

## Durability of concrete produced with proven internal curing agents subjected to the action of chloride

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### ABSTRACT

The procedure to promote internal curing is one of the strategies to reduce the shrinkage process of high strength concrete with low water/cement ratio. Various materials have been used as curing agents, such as porous materials, which must meet the minimum requirements defined by ASTM C1761. This research sought to contribute to the development of new ways to inhibit the degradation of reinforced concrete structures through the use of ceramic shell and expanded clay, used as an internal curing agent in concrete subjected to the action of chlorides. Based on the results found, it is possible to verify that the selected aggregates, mainly the ceramic shell, contributed to the improvement of the mechanical properties and durability of the concretes in the evaluated conditions.

**Keywords:** internal curing; porous materials; chlorides action; durability.

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#### Contribution of each author

In this work, the author J. G. Berton contributes with the original idea in 50%, drafting of the work in 90%, experimentation in 90%, collection of data in 100% and discussion of the results in 75%. L. C. P. Silva Filho contributes the original idea in 50, drafts the work in 10%, experiments in 10% and discusses the results in 25%.

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## **Durabilidade de concretos produzidos com agentes de cura interna submetidos à ação de cloretos**

### **RESUMO**

O procedimento para promover a cura interna é uma das estratégias para reduzir o processo de retração do concreto de alta resistência com baixa relação água/cimento. Vários materiais têm sido utilizados como agentes de cura, como materiais porosos, que devem atender aos requisitos mínimos definidos pela ASTM C1761. Esta pesquisa buscou contribuir para o desenvolvimento de novas formas de inibir a degradação de estruturas de concreto armado por meio da utilização de casca cerâmica e argila expandida, utilizadas como agente de cura interna em concretos submetidos a ação de cloretos. Com base nos resultados encontrados, é possível verificar que os agregados selecionados, principalmente a casca cerâmica, contribuíram para a melhoria das propriedades mecânicas e durabilidade dos concretos nas condições avaliadas.

**Palavras-chave:** cura interna; materiais porosos; ação de cloretos; durabilidade.

## **Durabilidad de hormigones elaborados con agentes de curado interno sometidos a la acción del cloruro**

### **RESUMEN**

El procedimiento para promover el curado interno es una de las estrategias para reducir el proceso de retracción del concreto de alta resistencia con baja relación agua/cemento. Se han utilizado varios materiales como agentes de curado, como materiales porosos, que deben cumplir con los requisitos mínimos definidos por la norma ASTM C1761. Esta investigación buscó contribuir al desarrollo de nuevas formas de inhibir la degradación de las estructuras de hormigón armado mediante el uso de caparazón cerámico y arcilla expandida, utilizados como agente de curado interno en hormigones sometidos a la acción de los cloruros. Con base en los resultados encontrados, es posible verificar que los agregados seleccionados, principalmente la cáscara cerámica, contribuyeron a la mejora de las propiedades mecánicas y durabilidad de los hormigones en las condiciones evaluadas.

**Palabras clave:** curado interno; materiales porosos; acción de cloruros; durabilidad.

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## 1. INTRODUCTION

According to NBR 6118 (ABNT, 2014), the durability of reinforced concrete structures can be defined as the capacity of the structures to resist the environmental influences foreseen and defined at the beginning of the project elaboration works, that is, the result of the interaction between the structure, the environment, the conditions of use and maintenance of the structures. Despite all the development of concepts about durability and useful life of structures and advances related to technology in civil construction, it is still possible to verify the early degradation of structures, characterized by pathological manifestations.

According to Pereira da Costa (2015), pathological manifestations in concrete structures are due to design errors, inadequate execution, use, changes in the environment, materials used, or it can come from a set of these factors. Researchers claim that most of these manifestations are the result of design and/or execution failures, which may be due to disregard of the environment in which the structure will be inserted, negligence of actions or even lack of knowledge of existing degenerative processes. According to Bolina, Tutikian and Helene (2019), the appearance of pathological manifestations in buildings can come from numerous factors and from different sources, being produced through simple or combined causes.

Among the factors linked to the occurrence of pathological manifestations, the porosity of the concrete is directly linked to the transport of aggressive agents in the concrete. Open porosity refers to open and interconnected pores, that allow the transport of aggressive agents in the material. According to CEB (1992) and Metha and Monteiro (2014), the pores that affect the transport of aggressive agents in concrete are capillary pores, which are formed due to water evaporation during hydration and curing processes, and macropores. For Silva (2011), the connection between these pores and tortuosity are fundamental properties for determining their structure, and only interconnected pores participate in the substance transport process.

More and more forms are sought to reduce the occurrence of pathological manifestations in reinforced concrete structures. One of the ways studied to search for concretes with greater durability and resistance are those related to the reduction of the water/cement ratio used in the mixture, linked to the internal healing technique that consists of introducing the so-called internal healing agents to the mixture, which are materials capable of act as water reservoirs, which act as regulators in order to keep the internal relative humidity high, and consequently can prevent or even reduce the effects caused by self-desiccation. These internal healing agents are normally porous materials capable of storing and releasing water quickly (CAETANO, 2019). Studies carried out by the author have shown the efficiency of internal healing agents in terms of autogenous retraction and permeability. Likewise, Cusson and Margeson (2010) and Thomas (2006) observed in their studies a reduction in the permeability of chlorides and water in mixtures with internal healing agents compared to concrete using only traditional aggregates.

Thus, the presente research seeks to evaluate the use of ceramic shell and expanded clay as internal healing agents in concrete subjected to the direct action of chlorides through accelerated tests. In this way, in addition to the use of these materials as healing agents through pore structure and mechanical performance tests, their effectiveness will be evaluated regarding the durability of concrete exposed to highly aggressive environments, through the performance of the accelerated corrosion test by modified immersion and chloride penetration.

## 2. EXPERIMENTAL PROGRAM

### 2.1 Materials, determination of the trait, molding and healing

In the same way as performed by Caetano (2019), the studies published by RILEM TC 255 - SAP Newsletter n°1 and ASTM C1761 (2015) were used as a basis for the production of concrete, which presents the requirements that materials must meet to ensure their potential as inner healers. For this research, the crushed waste of the ceramic shell for investment casting, called just ceramic shell, and expanded clay, as shown in Figure 1 below, were used as internal healing agents. CP-V ARI cement was used, given its high clinker content. Fine sand was used as fine aggregate.



Figure 1. Ceramic shell and expanded clay used

Both materials, in addition to the sand used, were tested according to the requirements of ASTM C1761 (2015), showing partial compliance with the minimum requirements established. The ceramic shell is in disagreement with regard to granulometry, unit mass and presence of organic impurities. As for expanded clay, the inconsistencies found are related to granulometry and organic impurities. The analyzed sand is within the established ranges for all analyzed parameters.

The mix defined for the mixture, based on RILEM, as previously mentioned, was defined as 1:1914 of cement and sand with a water/cement ratio of 0.30 and an addition of 10% in mass of silica fume. The proportion of water to be reserved by the aggregate to be used in the concrete hydration process was determined based on the Powers model, considering that for the mix used, it is necessary to add 0.066 kg of water for each 1 kg of cement used. The following presents the quantity of materials used, adapted by the authors based on RILEM TC 255 – SAP Newsletter n°1.

Table 1. Quantity of materials as per RILEM 225 - SAP

Material	Quantitative RILEM TC 225 -SAP (kg/m <sup>3</sup> )
Cement CEM I 42.5 R (Schwenk)	700
Active silica (Elkem, grade 917-U)	70
Water (a/c=0,3)	210
Aggregate (sand (0/4))	1218
Quartz sand (0,06/0,2)	121,8
Superplasticizer - 1,8% of cement	12,6

The internal healing agents were used as partial substitutes for the fine sand used, the theoretical values being used based on the studies carried out by Caetano (2019), with a volume replacement of 45% for the ceramic shell and 67% for the expanded clay.

Previously to mixing the materials, the porous materials (ceramic shell and expanded clay) were immersed for 24 hours in the total amount of water used in the mixture, leading to saturation of the aggregate. After 24 hours, the specimens belonging to each of the traits were molded in a 20 L mortar, as shown in Figure 2. Once molding was completed, all specimens were taken to wet curing for 28 days.



Figure 2. Materials and mortar used

### 2.1 Absorption and void ratio

To determine the void ratios, absorption and specific mass of the microconcretes studied, the parameters described by NBR 9778 (ABNT, 2005) were used. In this way, three specimens of 10 x 20 cm (diameter x length) of each of the studied traits were molded. Figure 4 shows two stages of the test, the first (a) consists of determining the mass of the test specimen and (b) the step in which the test specimens are submerged and brought to a boil for a period of 5 hours.



Figure 3. Steps for carrying out the absorption test and void ratio

### 2.2 Compressive strength

For the analysis of the compressive strength of the concrete produced, the procedure described in NBR 12041 (ABNT, 2012) - High mechanical resistance mortar for floors - Determination of the resistance to simple compression and traction by diametral compression was adopted, since the microconcrete produced present a reduced granulometric distribution similar to that found in mortars, due to the non-use of coarse aggregate.

In this way, 4 specimens of each of the traits were molded for each of the ages tested, 3, 7 and 28 days. The rupture of 4 specimens was also carried out at 75 days, the age at which the chloride penetration tests ended for the purpose of evaluating the resistance on this date. Figure 5 shows the execution of the compressive strength test.

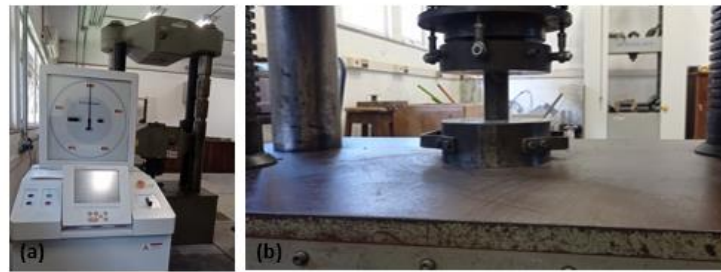


Figure 5. Compressive strength test

### 2.3 Modulus of elasticity

To carry out the modulus of elasticity test of concrete, the parameters set out in NBR 8822 (ABNT, 2002) were followed. In this test, each of the specimens was subjected to 4 loading cycles, with 30% of the value corresponding to the highest compressive strength result of the specimens tested being used in each cycle as the upper stress level. The displacements between the control sections were measured with the aid of LVDTs (Linear Variable Differential Transformer), which were previously positioned on the test specimens, as shown in Figure 6. The values referring to load and displacement are monitored, acquired and stored with aid of a QUANTUN type data acquisition system, using the CATMAN EASY software for visualization and control of the entire test performed.



Figure 6. Elastic modulus test

### 2.4 Penetration of chlorides

To perform the chloride ion penetration test, the parameters established by ASTM C1202 (2022) were followed. The test uses concrete samples with dimensions of 9.5 cm in diameter and 5.1 cm in height, subjected to the application of an electrical stimulus of  $60V \pm 0.1V$  for 6 hours. These cylindrical samples were coupled in half-cells of acrylic, in which the faces were exposed to two different solutions, in one face sodium hydroxide (NaOH) in a concentration of 0.3N, while the other half-cell receives a solution of chloride of sodium (NaCl) with a concentration of 3% in mass. In this way, 10x20 cm specimens were molded and cut and only the 5.1 cm high central layer was used. After cutting, the slices still went through a pre-conditioning. Figure 7 shows the scheme for carrying out the test.



Figure 7. Chloride penetration test

With the measurements of the electric currents during the 6 hours of the test, it is possible to analyze the total passing charge, which according to the referred standard is determined by the following Equation, where Q corresponds to the total passing charge, in Coulombs,  $I_0$  is the measured current immediately after application of voltage, in Amperes and  $I_t$  is the current measured in intervals “t” minutes, in Amperes.

$$Q = 900(I_0 + 2I_{30} + 2I_{60} + \dots + 2I_{300} = 2I_{330} + 2I_{360}) \quad (1)$$

The value corresponding to the passing load is also corrected, shown in the equation below, since the samples have a diameter different from 95 mm.

$$Q_s = Q_x \left(\frac{95}{X}\right)^2 \quad (2)$$

Thus, with the total through load results, it is possible to classify the concrete according to the risk of chloride ion penetration, according to the parameters described by ASTM C1202 (2022), which are shown in Table 2 below.

Table 2. Classification according to the risk of chloride ion penetration

Risk of penetration of chloride ions	Through Load (Coulomb)
High	> 4000
Moderate	2000 a 4000
Low	1000 a 2000
Very low	100 a 1000
Despicable	<100

### 3. RESULTS AND DISCUSSION

This topic presents the results of each of the tests carried out together with their analysis and interpretation. All data displayed in the following topics were treated in order to eliminate spurious values with the help of Excel software.

#### 3.1 Absorption and void ratio

The absorption test according to NBR 9778 (ABNT, 2005) is based on the process in which water is transported into the interior of the concrete and tends to occupy all spaces present in a sample, that is, it is the process in which water occupies the total pores of a sample. Thus, water penetration is forced to dissipate in all directions, allowing the evaluation of the total amount of pores existing in the microconcrete that has been analyzed. Figure 8 shows the graph with the average values of absorption and void ratio determined based on the parameters described by the regulations.

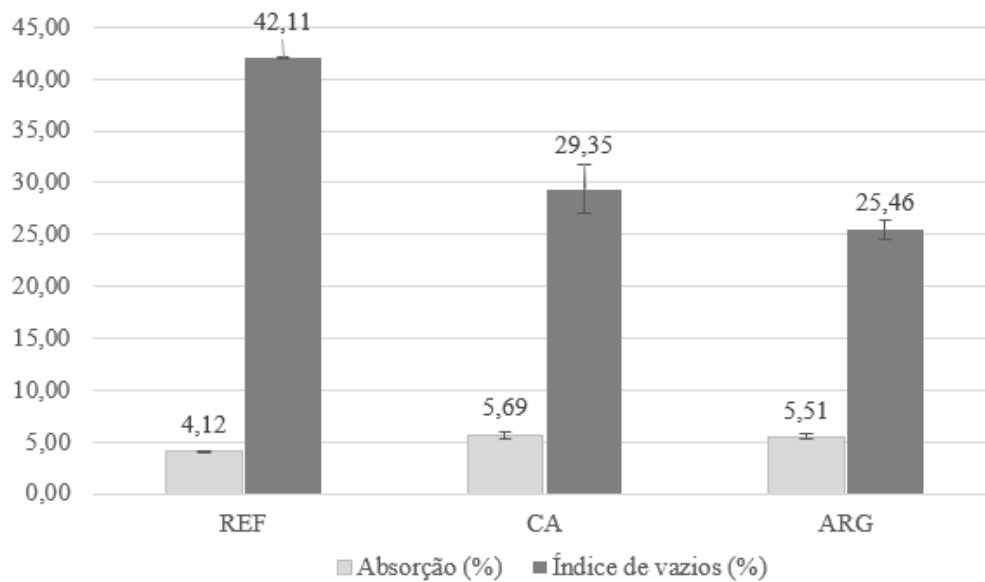


Figure 8. Mean absorption values and void ratio

Observing Figure 8, it can be noted that the microconcrete belonging to the reference mix showed a lower water absorption and a higher voids index when compared to the other two mixes analyzed. The microconcrete with ceramic shell (CA) incorporated into the mixture showed a reduction in the void ratio of 30.30% in relation to the reference mix (REF), while the concrete produced with expanded clay (ARG) showed a reduction of 39.54% also in relation to the reference trait (REF). According to the results found by studies carried out by Kim et al. (2018), Caetano (2019) the decrease in the void ratio of these concretes may have been due to the release of water for internal healing, generating concretes with less pores and less interconnectivity of pores. On the other hand, the absorption values showed a slight increase for both analyzed traits.

### 3.2 Compressive strength

Next, in Figure 9, the values related to the compressive strength of the concrete produced according to the tested ages are presented.

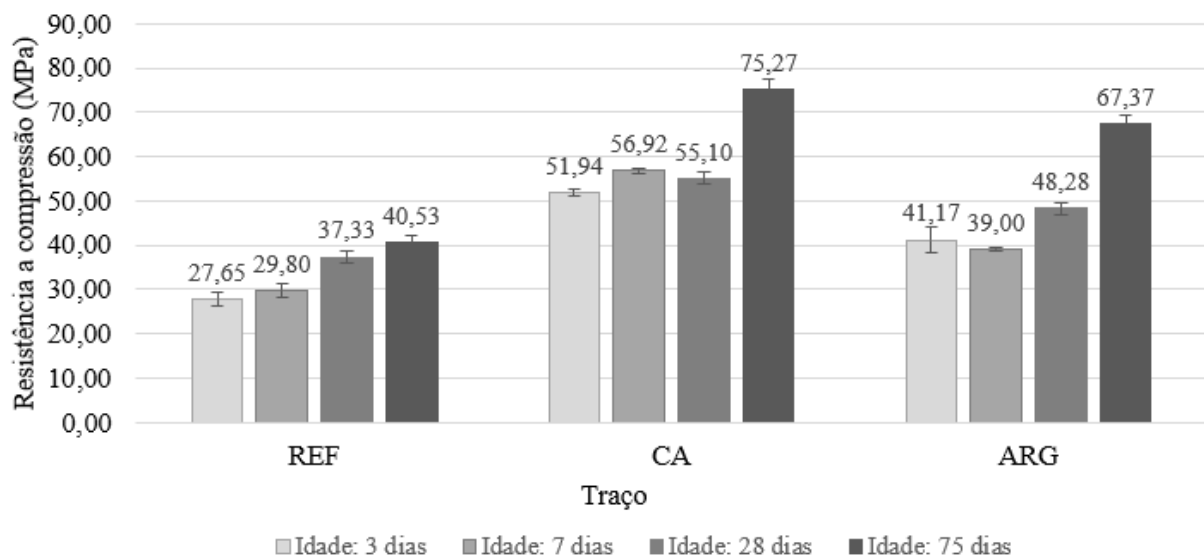


Figure 9. Compressive strength by age



With the analysis of Figure 8, it is possible to see that the additions of ceramic shell (CA) and expanded clay (ARG) provided a resistance gain in relation to the reference mix (REF). It is believed that this improvement in mechanical performance is due to the increase in hydration reactions of concrete due to the internal healing of the same provided by these materials.

In this work it can be seen that the mix with ceramic shell (CAR) showed an increase in resistance in the order of 40% for the ages of 3, 7 and 28 days, compared with the reference trait in the order of 30%, 24.18% and 29.9% for the ages of 3, 7 and 28 days, respectively. When performing the comparison of resistances between the groups with addition of ceramic shell (CAR) and expanded clay (ARG), it is noticed that the ceramic shell showed better results, with resistance greater than the expanded clay in the order of 20.6% , 28.17% and 11.7% for ages 3, 7 and 28 days, respectively. When analyzing the mix with the addition of ceramic shell (CA) and the mix with the addition of expanded clay (ARG), it can be seen that at the age of 75 days there was an increase of 36.6% and 39.54%, respectively, in relation to the age of 28 days. It is believed that this behavior occurred due to the occurrence of greater hydration of the cement matrices given by internal healing, according to the work carried out by De La Varga (2012) and Bayard, Schindler and Barnes (2010).

### 3.3 Modulus of elasticity

The modulus of elasticity is a property that reflects the deformation capacity of molded specimens. The test was carried out in accordance with the standards recommended by NBR 8522 (ABNT, 20008). In Figure 10, it is possible to visualize the results of modulus of elasticity for each of the three groups of microconcrete analyzed.

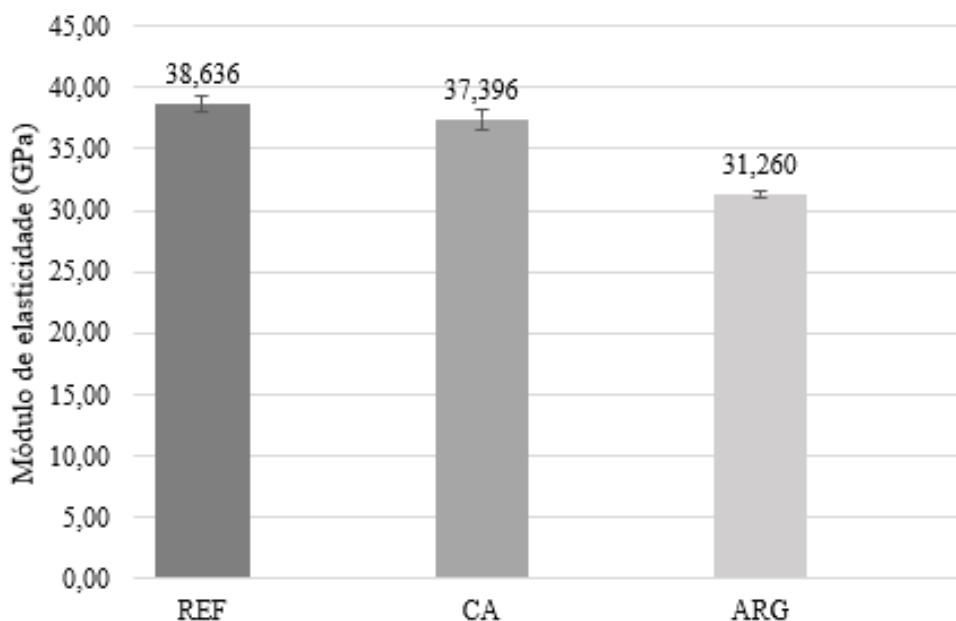


Figure 10. Modulus of elasticity by age

Based on the results, it can be seen that there was a reduction in the modulus of elasticity for the concretes with the addition of lightweight aggregates for internal healing, as expected. The presence of porous aggregates may have negatively affected the modulus of elasticity for both the expanded clay and the ceramic shell. For the group with ceramic shell the reduction was 3.21% while for the expanded clay the reduction was 19%.

### 3.4 Penetration of chlorides

The chloride penetration test is an accelerated type test, carried out in a stationary way, where the passage of electric current is monitored during a period of 6 hours. On the date of the test, the specimens were 75 days old.

During the entire test, the temperature of the solution present in each of the acrylic half-cells was monitored, not reaching a temperature of 90°C. however, the reference trait after reaching 4 hours of testing showed overheating, with the solutions reaching temperatures close to 90°C, causing the test to be terminated. Several authors such as Pereira da Costa (2015) noticed the same type of behavior during the test, and it is believed that this occurs due to the high voltage that is applied to the sample, which can lead to an increase in temperature, resulting in an increase in load. passerby. Figure 11 shows the graph with the load passing through the mixtures up to 4 hours of testing, thus allowing a better comparison between the results achieved.

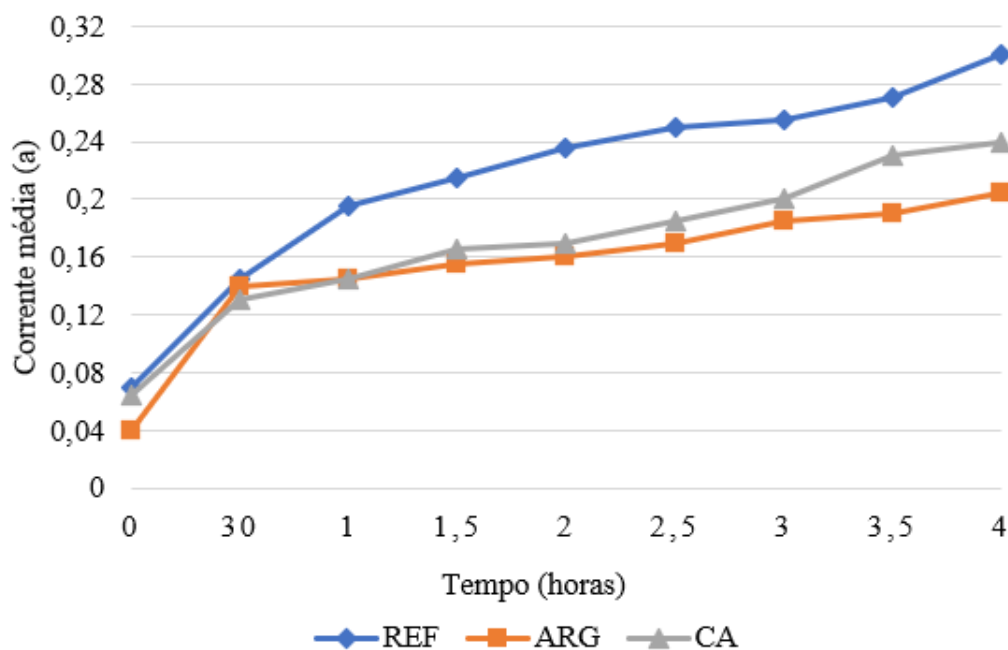


Figure 11. Average currents obtained during 4 hours of testing

The average results of passing load, in Coulombs, allow the verification of the penetrability of chloride ions in concrete through the table presented by ASTM C1202 (2022). Thus, Table 4 presents the average charges obtained for each of the tested groups and the probability of penetration of chloride ions.

Table 4. Average through loads and penetrability classification

Trace	Q media (Coulombs)	SD	CV %	Penetrability of chloride ions
Reference	308,66	66,05	4,67	Very low
Expanded clay	222,56	75,24	2,96	Very low
Ceramic Shell	243,27	74,09	3,28	Very low

Based on Figure 11 and Table 4, it can be seen that during the 4 hours of testing carried out for all the traces analyzed, the through charge values were low, thus all classified as very low risk of penetrability of chloride ions. It can also be seen that the mix with the lowest tendency for chloride penetration was the mix with the addition of expanded clay.

According to the results found by Zhang and Gjørv (1991), Bentz (2009) and Thomas (2006), the addition of lightweight aggregates that promote internal healing to the concrete mix reduces the permeability of the mixture. The authors believe that this behavior is due to internal healing, which promotes an increase in cement hydration products, making the matrix denser and consequently reducing the permeability of concrete. The behavior is consistent with what was found in this work, when comparing the test time performed with all specimens.

However, when performing an analysis with the total test time, shown in Figure 12 below, thus analyzing the 6 hours of testing, the results presented are different, and for this analysis the mix with ceramic shell (CAR) showed a result 11.06% higher than the reference mix (REF) and 9.50% higher than the mix with the addition of expanded clay (ARG). The same performance was verified by Caetano (2019), in which the trace with ceramic fence presented a 64% higher through load result than compared to its reference trace. In this way, it is possible to perceive that the researches with lightweight aggregates added to the concrete, seeking to promote the internal healing, present, in most cases, a better behavior or close to the reference regarding the penetration of chloride ions in the concrete. It is believed that the difference occurred due to the difference in test time between the analyzed traits.

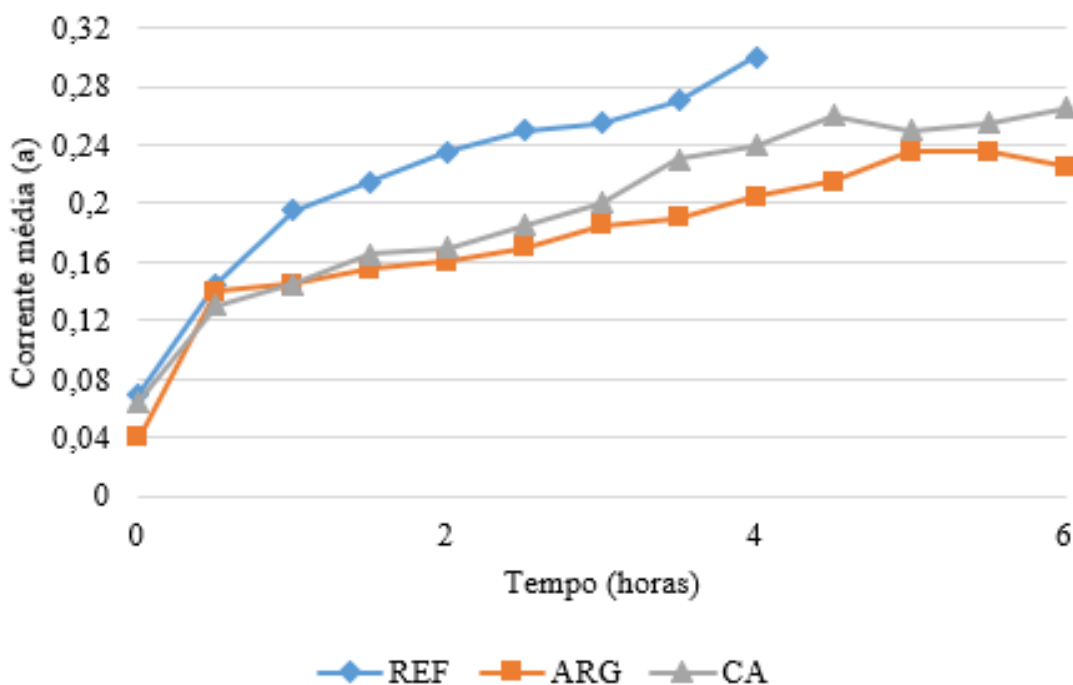


Figure 12. Average currents obtained during 6 hours of testing

#### 4. CONCLUSIONS

The analysis of the experimental data obtained during the development of this study shows that both the ceramic shell and the expanded clay showed good results regarding the durability and action of chlorides when compared with the reference mix, proving the effectiveness of using these materials as internal healing seeking greater durability of structures.

The use of internal healing agents promoted a reduction in the values related to the voids index, proving that there was a greater hydration of the cementitious matrix, generating concrete with a smaller amount of pores and less interconnectivity of pores. It is believed that this factor led to a lower penetrability of chlorides in the mixture, especially in the mixture with the addition of expanded clay. There was also an increase in the compressive strength of concrete with the

incorporation of lightweight aggregates, especially at 75 days. It is believed that the fact occurred due to the late hydration of the concrete suffering the positive influence of internal healing. However, there was a slight reduction in the modulus of elasticity of the concrete. The results presented are in line with those verified by Caetano (2019), in which the internal healing agents presented behavior similar to those found in this work.

In this way, it is possible to confirm that the use of ceramic shell from investment casting and expanded clay, in particular, have a high capacity to act as internal healing agents, improving both mechanical properties and those related to the durability of concrete, under the conditions evaluated in this study.

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