

BETON ARMAT, DE ÎNALTĂ PERFORMANȚĂ CU REZISTENȚA LA COROZIUNE ÎN MEDII AGRESIVE INDUSTRIALE HIGH PERFORMANCE REINFORCED CONCRETE, CORROSION – RESISTANT INTO AGGRESSIVE INDUSTRIAL ENVIRONMENTS

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Reinforced concrete structures exposed to aggressive industrial environments suffer different accelerated deterioration mechanisms. In chlor-alkali production plants, the industrial environment contains high concentrations of carbonates and chlorides, damaging the structures and causing constant stoppages and repair processes on the structure of reinforced concrete. In this study, the effect of using internal curing on high performance concrete with low water/binder ratios was studied, implementing pumicite as internal curing agent, chemical additives and replacing Portland cement with pulverized fly ash. Durability measurements conducted were permeability of chloride ions, and carbonation depth under industrial and laboratory conditions. Results showed that there is a reduction of the aggressive agents transport mechanisms when internal curing is applied. However, measurements at long ages must be done to evaluate the synergistic effect of the agents contained in the industrial environment on the metal reinforcement.

Keywords: Durability, Corrosion, Internal Curing, Composite, Structural Application.

1. Introduction

Continuous production processes are required to obtain primary raw materials such as sodium carbonate (Na_2CO_3), due to the permanent demand of these supplies. Equipment and devices that produce these materials consist of reactor towers that are supported primarily by steel beams and reinforced concrete structures. The deterioration of reinforced concrete structures exposed to aggressive industrial environments is a problem that has sought solutions in preventive maintenance through the use of expensive coatings with a complex application process or cathodic protection [1-2]. The use of special coatings and constant repair are the mechanisms used to increase the useful service life of structural elements inside these productive plants. However, in particular aggressive environments, such structures are highly susceptible to chemical attack by the compounds contained in local industrial environment [3]. Carbonation, chloride ions attack, and sulfates are conditions that are consistently present in industrial processes and decrease the equipment service life. Most of the problems associated with these deterioration mechanisms

have been evaluated in infrastructure elements of cities mainly near sea environments and/or roads and bridges where the presence of deicing salts promotes accelerated deterioration. In the latter situations, there are currently different methodologies in which deterioration can be evaluated, so that the elements corrosion progress can be predicted and evaluated. [4-12].

However, there is a lack of information about aggressive industrial environments, such as in the production of carbonates where local environmental conditions cause accelerated deterioration of the reinforced concrete structure, forcing constant stoppages and repairs.

The use of alternative building materials was proposed as a way of decreasing the corrosion agents transport mechanisms that cause deterioration [2,13]. As a solution, test concretes made of alternative material replacements were proposed, which were exposed to actual industrial conditions and then their performance was compared to laboratory conditions experiments.

1.1 Industrial Environment (In Situ)

Production of Na_2CO_3 uses a solution of sodium chloride (NaCl) and ammonia (NH_3).

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Limestone (CaCO_3) is decarbonated at high temperature and CO_2 is injected into the abovementioned salt solution. The process operates by passing these minerals through reactors towers to obtain alkaline compounds. CaCl_2 and CO_2 are generated as main byproducts. CO_2 is reutilized to produce more Na_2CO_3 . However, most of the CaCl_2 is confined. In equation 1 the overall above described process is shown. [14] The plant are shown in Figure 1 respectively. Inside the facilities is constantly precipitating CaCl_2 , so that the components and structures that are part of the process are constantly exposed to critical conditions of deterioration.



Under these circumstances, the useful service life of reinforced concrete structures will depend of the characteristics that decrease aggressive agents progression through them. High concentration of carbon dioxide CO_2 and moisture promote the formation of carbonic acid, which penetrates the concrete pore solution [13,15,16]. Furthermore, CaCl_2 permeates in the same way and when being in contact with the surface of the reinforcing steel form ferric chloride FeCl_3 produces pitting and eventually loss of structural load capacity. In Figure 2, different structural metallic elements damaged due to produced salts and constant exposure to high concentration of CO_2 in the process are shown. In the upper left image, concrete surface detachment due to internal tensions prompted by corrosion products is shown; upper right image, the reinforcing steel is exposed due to the chloride ions effect; lower left image, a slab with reinforcement fully exposed is shown; lower right image, structures with an excess of sodium carbonate residues (NaCO_3)

over the structural elements surface damaged by carbonation are shown. Permeability reduction in the concrete microstructure can be achieved with concrete production with low water/binder ratio (w/b). The use of superplasticizers, shrinkage reducers, and recently by adding byproduct materials; coupled with internal curing techniques, [17-19] lead to a high performance concrete (HPC) development which could reduce transport mechanisms increasing durability. However, it is now a technological advantage to increase density in cementitious matrices by developing pozzolanic reaction and reducing the possibility of alkaline leaching. This concrete densification is the result of the reaction which leads to a higher resistance at prolonged ages. The benefits are increased if the cementitious mixtures contain more than one supplementary cementitious addition.[20-25] Internal curing in HPC is an important feature in the resistance development in concrete with low w/b ratio[19]. Besides, the use of additions and internal curing can reduce autogenous shrinkage phenomena that cause cracking and consequent reduction of the element life internal curing is carried through lightweight aggregate (LWA), which is finely dispersed in order to reduce permeability. The concept is to use LWA with the same size of normal fine aggregate increasing curing times [26-31].

The aim in this study is to use mixtures of HPC as an alternative to increase the elements useful service life, using internal curing agents and pore viscosity modifiers.

The chemical composition of the chlor-alkali process residues are reported in Table 1 in which high content of carbonates and chloride compounds is observed.



Fig. 1 - Appearance of the surroundings of the production plant (precipitating CaCl_2).



Fig. 2 - Structural elements deteriorated due to corrosion processes active during production.

Table 1

Chemical composition of waste in sodium carbonate production damaging to the reinforced concrete

	CaCO ₃	Ca(OH) ₂	CaCl ₂	CaSO ₄	NaCl	Mg(OH) ₂
%wt	43.63	11.09	9.38	2.64	25.64	7.82

Table 2

Raw material chemical analysis by X-ray fluorescence technique

Raw Material	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	K ₂ O	CaO	Cr ₂ O ₃	Fe ₂ O ₃
LWA	1.71	0.43	14.36	72.02	0.04	0.05	2.74	0.06	3.71
PFA	0.64	0.84	25.01	63.78	0.02	0.01	2.29	0.05	4.99
OPC	0.37	1.68	5.84	20.71	0.12	0.01	65.81	0.07	2.35

2. Experimental Methodology

2.1. Description of the proposed repair materials.

High performance concrete are mixtures of cement, chemical and minerals admixtures, as well as the use of improved curing methodologies to develop the building material properties, with low w/b ratios an improved internal curing methodology is proposed to use as repair material, in which pulverized fly ash (PFA) and lightweight aggregate pumice (LWA) are combined, which is then saturated in shrinkage reducing admixture (SRA). The lightweight aggregate was immersed in a vessel containing a solution water and chemical admixture (This water is considered in the analysis of proportioning) so that the LWA absorbed the solution, it was subsequently added in the mixing process as partial replacement of the normal weight aggregate (FLA).

Raw materials were selected to produce the materials that are commercially available, such as ordinary Portland cement (OPC) with a $d_{50} = 30\mu\text{m}$, fly ash (PFA) with a $d_{50} = 75\mu\text{m}$, fine aggregate of

normal weight (FLA), coarse aggregate of normal weight (CA), lightweight aggregate pumice (LWA). Additives, such as the commercial super- fluidified admixture (HRWRA) and the commercial shrinkage reducing admixture (SRA).

2.2 Characterization of raw material for making concrete for repair structures

Raw materials were characterized measuring oxides content obtained by X-ray fluorescence technique (XRF) using a X EPSILON 3-XL equipment before making the concrete mixtures. The LWA was a silicoaluminous compound with low alkali content. According to ASTM C618-89[32], PFA was classified as class F, with low content of calcium oxide (CaO). Chemical composition of ordinary portland cement is shown in Table 2.

A peculiarity of the LWA was characterized by electronic microscopy using a JEOL JSM-6490LV microscope. In Figure 3, the internal porosity and the morphology of LWA are shown. By this analysis, it was registered pores with diameters ranging from $0.70\mu\text{m}$ to $2.8\mu\text{m}$ with an

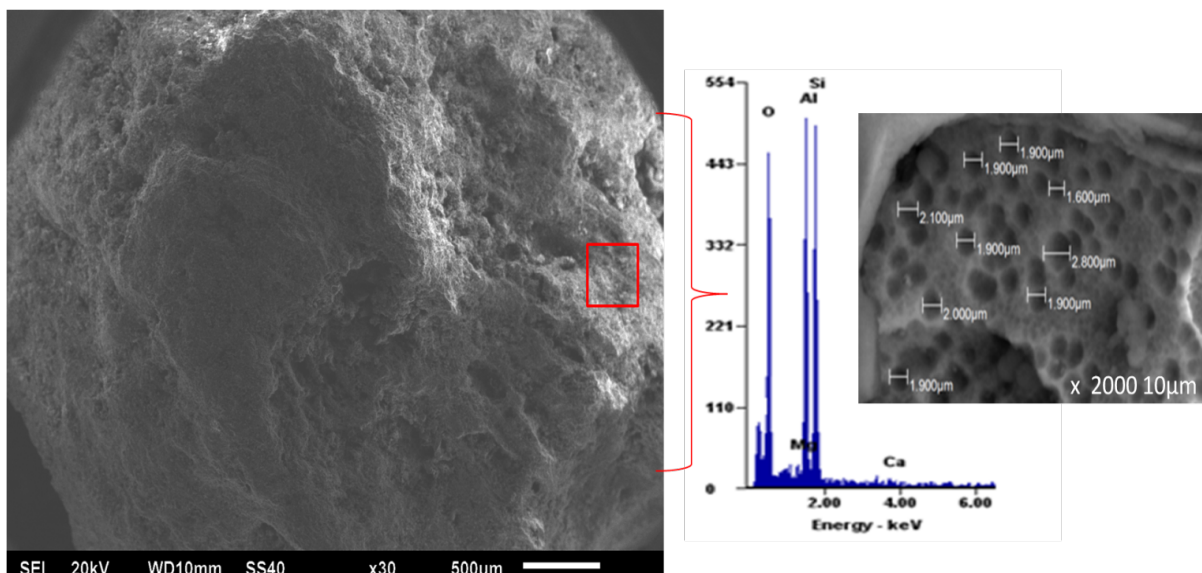


Fig. 3 - The pore size ranges around 2 μm.

Concrete mixtures proportions in kg/m³ for repair damaged structures

Table 3

Mixture	R1	IC1	IC2	IC3	IC4
OPC	422	421	424	423	338
PFA	-	-	-	-	85
Water	168	168	166	168	168
CA	789	788	788	791	786
FLA	980	800	783	786	781
LWA	-	117	117	117	117
SRA	-	0.0	6.36	19.5	19.3
HRWRA	1.13	1.13	1.13	1.13	1.13
w/cm	0.40	0.40	0.40	0.40	0.40

Description of elaborated concretes

Table 4

R1	Concretes with normal weight aggregates, ordinary Portland cement, Water-Reducing Admixture HRWRA
IC1	R1 concrete but with a partial substitution of 20% of lightweight aggregate LWA by fine aggregate of normal weight (fine lightweight aggregate)
IC2	IC1 concrete with Shrinkage-Reducing Admixture in the mixing process.
IC3	IC2 concrete with the lightweight aggregates LWA saturated with Shrinkage-Reducing Admixture
IC4	Concrete with limestone aggregates, partial substitution of ordinary Portland cement by pulverized fly ash at 20%, water, substitution of the fine aggregate by LWA saturated SRA.

average pore diameter of 2 μm. The pores of this aggregate can be seen, which allows up to 35% desorption of aqueous solutions. LWA were saturated with an aqueous solution with SRA with a proportioning 1:1 in volume.

A w/c ratio of 0.4 was utilized in the production of the high-performance concretes with a cement consumption of approximately 400 Kg/m³. The proportioning mixes used are shown in Table 3.

The concrete mixtures were prepared according to conditions shown in Table 4. The specimens were cured for 28 days under controlled humidity conditions and a temperature of 25 °C.

After this time, the samples were exposed to test environment, after that durability tests were performed.

The concrete specimens made were exposed to accelerated deteriorating conditions in the laboratory and to direct exposure in the production facilities of soda powder, as shown in Figure 4. The tests performed to evaluate the proposed material quality were compression tests according to ASTM C39-16b[33] in cylindrical molds of 10 x 20 cm. Permeability tests that would show the new material performance to reduce the entry of aggressive agents were rapid permeability of chloride ions, according to ASTM C 1202-12[34]



Fig. 4 - Specimens exposed to industrial environment.

and accelerated carbonation test. The samples exposed to industrial environment were cylindrical specimens of 7.5 x 15 cm, in which the carbonation advance and the entry of chloride ions were measured.

3. Results and Discussion

The result of performing the LWA additions with PFA in the HPC is shown in Figure 5a and Figure 5b, where the effect of adding SRA as internal curing agent is observed. The samples show unreacted PFA particles in the cement paste, so the effect of SRA may be to delay the pozzolanic reaction and consequently increasing the compression resistance development time.

3.1. Compressive Strength

The results obtained in mechanical tests see Table 5. The internal curing mixture showed a better performance in the compression test compared to the reference mixture. The use of SRA in the mixture IC2 improved the properties with respect to mixture IC1, IC3 and IC4. The use of PFA in IC4 mixture did not improve the mechanical properties. The objective of adding PFA was to study whether the pozzolanic effect could contribute to the development of mechanical resistance. Although LWA is a porous aggregate, its use does not reduce the specimen resistance significantly. See Figure 6a. However, all conditions exceeded the 400 kg/cm² specified for the portland cement used. One of the most relevant characteristics of the concrete mixtures in

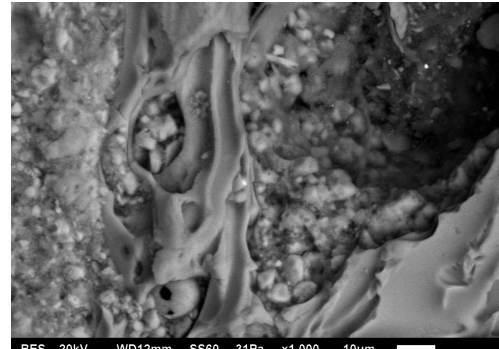


Fig. 5a - Densification of cement past into concrete IC2.

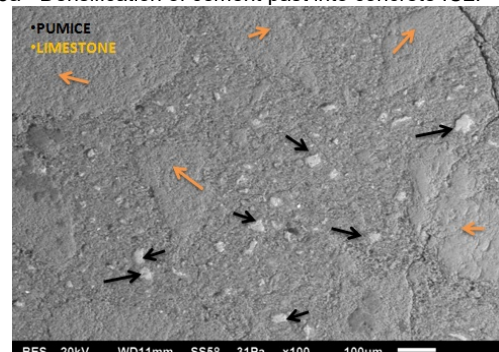


Fig. 5b - Densification of cement past into concrete (IC2).

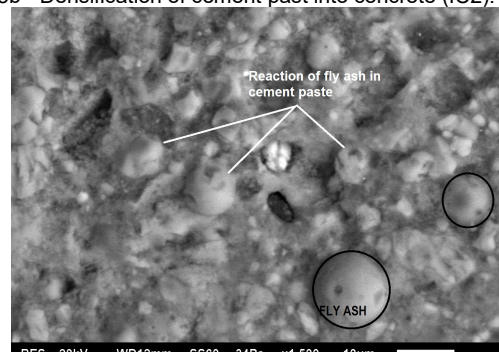


Fig. 6a - Interaction of fly ash and cement hydration gels (IC2).

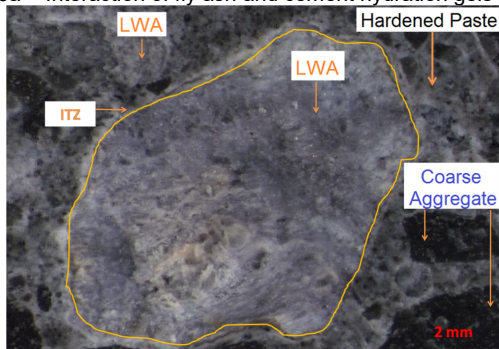


Fig. 6b - The aggregate is outlined and it is observe the effect of cement paste hydrated by the LWA (IC2).

Results of mechanical test of compressive strength

Table. 5

Mixture	Compressive Strength (MPa)			
	14 days	28 days	90 days	180 days
R1	34	40	47	49
IC1	37	46	49	50
IC2	36	50	51	52
IC3	39	43	47	49
IC4	31	35	44	45

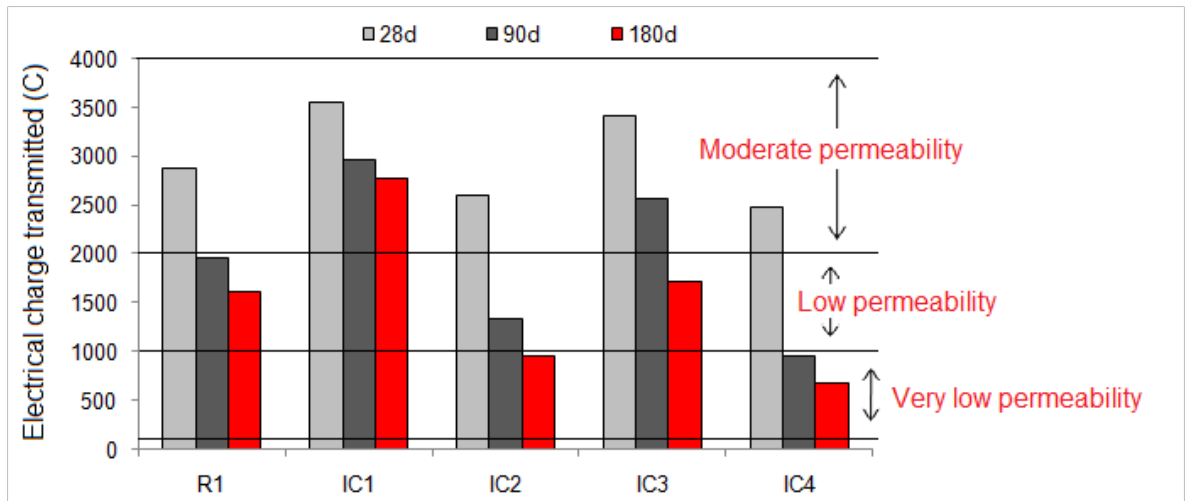


Fig. 7 - Electrical charge (C) of chloride ions transmitted through the cementitious matrix.

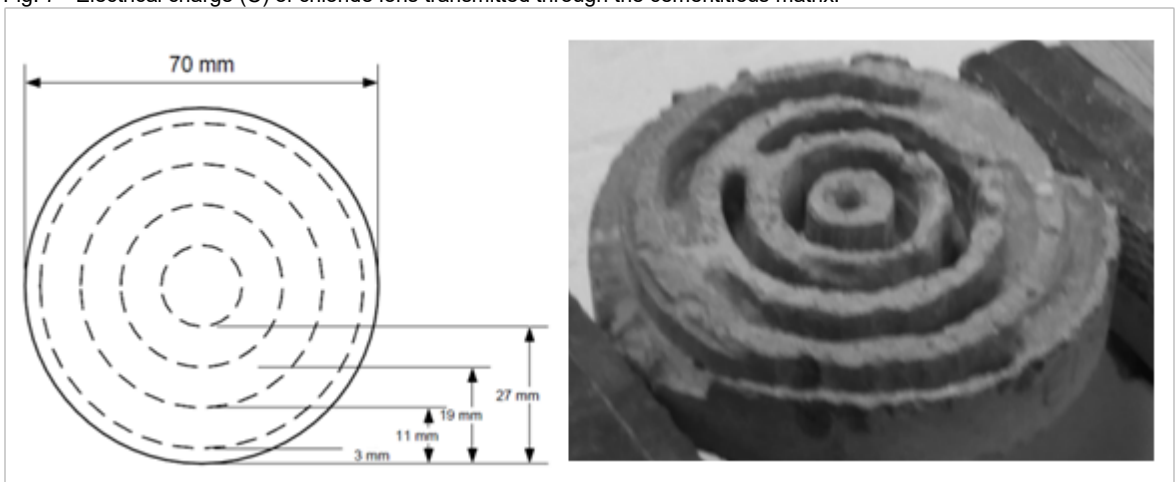


Fig. 8 - Concentric circles to extract the powder on the perimeter of the sample, the last corresponding to the chloride content in the surface of the steelbar.

which LWA was used, is that the paste densified the interfacial transition zone (ITZ) between the paste and the LWA, which in normal weight aggregates is identified as a low density zone. A detail of how the paste densified this zone can be seen in Figure 6b. It was observed that even the paste reacted inside the LWA due to its high porosity. As indicated in table 5 in IC4 mixture the partial substitution (20%) of PFA by OPC did not influence the increase in compressive strength at the tested ages. Showing lower resistance with an average of 17% at 14 days, 24% at 28 days, 10% at 90 days and 10% at 180 days with respect to the mixtures where the internal curing technique was used (IC1, IC2, IC3).

3.2. Chloride ion penetration depth

Rapid chloride ion permeability is a standard method directly related to the cementitious matrix permeability. The results showed a decrease in the penetration with which these ions travel inside the

samples obtained from the test cylinders. The lower the current in the tests, the lower the permeability under the conditions of this test, see Figure 7. IC4 and IC3 condition shows an electric charge up to 80% lower than a reference concrete, so that the SRA agent helps to avoid the cracks that damage the elements durability. The reference concrete behaves as a normal concrete, in which permeability is influenced by its microstructure, and in which it may be cracking due to contractions during the hydration reaction that increases the permeability. The concrete mixtures made were also exposed to industrial environment and measurements of the change in the concentration of chloride ions per kilogram of cement at 4 different depths, 3, 11, 19 and 27 mm are shown in Figure 8 at 150 days of exposure. The chloride concentration is higher as measured on a point closest to the surface and it decreases at deeper penetration. The depth at which the reinforcement is commonly located in a concrete is

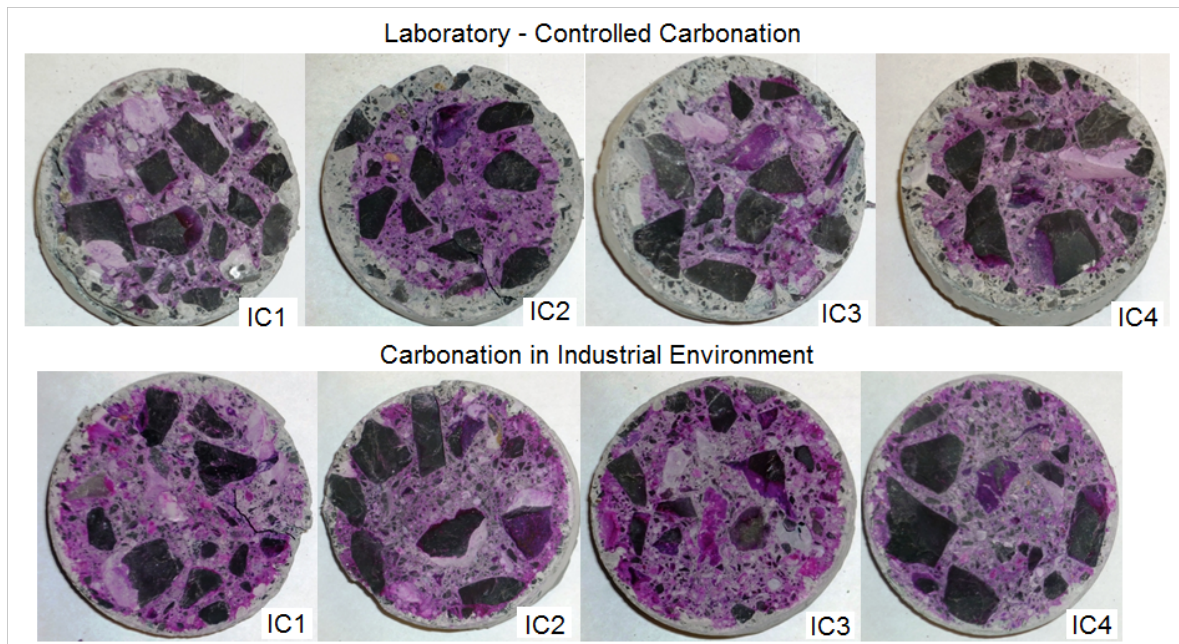


Fig. 9 - Carbonation depth at 180 days between 2 exposure conditions. The indicator dye shows an area where the concrete pH has not been modified due to carbonation.

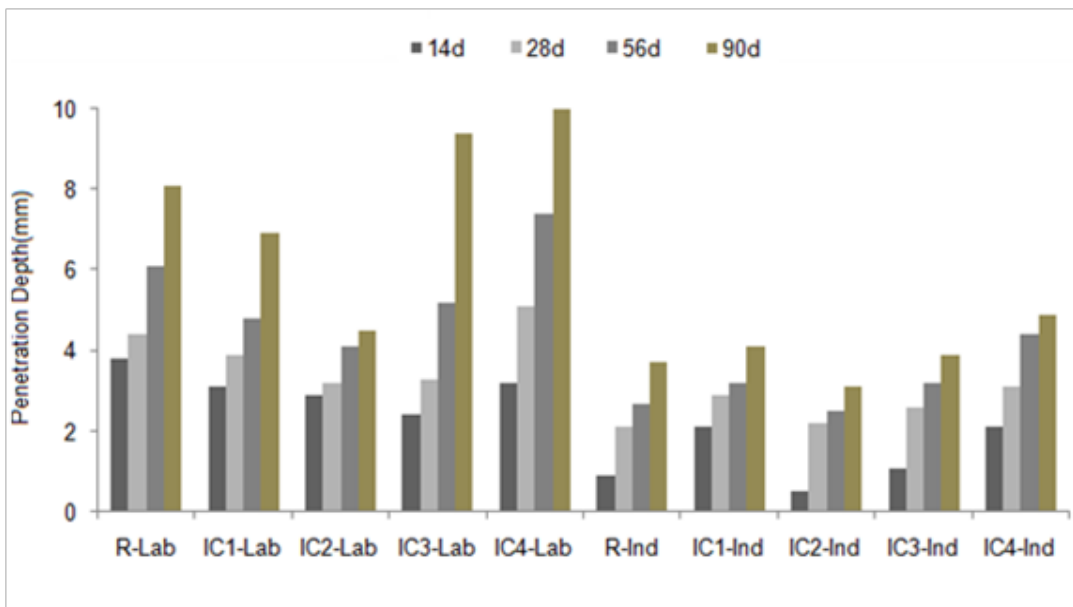


Fig. 10 - Carbonation penetration of concrete exposed to industrial environment and in accelerated carbonation chamber.

around 25 mm. At this distance, the three conditions showed a low concentration, less than 2% chloride per cement kg. The test concrete mixtures showed a better performance at longer age respect to the reference. This behavior is related to resistance development at long ages. However, the critical chloride concentration must be determined to see the response of the reinforcement steel.

3.3. Concrete carbonation

The concrete mixtures were exposed to laboratory chamber to a controlled environment of 4% CO₂ and a relative humidity of 40% and to industrial environment at the soda ash plant. The environmental conditions in the industrial

installations constantly vary. The climate is arid and local conditions may be easily affected by other factors, such as wind, pipe leaks and high content of residues in the environment, such as calcium chloride (CaCl₂) and sodium carbonates (Na₂CO₃). Cross sections of the cylinders exposed to industrial and laboratory conditions are shown in Figure 9. The progress of the carbonation was larger observed in the reference samples. The use of internal curing and the low w/b ratio helped to reduce the concrete carbonation. The IC2 mixtures showed better performance in laboratory and industrial environments in comparison with carbonation of the reference mixtures and IC1, the IC3 mixture showed a high carbonation in the studied environments, this was attributed to CO₂

ingress through the pores of the LWA. Not all internal pores of the LWA were sutured with SRA. The phenolphthalein test helped to deduce this this observed effect, the addition of PFA in IC4 does not promote the matrix densification probably fly ash has a filler effect and not a pozzolanic effect, possibly inhibited by the use of SRA. Therefore the use of SRA in mixtures IC3 and IC4 is not a contributor in the carbonation resistance in the studied environments. The carbonation depth test results indicated that the exposure conditions in the laboratory may not be representative of the industrial environment and they should be taken only as reference regarding the carbonation mechanisms behavior. In the chamber of accelerated carbonation, the concentration of CO₂, temperature and humidity are constantly controlled. It is well known that the carbonation process occurs between 40 and 60% of relative humidity. However, these conditions are not necessarily continuously present in the plant because the changes in humidity are common due to the microclimates of each process area. in Figure 10 shows how carbonation within the industrial (Ind) facilities is lower than the measured in the laboratory (Lab) samples.

4. Conclusions

During the process of obtaining sodium carbonate, gases and byproducts material are the main sources of the reinforced concrete structures deterioration. CO₂ carbonates the structures, and the calcium chloride byproduct produced breaks in the passive layer of the reinforced steel, triggering different deterioration mechanisms, and promoting long term structural damage. The research materials used in the proportioning to make the test concrete mixtures were byproducts that have been used in the literature to develop HPC. LWA used has the function of supplying water to perform the internal curing concrete mixtures and thus reduce the cracking due to drying effects. The use of supplementary materials, such as PFA, is very useful to reduce the consumption of cement without losing a significant mechanical strength. The use of LWA as curing agent, proved to be a good technique for water desorption inside the cementitious matrix, reducing permeability, and minimizing the effect of deleterious agents. As for the Mechanical properties, the internal curing in IC1 and IC2 showed an increase of 5% in comparison with R1. The variation of the resistance in the IC3 mixture is attributed to the fact the SRA was not homogeneously mixed, by the method used. In the IC4 mixture lower resistance were obtained possibly because the SRA inhibits the pozzolanic effect of the PFA. Additionally, the quantity of Portland cement was less, explaining the decrease of compression resistance. Laboratory testing did not reflect the real conditions

in industrial environment. However, the measurements tendency is similar. The test concrete mixtures have less carbonation depth and a lower income of chloride ions in its microstructure than reference. This is related to the fact that they are denser concrete mixtures. The hydration reaction occurs in the ITZ and hydration products inside the LWA were observed. The internal curing and the shrinkage reducing additive decrease permeability to the agents that promote corrosion. Thus, they are viable, durable and sustainable repair alternatives in structures exposed to aggressive environments. However, the synergistic effect of the corrosion promoting agents on reinforced concrete must be monitored in industrial environment to ensure the extension of the reinforced concrete structures service life.

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