

Surface treatment of reinforced concrete in marine environment: Influence on chloride diffusion coefficient and capillary water absorption

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ABSTRACT

There are currently many types of protective materials for reinforced concrete structures and the influence of these materials in the chloride diffusion coefficient still needs more research. The aim of this paper is to study the efficacy of certain surface treatments (such as hydrophobic agents, acrylic coating, polyurethane coating and double systems) in inhibiting chloride penetration in concrete. The results indicated that all tested surface protection significantly reduced the sorptivity of concrete (reduction rate > 70%). However, only the polyurethane coating was highly effective in reducing the chloride diffusion coefficient (reduction rate of 86%).

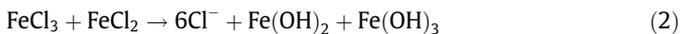
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1. Introduction

Chloride ions destroy the passive steel layer but, unlike carbonation, there is no overall drop in pH. Chlorides act as catalysts to corrosion when there is sufficient concentration at the rebar surface to erode the passive layer. These ions are not consumed in the process and allow the corrosion process to quickly proceed [1]. Corrosion involving chloride attack is thus a continuous process because chloride ions are not consumed as explained by the chemical reactions represented in Eq. (1) and Eq. (2) [2].



for hydrolysis



After the second reaction, chloride ions remain free to react again. Consequently, the corrosion caused by chloride ions is one of the most dangerous and deleterious processes in reinforced concrete. One of the main mechanisms governing chloride ion penetration in a reinforced concrete structure is diffusion, which is the transport stimulated by a difference in concentration of the species consid-

ered. However, there are other important mechanisms such as capillary suction and permeability that can act on the chloride ingress in a reinforced concrete structure.

Capillary absorption is an important mechanism related to the chloride ingress into concrete. Non-saturated concrete in contact with a salt solution will absorb this solution by capillary forces [3]. The simultaneous action of diffusion and capillary suction thus causes a mixed transport mode, which is present in most exposure conditions to which reinforced concrete structures are subjected.

The initial mechanism appears to be suction, especially when the surface is dry and salt water is rapidly absorbed by dry concrete. Next, capillary movement of the salt water through the pores is followed by a more substantial diffusion.

A possible means of extending the service life of reinforced concrete structures submitted to chloride ion attack is by surface protection of the concrete, which inhibits chloride penetration by diffusion and absorption of contaminated water. The influence of the surface protection in the chloride diffusion coefficient still needs more research; however, some studies can be found in the literature [4–14]. Moreover, Al-Zahrani et al. [15] concluded that physical properties like water absorption and chloride permeability correlates well with the accelerated corrosion performance of four coating systems studied.

The surface protection materials for concrete can be classified into three groups: pore liner (makes the concrete water repellent), pore blocker (reacts with certain soluble concrete constituents

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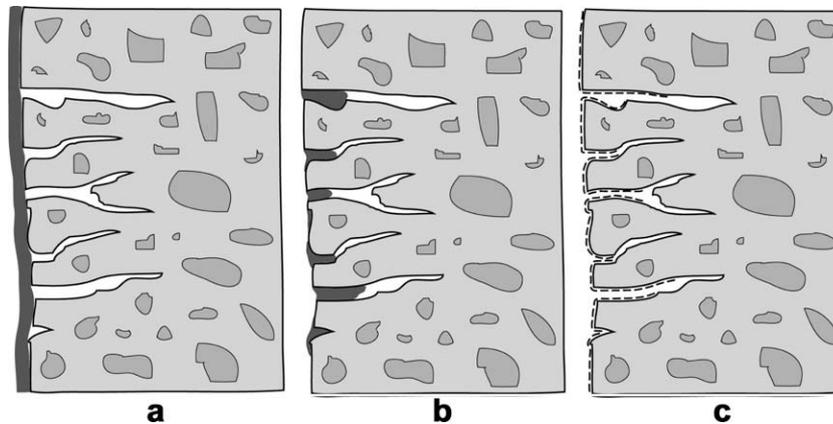


Fig. 1. Groups of surface treatments: (a) coating and sealers (b) pore blocker (c) pore liner (adapted of [16]).

and forms insoluble products) and coatings (form continuous film on concrete surface). Fig. 1 illustrates these three groups of protection.

The aim of this paper is to study the efficacy of certain surface treatment in inhibiting chloride penetration in concrete. Consequently, chloride diffusion coefficient and absorption were determined as these are the two main forms of chloride contamination of a reinforced concrete structure. The materials used in this work are hydrophobic agents, coatings and double systems (water repellent + acrylic coating). The results presented are part of Medeiros' [17] doctoral thesis.

It is obvious that there are other exposure environments that are important in specific conditions. Maslehuddin et al. [18] specifies the exposition to acid, sulfate and sulfur fume as other types of exposition to be studied in special cases; however, this work does not approach these subjects.

2. Experimental procedures

The specimens used as substrate for the application of the protection systems were cylindrical (100 mm diameter and 200 mm length). Four specimens were molded for each type of test and studied case.

2.1. Concrete of substrate

The concrete of the substrate was produced with a Brazilian Portland cement designated CII E-32, which is equivalent to ASTM C 595 [19] (Slag-modified Portland cement). This cement has blast furnace slag addition and is one of the most widely used cements in the state of São Paulo, Brazil. The coarse aggregate was dense, crushed granitic stone and the fine aggregate was natural siliceous sand.

The mix proportion of the concrete was of 1.0 (Portland cement): 1.8 (fine aggregate): 2.7 (coarse aggregate) and the water/cement ratio was equal to 0.52. This substrate had a compressive strength of 32 MPa after curing in water for 28 days. The mortar content (α) was 51% and the cement consumption (C) was equal to 358 kg/m³. After mixing, a vibrating table was used to ensure appropriate compaction.

Curing was conducted by conditioning the specimens in a wet chamber with relative humidity of 100% and 24 °C temperature for 91 days. This curing period corresponds to the stabilization level of the graphic compressive strength versus time, as shown in Fig. 2. The curing period was chosen to eliminate the influence of an additional curing that could occur in specimens exposed to water, which could particularly affect the control series.

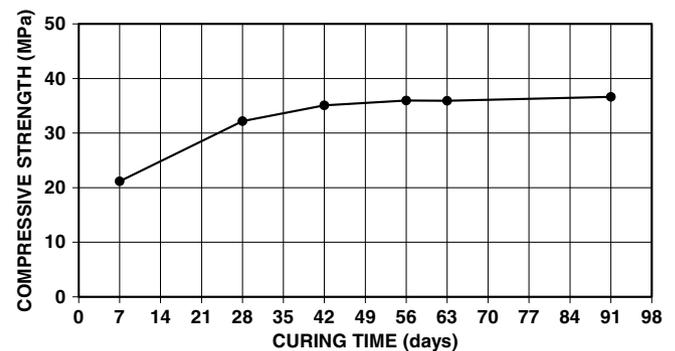


Fig. 2. Compressive strength versus curing time (1:4.5; α = 51%; cC = 358 kg/m³).

2.2. Surface protection materials

The surface treatment systems investigated in this study are presented in Table 1. Table 2 shows the curing type, number of coatings and the manufacturer's recommended consumption rate for each surface treatment material.

The manufacturer guidelines for the amount of material per unit of area were followed in all cases. Before applying the protective products, the specimens were kept in an oven at 60 °C until constant mass was obtained. Afterwards, they were kept in a chamber (23 ± 2 °C and 50 ± 4% HR) to cool down before the application of the products. Then, the surface protection materials were applied on one of the circular surfaces of specimens. Seven days after the application of protection, the specimens were tested.

2.3. Capillary water absorption

The performance of concrete subjected to marine environments is primarily a function of the penetrability of the pore system. Capillary suction is mainly controlled by the concrete surface properties, and the application of surface protection materials influences this issue.

This test was used to determine the water absorption of surface concrete treated by different materials recommended to protect reinforced concrete from a marine environment. This test was conducted in accordance to standard DIN 52617/87 [20]. Cylindrical specimens of 200 mm in length and 100 mm diameter were used. The lateral side of the specimens was sealed with silicon up to 3 cm in height so that only one circular face of the specimen was exposed to water. The water level during the test was kept constant and 5 mm above the surface of the specimen in contact with water.

Table 1
Characteristics of surface treatment systems

System	Product	Density (g/cm ³)	Viscosity (temperature = 20 °C)	Setting time (h)
Single A	Silane/siloxane dispersed in water (A)	1.0	10 CPs	2 a 6
Single B	Silane/siloxane dispersed in solvent (B)	0.78	8 CPs	2 a 6
Single C	Acrylic dispersed in solvent (C)	0.90	45	1
Single D	Polyurethane coating (D)	0.95	32	8
Double A + C	(A + C)	–	–	–
Double B + C	(B + C)	–	–	–

Table 2
Curing type and manufactures recommended rate

System	Product	Consumption rate (g/m ² /coating)	Number of coatings	Cure type
Single A	Silane/siloxane dispersed in water (A)	300	1	Water loss + reaction
Single B	Silane/siloxane dispersed in solvent (B)	150	2	Solvent loss + reaction
Single C	Acrylic dispersed in solvent (C)	150	2	Drying
Single D	Polyurethane (D)	180	2	Reaction
Double A + C	(A + C)	Indicated in (A) + indicated in (C)		Water loss + reaction + drying
Double B + C	(B + C)	Indicated in (B) + indicated in (C)		Solvent loss + reaction + drying

The weight of specimens was monitored in a period of time (0–16 days) throughout the contact with water.

Sorptivity, the volume of water penetrating per unit of area and time, was calculated to facilitate interpreting the results. In the present work, the time adopted to calculate sorptivity was 16 days. Sorptivity, S , is obtained empirically from the slope of the cumulative volume of water absorbed per unit of area of inflow surface versus square root of time, as represented in Eq. (3).

$$\text{Sorptivity} = \frac{V_w}{A_c \sqrt{t}} \quad (3)$$

where, V_w is the volume of water absorbed by the specimen (mm³), A_c is the cross-sectional area of each specimen (mm²) and t is the time of exposure (h).

2.4. Pipette absorption test

A similar configuration to Karstens' permeability test was adopted [21]. After molding and curing time of the cylindrical specimens (100 mm diameter and 200 mm length), 50-mm thick slices were cut from the base of the specimens. This test was carried out on the surface of the specimen's base. Fig. 3 shows the slice and the surface tested.

The apparatus shape differed from the one Karstens initially proposed because the former is only appropriate for horizontal surfaces. The apparatus was fixed on the concrete surface using sili-

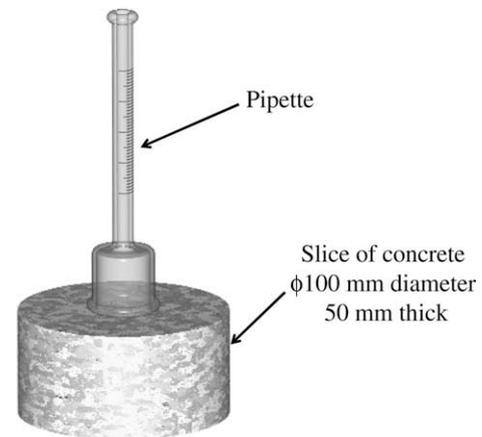


Fig. 4. Apparatus of pipette absorption test attached to concrete slice.

con, as shown in Fig. 4. The volume of water absorbed was measured as a function of time.

For this test, the pressure varies with time. It is equal to 120 kgf/m² (1176 Pa) in the beginning of the test and, as the water level drops, it can be reduced until it equals 80 kgf/m² (784 Pa).

2.5. Rapid chloride penetration test

This test was conducted according to standard ASTM C1202/97 [22]. After the curing time, each cylindrical specimen (100 mm diameter and 200 mm length) was cut into two 50 mm-wide slices from the central nucleus of the original specimen.

After the drying procedure, the protection materials were applied on only one of the circular surfaces of the specimens. Afterwards, the specimens were vacuum-saturated for 24 h. This procedure consisted in conditioning specimens in a vacuum desiccator and applying a vacuum pressure of 1 mmHg (133 MPa) for 3 h. Then, the desiccator was filled with de-aerated water until all specimens were immersed, and the vacuum pressure was maintained for one additional hour. Afterwards, the vacuum pump was turned off, and the specimens were maintained immersed for 20 h.

Seven days after the application of the surface treatment, the 50 mm-thick specimen was placed between two acrylic cells, each cell with 280 cm³ solution volume. One cell was filled with 0.3 N

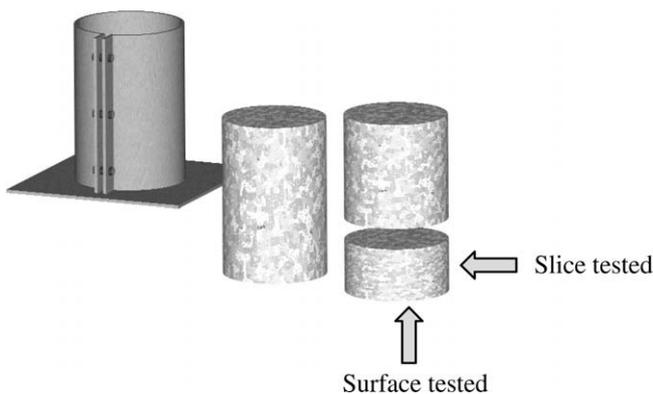


Fig. 3. Concrete slice and surface tested in the pipette absorption test.

NaOH aqueous solution and the other with 3.0% NaCl aqueous solution. The cells were connected to a 60 V power source for 6 h, in which the electrode immersed in NaOH aqueous solution was the anode and the electrode immersed in 3.0% NaCl aqueous solution was the cathode. The schematic presentation of the chloride migration test cell is illustrated in Fig. 5.

After 6 h under test, the total charge passed through the specimen (in coulombs) was obtained by integrating the current passed through the specimen during the test period.

2.6. Estimation of chloride diffusion coefficients

The method used to estimate the chloride diffusion coefficient followed the procedure described by Luping and Nilsson [23], which involves measuring the depth of color change of a freshly broken concrete surface in the direction of the chloride flow using 0.1 M AgNO₃ aqueous solution [24] and in the application of Eq. (4) in a migration test.

$$D = \frac{RT}{zFE} \frac{x_d - ax_d^b}{t} \quad (4)$$

where D is the diffusion coefficient (cm²/s), R is the gas constant (J/molK), T is the temperature (K), z is the valency of the ion, F is the Faraday constant (J/Vmol), E is the field density (V/m), x_d is the penetration depth (m), t is the test time (s), a and b are test constants. For chloride ion $z = -1$ and, when $E = -600$ V/m and $T = 298$ K, then, $a = 1.061$ and $b = 0.589$. The model used is based on Fick's second law because the test is developed in a non-steady state condition.

Fifty millimeter-thick slices were obtained by sawing two slices of the mid-portion of the cylindrical specimen (100 mm diameter and 200 mm length). After drying the concrete slices, the surface protection materials were applied on only one of the circular surfaces of the specimens. Seven days after the application of protection, the specimens were submitted to the same procedure described in 2.5. The differences were that the power source applied was 30 V, and the test time was equal to 30 h.

Next, the slices were broken, and the depth of chloride penetration was determined as illustrated in Fig. 6. Andrade et al. [25], comparing methods for determining the chloride diffusion coefficient in non-steady state using the accelerated chloride migration test, indicated that the Luping and Nilsson procedure [23] presents better reproducibility of results, thus justifying the test method here presented.



Fig. 6. Colorimetric method being applied.

3. Results and discussion

3.1. Water absorption

Fig. 7 shows the capillary water absorption test results. It is possible to verify that all the protection systems tested in this work are highly efficient in inhibiting the water absorption by capillary forces. However, the results indicated that the double systems (Double A + C and B + C) and the polyurethane coating (Single D) are more effective in inhibiting water penetration in concrete than the other products studied. This is important because penetrating water can be a vehicle for chloride penetration as it influences the kinetics of the corrosion process in case the steel reinforcements are already depassivated.

Further evidence of the efficiency of the protection systems tested is shown in Fig. 8. This figure presents results of sorptivity, which is the volume of water that penetrates per unit of area and time. The sorptivity reduction rates of each protection system are also shown in Fig. 8. The results clearly show that all the protection systems highly reduced the sorptivity (more than 70%), indicating high efficiency in inhibiting water penetration in concrete.

In the case of the double systems (Double A + C and B + C), when one of the materials of the double system fails, is that the other continues to protect the concrete. On the other hand, the polyurethane coating (Single D) is more efficient than the other single systems because this type of polymer results in a more closed

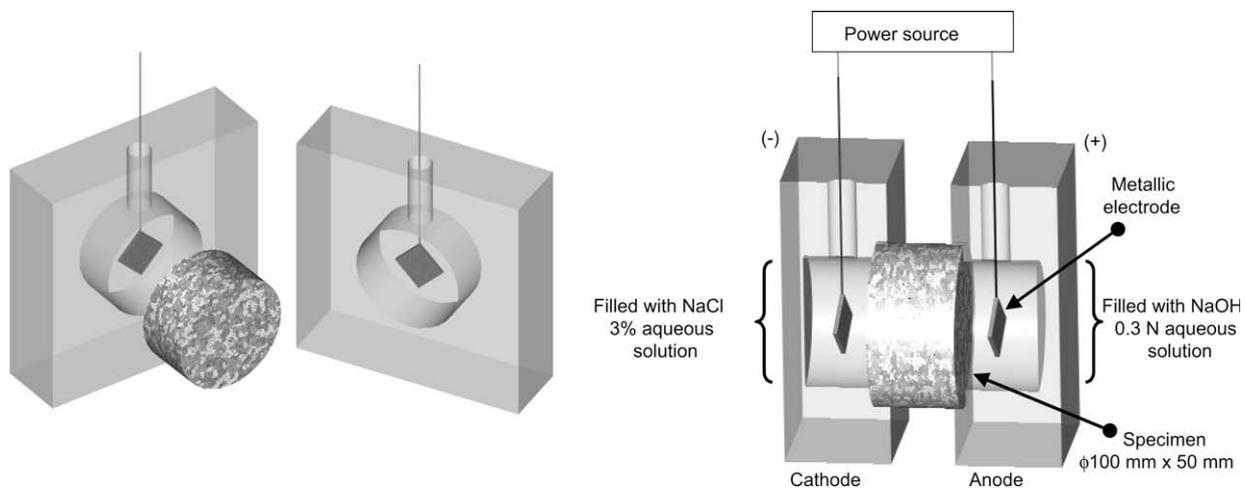


Fig. 5. Experimental arrangement of chloride migration test cell.

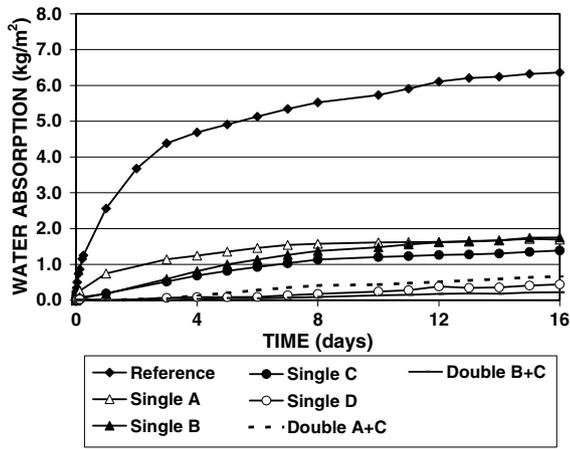


Fig. 7. Capillary water absorption of treated and untreated concrete surfaces.

the capillary water absorption test results. Also in this test, the polyurethane coating (Single D) presented the highest level of protection and the silane/siloxane dispersed in water (Single A) presented minor efficiency compared to other materials. However, in general, all the protection systems significantly reduced the absorption of the concrete.

The difference in the trend of results of the two tests occurs because the water penetration mechanism is not the same; in the pipette absorption test, the mechanism is permeability while in the absorption test it is capillarity. In this way, it is important to consider the mechanisms of water penetration occurring in the practical case to specify what type of absorption test is to be used. This calls attention to the importance of knowing the water exposure conditions that the concrete is submitted to before the specification of the efficient material for each case. Whether the structure is being submitted to water permeability or capillary absorption has to be verified. If there is water pressure, this pressure needs to be considered and, in general, high-thickness materials are recommended.

molecular chain in comparison to the other materials included in this work.

3.2. Pipette absorption test

Fig. 9 shows the results of this test. It is important to verify that in these test conditions, the double systems (Double A + C and B + C) presented efficiency similar to the silane/siloxane dispersed in solvent (Single B) and of the acrylic film (Single C), in contrast to

3.3. Diffusion coefficients

Fig. 10 presents the results of chloride diffusion coefficient determined by the method proposed by Luping and Nilsson [23]. In this experiment, the concrete is saturated; therefore, the ion movement caused by absorption and permeability does not exist. Thus, the values of chloride diffusion coefficient presented in Fig. 10 are for saturated concrete.

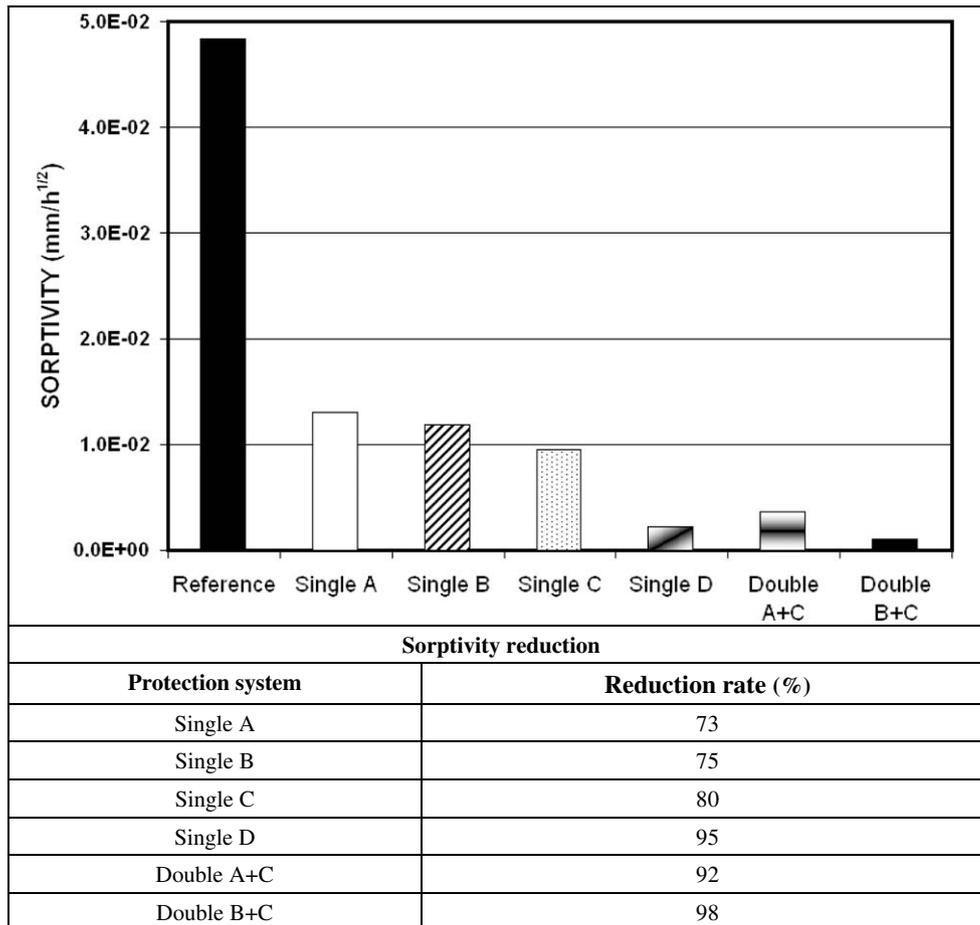


Fig. 8. Sorptivity of treated and untreated concrete specimens.

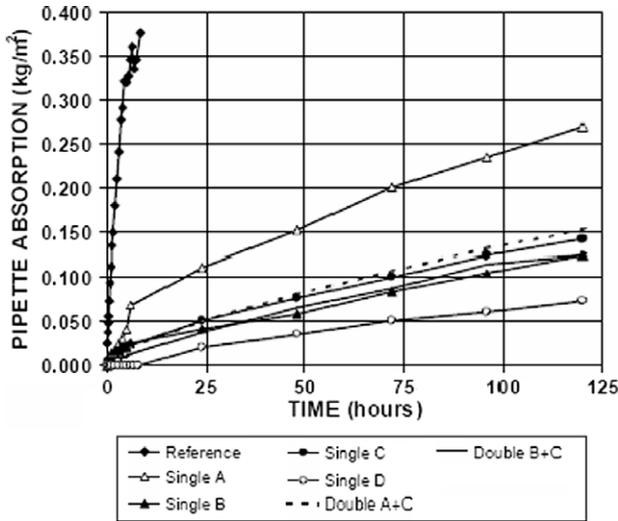


Fig. 9. Pipette absorption of treated and untreated concrete surfaces.

Generally, the chloride diffusion coefficient for the treated concrete surfaces was clearly reduced. This reduction increases the structure service life when exposed to a marine environment. However, there is a large difference in efficiency depending on the treatment used.

It is important to emphasize that, except for the polyurethane coating (Single D), the double systems (water repellent + acrylic coating) are far more advantageous as compared to the single systems due to the double barrier formed in this category of protection. When the chloride ion passes the acrylic topcoat layer, there is still the silane/siloxane treatment, which has some capacity to inhibit the chloride ion penetration inside the concrete. This is illustrated by the fact that the sum of the reduction rate of the chloride diffusion coefficients of the single systems is almost equal to the reduction rate of the double systems (Single A + Single C = 29% approximately to 33%; Single B + Single C = 37%, approximately to 41%). It should be stressed that this does not lead to the conclusion that the reduction rate of the double systems is equal to the addition of the reduction rate of each single system that forms the double system. A specific experiment should be conducted to prove this. However, it is important to report this fact to stimulate future works on this subject.

The double systems (Double A + C and B + C) reduced the chloride diffusion coefficient by more than 33%. The polyurethane coating (Single D) was the most efficient protection, reducing the chloride diffusion coefficient by 86%. On the other hand, the silane/siloxane dispersed in water (Single A) reduced the chloride diffusion coefficient by 9%, indicating that this material does not greatly influence chloride penetration. The silane/siloxane dispersed in solvent (Single B) and the acrylic coating dispersed in solvent (Single C) presented 17% and 20% chloride reduction rate, respectively.

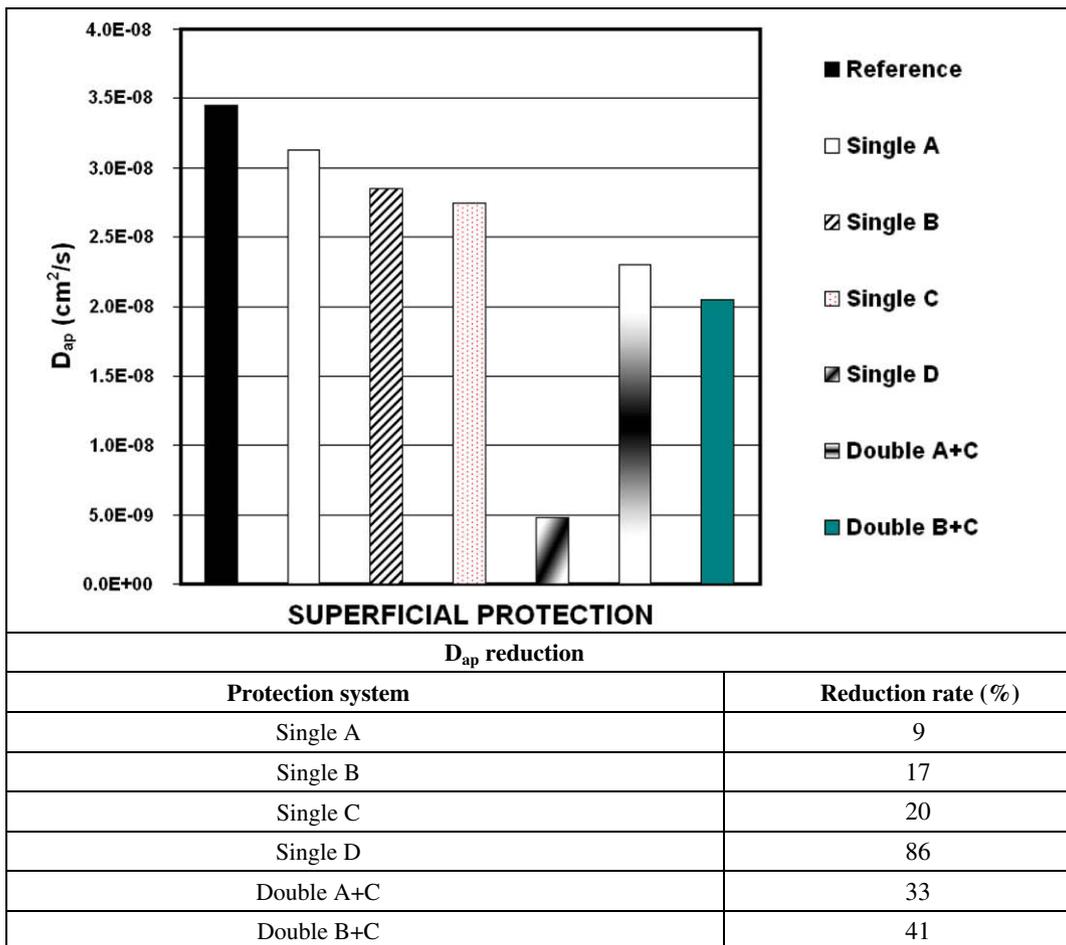


Fig. 10. Chloride diffusion coefficients in treated and untreated concrete systems.

It is important to observe that the silane/siloxane (Single A and Single B), in addition to reducing the penetration of the water in the concrete for water repellence, renders the diffusion of chloride ions more difficult. In the case of the silane/siloxane dispersed in solvent (Single B), this reduction in the chloride diffusion coefficient is of the same order of magnitude as that of acrylic coating (Single C).

The value of the chloride diffusion coefficient is not a measure that allows easy understanding of the advantage produced by the protection system. Fig. 11 was thus designed to facilitate the interpretation of the chloride diffusion coefficients results. This type of representation allows comparing the different types of treatment systems through a relation between the depth of chloride penetration and the reinforced concrete service life.

For constructing Fig. 11, Fick's second law of diffusion was used (Eq. (5) and (6)).

$$CP = 2(z)\sqrt{Dt} \tag{5}$$

$$\text{erf}(z) = 1 - \frac{C_{Cl} - C_0}{C_s - C_0} \tag{6}$$

Where D is the chloride diffusion coefficient (cm^2/year), t is the service life (years), $\text{erf}(z)$ is the Gauss error function, CP (chloride penetration) is the depth at which the chloride concentration reached

the threshold for reinforcement depassivation (cm), C_0 is the initial chloride concentration, C_s is the surface chloride concentration (%), C_{Cl} is the chloride concentration in depth and time (%), and C_{dep} is the chloride concentration threshold to depassivate the reinforcement (by weight of cement).

To design Fig. 11, it was necessary to fix some parameters, such as: $C_s = 1.8$ and $C_{dep} = 0.4\%$ by weight of cement.

Fig. 11 shows that the estimated service life for the reinforced concrete with 4 cm cover (for the concrete used in this work) is 4.5 years. Using the best of the protection systems in this work (Single D), the estimated service life of the reinforced structure increased to 35 years (7.8 times greater than the reference series). This evaluation is applied only for the conditions established to determine the chloride diffusion coefficient in this work (specimens saturated and directly exposed to 3.0% NaCl aqueous solution). It is important to emphasize that there are other factors that must be considered to improve the service life prediction when non-saturation conditions exist. For example, in his work Han advocates the need to consider the influence of the evaporable water content in the calculation of the chloride diffusion coefficient [26]. This explains why the service life values esteem for the reinforced concrete in the conditions of this work were so small (equal to 4.5 years and 35 years to untreated and treated concrete, respectively).

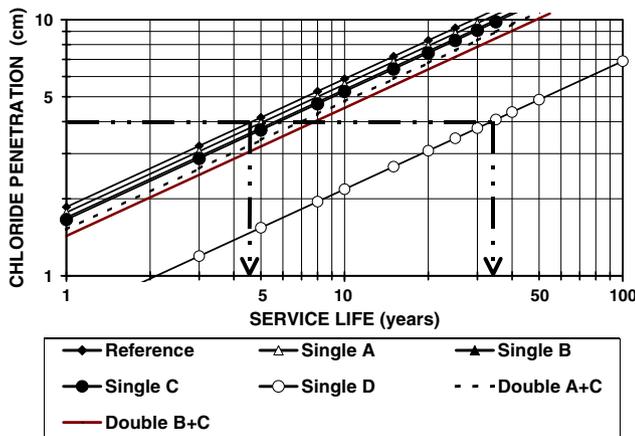


Fig. 11. Relation between estimated service life and chloride penetration (depth where 0.4% by weight of cement was reached).

3.4. Rapid chloride penetration test

Fig. 12 presents the charge passed results according to the ASTM C1202/97 test procedure [22]. The results indicated that the polyurethane coating (Single D) and the silane/siloxane dispersed in solvent + acrylic coating (Double B + C) have the greatest capacity to inhibit ion penetration in concrete.

It is important to emphasize that the results of charge passed do not exclusively refer to the movement of chloride ions through concrete because in a migration cell there are other ions moving. However, the results of this type of test can be used qualitatively. Moreover, Fig. 13 confirms the existence of a significant correlation between chloride diffusion coefficients and the charge passed results. The value of $R^2 = 0.91$ together with the trend of the results indicates a direct relation between the charge passed and the chloride diffusion coefficient. This interdependence of results exists, although the charge passed values relate to the transport of all ions independently of their being chlorides or not.

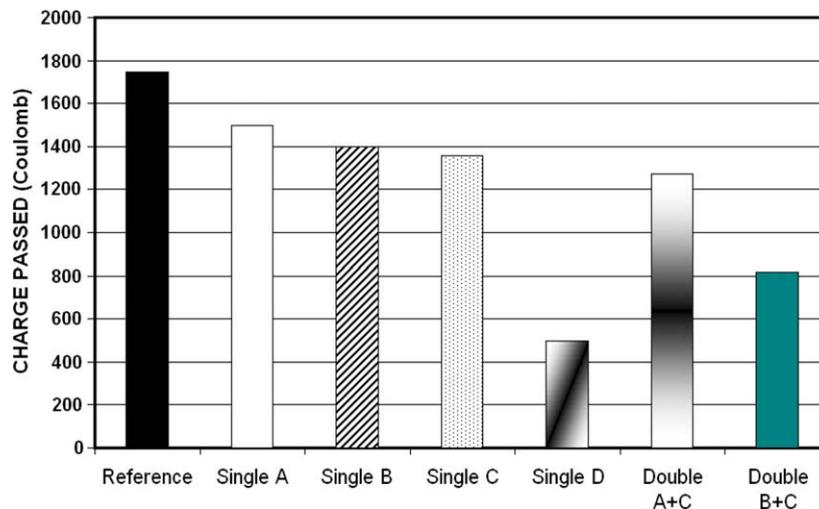


Fig. 12. Charge passed in treated and untreated concrete systems.

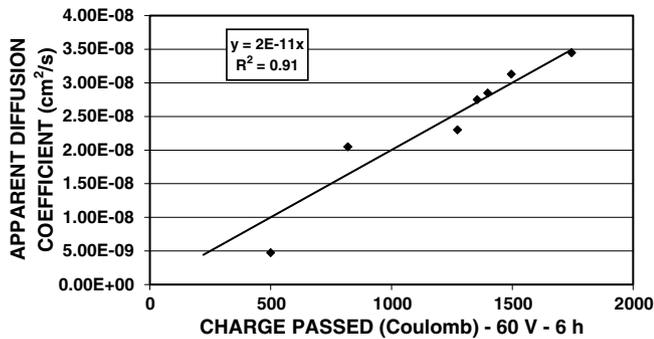


Fig. 13. Correlation between charge passed and apparent diffusion coefficient.

4. Conclusions

The chloride contamination in a reinforced concrete structure exposed to a marine environment is one of the most challenging forms of degradation to amend. The ingress of these ions in concrete occurs by chloride diffusion or penetration of contaminated water. From the research presented herein, the following conclusions can be derived:

1. The concrete surface protection can be an efficient means of inhibiting chloride penetration in concrete. Consequently, protection systems must be used to increase the service life of reinforced concrete in environments influenced by salt spray, such as buildings in coastal cities/towns.
2. All surface treatments reduced the capillary water absorption compared to the reference (minimum = 73% and maximum = 98%).
3. The results indicated that the most efficient protection system could reduce the chloride diffusion coefficient of concrete by 86%.
4. It is important to emphasize that the results indicated that the use of a high efficiency protection system could extend the service life of a reinforced structure by 7.8 times; however, this level of increase in service life is related to the test conditions adopted in this work. Different results can be obtained under other test conditions. Moreover, Castellote and Andrade stress that several models for service life prediction have been analyzed in different workshops within the work of RILEM TC 178-TMC and that numerous uncertainties remain, which prevent, at the present state of the art, from making accurate long-term predictions from short-term experiments [27].
5. Graphs (like Fig. 11) that relate service life with depth where the chloride concentration reached the threshold for reinforcement depassivation are the most efficacious means of presenting results of chloride diffusion coefficients. This type of representation is made possible with the use of the diffusion theory developed by Fick. It is important to emphasize that the use of the Fick's second law solution and the Gauss error function to estimate the service life of reinforced concrete date back to the 1970s; nevertheless, here the easiness to elaborate the representation of Fig. 11 is detached.
6. Although the results of the test procedure according to ASTM C1202/97 [22] are influenced by the movement of many other ions in addition to chloride ions, there is a significant correlation ($R^2 = 0.91$) between the chloride diffusion coefficient and the values of total charge passed.
7. It was clear that hydrophobic agents (Single A and Single B) reduce sorptivity, which is the main protection mechanism of this treatment. Moreover, the results indicated that this

type of product could reduce chloride ingress in the concrete. The reduction rate of the chloride diffusion coefficient of the silane/siloxane dispersed in solvent (Single B) was similar to that presented by the acrylic coating (Single C), for example.

8. The double systems (Double A + C and B + C) presented greater capacity for reducing the capillary water absorption and chloride diffusion than the simple protection systems, with exception of the polyurethane coating (Single D), which is acknowledged as a high efficiency material.
9. The polyurethanes were the surface protection products that presented the best efficiency in reducing the chloride penetration. Yet it is important to point out that this result cannot be universally considered because the efficiency of the treatment does not depend only on the type of resin that composes it. This is in accordance to Vipulanandan and Liu [28], that affirm that for one exact type of resin the efficacy to reduce the chloride penetration can be highly distinctive.
10. The water absorption and pipette absorption tests results showed that is very important to know the water exposure conditions of the reinforced concrete structure before the specification of the surface protection system.

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