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Reducing Water and Chloride Penetration Through Silicate Treatments for Concrete as a Mean to Control Corrosion Kinetics

M. H. F. Medeiros[1,*], *P. Castro-Borges*[2], *D. M. Aleixo*[3], *V. A. Quarcioni*[4], *C. G. N. Marcondes*[5] *and P. Helene*[6]

[1] Civil Construction Department, Federal University of Paraná (UFPR), Prédio do CESEC, Centro Politécnico, Jardim das Américas, Curitiba, Paraná, Brasil - CEP 81531-980
[2] Centro de Investigación y Estudios Avanzados del IPN Unidad Mérida, Km 6 Antigua Carreta a Progreso, CP 97310, Cordemex, Mérida, Yucatán, México
[3] Instituto de Pesquisa Tecnológicas do Estado de São Paulo (IPT), Avenida Professor Almeida Prado, 532 - Laboratório de Materiais de Construção Civil - Prédio 1 – Térreo,
[4] Civil Engineering, Instituto de Pesquisa Tecnológicas do Estado de São Paulo (IPT), Avenida Professor Almeida Prado, 532 - Laboratório de Materiais de Construção Civil - Prédio 1 - Térreo - Butantã - 05508-901 - Sao Paulo, SP – Brasil,
[5] Civil Construction Department, Pontifícia Universidade Católica do Paraná (PUC-PR), Prédio do CESEC, Centro Politécnico, Jardim das Américas, Curitiba, Paraná, Brasil - CEP 81531-980,
[6] Department of Civil Construction Engineering, Universidade de São Paulo (USP), Universidade de São Paulo, Escola Politécnica, Departamento de Engenharia de Construção Civil - Av. Prof. Almeida Prado, Travessa 2, nº 83 - Cidade Universitária - 05508900 - Sao Paulo, SP – Brasil,
[*] E-mail: medeiros.ufpr@gmail.com

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There are currently many types of protective materials for reinforced concrete structures and the influence of these materials in the chloride diffusion coefficient and water penetration still needs more research. The aim of this work is to analyze the contributions regarding the typical three surface concrete protection systems (coatings, linings and pore blockers) and discusses the results of three pore blockers (sodium silicate) tested in this work. To this end, certain tests were used: one involving permeability mechanism (low pressure-immersion absorption), one involving capillary water absorption, and the last, a migration test used to estimate the effective chloride diffusion coefficient in saturated condition. Results indicated reduction in chloride diffusion coefficients and capillary water absorption, therefore, restrictions to water penetration from external environmental. As a consequence, a reduction of the corrosion kinetics and a control of the chloride ingress are expected.

Keywords: Concrete, sodium silicate, marine environment, chloride, diffusion coefficient

1. INTRODUCTION

The use of superficial protection for concrete is a possible way of increasing the service life of reinforced concrete structures exposed to marine environments. This type of protection inhibits the penetration of aggressive agents by diffusion and capillary absorption.

The surface protection materials for concrete can be classified into three groups: Pore liners (make the concrete water-repellent), pore blockers (react with some of the soluble concrete constituents and form insoluble products) and coatings (form continuous film on the concrete surface). Figure 1 illustrates these three groups of superficial protection.

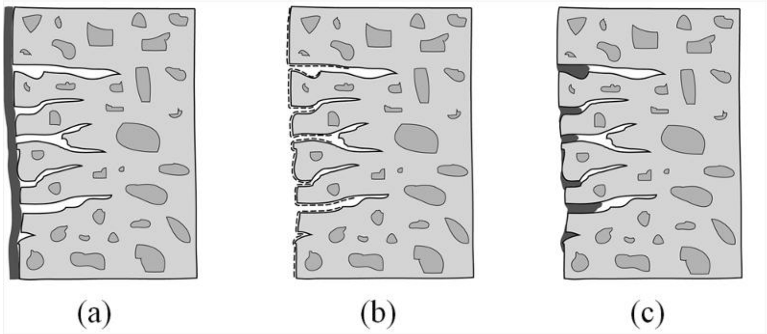


Figure 1. Groups of surface treatments: (a) coatings and sealers, physical barrier (b) pore liners, water repellent (c) pore blockers [1].

Coatings and sealers present the advantage of providing a physical barrier on the concrete surface, which isolates it from the aggressive agents of the external environment. These coatings require a homogeneous and smooth substrate with pores of 0.1 mm width at most [2]. This means that they are capable of covering a 0.1-mm wide crack. However, the film breaks if the concrete structure cracks after painting. Moreover, coatings do not allow the concrete to dry if it is wet. This can cause the deterioration of the film, causing the formation of bubbles due to the vapor pressure of the internal humidity. From the architectural point of view, this group of surface treatment modifies the aesthetics of the structure adding brightness or color to the concrete surface, which is sometimes desired [2].

This group of surface treatment (coating and sealers) has been intensely studied in the last fifteen years. Delucchi et al. [3] studied the importance of the parameters E (modulus of the material) and η (viscosity) in the crack-bridging ability of the coating. Seneviratne et al. [4], using mechanical thermal analysis, suggested that the most successful coating is able to maintain its elastomeric properties over the required period of exposure and over a wide range of operational temperatures. Uemoto et al. [5] showed a correlation between the paint pigment volume content and water permeability. Al-Zahrani et al. [6] showed that the accelerated corrosion performance of the four coating systems studied correlates well with the performance results obtained from the physical properties, in particular, water absorption, water permeability, and chloride penetration. Medeiros and Helene [7] suggested that the determination of the chloride diffusion coefficient allows a quantitative comparison of the protection systems and, therefore, needs to be made possible in migration tests. This kind of protection is probably the most used by the construction industry and that would be the reason why they are widely studied.

Water repellents or hydrophobic agents always result from silicon resins that are chemically bound to the concrete base. Currently, the most commonly used are silanes, siloxanes oligomerics and a mixture of these two components [8].

Chemically, silanes are formed of small molecules that have one silicon atom and siloxanes are short chains of a few silicon atoms in which the molecules have alkoxy groups (organics) connected to the silicon atom. Silanes and siloxanes react with the silicate of the concrete, forming a stable bonding [9] (Figure 2). The authors aforementioned showed that the penetration of the hydrophobic agent is better in finished faces than in formwork faces, due to the higher permeability of the second. Jacob; Hermann [10], Batista [11] and Moriconi et al. [12] presented an ample review on surface hydrophobic agents. The use of hydrophobic agents in the construction industry is increasing at an interesting rate and they have been reasonably studied. However, there are some items that are still being studied, such as the influence of the water penetration mechanism (capillary suction and permeability) in the efficacy of the hydrophobic treatment [8].

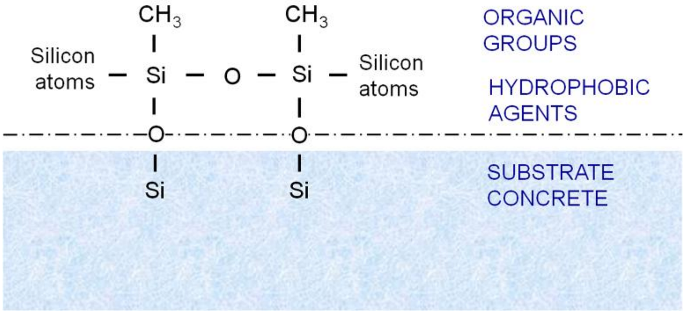


Figure 2. Chemical bond of the hydrophobic agent with concrete (adapted from Vries and Polder [9]).

Pore Blockers, such as sodium silicate, are used in the cement industry for some applications. For example, as slag cement activator; as treatments of vegetal fibers used for cement reinforcement, as surface treatment for concrete and as hardening/densifying of surface concrete floors [13, 14, 15, 16, 17].

Although this group of treatment has been known for several years, little technical and scientific information about this subject has been published. On the other hand, many products of this type are commercialized and indicated for the protection of concrete surfaces. Thompson et al. [18] explained the 3 theories on how silicates act to improve the performance of concretes:

- SiO_2 precipitating in the pores.
- Silicates forming an expansive gel similar to that formed in alkali silica reactions that fills the pores in the concrete by swelling.
- Silicates reacting with excess calcium present in the near surface region of the concrete to form relatively insoluble calcium-silicate hydrates.

The latter theory is currently the most accepted. In this way, theoretically, the pore blockers are products composed by silicate, which penetrate the superficial pores of the concrete and react with portlandite forming C-S-H. Sodium silicate is the mostly used silicate applied in this way.

According to Thompson et al. [18], Eq. 1 represents what happens when the sodium silicate solution penetrates the pores of the concrete.

$$Na_2SiO_3 + yH_2O + xCa(OH)_2 \longrightarrow xCaO \cdot SiO_2 \cdot yH_2O + 2NaOH \qquad \text{(Eq. 1)}$$

In this way, this treatment forms a less porous layer in the concrete surface modifying the water penetration into the concrete.

This treatment requires that sodium silicate reacts with portlandite [$Ca(OH)_2$]. Therefore, it is easy to understand that, in carbonated concrete, the reaction above fails to occur. This is why in the superficial layer the $Ca(OH)_2$ already reacted with CO_2 and originated $CaCO_3$. In this case, it is necessary to make an impregnation with hydroxyl ions before the application of sodium silicate. This explains why Kagi and Ren [19] affirmed that a significant reduction in the permeability of concrete due to silicate solution impregnation can only be achieved if the concrete is very fresh and repeated impregnations are carried out to fill all the concrete capillaries. Another contribution of Kagi and Ren [19] is related with water vapour permeability of the substrate which is not significantly affected by silicates because they only cover capillary walls without blocking them fully. Ibrahim et al. [20] focused their work on measuring the time taken for reinforced concrete specimens coated with sodium silicate to crack by corrosion and concluded that the results were similar to that of the uncoated concrete specimens. However, their results are not in accordance to those of Thompson et al. [18] regarding water absorption. It is important to emphasize that Ibrahim et al. [20] do not inform the concentration of sodium silicate solution used in their study. The use of a low concentration may have influenced their results. Thompson et al. [18] compared some concentrations of sodium silicate solutions and the value around 26% presented greater efficiency.

It is important to emphasize that the pore blocker should be applied after the structure is in service. This is after it has taken all the projected loads and cracks and microcracks have been produced. However, environmental effects can cause more cracks. If there are cracks (large enough to be visible) before the application of the silicates, it is necessary to repair them prior the treatment application. If the cracks (large enough to be visible) appear after the silicates treatment, they need to be repaired immediately because the low porosity layer formed by the treatment is ruptured. This is one of the main problems of the surface treatment (Pore liners, pore blockers and coatings): they do not absorb the deformation of the concrete of substrate. This means that the formed protection layer breaks if the concrete cracks, causing the prompt imperfection of the protection system.

It is important to remember that, independently of the crack width, the remaining superficial pores will still be smaller than if they do not have the pore blocker on the surface. Then, the environment attack will be isolated to the cracked area and the other areas will keep being protected.

The use of pore blockers is wide and their application is contributing to many of the several concerns about service life prediction since they can be applied as preventive or corrective methods. The service life approach has significantly changed during the last years [21] and this is mainly due to the recent introduction of more quantitative parameters than a few years ago. These parameters include new concepts such as serviceability, functionality, security, reliability and durability.

This work focuses on some additional contributions to the study of pore blocking systems in the sense that they can constitute a useful tool in the field of protection of new reinforced concrete structures. However, based on the real concerns promoted by their application, the main purpose of this paper is to indicate a way to estimate the influence of silicate surface protection in the service life of a reinforced concrete structure exposed to chloride contamination. This type of results presentation was made possible with the use of the diffusion theory developed by Fick.

2. EXPERIMENTAL PROCEDURES

2.1. Substrate concrete:

The substrate concrete was produced with a Brazilian Portland cement designated CPII E-32, which is equivalent to ASTM C 595 (Mehta and Monteiro [13]) (Slag-modified Portland cement). This cement has blast furnace slag admixture and is one of the most widely used cements in the state of São Paulo, Brazil. The chemical composition and physical properties of the cement are listed in Table 1. Coarse aggregate was a dense, crushed granitic stone. Fine aggregate was natural siliceous sand.

The mix proportions details are summarized in Table 2. After mixing, a vibrating table was used to ensure appropriate compaction. The curing adopted was conditioning the specimens in wet chamber with relative humidity of 100 % and 24^{o}C temperature for 91 days. This curing period was chosen to produce a high hydration level and eliminate the influence of an additional curing that could occur in the specimens exposed to water. This could especially affect the control series.

2.1.1. Surface protection materials

Table 3 shows a summary of the protection products studied in this work and descriptions of each of them are presented below.

- Commercial silicate - Treatment composed of silicate commercialized specifically for the protection of concrete surfaces. According to the manufacturer, it creates a surface barrier against the ingress of water and contaminants such as chloride ions.
- Sodium silicate (25%) - Sodium silicate solution elaborated from solid sodium silicate dilution in water with concentration equal to 25%. This concentration was chosen because it is approximately the solid content of the commercial product indicated specifically for the protection of concrete [Commercial silicate].
- Liquid sodium silicate - Liquid sodium silicate, known as water glass or liquid glass. This product was chosen because it is a product commercialized for many purposes and is formed solely by sodium silicate.

Before the application of the protective products, the specimens were kept in an oven at 60^{o}C until constant mass was obtained. After that, they were kept in a chamber (23±2 ^{o}C and 50±4 % HR) to cool down before the application of the products. Seven days after the application of protection, the specimens were tested.

The treatments were applied in a single coating with consumption rate of solution of 250 g/m^2. One coating for all cases was used because this is the manufacturer's recommended consumption rate of the commercial treatment, the only product of this work commercialized specifically for the protection of concrete surfaces.

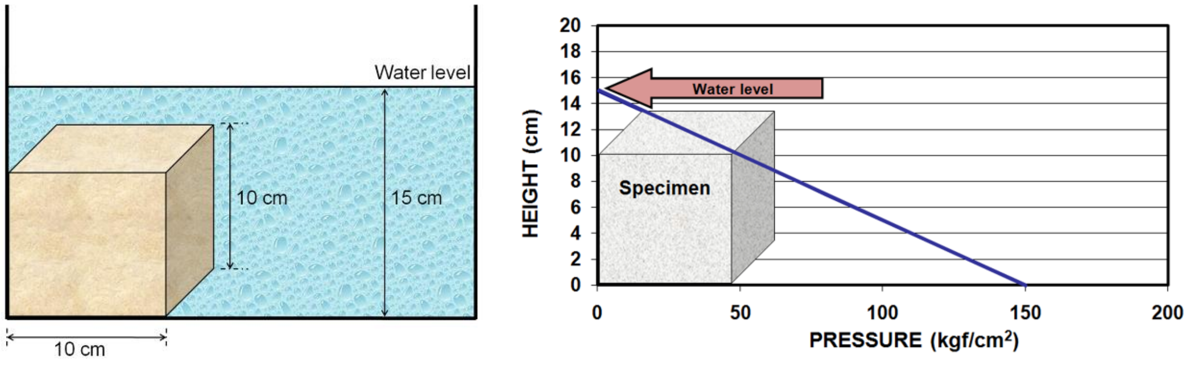
Pore blockers are being suggested in this work as a preventive procedure. This is why the tests were all carried out on "virgin" specimens. However, this product is also indicated to repair (old reinforced concrete structures), but in this case it can be necessary to apply another product before the silicate treatment. This happens because the silicate needs portlandite to react and form insoluble products inside the pores of the surface layer.

The following items show information about the test procedures used in this work.

2.1.2. Immersion absorption

This test was conducted in accordance to standard ASTM C642/97. Cubic specimens with 10-mm edge lengths were used. The immersion absorption was measured by weighting the specimens along the first contact with water up to 30 days. 4 specimens were used in each case studied.

The water level in the test recipient was fixed at 15 cm, assuring that the variation of the water pressure on the specimen was between 150 kgf/m^2 (at the base) and 50 kgf/m^2 (at the top), as shown in Figure 3.



(a) (b)

Figure 3. Variation of pressure in the specimen during the immersion absorption test.

2.2. Capillary water absorption

This test was conducted in accordance to standard DIN 52617/87. Cylindrical specimens of 20 cm in height and 10 cm in diameter were used. The lateral side of the specimens was sealed with silicon up to 4 cm in height so that only one circular face of the specimen was exposed to water. This care was taken to induce water penetration only to occur by the circular face of the test. Figure 4 illustrates a specimen with the lateral side partially sealed and the test in progress. The weight of specimens was monitored in a period of time (0–30 days) along the contact with water. 4 specimens were used in each case studied.

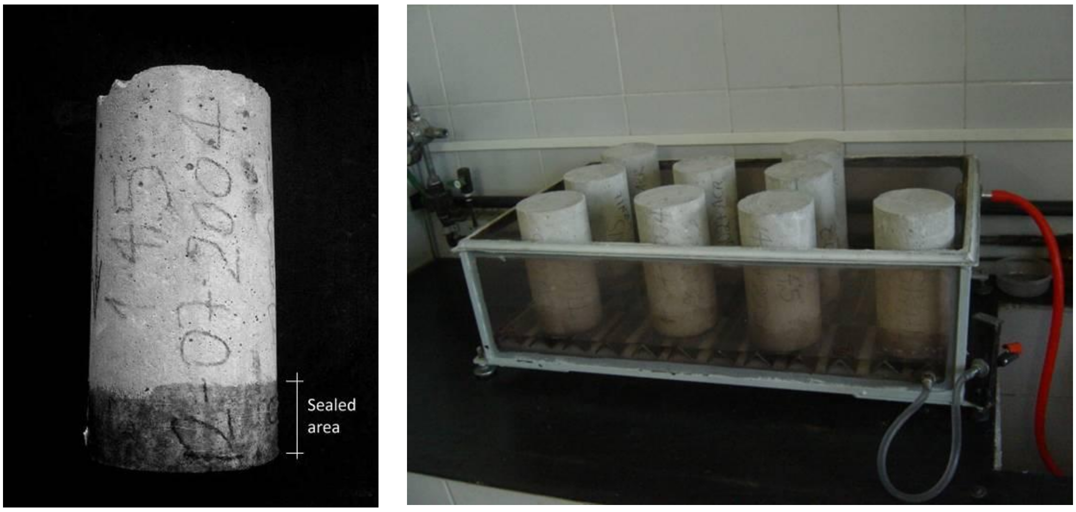


Figure 4. Capillary water absorption test.

2.3. Estimation of chloride diffusion coefficients

This test procedure is based on the first Fick's law, steady-state conditions. To elaborate this experiment, some works of reference were used [22, 23, 24, 25, 26, 27].

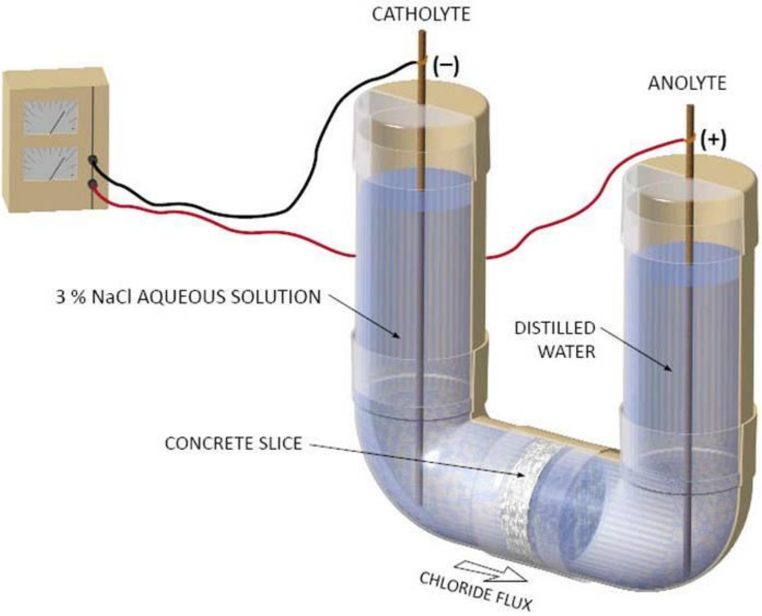


Figure 5. Experimental arrangement of steady-state migration test (voltage = 12 V).

The test was conducted in 25-mm thick concrete slices obtained by sawing the mid-portion of the cylindrical specimen (100 mm diameter and 200 mm length). 3 concrete slices of each case were used in this test and these slices were extracted from the same specimen, as suggested previously [28]. The method consisted in positioning the concrete slices between two PVC cells. One compartment was filled with distilled water (anolyte) and the other with 3.0 % NaCl aqueous solution (catholyte). The voltage applied was 12 V and two copper rebars each 35 cm in length and 0.6 cm in diameter, were used as electrodes. The volume of each compartment was of 3 liters and, periodically during the experiment, the chloride ion concentration in anolyte cell was monitored by removing small volumes of the solution and analyzing them by potentiometric titration with $AgNO_3$. The schematic presentation of the accelerated chloride migration test cell is illustrated in Figure 5.During the test, the steady-state condition is reached when the chloride concentration in the positive cell (anolyte) varies linearly with the time. This indicates a constant flux, which is the basic definition of the steady-state condition. In this way, the chloride ion concentration in the anolyte was monitored in a period of time along the steady-state migration test until the chloride steady-state flux was attained, and this was used to estimate the diffusion coefficient in accordance with Eq. 2 (Nernst–Planck equation). The result of this procedure is a graph of the chloride concentration versus time, as illustrated in Figure 6.

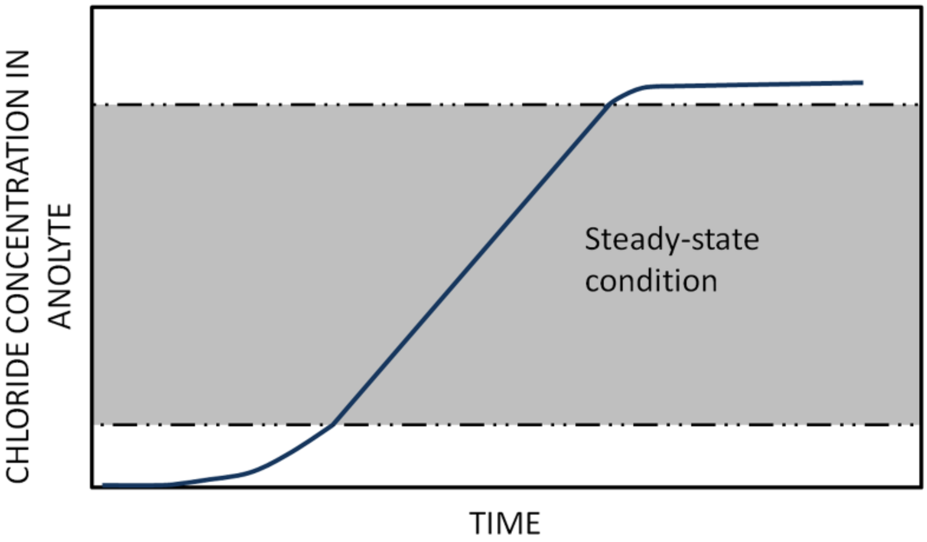


Figure 6. Arrangement of variation of the chloride concentration in the anolyte (positive cell).

$$J = \frac{zF}{RT} DC \frac{dE}{dx} \qquad \text{(Eq. 2)}$$

Where: J = chlorides flux (g/m^2s); D = diffusion coefficient (m^2/s); C = anolyte concentration (g/m^3); x = thickness of the sample (m); z = ion valency ; F = Faraday constant (J/Vmol); R = gas constant (J/molK); T = temperature (K); E = voltage (V).

3. RESULTS

3.1. Immersion absorption

Figure 7 presents immersion absorption results for the untreated and silicate treated concrete. It is clear that the silicate treatment has little influence in the immersion absorption. All specimens reached the mass stabilization due to water absorption at about 8 hours of test.

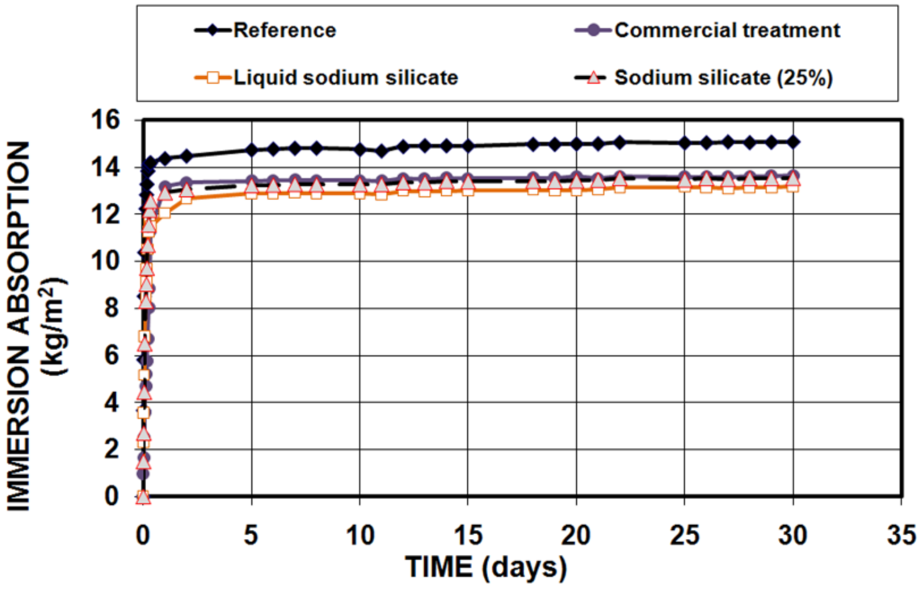


Figure 7. Immersion absorption of untreated and treated concrete specimens.

However, it is important to emphasize that the stabilization platform in the case of the concrete treated with the silicate solutions was a little below that of the platform of the reference concrete. This is caused by the filling of the pores localized in the superficial layer of the concrete. In general, the three silicate treatments presented similar effect in this type of test.

3.2. Capillary water absorption

Figure 8 presents results of capillary water absorption. In the conditions of this test, the treatments showed great effect, significantly reducing the concrete absorption. The water absorption by capillary suction is the most common mechanism of water penetration in reinforced concrete structures [29]; therefore, it can be considered that this effect is a factor that contributes to the durability of reinforced concrete. If the treatment inhibits water penetration, this means that the salt spray will enter with more difficulty. Moreover, if the corrosion process is already installed, its kinetics will be reduced by the restriction of water access.

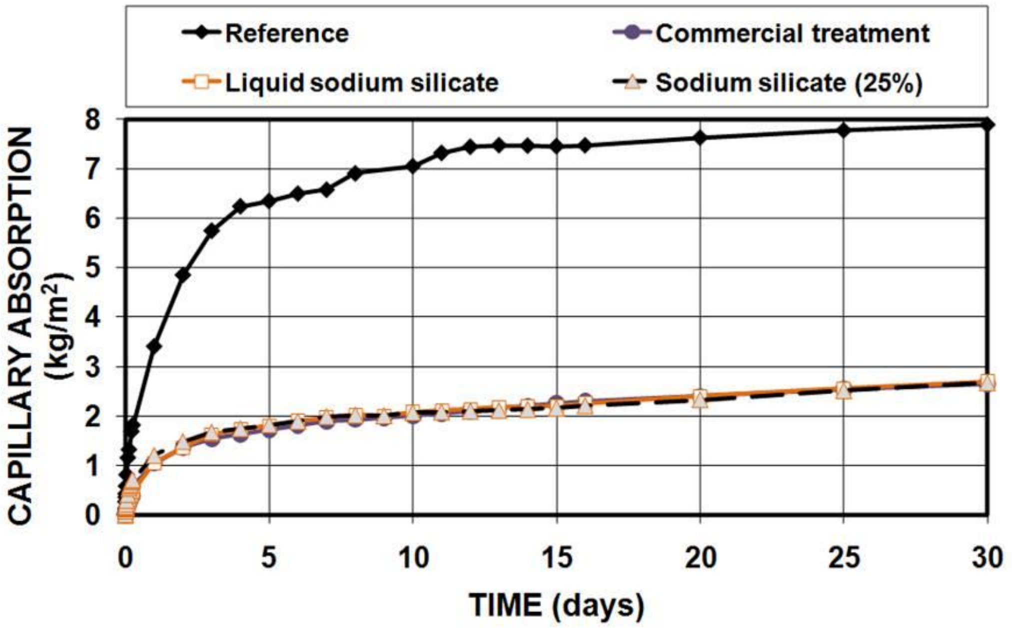


Figure 8. Capillary water absorption of untreated and treated concrete surfaces.

The results indicate that there is a large reduction in capillary water absorption of concretes treated with the three silicates tested in this work. Furthermore, the efficiency of each product tested was the same, as shown in Figure 8.

It is important to emphasize that the reduction in capillary water absorption restricts water penetration from external sources. This fact causes a reduction in the kinetics of the corrosion process (when the steel corrosion is already installed) and controls the chloride contamination of the concrete, because these ions penetrate the concrete dissolved in an electrolyte (generally water). Moreover, the durability of the products formed is a gap about this type of treatment. It is important to know if the products formed break down or alter their composition over time. In sum, more researches about the silicate treatment are necessary.

3.3. Diffusion coefficients

Figure 9 shows the variation of the amount of chloride ions along time in the anolyte (positive cell). This graph allows a qualitative evaluation of the treatments related to the capacity of reducing the penetration of chloride ions through the untreated and treated concrete. However, the most appropriated way to interpret this type of results is to transform them into quantitative results through the determination of the chloride diffusion coefficient. For this, the results of Figure 9 were used to determine the flux of chlorides through the untreated and treated slices of concrete and Eq. 2 (Nernst–Planck equation) was used to determine the chloride diffusion coefficient.

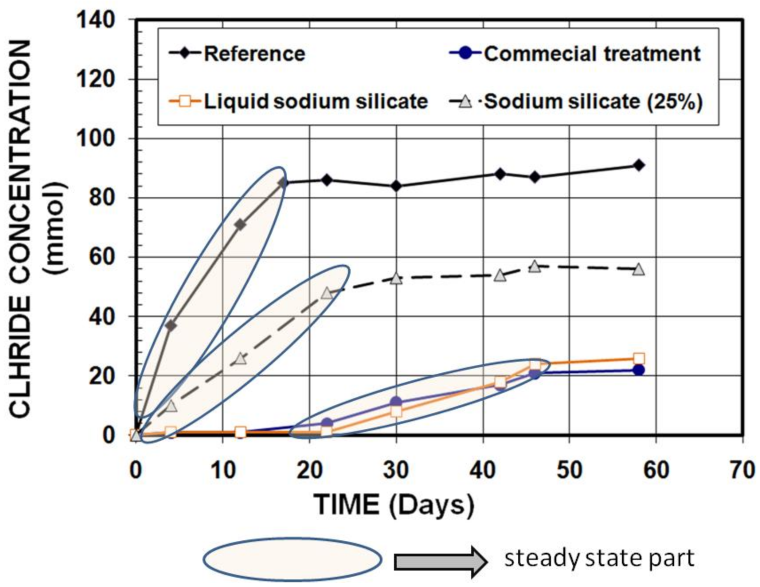


Figure 9. Variation of the chloride concentration in the anolyte along time (positive cell).

In this way, Figure 10 shows the chloride diffusion coefficient for untreated and treated concrete. The data show that the treatment with sodium silicate reduces the chloride diffusion by between 64 % and 88 % through the concrete, confirming that this type of treatment is capable of protecting the concrete against chloride ions contamination.

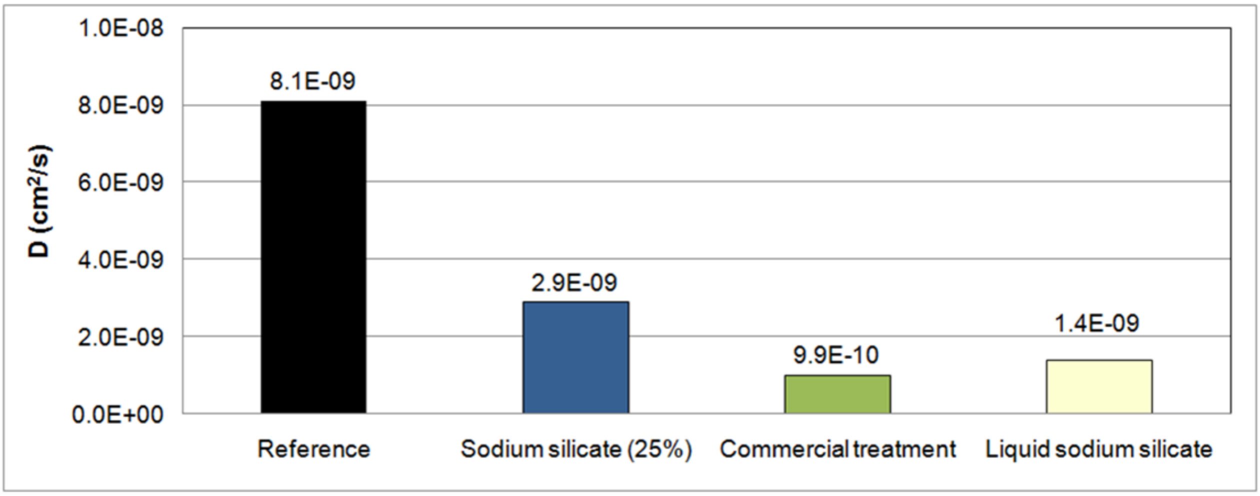


Figure 10. Chloride diffusion coefficients of untreated and treated concrete surfaces.

The difference in ability to block chloride can be attributed to the real concentration of sodium silicate of each treatment. Only the product sodium silicate (25%) has the sodium silicate concentration confirmed, because the solution was made by the authors of this paper. The other two treatments are products ready manufactured and its real sodium silicate concentration is not known. The amount of silicate of these products was estimated by the solid content.

4. DISCUSSION

Service life prediction is one of the areas that need greater advancements in the durability of reinforced concrete. Currently, many models developed in this area exist; however, their validation still needs to be studied and disseminated.

In this work, a service life prediction based on the second Fick's law of diffusion is presented. The method proposed in this work allows correlating service life with concrete cover, which can be a useful tool to design reinforced concrete structures. It is important to verify that this is an application based exclusively on the chloride diffusion phenomenon. Its extrapolation to other penetration mechanisms such as absorption, convection, or a mixture of these must be careful. It is necessary to conduct more research on these phenomena to develop a way of making these considerations. However, the application proposed in this work is valid because it allows a quantitative comparison between different materials and supplies service life estimations, although the model still needs improvements.

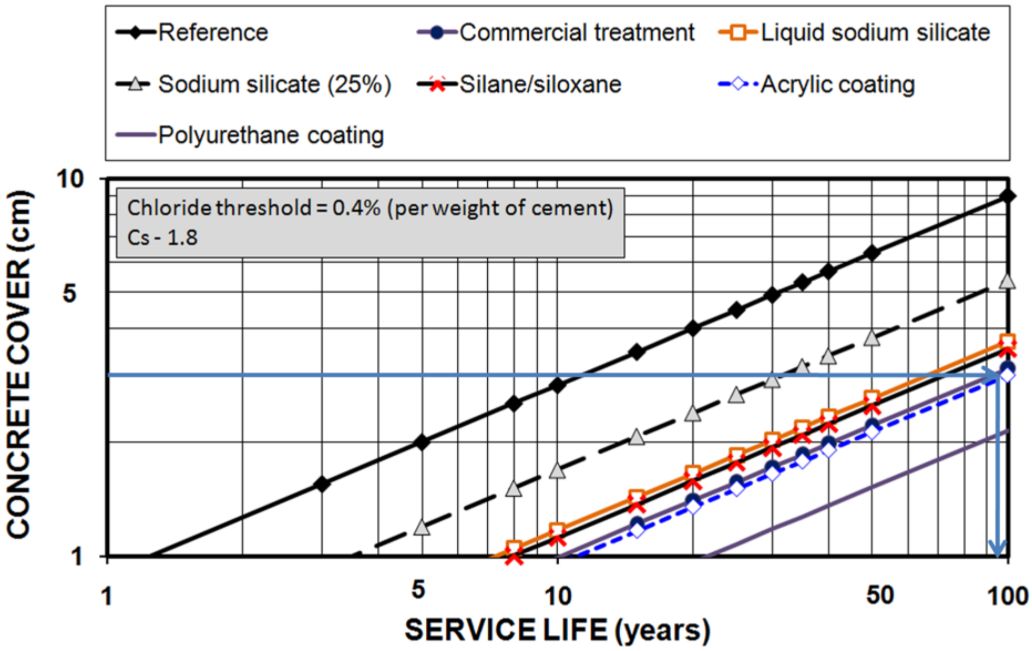


Figure 11. Service life X Concrete cover.

Helene [2] presents Eq. 3 and Eq. 4 based on the second Fick' law. These equations were used in this work to analyze diffusion coefficients results, allowing the construction of a plot that correlates the chloride penetration depth with the reinforced concrete structure service life (Figure 11).

$$x=2(z)\sqrt{Dt} \qquad \text{(Eq. 3)}$$

$$erf\,(z)=1-\frac{C_{Cl}-C_0}{C_S-C_0} \qquad \text{(Eq. 4)}$$

Where: D is the chloride diffusion coefficient (cm^2/year), t is the service life (years), erf (z) is the Gauss error function, x is the depth where the chloride concentration reached the threshold for reinforcement depassivation (cm), C0 is the initial chloride concentration, CS is the surface chloride concentration (%), CCl is the chloride concentration in depth x and time t (%).

It is important to emphasize that the service life predictions presented in this work are only valid in the conditions below.

- The reinforced concrete structure is at the beginning of its service life (starting from the initiation period);
- The fastest degradation agent is chloride attack;
- No accidental external factors have affected the properties of the concrete cover.

Previous results indicated a clear trend of reduction of the chloride penetration when the sodium silicate treatment was used. The result of this is the rise in the service life of the reinforced concrete structure treated with this type of product.

The chloride diffusion coefficient results for each case studied and the second Fick's law were used to elaborate graphs that correlate the service life (years) with the depth in which the chloride concentration reached the threshold for reinforcement depassivation (cm). Figure 11 shows this type of result for each treatment studied. Although chloride threshold levels for corrosion initiation have been widely reported for several environments [30, 31], a level of 0.4% per weight of cement (pwc) was chosen in order to compare data with other authors. In spite of the well known environment cycles of chloride concentration on concrete surface [32], a restriction for this work was the selection of a fixed surface chloride concentration (Cs) of 1.8.

To make a comparison of sodium silicate products with other protection systems possible, in Figure 11 results of a pore liner (water repellent - silane/siloxane dispersed in solvent) and two coatings (1 acrylic coating and 1 polyurethane coating) were included. These products are not the focus of this work and were presented in this item to allow a comparison of the silicate treatment with other groups of protection.

In Figure 11, it is clear that for concrete cover equal to 3 cm, the service life of the untreated reinforced concrete structure is about 12 years and can reach 90 years depending on the silicate protection system applied on the surface of the concrete.

Figure 11 shows that a sodium silicate treatment can have the same efficiency of a water repellent (silane/siloxane) and an acrylic coating. However, the polyurethane coating presented greater efficiency than the other treatments tested in this work.

Figure 11 is obviously limited to cases as that presented here with its own limitations. However, it is necessary to emphasize that the initiation period established here corresponds to that of stage 4, as mentioned by Castro-Borges and Helene [21]. This is a period of time from the in-service day until the day on which the aggressiveness of the environment loads result in direct deterioration of the steel/concrete.

It is important to emphasize that the reduction in capillary water absorption restricts water penetration from external sources. This fact causes a reduction in the kinetics of the corrosion process (when the steel corrosion is already installed) and controls the chloride contamination of the concrete, because these ions penetrate the concrete dissolved in an electrolyte (generally water). Moreover, the durability of the products formed is a gap about this type of treatment. It is important to know if the products formed break down or alter their composition over time. In sum, more researches about the silicate treatment are necessary.

5. CONCLUSIONS

The conclusions expressed here apply to the characteristics of this work and materials. Any extrapolation to other materials or circumstances must be carefully conducted.

The treatment of the concrete surface using sodium silicate reduced the chloride diffusion coefficient. The results indicated that the concrete with this type of protection can extend its service life significantly, when the concrete is exposed to chloride contaminated solutions. This conclusion is valid only if the fastest degradation process is chloride attack instead of no accidental external factors that could affect concrete cover properties.

The results indicated that the surface treatment using sodium silicate reduced the immersion water absorption of the concrete. However, it was clear that the capillary water absorption of treated concrete was highly reduced. It indicates that this type of product does not make concrete impermeable and it is not adequate to be applied in water ponding conditions, like swimming pools and water tanks.

The treatment with sodium silicate was compared with other groups of treatment (one pore liner and two coatings). The conclusion was that the treatment with sodium silicate can increase the service life in the same way as silane/siloxane pore liner and to an acrylic coating; however, the protection with polyurethane coating was more efficient than that with sodium silicate.

This work proposes an interesting way to interpret chloride diffusion coefficient results. The possibility of making service life predictions, easiness of interpretation and correlation between concrete cover and service life are the advantages of this method. In this way, this interpretation procedure can become an important tool to predict the service life of reinforced concrete structures. However, it is important to observe that this is an application based exclusively on the chloride diffusion phenomenon. How to consider these other phenomena still is unknown.

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