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Surface skin protection of concrete with silicate-based impregnations: Influence of the substrate roughness and moisture



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HIGHLIGHTS

• Silicate-based impregnations for concrete superficial protection.

• Influence of the substrate roughness and moisture on impregnation performance.

Concrete substrate condition influences performance of silicate impregnation.

• The above mentioned influence is dependent on the property at stake.

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ABSTRACT

Silicate-based impregnations are often used to protect concrete against aggressive external actions. However, the understanding of several aspects concerning this type of impregnations is still rather limited, including the influence of the concrete substrate on their performance. This paper presents results of an experimental study about (i) the efficacy of silicate-based impregnations to protect concrete elements, and (ii) the influence of the concrete substrate's characteristics on the performance of such superficial protection. Concrete specimens with two different water/cement ratios (0.40 and 0.70) were produced and, prior to the application of the impregnation, were prepared following different procedures that created (i) three different surface roughnesses (no surface preparation, 160 bar water jet and needle scalers) and (ii) three different moisture contents (3.0%, 4.5% and 6.0%). The performance of unprotected and protected concrete specimens was assessed by means of the following procedures, indicated in EN 1504-2 standard; (i) product penetration depth; (ii) water absorption by immersion; (iii) abrasion resistance; (iv) impact resistance; and (v) bond strength. Results obtained show that the silicate-based impregnation was effective in improving the resistance to water penetration and abrasion resistance, but did not improve the resistance to impact. The surface roughness and the moisture content at the instant of the application of the surface protection proved to influence the performance of the impregnation product, however such influence was dependent on the property at stake.

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1. Introduction

In the last few decades, the deterioration of reinforced concrete (RC) structures has become a major problem in most countries. This concern is attested by the increasing number of RC structures presenting premature deterioration, which is leading to a substantial growth of financial costs associated to their rehabilitation [1]. Environmental agents can produce different types of physical, chemical and mechanical damage in RC structures [2]. The causes of deterioration and the degradation mechanisms of RC structures

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http://dx.doi.org/10.1016/j.conbuildmat.2014.07.071 0950-0618/© 2014 Elsevier Ltd. All rights reserved. are presently reasonably well understood and have been described in more or less detail in the technical literature (e.g. [3]).

In order to extend the durability of both new and existing concrete structures, several kinds of surface treatments can be adopted. In general, the surface treatments are classified into three groups, illustrated in Fig. 1: (i) hydrophobic impregnations that produce a water repellent surface generally with no pore filling effect; (ii) impregnations, which reduce the surface porosity by filling totally or partially the concrete pores; and (iii) coatings that produce a continuous protective layer along the concrete surface [4,5]. Some of these surface treatments can penetrate inside the concrete pores and react with the hydration products of concrete, reducing the surface porosity and increasing the superficial



Fig. 1. Surface treatments classification: (a) hydrophobic impregnations, (b) impregnations, (c) coatings (adapted from [5]).

strength. In some cases, they have a pore lining effect or form a continuous layer at the concrete surface, thus acting as a barrier between the environmental agents and concrete, preventing and/ or delaying the penetration of aggressive agents, such as moisture, chloride ions, carbon dioxide and sulphates.

Several factors must be considered when selecting a commercial surface treatment product, namely the substrate condition, the moisture content, the required durability for the protection system, requirements concerning the application process and budgetary aspects [6–8]. However, the surface treatments available for concrete protection can provide different levels of protection, even those that exhibit similar generic chemical composition [6,9,10].

In recent years, two types of surface impregnation treatments have been used more frequently in civil engineering applications: (i) silane- and siloxane-based (water repellents), and (ii) silicatebased (pore blockers, also known as "waterglass"). In the first type of impregnations, the active ingredient product produces a thin hydrophobic layer on the pores, while in the second type, the reaction product can block the pores, strengthening the concrete surface [11,12]. Although silicate-based impregnations are relatively often applied, a review of the technical literature (presented in the next section) shows that the knowledge about their performance and behaviour is still rather limited, particularly when compared with silane-based impregnations.

The present paper aims at improving the understanding about the protection of concrete substrates with silicate-based impregnations. In particular, this work aims at evaluating the influence of the concrete substrate in their performance, particularly in what concerns the type of concrete and the roughness [13] and moisture content [14] of the substrate prior to the application of the impregnation.

2. Literature review

One of the first studies addressing the performance of silicate sealers on concrete is the one by Thompson et al. [11]. The authors evaluated the performance of two different aqueous sodium silicates in protecting (i) commercial paving blocks and (ii) concrete produced in the laboratory with a water/cement (w/c) ratio of 0.48. Results obtained from water absorption, abrasion resistance and chloride penetration tests showed that the tested sodium silicate products were only moderately effective.

Ibrahim et al. [15,16] compared the performance of different types of surface treatments (sodium silicate, silicon resin solution, silane/siloxane, alkyl alkoxy silane, silane/siloxane with acrylic top coating and two-component acrylic coating) in preventing the deterioration of concrete with a w/c ratio of 0.45 due to sulphate attack, carbonation and chloride penetration. The best performance was obtained by the silane/siloxane protection with acrylic top coating, with the sodium silicate impregnation providing the less effective protection. The sodium silicate impregnation reduced the concrete's carbonation depth after five weeks by approximately 50% compared with unprotected concrete, however the same effectiveness was not obtained in reducing chloride diffusion coefficient as well as in maintaining compressive strength of concrete immersed in a sulphate solution (330 days) [16].

Dai et al. [10] evaluated the influence of surface treatments in protecting reinforced concrete structures located in humid subtropical marine environments. Two families of products were analysed: (i) four types of silane-based water repellent agents and (ii) two types of sodium silicate-based pore blockers. In this study, concrete specimens with a w/c ratio of 0.68 were exposed for 1 year to cyclic sea water shower under an outdoor environment of accelerated dry/wet cycles. The results obtained revealed that sodium silicate-based impregnations were not efficient in preventing water absorption and chloride penetration into the concrete, in contrast with the silane-based products.

Mirza et al. [17] compared the performance of several surface treatments, namely 28 silanes, 13 siloxanes, 12 cement-based sealers, 2 epoxies resins, 2 acrylic resins and 1 silicate, in protecting concrete structures with w/c ratios of 0.55 and 0.70 at low temperature. In that study, in which the surface protections were applied and cured for 14 days at a temperature of only 4 °C, the best performance was provided by silane and siloxane family impregnations; as in the preceding studies, silicates presented a poor performance in terms of water absorption and water vapour transmission capacity.

Recently, Pigino et al. [18] studied the characteristics and performance of ethyl silicate for the surface treatment of concrete with w/c ratios of 0.45 and 0.65. After the treatment, both concretes showed a significant decrease in capillary suction, chloride diffusion coefficient and carbonation depth, indicating an interesting potential of this specific type of silicate. In this study, the changes in colour and brightness presented by the concrete over time were also analysed, an aspect that may be relevant in some outdoor applications due to aesthetical reasons.

As aforementioned, only a relatively limited number of studies were performed to characterise the performance and mechanisms of action of silicate-based impregnation products in protecting concrete elements, especially focusing their potential efficacy in changing the concrete surface skin. The literature review shows that the efficacy of this kind of impregnations in protecting concrete against the ingress of water, chloride and carbonation is much lower compared to other products, namely the hydrophobic impregnations. However, other physical principles of action of those products were still not properly addressed. In addition, there are still several aspects concerning this type of concrete surface protection whose understanding is still insufficient. Among those aspects, according to the best of the authors' knowledge, the influence of the concrete substrate, namely the type of concrete, as well as the substrate condition, in terms of surface roughness and moisture content, is still not documented in the literature.

3. Materials and methods

3.1. Experimental programme

The experimental programme comprised the production of two different types of concrete specimens, with w/c ratios of 0.40 and 0.70, a part of which was protected with a commercial silicate-based impregnation product. These types of concrete comprise low and high w/c ratios, thus allowing to assess the influence of the concrete compactness on the efficacy of the impregnation.

Both types of concrete specimens were prepared using three different surface roughnesses (series R), which were obtained by the following procedures: (i) no surface preparation, and surface roughening produced by either (ii) applying a 160 bar water jet or (iii) using a needle scaler. These surface preparation procedures, frequently used in concrete substrates, were selected for providing different surface roughnesses, thus allowing to assess the influence of this parameter on the performance of the impregnation.

Also the moisture content of the concrete specimens was controlled, with the following three classes being tested (series H): surface water saturated and surface dried until two different levels of moisture content were attained. The moisture levels were defined taking into account a range of possible situations likely to be found on site, varying from wet environments (e.g. recently cast constructions or in moist/wet locations) to dry environments (representative of existing constructions in dry locations). This allowed evaluating the influence of the substrate moisture on the efficacy of the silicate-based impregnation.

The performance of the impregnation product was evaluated through the following tests indicated in EN 1504-2 [5], deemed adequate for the assessment of the physical resistance improvement of the protected concrete: (i) penetration depth; (ii) water permeability; (iii) abrasion resistance; (iv) impact resistance; and (v) bond strength. For each property and experimental series (type of concrete, surface preparation and moisture level), three specimens were tested. All tests were performed in laboratory conditions, with a temperature of approximately 23 °C.

3.2. Materials

Table 1 presents the composition of the two types of concrete used in the experimental campaign with w/c ratios of 0.40 and 0.70 (MC(0.40) and C(0.70), respectively), the latter composition, with a low binder content, leading to a much more porous concrete. The compositions of the two mixes were prepared according to EN 1766 [19]. The cement used on both concrete compositions was type I/42.5R.

Concrete slabs with geometry of $300 \times 300 \times 40 \text{ mm}^3$, cubes with $150 \times 150 \times 150 \text{ mm}^3$ and cylinders with 300 mm of height and 150 mm of diameter were manufactured according to standard procedures and then cured in a moist chamber at $(21 \pm 2) \,^{\circ}$ C for 28 days. The cubes and the cylinders were tested in order to determine respectively the compressive and splitting tensile strengths at 28 days of age – results are presented in Table 1.

After the curing period, concrete specimens were obtained by cutting the slabs with a diamond blade, according to the dimensions defined by the tests to be performed: (i) $220 \times 70 \times 40 \text{ mm}^3$ (water permeability for series R and impact resistance); (ii) $70 \times 50 \times 40 \text{ mm}^3$ (water permeability for series H); (iii) $110 \times 110 \times 150 \text{ mm}^3$ (abrasion resistance); and (iv) $300 \times 150 \times 40 \text{ mm}^3$ (bond strength). The corners of the specimens used in the abrasion resistance tests had to be chamfered, so that they were compatible with the Taber abraser used (c.f. Section 2.5.3).

The superficial protection product used is this study was a silicate-based impregnation mixed with an acrylic resin. This product is commercially available and relatively often applied to protect concrete substrates. Table 2 reports the characteristics of that product that were analysed according to the following identification tests, referred in EN 1504-2 [5] and described on the normative documents also indicated: (i) chemical identification by FTIR; (ii) density, ISO 2081-1 [20]; and (iii) non-volatile matter content, ISO 3251 [21]. Table 2 also reports the freezing point and the total curing period of the impregnation product, indicated in the technical data sheet.

3.3. Surface preparation and characterisation

As already mentioned, the concrete specimens were grouped in two different series: (i) series R, in which specimens presented different roughness, but similar moisture content prior to the application of the silicate-based compound; and (ii) series H, in which specimens had a surface with similar roughness, but different moisture content.

3.3.1. Series R (roughness)

For series R, the surface of the concrete specimens was prepared using different mechanical processes chosen to produce three different roughness conditions: (i) a concrete surface that had not been in contact with the formwork and without any

Table 1

Concrete compositions (in kg/m³) and compressive and tensile strengths at 28 days of age (average ± standard deviation).

| Materials | MC(0.40) | C(0.70) |
|--|---------------|---------------|
| Limestone coarse aggregate 1 (4–10 mm) | 850 | 600 |
| Limestone coarse aggregate 2 (20 mm) | - | 400 |
| Siliceous river sand | 900 | 900 |
| Cement | 455 | 260 |
| Water | 182 | 182 |
| Compressive strength (MPa) | 56 ± 2 | 30 ± 2 |
| Tensile strength (MPa) | 4.2 ± 0.4 | 2.7 ± 0.2 |

Table 2

Main physical and chemical characteristics of the silicate-based impregnation.

| Property | Results |
|---|--|
| Chemical identification Density, 20 °C Non-volatile matter content at 125 °C Freezing point ¹ (°C) Total curing period ¹ (h) ¹ Manufacturer's technical sheet | Acrylic polymer and silicates 1.080 g/cm ³ 35.04% -2 4 to 6 |
| manufacturer 5 teenmeur sneet | |

preparation (R0); (ii) concrete surface roughened by using a water jet with 160 bar (R1) (Fig. 2a); and (iii) concrete surface roughened with a needle scaler (R2) (Fig. 2b).

To evaluate the surface roughnesses produced by the above mentioned procedures, two alternative techniques were used: (i) surface texture determination by moulding plasticine, according to ISO 4287 [22] (Fig. 3a); and (ii) roughness index determination by spreading sand, according to EN 1766 [19] (Fig. 3b). For the first method, the following procedure was carried out: (i) moulding plasticine $(40 \times 40 \text{ mm}^2)$ against the concrete surface (covered by a sheet of tracing paper); (ii) cutting the moulded plasticine with a scalpel to produce several cross-sections; (iii) digitalizing the cross-sections with a high-precision scanner; (iv) and computing the profiles coordinates and average roughness by using the *BuildingLife* software [23] and according to the geometrical product specifications provided in ISO 4287 [22] standard. For the second method, the determination of the surface texture involved the following steps: (i) positioning 2.5 ml of sand in the centre of the test specimens; (ii) spreading the sand with a disc, applying circular movements without pressure; and (iii) measuring the diameter of the resulting circle.

All specimens of series R were conditioned in order to stabilize their moisture content at about 5.0 \pm 0.5% (corresponding to level H2 of series H, cf. Section 2.3.2), as suggested in EN 1504-2 [5]. Therefore, the specimens were conditioned in a laboratory environment (temperature of 21 \pm 2 °C and relative humidity of 60 \pm 10%) or placed in an oven at 60 °C until their moisture content stabilized at the target level, as estimated using the calculation procedure described in EN 13579 [24].

3.3.2. Series H (moisture)

Concerning series H, prior to the application of the silicate-based compound, three different levels of moisture content were defined: (i) very dry (H1); (ii) an intermediate moisture level (H2); (iii) and water saturated (H3).

Before implementing the moisture content stabilization procedure, the concrete specimens were subjected to the 160 bar water jet surface preparation, corresponding to roughness R1 (Fig. 2a), described in Section 3.2.1.

The procedure for stabilizing the moisture content of the concrete specimens was based on the recommendations provided in EN 13579 [24] and described above (*c.f.* Section 2.3.1). Such methodology was duly adjusted to the need of increasing or decreasing the moisture content. In the former case, specimens were immersed in water or placed in a humidity chamber until their mass increased up to the predefined value. In the latter case, specimens were dried in an oven at 60 °C or in a conditioned room at (21 ± 2) °C and $(60 \pm 10)\%$ – the option for a temperature of 60 °C instead of ambient temperature was set in order to attain the lower and intermediate target moistures in due time. During the process of moisture content stabilization, the relative moisture analyser (used in the *Search* mode), which is based on electrical conductivity principles. These qualitative measurements were performed as an additional validation procedure regarding the moisture contents determined based on specimens weighing. In average, following the calculation procedure indicated in EN 13579 [24], the specimens from H1, H2 and H3 groups registered moisture contents of about 3%, 4.5% and 6%, respectively.

3.4. Application of the impregnation product

The silicate-based impregnation product was applied with a brush in the prepared surface of the test specimens, taking into account the quantities recommended by the manufacturer in the technical sheet, $110-215 \text{ g/m}^2$. After the product application, all specimens were kept in a conditioned room to ensure their drying in a controlled environment with temperature of 23 °C and relative humidity of 50%.

3.5. Tests to assess the performance of the impregnation product

3.5.1. Product penetration depth

The penetration depth was first evaluated following the procedures defined in EN 1504-2 [5]. In a first stage, the specimens treated with the impregnation product were fractured in two parts and the fracture surface was sprayed with water. The impregnated depth was taken as the dry zone (Fig. 4a).

Because the procedure described above proved to be inefficient in providing a measure of the penetration depth of the silicate-based compound, in a second stage the authors decided to use an alternative procedure, which consisted of adding a

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Fig. 2. Surface preparation using: (a) water jet; and (b) needle scaler.



Fig. 3. Surface roughness measurement using two alternative techniques: (a) pressing a plasticine mould against the concrete surface (ISO 4287); (b) spreading sand over the concrete surface (EN 1766).

 E_{t}

red pigment (*KVK Aquadisperse* FG - EP (04/87)) soluble in water to the impregnation product (Fig. 4b). Then, the mixture was applied in the specimens, according to the procedure specified in Section 2.4. These tests were performed only for specimens of series H, with a moisture content of about 5% (as specified in EN 13579 [24]) and roughness R1. According to EN 1504-2 [5], the penetration depth of impregnation products in concrete type C(0.70) should be at least 5 mm.

3.5.2. Water permeability

The water permeability was measured by using the experimental procedure described in the standard EN 1062-3 [25]. Prior to testing, the specimens were sealed with an epoxy coating in all faces, expect the one in which the impregnation product had been applied. The test consisted of immersing in demineralised water the surface of the specimens (previously treated), about 10 mm below the surface of the water (Fig. 4b). After predefined periods of time (10 min, 30 min, 1 h, 2 h, 3 h, 6 h, 24 h, 30 h, 48 h, 72 h and then every two days until attaining a saturation tendency), the specimens were dried with an absorbent cloth and then weighed.

The water transmissibility coefficient corresponding to the first 24 h (w) was calculated using equation (1), where Δm_{24} is the mass variation of a specimen between 0 h and 24 h of immersion (kg) and A is the specimen area (m²):

$$W = \frac{\Delta m_{24}}{\sqrt{24} \times A} [kg/(m^2 \cdot h^{0.5})]$$
(1)

3.5.3. Abrasion resistance

The abrasion resistance tests were carried out in accordance with EN 5470-1 [26] only in specimens from series H. A Taber abraser was used and, according to EN 1504-2 [5], H22 abrasive wheels were used with masses of 1 kg, which were attached to each arm of the abraser (Fig. 4c).

According to EN 1504-2 [5], 1000 cycles should be executed on each specimen and an improvement of at least 30% in the abrasion resistance of concrete type C(0.70) should be expected due to the application of the impregnation product.

However, after about 200 cycles it was observed that the treated surface layer had been completely abraded. Therefore, instead of the 1000 cycles referred in EN 1504-2 it was decided to consider only the first 100 cycles, after which the specimens were weighed. Based on the measured values, the percentage reduction of mass loss of protected specimens compared with that of non-treated specimens was computed (R).

3.5.4. Impact resistance

The impact resistance tests on both unprotected and protected specimens were carried out according to the standard EN ISO 6272-1 [27]. A test device was used to drop a mass of 1.973 kg with a sphere of 20 mm diameter at the end (Fig. 4d).

According to EN 1504-2 [5], a load class II (10 N m) was set. Preliminary tests were carried out for classes I (4 N m) and III (20 N m), the latter having caused the fracture of the concrete samples. The drop height of the mass (h = 0.517 m) was determined from the required gravitational potential energy ($E_p = 10$ N m), the mass of the sphere (m, in kg) and the gravity acceleration (g, in m/s²), using Eq. (2):

$$h_{p} = m \times g \times h[N m]$$
 (2)

3.5.5. Bond strength

The bond strength of the concrete substrate treated with the silicate-based product was determined from pull-off tests, carried out according to the standard EN 1542 [28].

Drills with 15 mm of depth and 50 mm of diameter were first performed on the specimens, using a core drilling machine. Metallic dollies with a diameter of 50 mm were glued to the surface of the concrete specimens with an epoxy adhesive. The dollies were connected to the pull-off equipment with a screw and then a tensile load was applied until failure (Fig. 4e).

The tensile load required to detach the dolly from the concrete specimen was measured and the type of failure was classified according to EN 1542 [28]. The bond strength (σ) was calculated from the following equation, where F_{max} is the maximum applied load and A_{dolly} is the dolly's cross-section area:

$$\tau = \frac{F_{\text{max}}}{A_{\text{dolly}}} \tag{3}$$

According to EN1504-2 [5], the bond strength for concrete type C(0.70) treated with impregnations must be higher than 1.5 MPa or 1.0 MPa for horizontal substrates subjected or not subjected to road traffic, respectively.

4. Results and discussion

4.1. Characterisation of surface roughness

Table 3 shows the results of the surface roughness evaluation. It can be seen that the two alternative methods provided very consistent results. In spite of the differences in the experimental techniques, results obtained follow the same general trend.

As expected, for the same type of surface preparation, concrete C(0.70) presents higher roughness index, due to its higher w/c ratio and hence the lower resistance of its superficial layer to the



Fig. 4. Tests to assess the performance of the silicate product: (a) depth penetration; (b) water permeability; (c) abrasion resistance; (d) impact resistance; and (e) bond strength.

Table 3 Characterisation of surface roughness based on test procedures defined in ISO 4287 and EN 1766 (in mm, average ± standard deviation).

| Type of | Plasticine Casts, ISO 4287 [22] | Sand Test, EN 1766 [19] |
|-------------------------|---------------------------------|-------------------------|
| concrete | (-) | (-) |
| MC(0.40).R ₀ | 0.014 ± 0.003 | 0.096 ± 0.003 |
| MC(0.40) R | 0.020 ± 0.008 | 0.154 ± 0.001 |
| $MC(0.40).R_2$ | 0.022 ± 0.003 | 0.164 ± 0.008 |
| C(0.70).R ₀ | 0.017 ± 0.006 | 0.133 ± 0.007 |
| C(0.70).R ₁ | 0.024 ± 0.013 | 0.181 ± 0.006 |
| C(0.70).R ₂ | 0.027 ± 0.005 | 0.207 ± 0.020 |

abrasive action caused by the water jet and the needle scaler superficial preparation procedures. For both types of concrete, the needle scaler technique (R2) provided higher roughness than the 160 bar water jet (R1), indicating a higher abrasive power of the needle scaler, when compared to the water jet. Compared with specimens with no surface preparation (R0), both techniques provided a considerable surface roughness increase.

4.2. Product penetration

As referred above, the visual procedure suggested in standard EN 1504-2 [5] was ineffective in detecting the presence of the silicate-based impregnation product in the concrete.

Nevertheless, the alternative procedure (cf. Section 2.5.1) that involved the use of a red pigment mixed with the impregnation product was successful in measuring the penetration depth. For concretes MC(0.40) and C(0.70), average penetration depths of respectively 0.075 mm and 2.00 mm were measured in specimens from series H (moisture content of about 5%) with roughness R1, based on 3 measurements performed on each specimen. In all specimens, the penetration was very uniform. As expected, the higher penetration depth was measured in the more porous concrete C(0.70), although the above mentioned values were much lower than the minimum penetration depth of 5.0 mm recommended by EN 1504-2 [5]. The penetration depth measured for the C(0.70) concrete was much higher than that reported by Dai et al. [10] for a similar concrete (w/c ratio of 0.68) and silicatebased impregnation. However, in this study, the penetration depth was measured after 1 year of exposure cyclic seawater showers under an outdoor environment.

4.3. Water permeability

4.3.1. Influence of the surface roughness

Fig. 5 plots the curves of the water absorption as a function of the square root of time of representative specimens from series R, illustrating the results obtained on the two types of concrete (MC(0.40) and C(0.70)), with three different surface roughnesses (R0, R1 and R2), either unprotected (O) or protected with the silicate-based impregnation compound (C). The slopes of the straight lines indicate the water transmissibility coefficient of the specimens, with higher slopes corresponding to higher speed of water penetration. The results of the water transmissibility coefficient after the first 24 h of immersion (w) are summarised in Table 4, together with the percentage reduction (R) of treated specimens compared to control specimens.

In what concerns the unprotected specimens, as expected, specimens made of concrete MC(0.40), presented a better performance in terms of water permeability (*i.e.*, were less permeable) compared to those made of concrete C(0.70). This result stems from the lower w/c ratio of the former type of concrete and thus its lower porosity. In addition, it can also be seen that in both types of concrete the speed of water transmission in unprotected specimens (O) consistently increases with the surface roughness, which also stems from the porosity increase caused by the surface preparation procedures, which facilitated the ingress of water.

The silicate-based compound provided significant water transmission reductions for both types of concrete and all surface roughnesses. In spite of the differences in the experimental procedures, the magnitude of the water absorption measured in this study is similar to those reported by Dai et al. [10] and Pigino et al. [18] from tests on concrete specimens protected by silicatebased impregnations. For concrete MC(0.40), the highest reductions in water transmission were obtained for the roughest concrete substrates, whose surfaces had been treated with 160 bar water jet (R1) and needle scaler (R2). For concrete C(0.70), the best performance of the impregnation product was also obtained for the highest surface roughness, but the influence of this parameter was less clear, as very similar water transmissibility reduction was obtained in surfaces without any surface treatment.

It is relevant to note that for both concretes MC(0.40) and C(0.70), the unprotected specimens without any surface preparation (R0.0) presented very similar results to the protected specimens with the different roughnesses. In other words, the application of the silicate-based impregnation basically counterbalanced the porosity increase caused by the surface preparation procedures, providing limited improvement when compared to unprotected specimens without surface preparation.

4.3.2. Influence of moisture content

Fig. 6 plots the water transmissibility curves of specimens from series H, showing the results obtained on specimens made of the two types of concrete (MC(0.40) and C(0.70)), unprotected (O) or protected with the silicate-based impregnation compound (C) applied with three different substrate moisture contents (H1, H2 and H3). The values of the water transmissibility coefficient after 24 h of immersion (w) are listed in Table 5, together with the percentage reduction (R) compared to reference specimens.

As for series R, the type of concrete proved to have a remarkable effect on the water absorption behaviour and also in the efficacy of the silicate-based impregnation. In concrete type MC(0.40), the surface protection provided reductions in the water transmissibility coefficient higher than 85% after 24 h of immersion (Table 5). In the more porous concrete C(0.70), those reductions were clearly lower, which stems from the higher porosity of this type of concrete and, consequently, from the lower ability of the impregnation product to fill totally the porous surface and seal the concrete surface. The influence of the concrete's quality in the water absorption behaviour is also shown in Fig. 6, particularly in what concerns the period of time necessary to attain a saturation tendency: in concrete C(0.70), most curves reflect saturation after an immersion period of about 100 h, while most specimens made of concrete MC(0.40) did not attain such saturation plateau during the whole test duration. Regardless of the moisture content, the more compact concrete most likely promoted the stagnation of the impregnation product at the surface (which is attested by its lower penetration depth) which in turn resulted in a more impermeable surface

For concrete type MC(0.40), the moisture content had only limited influence on the performance of the impregnation product – percentage reduction of the water transmissibility coefficient ranged between 86% and 91%. Results obtained for moisture contents H1 and H2 were virtually identical, with an additional

Table 4

Water transmissibility coefficient after 24 h of immersion (w) of series R and percentage reduction compared to control specimens (R).

| Identification | MC(0.40) | | C(0.70) | |
|----------------|---|-------|----------------------|-------|
| | w (kg/m ² h ^{0.5}) | R (%) | $w (kg/m^2 h^{0.5})$ | R (%) |
| R0.0 | 0.068 ± 0.013 | - | 0.301 ± 0.082 | - |
| RO.C | 0.045 ± 0.005 | 33.4 | 0.113 ± 0.006 | 62.5 |
| R1.O | 0.252 | - | 0.415 | |
| R1.C | 0.052 ± 0.008 | 79.7 | 0.31 ± 0.066 | 25.3 |
| R2.0 | 0.426 | - | 0.831 | - |
| R2.C | 0.056 ± 0.003 | 86.8 | 0.258 ± 0.056 | 69.0 |

improvement having been obtained for moisture content H3. For concrete type C(0.70), results obtained for moisture contents H1 and H2 were very similar to those obtained for the reference specimens. As for concrete MC(0.40), the best performance in concrete C(0.70) was provided (in this case, by far) by moisture content H3. The improved performance observed for specimens with moisture content H3 from both concrete compositions MC(0.40) and C(0.70) is attributed to the saturation of the pores: the increased moisture content of the substrate promoted the stagnation of the impregnation product at the specimens' surface.

4.4. Abrasion resistance

Fig. 7 presents the results of the abrasion resistance test for specimens from series H, after being subject to 100 cycles in the Taber abrader. Results are plotted in terms of mass loss exhibited by protected specimens compared to unprotected specimens. As already mentioned, after 200 cycles the silicate-based impregnation layer was no longer visible, hence no further cycles were performed.

The results obtained show that up to 100 abrasion cycles the silicate-based impregnation provided a relatively high degree of abrasion resistance, particularly in concrete type MC(0.40), for which the mass loss reduction compared with the unprotected concrete was higher than 75%. Even though there was considerable scatter in some test series of concrete type C(0.70), it seems that higher moisture contents of the substrate at the moment of application reduce the efficacy of the impregnation as far as the abrasion resistance is concerned. This result may stem from the fact that for higher moisture levels, the superficial pores are filled with water, causing the stagnation of the impregnation product at the surface, preventing it from penetrating in depth and chemically reacting with concrete. This stagnation, which had proved to improve the performance in terms of water permeability, has a detrimental effect in terms of abrasion resistance.



Fig. 5. Water transmissibility in series (a) MC(0.40).R and (b) C(0.70).R.



Fig. 6. Water transmissibility in series (a) MC(0.40).H and (b) C(0.70).H.

Table 5 Water transmissibility coefficient after 24 h of immersion (w) of series H and percentage reduction compared to control specimens (R).

| Identification | MC(0.40) | | C(0.70) | |
|----------------|---|-------|---|--------|
| | w (kg/m ² h ^{0.5}) | R (%) | w (kg/m ² h ^{0.5}) | R (%) |
| 0 | 0.347 ± 0.022 | | 0.618 ± 0.021 | - |
| H1.C | 0.049 ± 0.010 | 85.8% | 0.529 ± 0.246 | 14.4% |
| H2.C | 0.049 ± 0.010 | 85.8% | 0.709 ± 0.056 | -14.7% |
| H3.C | 0.031 ± 0.000 | 91.0% | 0.099 ± 0.034 | 84.0% |

Since the studied product could not penetrate deep enough to strengthen also the inner concrete, the minimum value required by the standard EN 1504-2 [8] for concrete type C(0.70) of 30% after 1000 abrasion cycles could not be attained. This suggests an insufficient performance of the silicate-based protection in light of the standard's requirements. On the other hand, the results obtained also show that as long as this impregnation product remains at the concrete surface, it provides a high level of abrasion resistance. Overall, the results obtained in show that the silicate based impregnation is effective in terms of abrasion resistance, but its durability is far below the requirements set in EN 1504-2.

4.5. Impact resistance

4.5.1. Influence of surface roughness

The influence of the surface roughness on the impact resistance of specimens from series R is illustrated in Fig. 8. The results are presented in terms of the diameter of the impact zone.



Fig. 7. Influence of the substrate moisture content in reduction of mass loss for concretes MC(0.40) and C(0.70) (average ± standard deviation, the horizontal line represents the limit specified in EN 1504-2).

As expected, specimens made of concrete MC(0.40) performed better than those made of concrete C(0.70) in what concerns the resistance to impact. Such better performance of the former type of concrete naturally arises from its lower w/c ratio and therefore its higher compactness. For unprotected specimens, it is also relevant to note that roughness type R0 (for which specimens still exhibit the slurry laitance of the concrete) provided better performance to impact in both types of concrete. The difference between the impact resistance of specimens with roughnesses R1 and R2 (with surface preparation) is not significant (taking into account the mean and standard deviation values).

By comparing the diameter of the impact site for specimens with and without protection, it is possible to conclude that the improvement provided by the application of the impregnation product was negligible for concrete MC(0.40) and moderate for concrete C(0.70). In this respect, the varying efficacy of the impregnation product in the different types of concrete stems from their w/c ratios. While in the more compact concrete MC(0.40) the potential beneficial effect of the impregnation product is not visible (because concrete already performs well), in the more porous concrete C(0.70) such effect is visible.

In what concerns the influence of the surface roughness on the impact resistance of protected specimens, it proved to have a slightly detrimental effect in concrete type MC(0.40) (similarly to the unprotected specimens and possibly for the same reasons, namely the competing effects stemming from the slurry laitance removal and the application of the protection layer), with no significant influence being observed in concrete C(0.70).

4.5.2. Influence of moisture content

Fig. 9 plots the diameter of the impact zone for specimens from series H.

Once again, regardless of the application of silicate-based impregnation, specimens made of concrete MC(0.40) present better impact resistance than those made of concrete C(0.70), attesting once again that the w/c ratio of concrete influences much more the impact resistance than the application of the impregnation product. In fact, for both types of concrete, the application of the impregnation product did not introduce a noticeable improvement on the impact resistance. Concerning the influence of the moisture content on impact resistance, for concrete MC(0.40) there seems to be a slight improvement with increasing moisture content (possible due to the above mentioned phenomenon of product retention at the surface). However, taking into account the scatter of the experimental results, it is not possible to draw definitive conclusions with this respect. Moreover, on concrete type C(0.70), such influence is not observed.



Fig. 8. Influence of the substrate roughness in the impact resistance for concretes (a) MC(0.40) and (b) C(0.70) (average ± standard deviation).



Fig. 9. Influence of the substrate moisture content in the impact resistance for concretes MC(0.40) and C(0.70) (average ± standard deviation).

4.6. Bond strength by pull-off

4.6.1. Influence of surface roughness

Fig. 10 plots the results of bond strength (or direct tensile strength in unprotected specimens) measurements on specimens from series R, corresponding only to valid failure modes, *i.e.* specimens in which bonding problems were observed were excluded from the analysis. Failure of the unprotected specimens (O, with no surface preparation and moisture content H2) always occurred in the concrete. In the protected specimens (C), in almost all specimens the loss of bond occurred at the interface of impregnated surface and inner concrete.

For unprotected specimens, the higher direct tensile strength of concrete type MC(0.40) compared to concrete C(0.70) naturally stems from the lower w/c ratio of the former concrete, which provides higher mechanical strength. In the protected specimens, such lower w/c ration also promotes a higher bond strength of the impregnated surface layers.

For protected specimens made of concrete MC(0.40), for all types of surface roughness, the bond strength was lower (in average, 56%) than the pull-off strength of the unprotected specimens. In opposition, for protected specimens made of concrete C(0.70), although the scatter in the results was relatively high, in average the bond strength was similar to the pull-off strength of the unprotected concrete.

Regarding the possible influence of roughness on the bond strength between the inner concrete and the silicate-impregnated surface layer, due to the high scatter in the experimental results, it is not possible to conclude about the existence of any type of relation between those two parameters. Yet, for specimens C(0.70), it is worth noting that the lowest average results were obtained for specimens having the lower roughness (R0), *i.e.* without any type of surface preparation.

4.6.2. Influence of moisture content

Fig. 11 illustrates the bond strength for specimens from series H. As for series R, failure in unprotected specimens (with no surface preparation and moisture content H2) occurred within the concrete core, whereas in the vast majority of protected specimens failure occurred at the interface between the inner concrete and the impregnated surface.

Similarly to series R, higher tensile strengths (in unprotected specimens) and higher bond strengths (in protected specimens)



Fig. 10. Influence of the substrate roughness in the bond strength by pull-off test for concretes (a) MC(0.40) and (b) C(0.70) (the horizontal line represents the limit specified in EN 1504-2).



Fig. 11. Influence of the substrate moisture content in the bond strength by pull-off test for concretes (a) MC(0.40) and (b) C(0.70) (the horizontal line represents the limit specified in EN 1504-2).

were measured in concrete type MC(0.40), due to its lower w/c ratio. Also in this series, the difference between the average bond strength of protected specimens (with different moisture contents) and the direct tensile strength of unprotected specimens was higher in concrete MC(0.40) compared to concrete C(0.70).

In what concerns the influence of the moisture content on the bond strength, for both types of concrete, although moisture level H2 provided the best performance for both types of concrete, taking into account the scatter of the results, it is not possible to draw any definitive conclusions.

5. Conclusions

This paper presented results of an experimental study about the influence of the concrete substrate on the performance of a silicatebased impregnation product. In particular, this study evaluated the influence of the type of roughness and moisture content at the moment of application on the efficacy of the impregnation. Based on the results obtained, the following main conclusions are drawn:

- 1. Regardless of the substrate properties and type of surface preparation, the silicate-based impregnation was effective in improving the performance in terms of water permeability and abrasion resistance. In opposition, the impregnation product did not improve the resistance to impact.
- 2. The abrasion resistance improvement mentioned above was observed only while the impregnation product remained at the concrete surface. In this regard, from a durability point of view, it is relevant to note that the impregnation was completely abraded after only 200 cycles, far below the 1000 cycles mentioned in the EN1504-2 standard.
- 3. The procedure indicated in standard EN 1504-2 for evaluating the penetration depth proved to be inadequate for the silicate based impregnation used in this study. The alternative procedure involving adding a pigment to the impregnation product appears to be feasible. As expected, a higher penetration was observed in concrete C(0.70) when compared to concrete MC(0.40), stemming from the higher porosity of the former type of concrete.
- 4. As expected, for all tests performed, specimens made of concrete MC(0.40) presented better performance than those made of concrete C(0.70), due to the lower w/c ratio of the former concrete, which influences the porosity and mechanical properties of the substrate and also the bond strength of the impregnated surface layer.
- 5. In terms of water permeability, for both types of concrete, the best performance among the protected specimens was obtained for specimens not subjected to any type of surface preparation

(roughness R0), especially for concrete type C(0.70). This means that there is no advantage in increasing the roughness of the substrate prior to the application of silicate-based impregnation products for water-ingress protection purposes. The highest moisture content improved the performance of the impregnation, especially for the more porous concrete, most likely because the increased moisture content of the substrate promoted the stagnation of the impregnation product at the specimens' surface, resulting in a more impermeable surface.

- 6. Regarding abrasion resistance, unlike water permeability, higher moisture contents reduced the efficacy of the impregnation. In this case, the stagnation of the impregnation product at the surface must have prevented it from penetrating in depth and chemically reacting with concrete.
- 7. As far as impact resistance is concerned, the surface roughness and the moisture content had only a marginal effect in the performance of the silicate-based impregnation.

As a final remark, this study highlights the importance of defining *a priori* the objectives of using silicate-based impregnations for concrete surface protection. These objectives should be considered when defining the most appropriate surface preparation procedures.

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