

## Durability analysis of reinforced concrete with loading induced cracks

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### ABSTRACT

The objective of this study is to verify the relationship between the action of loads inducing cracks and the durability of reinforced concrete. Prismatic specimens were produced and for two years these samples were subjected to artificial salt spray, under the action of different types of loading and unloaded (reference), with moist curing for 7 days. Chloride penetration tests and microstructural analysis were carried out. It was observed that loading did not influence the results of chloride penetration. However, it was observed that in the micrographs and microanalysis of the cracked samples the clearer formation of deterioration products and possible microorganisms, compared to the samples that did not suffer loading.

**Keywords:** durability; loading; cracking; chlorides; microscopy.

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#### Contribution of each author

In this work, the author M. P. Costa Junior contributed with the activities of conceptualization, development, results and discussion, writing and preparation of the original text; S. M. M. Pinheiro contributed to the activities of conceptualization, development, results and discussion.

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## **Análise da durabilidade de concreto armado com fissuras induzidas por carregamento**

### **RESUMO**

O objetivo desse trabalho é verificar a relação entre a ação de carregamentos induzindo fissuras e a durabilidade do concreto armado. Foram produzidos corpos-de-prova prismáticos e durante o período de dois anos estas amostras foram submetidas à névoa salina artificial, estando sob a ação de carregamento central permanente, carregamento central de curta duração e sem carregamento (referência), sendo realizados ensaios de penetração de cloretos e análise microestrutural, além do mapeamento das fissuras. Verificou-se que o carregamento não influenciou nos resultados de penetração de cloretos, porém, nas micrografias e microanálises das amostras fissuradas observou-se a formação de produtos de deterioração e possíveis microorganismos, em comparação aos corpos-de-prova que não sofreram carregamento.

**Palavras-chave:** durabilidade; carregamento; fissura; cloretos; microscopia.

## **Análisis de durabilidad del hormigón armado con fisuras inducidas por la carga**

### **RESUMEN**

El objetivo de este trabajo es verificar la relación entre la acción de cargas que inducen fisuras y la durabilidad del hormigón armado. Fueron producidos modelos de prueba (especímenes) prismáticos y durante dos años estas muestras fueron sometidas a niebla salina artificial, bajo la acción de una carga central permanente, carga central a corto plazo sin carga (referencia), con un curado de 7 días. Se realizaron pruebas de penetración de cloruros y análisis microestructurales, además del mapeo de fisuras. Se encontró que la carga no influyó en los resultados de penetración de cloruros, sin embargo, se observa que las micrografías y microanálisis muestran una formación de productos de deterioro y posibles microorganismos, en comparación con las probetas que no sufrieron carga.

**Palabras clave:** durabilidad; carga; craqueo; cloruros; microscopía.

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## 1. INTRODUCTION

The causes of the deterioration process of reinforced concrete are directly related to the action of aggressive agents. Among the various existing agents (carbon dioxide, chloride ions and sulphate attack), there is the deleterious action of chlorides and carbon dioxide (CO<sub>2</sub>), which have been extensively studied in recent years and which are still a great challenge for the good performance of reinforced concrete structures. Thus, the structures that are exposed to the marine and urban environment demand a minimum quality of the material to ensure its design life and durability (Andrade, 2005; Cascudo, 2005; Silvestro et al., 2021).

In addition to chemical causes, the causes of the mechanical deterioration process are highlighted, such as overload and cyclical loads; whose main symptom is concrete cracking. These cracks must be controlled for three main reasons: durability due to the risk of corrosion of the reinforcement, aesthetic appearance, and functional requirements such as hygiene (proliferation of fungi, microorganisms, etc.) and permeability to gases and water (Ghali and Favre, 1994; Hearn and Figg, 2001).

Under natural exposure conditions, the durability of concrete is controlled by its ability to prevent the transport of ions and fluids. Concrete is often subject to several types of stress (thermal, mechanical, etc.) that generate tensile stresses that exceed the material's resistance, generating cracks, which can affect the transport of aggressive agents to the mixture (Lim et al., 2000; Hearn and Figg, 2001; Mehta and Monteiro, 2014).

Cracks manifested due to the action of external loads can act as an important factor for the entry of aggressive agents such as chloride ions and CO<sub>2</sub> (carbonation). However, studies show that cracks are not the biggest factor for deterioration of the structure by corrosion (entry of aggressive agents) if they do not exceed the openings stipulated by international standards and NBR 6118 (ABNT, 2014). In this case, the quality of the covering concrete and the nominal covering itself are more relevant to its durability (Konin et al., 1998; Helene and Diniz, 2001; Alexander et al., 2001; Cascudo, 2005).

Regardless of loading, the characteristics of cracks (connectivity, opening, width, length) play a fundamental role in the durability of concrete structures. In this sense, NBR 6118 (ABNT, 2014) establishes the maximum characteristic opening for cracking (identified in the standard as  $W_k$ ) and protection of reinforcement in terms of durability. This standard defines the maximum crack opening of 0.4mm for reinforced concrete, which varies depending on the class of environmental aggressiveness, the type of concrete structure and the combinations of service actions.

The penetration and diffusion of chloride ions can occur through the crack as shown in Figure 1.

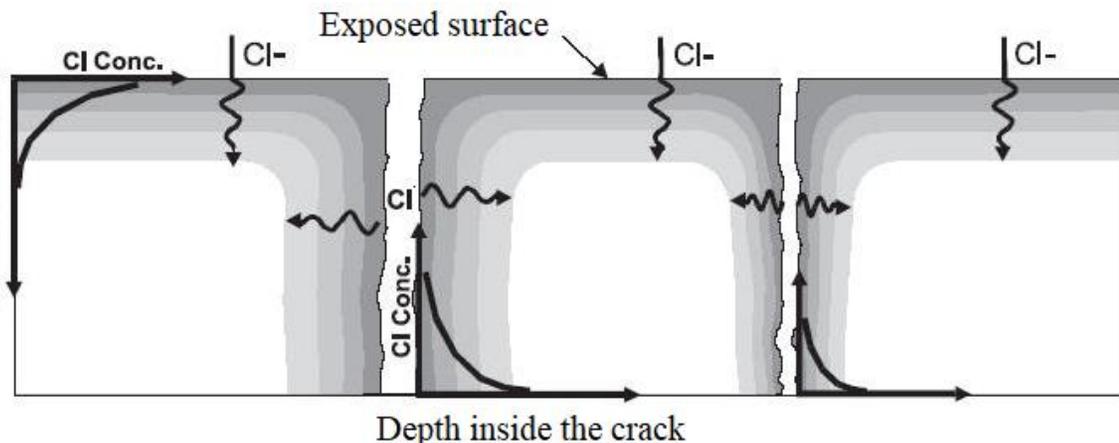


Figure 1. Penetration of chlorides on the surface and between cracks (Ismail et al., 2006).

Cracks act as a gateway for aggressive agents, which have a significant effect on the diffusion of chlorides, as they facilitate the movement of these agents through concrete, the intensity of which is directly dependent on crack opening (Wang et al., 2016).

When the chloride ions are inside the crack, they can penetrate at different depths, starting from the surface inside, becoming dissolved in the aqueous phase of the pores, forming the free chlorides, which can trigger the process of material deterioration. It is also observed that the concentration of chlorides is high on the exposed surface of the concrete. However, when it penetrates the crack opening, it decreases with the increase in depth from the surface of the material (Ismail et al., 2006; Figueredo, 2005; Win et al., 2004).

In addition to free chlorides in concrete pore solutions, you can also find them: chemically combined with CSH, or as chloroaluminates, physically adsorbed to pore walls or free in concrete pore solutions (Romano, 2009; Crauss, 2010).

Of the chloride ions that penetrate the concrete, part binds to tricalcium aluminate ( $C_3A$ ) forming mainly calcium chloroaluminate, also known as Friedel's salt -  $C_3A.CaCl_2.10H_2O$ , which is incorporated into the hydrated cement phases. Another part is absorbed on the surface of the pores and the rest is dissolved in the aqueous phase of the pores, which form the free chlorides that are dangerous and cause damage to the structure (Helmuth and Stark, 1992; Figueredo, 2005; Crauss, 2010).

In general, there will always be a state of balance between the three forms of occurrence of these ions, so that there will always be a certain content of free  $Cl^-$  in the liquid phase of the concrete (Helmuth and Stark, 1992; Fortes and Andrade, 1995; Cascudo, 1997). Cements with low levels of  $C_3A$  have less ability to immobilize chloride ions by the formation of hydrated calcium chloroaluminate. With the formation of this compound, there is a decrease in the concentration of free chloride ions in the aqueous solution of the concrete pores.

The penetration of chlorides in the form of free chlorides depends on factors such as the type of positive ions (cations) associated with the chlorides, the moment of access to the concrete before or after it hardens, the presence of another negative ion (anion) such as sulfate, the type of cement used in the production of concrete, the quality of concrete production and curing, the ambient humidity, the water/cement ratio, the carbonation state and the consumption of cement per  $m^3$  of concrete. Analyzing the resistance to the penetration of chloride ions, Leng et al. (2000) and Oh et al. (2002) found that the diffusion of chloride ions increases with the increase in the water/cement ratio, and cements with fly ash and blast furnace slag have high resistance to diffusion (Helene, 1997; Song et al., 2008; Lawrence, 2006).

In this context, this article presents an analysis of the durability in reinforced concrete under different loading conditions, during a period of 24 months. Depth of penetration of chloride ions was analyzed, in addition to the microstructural analysis.

## 2. PROCEDURE

The experimental program was carried out following these steps: characterization of the material and dosage of the specimens. After dosing, two types of specimens were molded (prismatic and cylindrical).

After molding and deformation, the prismatic specimens underwent wet curing for 7 days, then different types of loads were applied and they were subjected to salt spray, in a natural environment until the test dates (ages 6, 12, 18 and 24 months). At these ages, chloride depths were evaluated, and Microstructural Analysis (SEM and EDS) was performed.

As for the cylindrical specimens, after molding, they were deformed and cured (wet curing) for 28 days. At the end of this period, tests of axial compression strength were performed.

The study ended with the analysis of the results and final considerations. All these steps will be

more detailed below.

As for the materials, the Portland cement used in this study was CP III 40-RS (Portland Blast Furnace cement), whose granular slag content of blast furnace in the cement can reach 75%. The following materials were used for the composition of the mixture: river sand (medium), available and used in the region of Campinas - SP, crushed stone 9.5 / 25 (B1 - basalt) with a maximum characteristic dimension of 19mm and polyfunctional base additive of lignosulfonate, with a specific mass of 1.18 g / cm<sup>3</sup>. In the production of the prismatic concrete specimens, a CA50 steel bar with a nominal diameter of 10 mm was used.

The experimental mixtures were chosen according to the dosage of the concrete used in this research, whose proportion of agglomerate; aggregates adopted was 1:5. In this sense, meeting this requirement, the adopted mix was 1: 2: 3 (cement: sand: gravel) (in mass), with a water / cement ratio of 0.42.

The choice of cement: sand: gravel ratio of 1: 2: 3 was due to its good mortar content. The water / cement ratio of 0.42 was adopted to obtain more resistant concrete when exposed to aggressive media, according to NBR 6118 (ABNT, 2014). The class adopted for the cast concrete in this study was C50. The mix and the water/cement ratio adopted in this study have been used and evaluated since 2000 within the research project on durability of the covering layer, at Unicamp's building materials laboratory.

The amount of additive used was necessary to maintain an adequate workability for the molding of the specimens on a vibrating table, due to the low water/cement ratio used. The consistency index, determined by the slump test ( $4 \pm 1$  cm), was found by means of a cone trunk (according to NBR 16889, 2020). This abatement value, although not widely used in current studies, was adopted for concrete, since it was desired to obtain a minimum workability in which the concrete was molded to produce prismatic specimens with mechanical vibration.

The consumption of materials used for the molding of the prismatic and cylindrical specimens can be seen in table 1.

Table 1. Specification of the materials used in the experimental study.

	<b>Cement (Kg/m<sup>3</sup>)</b>	<b>Additive (Kg/m<sup>3</sup>)</b>	<b>Sand (Kg/m<sup>3</sup>)</b>	<b>Gravel (Kg/m<sup>3</sup>)</b>	<b>Water (Kg/m<sup>3</sup>)</b>
1 : 2 : 3 : 0,42	398	2,4	796	1194	167

Cylindrical specimens were used to characterize the concrete, evaluating its mechanical property (resistance to axial compression) (Table 2).

Table 2. Series of cylindrical specimens.

<b>Series</b>	<b>Type or curing</b>	<b>Date of test</b>
M6um	Immersed until the test date	28 days
M12um		
M18um		
M24um		

The mixture used was mechanical, using a concrete mixer with an inclined axis. For each series, 4 cylindrical specimens with 10 cm in diameter and 20 cm in height were molded and tested at the age of 28 days, according to NBR 5739 (ABNT, 2018). The specimens were molded in two layers, on a vibrating table, in the time necessary to allow adequate compaction of the concrete in the mold, according to NBR 5738 (ABNT, 2015).

After molding, the specimens were covered with plastic canvas until the moment of deformation, which occurred 48 hours after the moment of molding. This release period was adopted due to the deformation of the prismatic specimens. Then, the specimens were subjected to immersed curing for 28 days.

Six prismatic specimens were molded for each age (6, 12, 18 and 24 months), 2 specimens of the sample without loading, 2 specimens of the sample under central loading of short duration and 2 specimens of the sample under permanent central loading, in the dimensions of 1.39x0.1x0.1m. The molds were defined in these dimensions because they are the same used in other research studies carried out at Unicamp's Building Materials Laboratory.

The mixing of the concrete to produce prismatic specimens was mechanical in a concrete mixer with an inclined axis. A CA50 steel bar with a nominal diameter of 10 mm ( $\varnothing 10$ ), with a nominal coverage of 30 mm was used, although steel corrosion is not the scope of this work. The coverage adopted (30 mm) was defined according to the study carried out by Martins (2001), in addition to the study by Midness and Young (1981), Illston (1994), Alexander et al. (2001) and Figueiredo and Nepomuceno (2004).

The prismatic specimens were molded two by two. To maintain coverage, 3 spacers of 30 mm were placed along the steel bar. Figure 2 shows the dimensions of the prismatic specimen and the position of the steel bar.

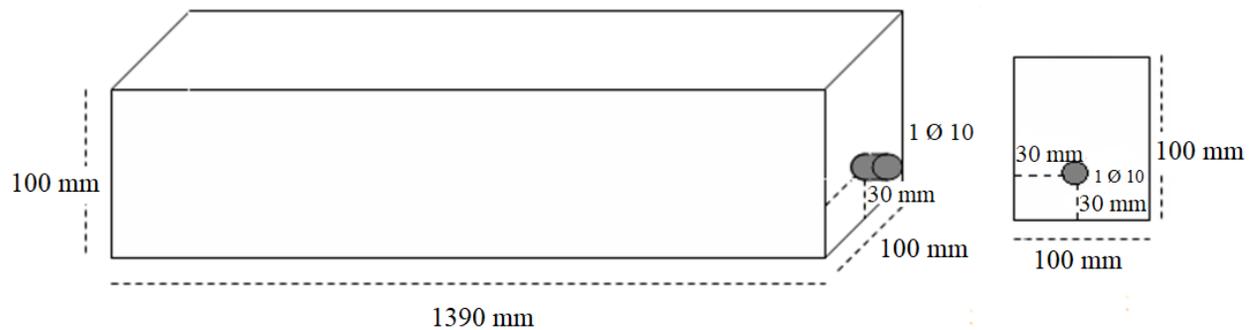


Figure 2. Scheme of dimensions of the prismatic specimens.

After molding, the prismatic specimens remained in the molds for 48 hours, covered with plastic canvas. This time was adopted because it was not possible to deform and mainly to transport these specimens before 48 hours, as they cracked during handling.

After this period, they underwent wet curing, when they were immersed in lime-saturated water until the age of 7 days. This period was defined based on the recommendations of Thomaz (2005), Castro (2003), Braun (2003) and ACI 308 (2016), which determined at least 7 days of curing, regardless of the type of cement adopted. In addition to the references, the age of 7 days of curing was adopted because this is the term used in Brazilian studies for curing structural pieces.

The way of loading the prismatic specimens was defined, which would be according to the following situations:

- Without loading (SC). It was adopted to serve as a reference in the comparison between the two other types of loading.
- Short-term central charging (CCCD). Application of a concentrated force  $P$  to the prismatic specimen until the first crack appears, and then removed. We opted for the choice of this load to check the performance of the concrete in a situation of cracks that may arise over the life of the structure due to the action of point loads, that is, cases in which there is a short-term overload on the structure, with the appearance of cracks. In this case, as the loading is of short duration, the crack may disappear when loading is removed, but stresses and internal micro-cracks in the material have already occurred.

- Permanent central loading (CCP). The prismatic specimens were loaded until the date of durability tests. This type of loading was chosen so that the prismatic specimen had superficial cracks and remained open throughout the exposure period, until the test dates. Thus, on the test dates, the influence of the crack on the depth of chloride penetration and on the microstructure of the concrete can be observed.

For the maximum crack opening, the limit established by the NBR 6118 standard (ABNT, 2014) for durability, related to cracking and protection of the reinforcement, according to the class of environmental aggressiveness, was adopted as a parameter. Thus, the maximum crack opening in the prismatic specimens, which were under constant loading, was between 0.3 mm and 0.4 mm (Figure 3). The openings of cracks were mapped over time, up to the test date of the specimens.



Figure 3. Measurement of the crack opening in the center of the prismatic specimen with a crack meter.

The prismatic specimens were under constant loading until the test dates. The diagram shown in Figure 4 shows the prismatic specimens supported by cylindrical concrete specimens (10 x 20 cm) with a central porch composed of two steel plates and two threaded bars (9.5 mm, 3 nuts, 3 washers and two steel plates of 8 x 300 x 100 mm) applying the load of 250 kgf, causing holes in the center of the beams, with openings of up to 0.3 mm (within the limits of the NBR 6118 standard). The loading was carried out by applying a torque of 0.5 Kgf·m, with a torque wrench, to the threaded bars and, consequently, the concentrated force was applied to the prismatic specimen causing the crack.



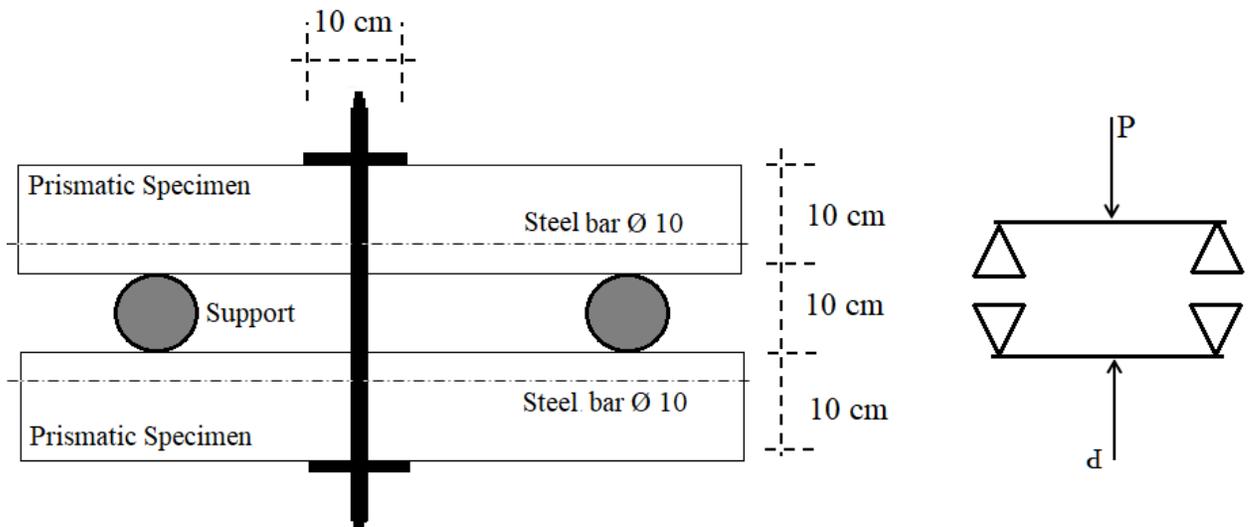


Figure 4. Permanent central loading (CCP) in the prisms.

Table 3 shows the sequence adopted for the samples of each molding series, under different loading conditions and durability test dates.

Table 3. Samples of each molding series, under loading conditions.

Sample	Sample Without loading	Sample under Loading Short Term Center	Sample under Permanent Central Loading	Date of trial
M6um	M6umSC	M6umCCCD	M6umCCP	06 months
M12um	M12umSC	M12umCCCD	M12umCCP	12 months
M18um	M18umSC	M18umCCCD	M18umCCP	18 months
M24um	M24umSC	M24umCCCD	M24umCCP	24 months

After a seven-day wet curing, the specimens passed through saline mist until the test age. The purpose of the mist was to simulate a saline environment. In that sense, a solution of NaCl was manually sprayed on the prismatic specimens until their moistening, with a periodicity of 3 times a week and three times a day.

The salt concentration in sea water is 35 grams of NaCl for each liter of water. In this sense, this amount of 35 g/l of NaCl was used for the solution simulating the salt spray.

The spraying of the chloride solution occurred until the testing dates for the prismatic specimens. The choice of this methodology was based on the study by Arya and Darko (1996), who carried out corrosion tests on concrete beams using the same procedure.

To determine the depth of penetration of chlorides, the cross section of the samples was broken, whose evaluation is carried out by spraying silver nitrate (0.1N solution) on the concrete surface, causing a photochemical reaction, where the free chlorides present in the concrete react with the silver ions of the silver nitrate solution to form a white precipitate. In areas where there are no combined chloride or chloride ions, there is the appearance of a brown color, the silver oxide, due to the reaction between the silver ions and the hydroxyls present in the pores of the cementitious material (Jucá et al., 2002; Real et al., 2015). Through this method, the objective is to evaluate whether the chloride front has reached the reinforcement. The steps of the test can be seen in Figure 5.

The cut in these prismatic specimens was carried out in the middle thirds, where you can see the compressed and stretched areas of the prismatic specimens.



Figure 5. Chloride penetration tests with silver nitrate spray.

For the microstructural analysis, samples were taken from the prismatic specimens immediately after they were cut or ruptured and before the durability tests were performed, so that the samples were not contaminated with silver nitrate solutions (Figure 6). In this study, only the results of microstructural analysis were presented at 6 months and 24 months, which were the first and last ages of trials, respectively.

The samples were taken from the covering areas, with a maximum depth of 1.5 cm. Samples were taken from cracked specimens, in the cracked area (the sample was taken exactly at the crack opening or the closest to it), always in the ruptured region of the prismatic specimen, to be observed by SEM. Since concrete is not a conductive material, the samples had to be metallized with gold. These samples were taken with a steel chisel and hammer. The observations were made at the National Laboratory of Luz Sincontron (LNLS), in Campinas, State of São Paulo.

The energy measure (EDS) was adopted in this study. In this case, there is the advantage of quick identification of the chemical elements present (Dedavid et al., 2007).

Area where the sample was removed for microstructure tests.

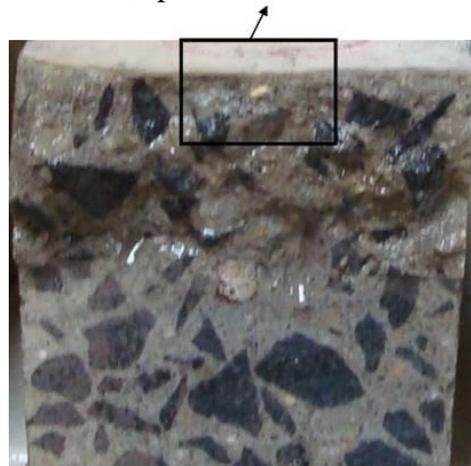


Figure 6. Place where the sample was taken for microstructure tests.

The results obtained were analyzed using descriptive statistics techniques (calculation of the mean, standard deviation) to characterize the variables (concrete properties and behavior regarding chlorides).

To determine statistically significant differences (the level of significance adopted was 5%)

between the averages of the results, parametric hypothesis tests -ANOVA and DUCAN (Montgomery, 1991) were used.

The stat graphics program was used to perform the statistical tests. The program built several tests to compare the chloride penetration averages between all samples. The F test of the ANOVA table verified if there are significant differences between the averages of the results, in relation to the types of cure and loading.

### 3. RESULTS

The results of resistance to axial compression at 28 days of age of the concrete, referring to the moldings made on the cylindrical specimens are shown in Table 4.

Table 4. Results of resistance to axial compression.

Concrete (cylindrical specimens)	Curing of cylindrical specimens	Age of rupture	Resistance to axial compression (MPa)	Standard deviation	Coefficient of variation
M6um	Immersed up to 28 days	28 days	66.2	4.3	6.6
M12um			50.2	2.6	5.3
M18um			59.7	4.1	7.0
M24um			56.8	2.6	4.6

As it can be seen in Table 4, the averages of the results of resistance to axial compression were in the range of 44 MPa to 66 MPa and the results decrease in this order: M6um, M18um, M24um, M12um. This difference in results may have been caused by several reasons, such as temperature and humidity at the time of molding, as well as the transportation, mixing, casting, densification and curing of the concrete used.

As for the variation coefficient, when using the parameter of the ACI 214R (2002) standard to verify how the variation attributed to sampling, sample preparation, curing and laboratory testing was, it is observed that, in the classification presented by this standard, the coefficient of variation of the M12um and M24um samples are considered good ( $<5.5$ ), with the exception of the M18um and M6um samples, which despite high resistance values, are considered weak ( $> 5.5$ ).

As verified in the literature, the compressive strength of concretes with the use of cements with high levels of slag tends to increase with time and the resistance gain can occur in longer periods. Studies, such as the one by Khatib and Hibbert (2005), point to an even greater growth after 28 days of age. In this sense, one can expect an even greater improvement in the resistance results of this study.

Throughout the period that the prismatic specimens submitted to permanent central loading were exposed in the environment, mapping of the cracks was performed, as shown in Figure 7 (6 months old) and Figure 8 (24 months old).

M6umCCP

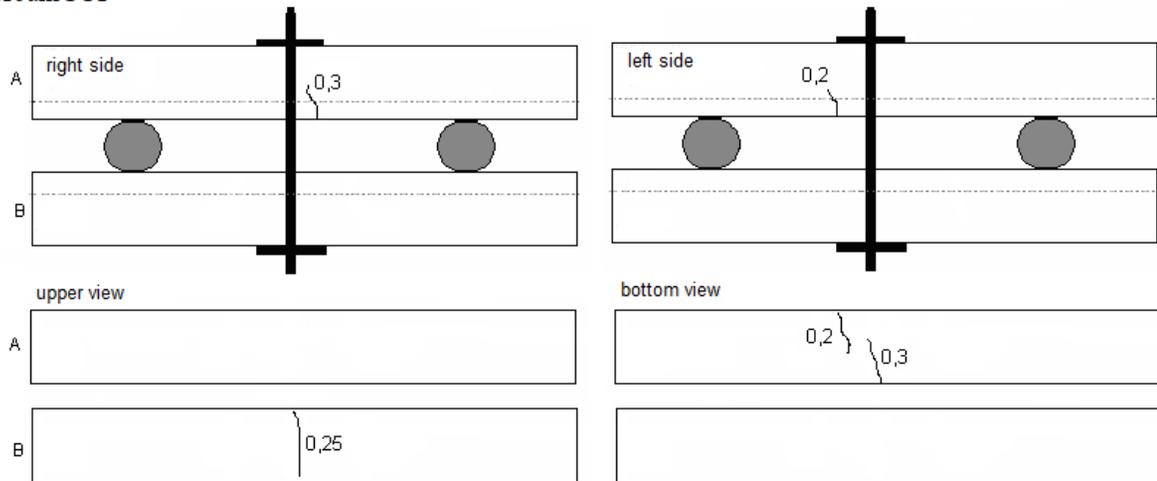


Figure 7. Mapping of cracks in concretes that have been under permanent central loading for 6 months (M6umCCP).

The M6umCCP concretes in Figure 7 had crack openings of 0.2 mm and 0.3 mm. Over the exposure period there was an increase of 0.3 mm to 0.4 mm and 0.25 mm to 0.35 mm.

M24umCCP

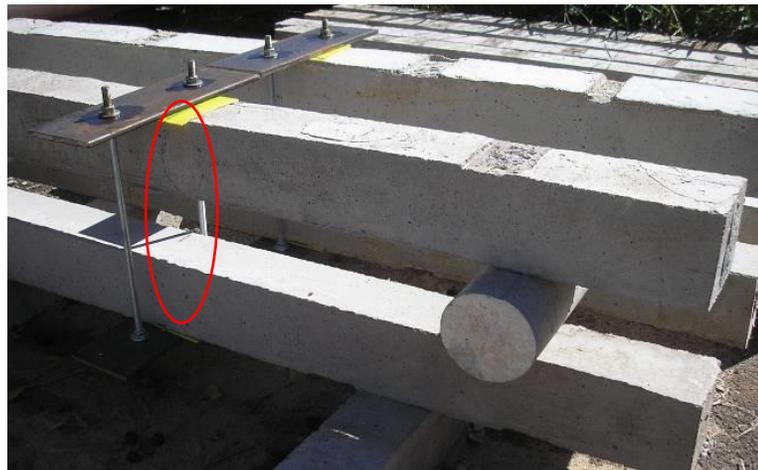
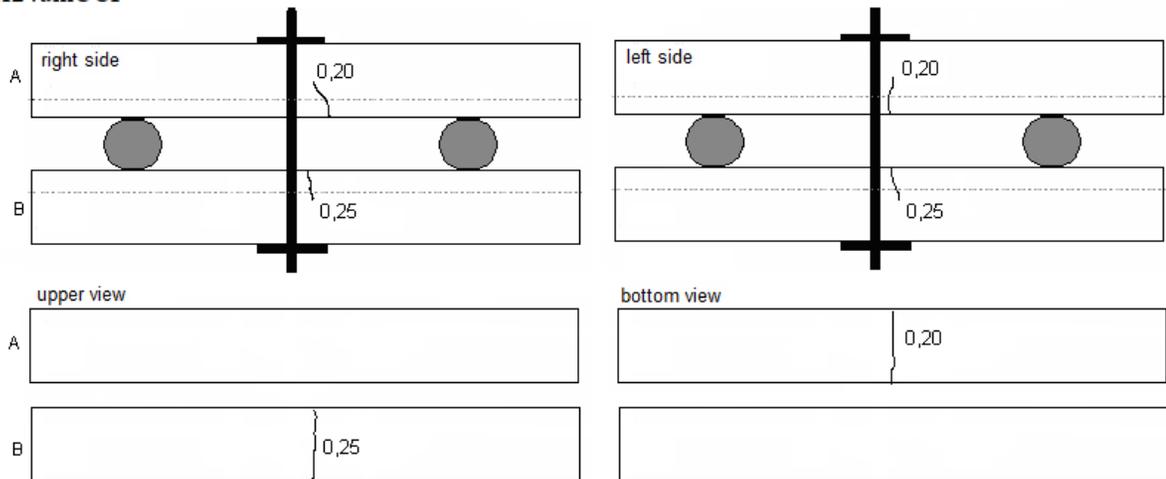


Figure 8. Mapping of cracks in concretes that have been under permanent central loading for 24 months (M24umCCP).

It was observed in the first six months of age that the gantry used for the application of the loading, as well as the monitoring of the crack opening was effective, since the crack opening was in the range of 0.3 mm and 0.4 mm, as outlined in the experimental program.

The cracks in the M24umCCP prismatic specimens (subjected to wet curing) had openings ranging from 0.20 mm to 0.25 m. At 24 months, the behavior of the fissures was like that of other ages. The initial openings were from 0.2 mm to 0.25 mm, reaching 0.4mm at the test ages.

Some factors may have caused the increase of crack openings in the specimens over time, among which we can highlight the load permanently applied to these concretes and the time they were subjected to this load. It was expected that the crack opening would not remain constant due to the material's own properties (such as creep), even with the support of the steel bar. The torque applied to the specimens may also have influenced the crack openings, because although the torque value has been defined experimentally, each concrete (at the ages of testing and curing) may present different responses, as observed in the mapping, in which some samples had larger crack openings. In studies carried out by Vidal et al. (2004; 2007) and François et al. (2006), which mapped crack openings resulting from loading, under saline environment for 14 and 17 years, respectively, two types of cracks can be observed: the transversal, induced by the loading action (bending); and the longitudinal ones in the beam that refer to the corrosion of the reinforcement, that is, cracks that coincide with the reinforcement.

### 3.1 Depth of chloride penetration

Table 5 shows the results of depth of penetration of chlorides in concretes without loading, under short-term central loading and under permanent central loading.

Table 5. Depth of penetration of chlorides under different loading conditions and without load application.

Sample	Average (mm)	Standard deviation
M6umSC	0.1	0.1
M12umSC	2.2	0.6
M18umSC	2.3	1.3
M24umSC	2.6	0.7
M6umCCCD	0.2	0.0
M12umCCCD	2.2	0.7
M18umCCCD	1.9	1.1
M24umCCCD	2.5	0.6
M6umCCP	3.3	1.8
M12umCCP	1.7	0.8
M18umCCP	0.3	0.0
M24umCCP	3.0	1.0

Table 6 shows the statistical comparisons between the three types of loading used in this study: one-off permanent loading - CCP, short-term loading - CCCD, without loading - SC, at the different test ages, for the results of chloride penetration.

Table 6. Comparison among the results of chloride penetration.

Concrete (concrete bodies) prismatic test)	Test Age	Difference between results of chloride penetration
M6umCCP – M6umCCCD	6 months	No (chloride penetration CCP > chloride penetration CCCD)
M6umCCP – M6umSC		No (chloride penetration CCP > chloride penetration SC)
M6umCCCD - M6umSC		No (chloride penetration CCCD > chloride penetration SC)
M12umCCP – M12umCCCD	12 months	No (chloride penetration CCP < chloride penetration CCCD)
M12umCCP – M12umSC		No (chloride penetration CCP < chloride penetration SC)
M12umCCCD – M12umSC		No (chloride penetration CCCD = chloride penetration SC)
M18umCCP – M18umCCCD	18 months	No (chloride penetration CCP < chloride penetration CCCD)
M18umCCP – M18umSC		No (chloride penetration CCP < chloride penetration SC)
M18umCCCD – M18umSC		No (chloride penetration CCCD < chloride penetration SC)
M24umCCP – M24umCCCD	24 months	No (chloride penetration CCP > chloride penetration CCCD)
M24umCCP – M24umSC		No (chloride penetration CCP > chloride penetration SC)
M24umCCCD – M24umSC		No (chloride penetration CCCD < chloride penetration SC)

At 6 months and 24 months, chloride penetration was higher in concretes subjected to permanent central loading compared to concretes under short-term central loading and without loading; however, in these cases there was no significant difference between the results. At 12 months of age, concretes with permanent central loading had lower chloride penetration values than concretes subjected to short-term loading and without loading, in which case there were also no significant differences between the results.

Over 24 months of age, the loading action, whether short-term or permanent, did not significantly influence the results of chloride penetration in almost all studied ages.

When comparing these results with those obtained in research by Vidal et al. (2004), Vidal et al. (2007) and François et al. (2006), it is observed that the time factor and the saline environment are fundamental for the loading and, consequently, the opening of cracks, influences the results of penetration of chlorides. Research such as that by Vidal et al. (2007), which left the samples exposed in environments under salt spray for longer than 10 years, obtained significant results only after 5 years of exposure. In this sense, longer periods should be considered in future research.

By analyzing the behavior of samples with different percentages of blast furnace slag, subjected to continuous loading, An Cheng et al. (2005) found that crack opening affects the time of onset of reinforcement corrosion. The specimens with the greatest crack opening were the ones that started the corrosive process. However, the amount of slag added to the mixture did not influence the results, that is, with the increase in the blast furnace slag content there was no decrease in the speed and propagation of reinforcement corrosion.

According to Ayra and Darko (1996), the frequency of the appearance of cracks has an influence on the intensity of corrosion that the structure is subjected to. The greater the number of cracks, the greater the intensity of corrosion in reinforced concrete. A fact to be highlighted, in this case, is the importance of the concrete cover to reduce this process. Likewise, this thickness can be as important a factor in this context as the incidence of cracks in the structure itself. Thus, attention is paid to the importance of studies on covering concrete, which makes it possible to obtain relevant information that contributes to the production of durable concrete.

As for the curing period adopted, Thomaz (2005) found that the wet curing time of 7 days is sufficient for the concrete to acquire the desired properties. However, this period depends on the type of cement and the water/cement ratio used. However, when using cements with mineral additions, longer curing time is necessary, as it is the case with cement with blast furnace slag,

whose hydration process is slower than that of ordinary cement (Çakir; Akoz, 2006; Furnas, 1997; Thomaz, 2005).

It is also observed in studies such as that by Castro (2003) and Braun (2003) that cements with a higher content of addition, such as CP III 32, require a longer curing period, compared to other types of cements.

### 3.2 Evaluation of the microstructure

At six months of age, only two types of samples were selected for microstructure tests. These samples were taken from the traction region of the prismatic specimens without loading (SC) and subjected to Permanent Central Loading (CCP). Both were in wet cure for 7 days before undergoing shipments. Figure 9 shows a sample without loading.

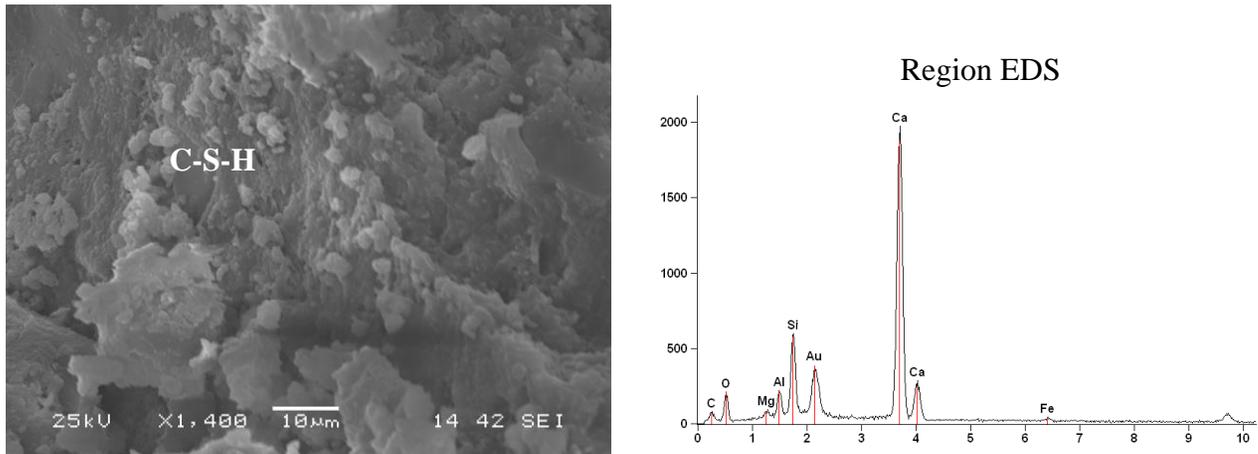


Figure 9. Concrete subjected to wet curing, without loading, at six months of age.

Figure 9 shows an area with dense and amorphous morphology (C-S-H Type III or IV), which is more common in this case because it is a sample at older ages. Cheng et al. (2005) observed in the concrete micrographs with different levels of blast furnace slag in high percentages, a denser structure, with few ettringite needles and with capillary pores smaller than 50 nm, which may have been filled by products like CSH. The Au peak appeared in the EDS in the region of Figure 9 may be due to metallization with gold.

The C-S-H phase occupies a volume between 50% and 60% of cement paste solids, being the main responsible for the properties of the paste, such as the axial compression resistance. Its structure depends on the temperature and the free space in the mixture for its hydration (Baroghel-Bouny, 1994; Irassar, 2004).

This phase can be found in the following morphologies: Type I - fibrous, usually in the form of “hedgehog”, when hydration is developing (early ages) out of the  $C_3S$  grain with sufficient available space; Type II - alveolar or reticulate, also called “honeycomb”, which occurs in conjunction with C-S-H Type I; Type III and IV - prominent at older ages, constitutes a dense and amorphous morphology, difficult to define and can constitute a good portion of the total hydrated products. The characteristic products in more advanced stages of hydration are C-S-H type III and IV and more  $Ca(OH)_2$  (Taylor, 1997; Ghosh, 2002).

According to Kurdowski (2014), there are 4 morphological forms of the C-S-H phase: fibrous, mesh, isometric and spherical conglomerate particles, belonging to the internal C-S-H and identified as a firm gel under an electron microscope.

Figure 10 shows the microstructure of the concrete subjected to permanent central loading in wet curing for 7 days, at six months of age; and Figure 11 shows the microanalysis in three points of this same concrete.

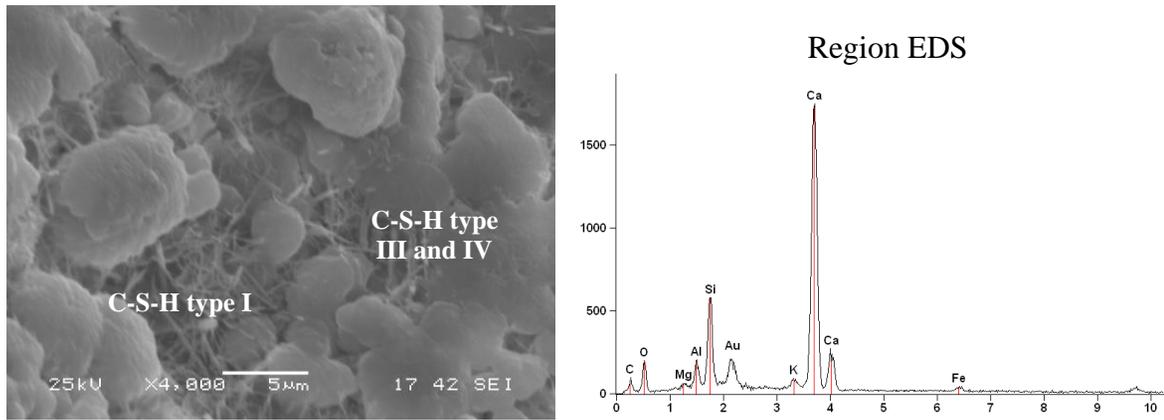


Figure 10. Concrete subjected to wet curing for 7 days and permanent central loading, at six months of age.

In the image of Figure 10 some needles are observed, but in the EDS the element sulfur (S) is not present to configure an ettringite. In this case, they can be C-S-H needles, with fibrous morphology (Type I) (Taylor, 1997; Ghosh, 2002). Phases C-S-H type III and type IV are also observed. In blends using blast furnace cement, the C-S-H fibrillar morphology of Portland cement without additions (Portland clinker) is gradually replaced by a different morphology, which Richardson (1999) calls “sheet” or “sheet type”. This author reports that this change in morphology is responsible for the better performance and greater durability of mixtures with this addition. Morphologies not yet observed at the age of 6 months of testing.

Figure 11 shows three selected points of the concrete subjected to wet curing for 7 days and permanent central loading. In the elementary composition by the EDS of the three points, the same elements Ca, Si, Al and Mg are found.

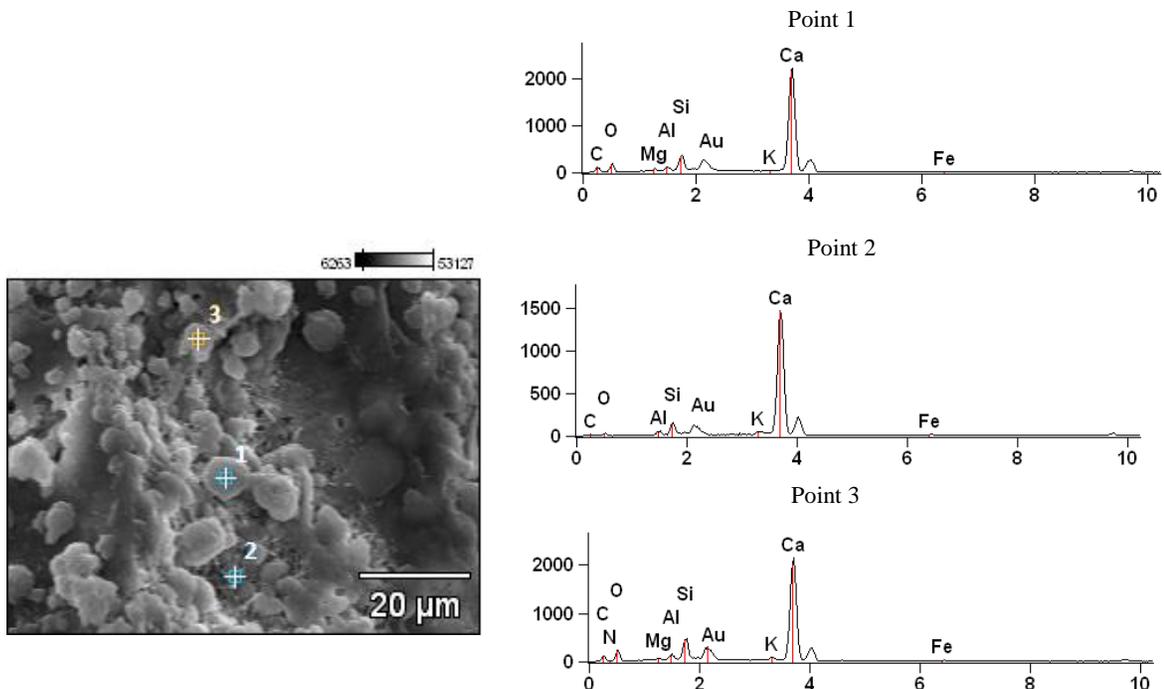


Figure 11. EDS microanalysis at three points of the concrete under permanent central loading (CCP).

In the microanalysis, we detected the presence of the elements Ca, K, Si, Al, Mg, O and C, which are typical of cement hydration products.

The C-S-H resulting from the hydration of Portland cement and blast furnace slag has similar morphologies; however, the slag grain has high percentages of Mg and Al (Richardson, 1999).

In Figure 12, a micrograph of a sample of unloaded concrete is presented, submitted to moist curing for 7 days, at 24 months of age.

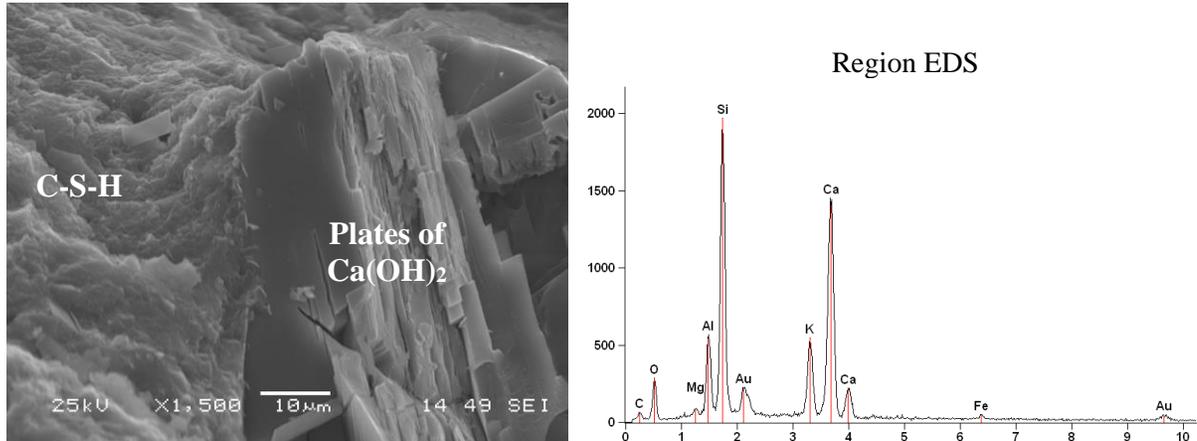


Figure 12. Micrograph and microanalysis of the unloaded concrete sample, at 24 months of age.

Figure 12 shows the presence of possible Ca(OH)<sub>2</sub> and C-S-H plaques. As verified by Baroghel-Bouny (1994) Mehta and Monteiro (2008), the phase corresponding to Ca(OH)<sub>2</sub> maintains the high alkalinity of the system, preserving the stability of the C-S-H and the concrete cover.

Ca(OH)<sub>2</sub> occupies a solids volume of 20% to 25% in the hydrated cement paste. Because of its composition with defined stoichiometry, they are formed in large crystals with hexagonal prismatic morphology. This morphology can also vary depending on the hydration temperature and the impurities present. Due to these factors, piles of large plaques can be formed.

This phase maintains the high alkalinity of the system, preserving the stability of the C-S-H and the reinforcement covering layer (Baroghuel-Bouny, 1994; Castro, 2003).

Figure 13 shows the micrographs and EDS of the concrete subjected to short-term central loading, at 24 months of age.

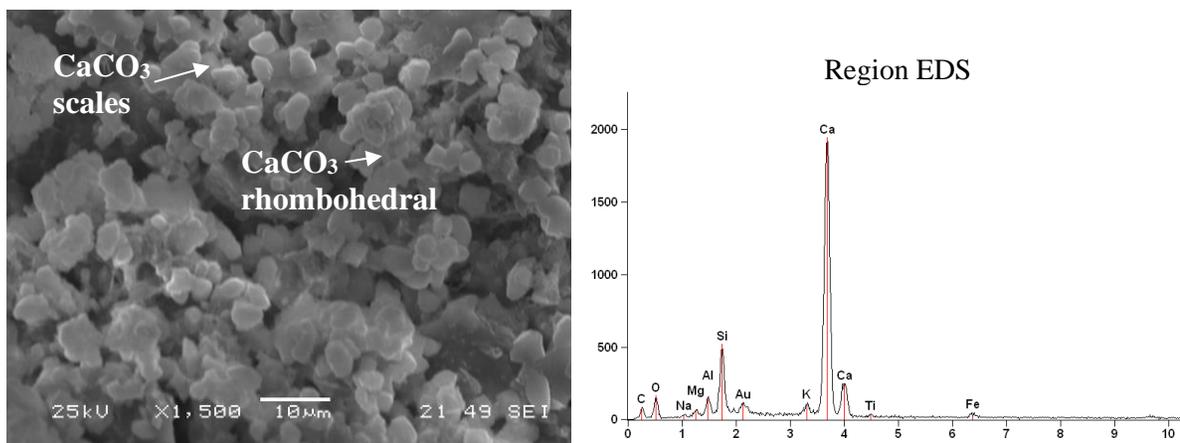


Figure 13. Micrograph and microanalysis of the concrete sample submitted to short-term central loading, at 24 months of age.

In Figure 13, it is possible to observe the formation of calcium carbonate in the form of rhombohedral crystals and in the form of scales, on a porous and possibly carbonated C-S-H. It is observed in the hydrated compounds formed, the presence of calcium carbonate ( $\text{CaCO}_3$ ), with different morphologies on C-S-H, which may also be in the process of being modified by the action of time, and due to its porosity. The porous C-S-H phase and the formation of  $\text{CaCO}_3$  indicated in Figure 13 was also observed by Sakar et al. (2001) in their study, with  $\text{CaCO}_3$  in the form of scales, however, in large quantities.

The salts dissolved in sea water are mainly chlorides and sulphates. In the case of chloride ions ( $\text{Cl}^-$ ), when in contact with alumina, the monochloroaluminate crystallizes in the form of unstable hexagonal plates. Chloride enters the crystalline network of hydrated silicates (C-S-H) and transforms the fibers into reticulated networks, making this phase more porous. Regourd et al. (1980) also observed the presence of  $\text{CaCO}_3$  in micrographs of concrete samples (hydrated Portland cement compounds) exposed to salt spray.

Figure 14 shows the micrograph and microanalysis of the concrete subjected to permanent central loading (CCP), at 24 months of age.

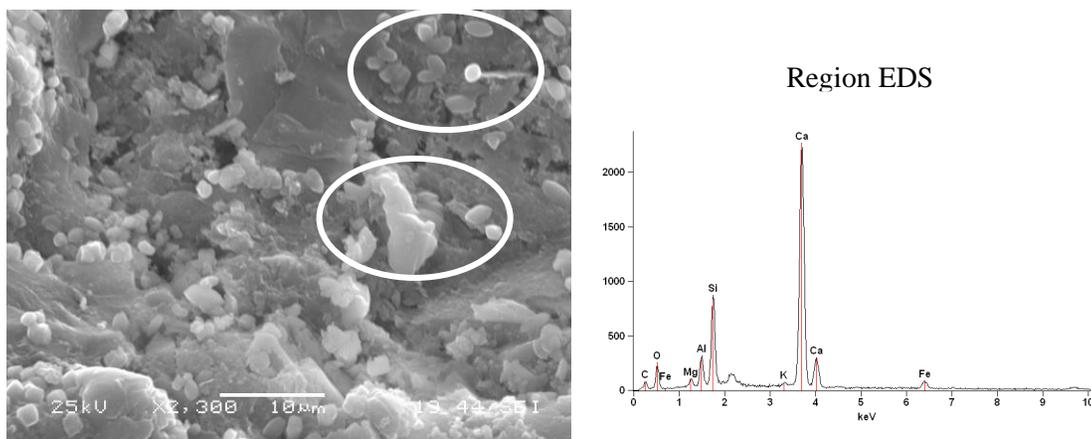


Figure 14. Micrographs and microanalyses of concretes with permanent central loading.

The sample in Figure 14 shows an area with a denser microstructure, with different  $\text{CaCO}_3$  morphologies. Some products that attracted attention, and that are highlighted in this micrograph, due to their morphology, can configure organic matter, probably microorganisms (Ribas Silva, 1996). However, to confirm this hypothesis, a microbiological analysis would be necessary, which was not performed because it was not the object of this study. In this case, the loading-induced cleft could have led to the entry of these microorganisms, since they were not observed in the SC and CCCD concretes.

When comparing the micrographs, as for the CCCD and CCP loads and without loading (SC), it is observed that the sample with greater compactness and less pore quantity is the reference (SC). As for the compounds formed, the carbonated C-S-H phase can be seen in the CCCD concrete micrographs. When subjected to permanent central loading, the C-S-H phase is dense with the formation of microorganisms, and the porosity in this case is not high, probably due to the wet cure at 7 days. And in the reference concrete (SC), there is the formation of  $\text{Ca(OH)}_2$ , as well as the dense and homogeneous C-S-H phase. Thus, the influence of cracks on the durability of the material is observed, since in the concrete with the  $\text{Ca(OH)}_2$  plates there is higher alkalinity of the system and the preservation of the CSH phase, consequently, of the covering layer (Baroghel - Bouny, 1994; Mehta, Monteiro; 2008).

## 4. CONCLUSIONS

From the results obtained from the penetration of chlorides, it was observed that the crack opening did not influence the entry of chloride ions into the structure, with no significant differences between the types of loading and the reference concrete (without loading).

Studies carried out in Europe on this subject use longer periods of exposure to the external environment to obtain more expressive results regarding the behavior of the crack in the penetration of chloride ions in concrete. In this sense, it is seen that for the studied concretes, a longer period would be necessary to find more significant results between the types of loading and without loading. Some factors may also have contributed to this result, highlighting in this case the use of cement with the addition of blast furnace slag, which is more resistant to attack by chlorides, compared to CO<sub>2</sub>. The salt spray used also does not seem to have contributed sufficiently so that there could be differences between the types of loading and without loading.

When comparing the crack opening tolerances of the Brazilian standard with international standards, it appears that the openings allowed for the Brazilian standard are larger. The climatic conditions in Europe, with temperature variations ranging from -5 °C to 30 °C as is the case in France for example, are quite different from those in Brazil, where there are variations that range from 20°C to 35°C. However, the technological control of the quality of materials and execution in Brazil tends to be less strict. Thus, the crack openings can be an aggravating factor in this context. On the other hand, it is observed in this study that in Brazilian climatic situations, up to the age of two years, there were no significant differences between the results of samples under the loading condition determined in this study, the reference one, that is, without loading.

Up to the age of 12 months, the formation of cement hydration products was observed in micrographs and microanalyses, mainly found in the C-S-H phases, in addition to Ca(OH)<sub>2</sub> crystals, ettringite and CSH needles and slag grains with different dimensions. The deterioration products (CaCO<sub>3</sub> and carbonated C-S-H) were found after 18 months of age, in loading and curing situations. However, only at 24 months it is more evident that, in concretes subjected to the types of loading (CCP and CCCD), the presence of CaCO<sub>3</sub> and carbonated C-S-H was observed and in the sample without loading, only phases of C-S-H and Ca(OH)<sub>2</sub> were found. However, in the concrete with crack opening (CCP) possible microorganisms were found, which may have entered through the crack.

From the microstructural point of view, it was observed that at 24 months the loading influenced the results of the studied samples, since in the concretes with crack opening, the presence of microorganisms in samples subjected to permanent loading was visualized under microscopy. These compounds were only found in concretes with crack opening (in permanent central loading). In this sense, it is seen that the fissure may have been a way for these microorganisms to enter, which may have consequences on durability, as verified in the literature.

Thus, it is observed that the crack (when constantly open) can influence the microstructure of the concrete over time, depending on the preferred path for the entry of aggressive agents and microorganisms. This fact was not observed in concretes with short loading and without loading.

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