS3 exposure environments (Figure 7 and 8). Some authors also reached the same results [1], [9]. In the literature, there are several different explanations for the decrease of the surface chloride concentration with the increase of w/c. Thomas and Bremner [10] consider that the decrease in surface chloride concentration with the increase of w/c ratio is related to the decrease in cement content, leading to a lower chloride binding capacity. Another factor that may explain the decrease in the surface chloride concentration with the increase in the w/c ratio may be the fact that the values of the surface concentration

are not real, but rather reflect the best adjustment to the Fick's curve, disregarding some cases in which the chloride peak concentration occurs inside the concrete and not at the surface.

In general, SLWAC with aggregates of higher porosity tended to present higher values of C_s . In general, the highest C_s occurred for SLWAC with Leca and Argex (the most porous aggregates) and the lowest C_s for NWC and for SLWAC with Stalite, both for exposure environment XS2 and XS3 (Figure 7 and 8).

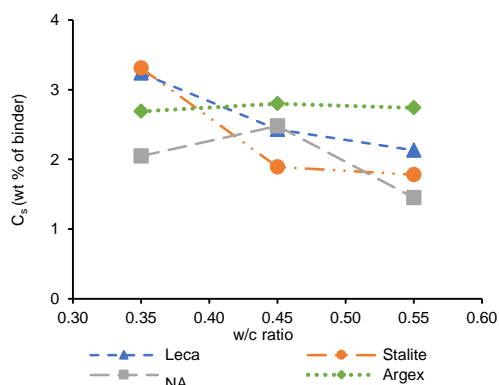


Figure 7 - Surface chloride concentration, C_s , of SLWAC and NWC with different types w/b ratios and aggregate, after 5 years exposure period to class XS2

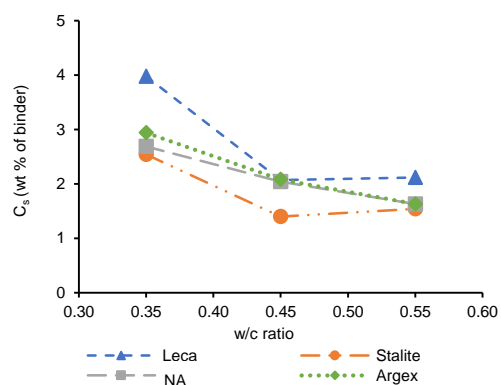


Figure 8 - Surface chloride concentration, C_s , of SLWAC and NWC with different types w/b ratios and aggregate, after 5 years exposure period to class XS3

Regarding the influence of the type of binder, in general, there was a similar variation in surface chloride concentration for both NWC and SLWAC with Leca.

For the concrete with incorporation of silica fume, the surface chloride concentration had an increase of 34% for SLWAC with Leca and 40% for NWC, in relation to CEM I. According to the literature, this increase was expected, since the addition of silica fume increases the ability to bind chlorides to the concrete surface [9], [11], [12].

The replacement of CEM I with different percentages of fly ash contributed to an increase of the surface chloride concentration of NWC and SLWAC with Leca. This increase for the addition of 30% of fly ash was 116% for SLWAC with Leca and 102% for NWC, in XS3 exposure environment. A possible reason for this fact could be that, for the same amount of binder mass, these have a higher binder volume, being directly related to the increase in binding capacity [1].

In the case of concrete with incorporation of 15% lime filler, there was a small decrease in the surface chloride concentration of 20% for SLWAC with Leca and 14% for NWC. Since the incorporation of lime filler additions reduces the binding capacity, the surface chloride concentration tends to be lower [6].

As expected, the surface chloride concentration tended to increase over the exposure period, both for NWC and SLWAC, regardless of the type of aggregate, binder and w/c ratio. Although there were some differences in the results of C_s over time, most of the authors obtained results in which the C_s of the different types of concrete increased over the exposure period, thus reinforcing the results obtained [2], [13], [14].

In general, it was not possible to observe a relation between C_s of the various types of concrete and the exposure conditions. The way chloride superficial concentration changes with time in function of environmental exposure was different for SLWAC and for NWC, it does not show a defined tendency in these types of concrete. Thus, is not possible to conclude about the influence of the environmental exposure in C_s evolution in these types of concrete.

3.4. Penetration parameter

The penetration parameter, K_{cr} , for the various types of concrete in the first year of exposure varied between 10.3 and 31.3 mm/year^{0.5} for the exposure environment XS2 and between 10.8 and 39.5 mm/year^{0.5} for the exposure environment XS3. After 3 years of exposure, this parameter varied between 7.4 and 23.5 mm/year^{0.5} and 10 and 36 mm/year^{0.5}, respectively. After 5 years of exposure, the penetration parameter varied between 12.17 and 31.09 mm/year^{0.5} and between 10.68 and 28.96 mm/year^{0.5} for exposure environments XS2 and XS3, respectively.

As expected, in general, the penetration parameter increased with the increase of the w/c ratio, regardless of the exposure conditions and types of concrete (Figures 9 and 10).

In general, the SLWAC with more porous aggregates (Leca and Argex) presented the highest penetration parameters, while the NWC and the SLWAC with Stalite presented lower K_{cr} values, due to the fact that the concrete with more porous aggregates had an increase of C_s , compared to the less porous ones, although they did not suffer a significant increase of D_{cl} .

For both SLWAC with Leca and NWC, the penetration parameter generally followed the same linearity relation according to the percentage of substitution of CEM I with additions. It should be noted that the K_{cr} values of the SLWAC with Leca were slightly higher than those of the NWC.

In general, the penetration parameter showed the same trend for the various types of concrete studied, and the K_{cr} values were slightly higher for the concrete exposed to environment XS2. Since, in section 3.3, it had already been mentioned that higher surface chloride concentrations were obtained in submerged concrete, it was expected that the penetration parameter would present slightly higher values.

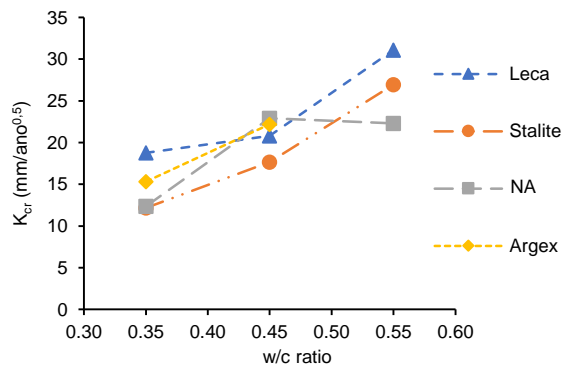


Figure 10 - Penetration parameter, K_{cr} of SLWAC and NWC with different types of aggregate and w/c ratios, after 5 years of exposure to class XS2

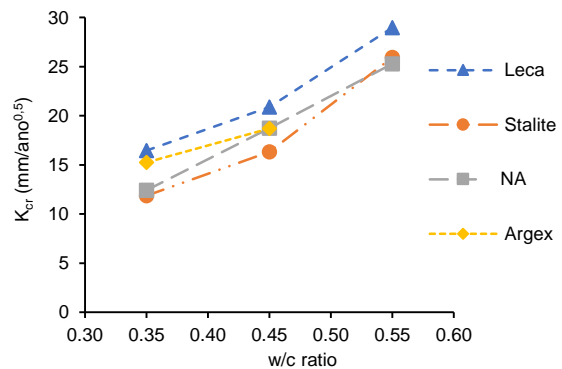


Figure 11- Penetration parameter, K_{cr} of SLWAC and NWC with different types of aggregate and w/c ratios, after 5 years of exposure to class XS3

4. Conclusions

This study involved an experimental campaign aimed to collect various samples of slabs placed in Cascais' bay exposed to two different environments (permanently submerged zones, XS2, and tidal and splash zones, XS3), and the determination of the chloride concentration, the chloride profiles and the respective diffusion coefficient, surface chloride concentration and a penetration parameter. The main conclusions resulting from the various parameters analysed are presented below:

- After 5 years of exposure, no signs of reinforcement corrosion were observed, and the maximum chloride concentration values obtained at a depth of 30 mm were 1.65 (% binder mass), for the environment XS2, and 1.20 (% binder mass), for the environment XS3. Possibly, the critical chloride content was not reached, although the value was above that suggested in the LNEC specification E465 (2007);

- Contrary to what was expected, most of the concretes presented greater chloride penetration in XS2 exposure environment than in XS3, regardless of the type of concrete;
- In general, the chloride penetration was higher for higher w/c ratios, regardless of the type of aggregate and binder;
- The type of aggregate influenced the penetration of chlorides in concrete, with SLWAC with more porous aggregates associated with a higher penetration of chlorides;
- The incorporation of lime filler impaired the resistance to chloride penetration, while the incorporation of fly ash and silica fume resulted in a better durability for chloride penetration;
- The D_{cl} was little influenced by the type of aggregate, highlighting only that SLWAC with more porous aggregates had a slightly higher D_{cl} ;
- The D_{cl} decreased with the increase of silica fume and fly ash in the cement, while with the incorporation of limestone filler increased this parameter;
- In general, the chloride diffusion coefficient decreased with the exposure period;
- C_s decreased with the increase in the w/c ratio, regardless of the type of aggregate and exposure condition;
- The highest C_s occurred for SLWAC with more porous aggregates;
- The C_s increased with the incorporation of silica fume and fly ash in the cement, while with the addition of lime filler decreased this parameter;
- Throughout the exposure period, in general, the surface chloride concentration had a tendency to increase, not having stabilized before 5 years of exposure;
- K_{cr} increased with the increase in the w/c ratio and presented higher values for more porous materials, following the same trend as D_{cl} ;
- With the incorporation of silica fume, the penetration parameter did not change significantly, while with the replacement of CEM I with fly ash decreased this parameter, and the incorporation of lime filler increased it, regardless of the type of concrete;
- K_{cr} showed a similar behaviour and the similar values for the two types of environmental exposure, being sometimes slightly higher in the environment XS2;

In sum, structural lightweight aggregate concrete with less porous aggregates reached a resistance to chloride penetration similar to normal weight aggregate concrete of the same composition. On the other hand, SLWAC with more porous aggregates presented lower resistance to chloride penetration than NWC.

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