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Comparison between surface and bulk hydrophobic treatment against corrosion of galvanized reinforcing steel in concrete

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ABSTRACT

The effectiveness of bulk hydrophobic treatment against corrosion of galvanized steel reinforcement in concrete specimens with w/c = 0.45 and w/c = 0.75 was compared with that of surface treatment, even in the presence of cracks 0.5 and 1 mm wide in the concrete cover. In this case surface hydrophobic treatments were applied both before and after cracking as a preventive and a restorative method against reinforced concrete deterioration, respectively. The obtained results in terms of water absorption, electrochemical measurements, chlorides penetration, and visual observations carried out on reinforced concrete specimens during the exposure to wet–dry cycles in 10% NaCl solution showed that bulk hydrophobization is the most effective treatment in improving the corrosion resistance of galvanized steel reinforcements in concrete also in the presence of cracks. Surface hydrophobization is very effective just in the first few exposure cycles to the aggressive environment and when used as a restorative method which is able to cancel the deleterious effect of cracks only 0.5 mm wide.

1. Introduction

Weathering is the most important harmful factor for building materials causing their deterioration, detectable through exfoliation, scaling, crumbling and reinforcement corrosion. Weathering is in general related to water penetration into porosity of materials, since water acts as the main carrier of aggressive agents, as chlorides for reinforcement corrosion, and water is essential for most deterioration reactions [1].

A water droplet in contact with concrete, that is a porous hydrophilic material, wets the solid surface by spreading itself and penetrates the cementitious matrix by means of capillary forces following the Washburn equation, $p = (2\,\gamma/r_c)\cos\sigma$, where γ is the liquid surface tension, r_c is the capillary pore radius, and σ is the contact angle. The contact angle is less than 90°, p is positive and water fills the pore spontaneously. Since the molecular attraction between water and the concrete pore walls may be lowered by using hydrophobic agents, such as those currently named silanes and siloxanes [2], surface hydrophobic treatments or the introduction of hydrophobic agents directly in the mixture should involve an increase in durability. Previous works have shown that hydrophobic admixtures protect steel reinforcement from corrosion only in sound concrete, while in cracked concrete it can induce a catastrophic corrosion [2]. This unexpected result has been ascribed to a greater oxygen diffusion through an unsaturated hydrophobic cementitious matrix that, in this way, can feed more quickly the cathodic reaction controlling the corrosion process [3]. However, if the reinforcement is galvanized, in which case it has been already experimented that passivation is mainly promoted by oxygen [4], the use of hydrophobic admixture always improves the corrosion resistance of galvanized steel reinforcements in concrete, even in the presence of concrete cracks, especially when high w/c ratios are used [5]. In this work, the effectiveness of bulk hydrophobic treatment against corrosion of galvanized steel reinforced in concrete specimens with w/c = 0.45 and w/c = 0.75 was compared with that of the surface treatment, even in the presence of cracks 0.5 and 1 mm wide. In this case surface hydrophobic treatments were applied both before and after cracking as a preventive and a restorative method against reinforced concrete deterioration, respectively.

2. Experimental

2.1. Materials

A commercial Portland-limestone blended cement type CEM II/A-L 32.5 R according to EN-197/1 was used. Natural sand with 6 mm maximum size and gravel with 11 mm maximum size were used as fine and coarse aggregates, respectively, to manufacture concretes. A 45% aqueous emulsion of an alkyl-triethoxy-silane was used as a hydrophobic silane-based admixture to manufacture bulk hydrophobic cementitious materials. A commercial hydrophobizing agent

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Table 1
Concrete mixture proportions (kg/m^3) and related compressive strengths (MPa).

Mixtures	w/c = 0.45	w/c = 0.45 hydrophobic	w/c = 0.75	w/c = 0.75 hydrophobic
Water	240	233	240	236
Cement	533	533	320	320
Silane	–	11.8	–	7.1
Sand	639	639	949	949
Gravel	847	847	716	716
R_c at 2 days	19.9	16.8	6.7	4.,9
R_c at 7 days	35.4	27.5	14.5	9.2
R_c at 28 days	39.2	32.3	25.6	19.8

based on siloxanic resin was used as a hydrophobic surface treatment on concrete specimens.

2.2. Specimens

Concrete mixtures with w/c of 0.45 and 0.75 were manufactured with and without silane as a hydrophobic admixture added at a dosage of 1% of active ingredient by cement weight. The proportions of the concrete mixtures are given in Table 1, where compressive strengths at 2, 7, and 28 days are also reported. In the presence of silane admixture, a reduction in the concrete compressive strength of about 18 and 23% was recorded with respect to that of non-hydrophobic concrete when a w/c ratio of 0.45 and 0.75 was used, respectively.

For each concrete type, prismatic specimens (70 × 70 × 280 mm in size) were produced (Fig. 1). These prismatic specimens were reinforced with a hot dip galvanized steel plate (210 × 40 × 1 mm) embedded at mid depth from a specimen side. The zinc coating, obtained by immersion in molten zinc, was 100 μm thick with an outer pure zinc layer about 20 μm thick. An electric cable, isolated through a PVC sheath, was connected to each steel plate by means of spot welding. The weld was protected by epoxy resin before casting the concrete mixture. Steel plates instead of usual rebars were used in order to facilitate the cracking of specimens and to modulate the crack width.

After casting, all the specimens were wet cured for 2 days (RH = 100% and T = 22 °C) and air dried for 1 month at RH = 60% and T = 22 °C. Then, some specimens for each concrete type were deliberately cracked by a flexural stress in order to open a crack 0.5 or 1 mm wide in the middle zone, with the crack apex reaching the steel reinforcement. In order to localize the crack formation at a predetermined point, a preformed V-notch, 10 mm deep and 14 mm wide, was produced on these specimens by placing a plastic rod on the bottom surface of the mold. Once the crack of required width, measured by an extensometer, was obtained its opening was maintained after the elimination of the flexure load by the insertion of small elements of hard polymeric material of suitable thickness. In order to make the oxygen and chloride flow unidirectionally and perpendicularly directed to the steel plate through the concrete cover, all the other sides of the specimens but the one containing the crack apex were epoxy coated.

The hydrophobic surface treatment, if any, was applied after curing of the concrete specimens by impregnating the specimens' surface until saturation with the commercial hydrophobizing agent. Some specimens were cracked by flexural stress (crack width of 1 mm and 0.5 mm) after an additional week. In order to estimate the efficiency of the hydrophobic surface treatment as a restorative method against deterioration, half of the specimens were coated after cracking. In order to estimate the efficiency of the surface coating as a preventive method against corrosion, the other half was coated before cracking. Table 2 explains the nomenclature used to identify the different cases studied.

2.3. Tests

2.3.1. Water absorption at atmospheric pressure

Water absorption tests at atmospheric pressure were carried out on concrete and mortar specimens according to the procedure reported in UNI 7699:2005 — Testing hardened concrete — determination of water absorption at atmospheric pressure.

2.3.2. Electrochemical measurements

After the curing period, the hydrophobic surface treatment and the cracking (about 45 days from the cast), all the specimens were exposed to wet–dry cycles (2 days wet followed by 5 days dry) in a 10% NaCl aqueous solution for four months.

The corrosion risk of the reinforced concrete specimens exposed to the chloride environment was evaluated by free corrosion potential measurements with respect to a saturated calomel electrode (SCE) as reference, while the kinetics of the corrosion process were followed by polarization measurements. The polarization resistance, in inverse relation with the corrosion rate, was measured through the galvanodynamic method (0.5 μA/s) by calculating the average value between the anodic and the cathodic branch ($\Delta V = \pm 5$ mV). An external graphite bar (210 × 40 × 10 mm) on the surface of the specimen was used as counter-electrode; the surface area of galvanized steel exposed to the environment was 210 × 40 × 2 mm^2. The electrochemical measurements reported in the following graphs were carried out every cycle at the end of the wetting period with the specimen, the reference electrode and the counter-electrode immersed in the chloride solution.

2.3.3. Chloride content and visual evaluation

In all cases, the free chloride content of the concrete in proximity of the steel reinforcement was evaluated by water extraction at the end of the exposure time to the chloride environment. The concrete powder was collected by a drill, at 0–1 cm from the galvanized plate and 1 cm from the notch initiating crack, from both the two sides of the crack. The powder was mixed with distilled water (water/powder = 12 by weight) for 24 hours, then the solution was filtered and analyzed by ion chromatography. The chloride content has been reported as % by

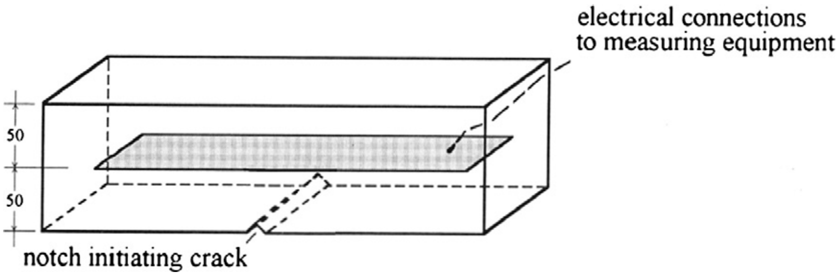

Fig. 1. Prismatic reinforced concrete specimens.

Table 2
Nomenclature used to identify the different cases studied.

Description	Nomenclature
No hydrophobic treatment, no crack	no-h
No hydrophobic treatment, crack 0.5 mm wide	no-h-0.5
No hydrophobic treatment, crack 1 mm wide	no-h-1
Bulk hydrophobic treatment, no crack	h-bulk
Bulk hydrophobic treatment, crack 0.5 mm wide	h-bulk-0.5
Bulk hydrophobic treatment, crack 1 mm wide	h-bulk-1
Surface hydrophobic treatment, no crack	h-sur
Surface hydrophobic treatment applied before crack 0.5 mm wide	h-sur-0.5
Surface hydrophobic treatment applied before crack 1 mm wide	h-sur-1
Surface hydrophobic treatment applied after crack 0.5 mm wide	h-0.5-sur
Surface hydrophobic treatment after crack 1 mm wide	h-1-sur

cement weight. The cement content of the manufactured concretes was calculated by the mix proportions reported in Table 1.

Finally, in order to validate and to complete the evaluation of the electrochemical behavior, the galvanized steel plates were extracted by splitting the concrete specimens after 16 wet–dry cycles in the chloride solution and the corrosion extent was assessed by visual observation. Zinc corrosion products were identified by X-ray diffraction.

3. Results and discussion

3.1. Water absorption at atmospheric pressure

Figs. 2 and 3 show the results of water absorption tests at atmospheric pressure obtained on concrete specimens with a w/c ratio of 0.45 and 0.75, respectively. It is evident that cracks in the concrete cover do not generally affect the test results of non-hydrophobic concretes and bulk hydrophobic concretes, probably because the surface area of the crack is much smaller with respect to the whole specimen surface in contact with water. At the end of the test, hydrophobic concretes with silane admixture at a dosage of 1% by cement weight absorb just 50% and 35% of water absorbed by the corresponding non-hydrophobic ones when w/c of 0.45 and 0.75 are used, respectively. The hydrophobic admixture due to its ability to decrease the molecular attraction between water and the concrete pore walls, without decreasing the concrete porosity [2], seems able to cancel the deleterious effect of a high w/c and concrete cover cracking up to 1 mm wide on concrete water absorption.

On the other hand, surface hydrophobic treatment seems to be effective just in the first days of full immersion in water but after longer water contacts surface hydrophobic concrete absorbs almost the same water absorbed by the non-hydrophobic one especially when high w/c ratios are adopted and the surface treatment is used as a preventive method against deterioration (before cracking). Moreover, when the hydrophobic surface treatment is used as a restorative method against deterioration (after cracking) it is able to hinder water penetration only in sound concrete or in a 0.5 mm cracked concrete but not in a 1 mm cracked concrete.

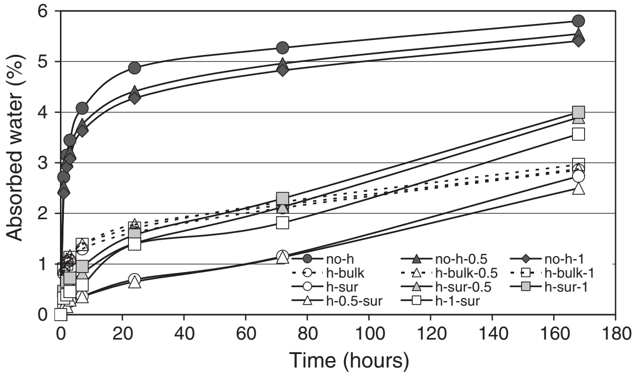

Fig. 2. Water absorbed by concrete specimens with w/c = 0.45, as a function of the test time.

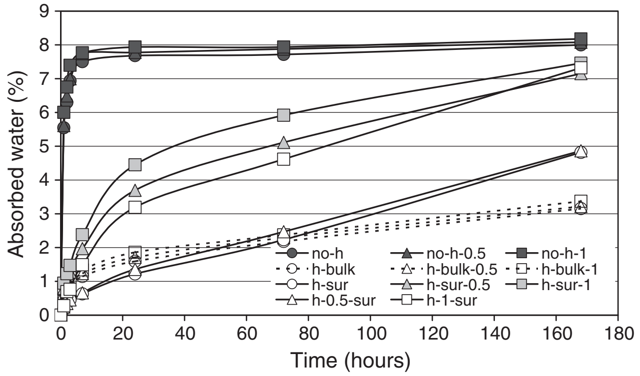

Fig. 3. Water absorbed by concrete specimens with w/c = 0.75, as a function of the test time.

3.2. Electrochemical measurements

The discussed electrochemical values are averaged among the measurements carried out on three specimens of each concrete type.

Just after the cast, the reinforcements embedded in the specimens assumed potentials values of about −1350 mV/SCE which are typical values for the zinc activation state from the cast time as long as the specimens were stored in their molds. After demolding the values rose after about 6 hours to −750 mV(SCE) in concretes with w/c = 0.45 and to −600 mV/SCE in concretes with w/c = 0.75. Although these potentials would represent active corrosion for steel they are not for zinc. This difference related to w/c is due to the greater porosity of the cementitious matrix with the higher w/c. The greater porosity allows a higher amount of oxygen to flow through the concrete cover, to reach the galvanized rebars and to induce a better zinc passivation. During the whole air curing period the potentials continue to become more positive (from −700 mV/SCE to −500/−400 mV/SCE). This behavior was observed regardless of the presence of the hydrophobic treatment and it is analogous to those already discussed in References [4,6] and [7].

Fig. 4 shows the free corrosion potential values of galvanized steel plates embedded in concrete specimens with w/c = 0.75 as a function of wet-dry cycles in the aggressive environment based on chlorides. After exposure to chlorides, in sound non-hydrophobic concrete the potentials assume active values of about −1000 mV/SCE reflecting a

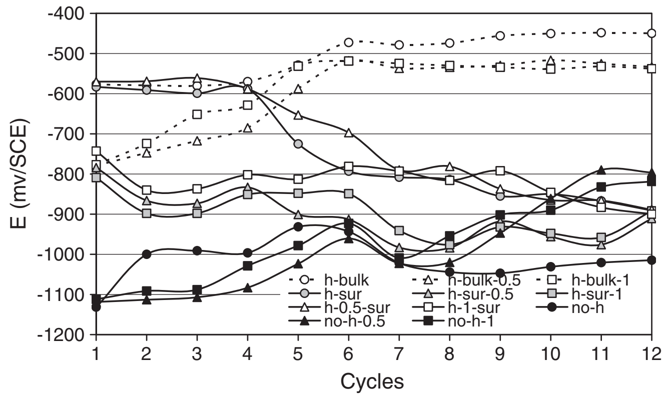

Fig. 4. Corrosion potential of the galvanized steel plates embedded in concrete specimens with w/c = 0.75, as a function of the test time.

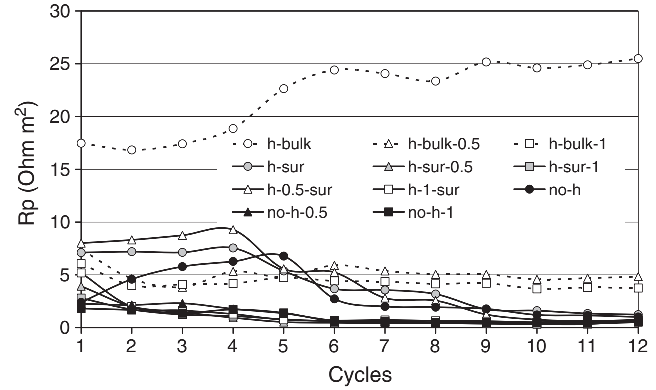

Fig. 5. Polarization resistance of the galvanized steel plates embedded in concrete specimens with w/c = 0.75, as a function of the test time.

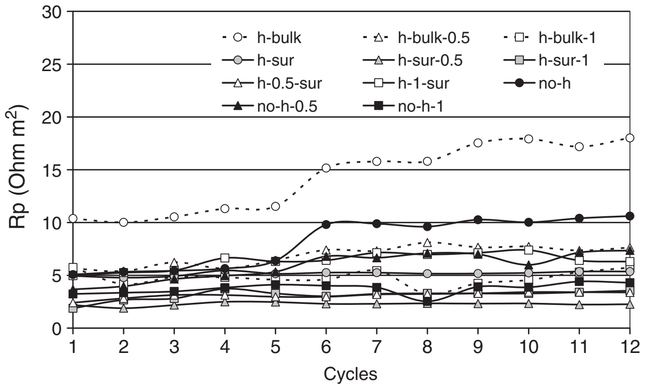

Fig. 7. Polarization resistance of the galvanized steel plates embedded in concrete specimens with w/c = 0.45, as a function of the test time.

generally great corrosion risk and they remain constant for almost the entire test period. Galvanized steel in the non-treated concrete does not passivate due to the lack of oxygen, that is the fundamental oxidizing agent for a fast and effective zinc passivation in concrete [3–5], in a well saturated cementitious matrix as that with a high w/c (0.75) after 2 days of immersion in a chloride solution. In the presence of concrete cracks, but without the hydrophobic treatment, the free corrosion potential after 7 cycles moves towards more anodic values (−800 mV/SCE, that represents active corrosion for steel) when, due to the chloride attack, the underlying steel becomes exposed at the intersection with the cracks and then it actively corrodes.

On the other hand, in hydrophobic concrete, whatever the type of hydrophobic treatment, just after the exposure to the aggressive environment the galvanized steel plates assume values of −600 mV/SCE in sound specimens or in after cracking surface treated specimens if the crack width is 0.5 mm, and −800 mV/SCE in all the other cases. This means the ability of the hydrophobic treatments to prevent water penetration especially in sound concrete or when used as a restorative method in 0.5 mm crack width. However, when bulk hydrophobic treatment is used, the free corrosion potentials move towards more passive values, indicating a very low corrosion risk, after a few wet-dry cycles. This is due to the great capacity of the hydrophobic admixture to repel water, even with the presence of concrete cracks, which favors faster oxygen diffusion through concrete, thus making galvanized reinforcement passivation faster and easier [3–5]. On the contrary, when surface hydrophobic treatment is applied as a preventive or as a restorative method against corrosion, the corrosion potential moves towards more active values due to the chloride attack to zinc and at the end of the test it reaches the same values detected in non-hydrophobic concrete. This is due to the lower capacity of the surface treatment to hinder water penetration for a long time also when applied as a restorative method against deterioration and in the absence of concrete cracks.

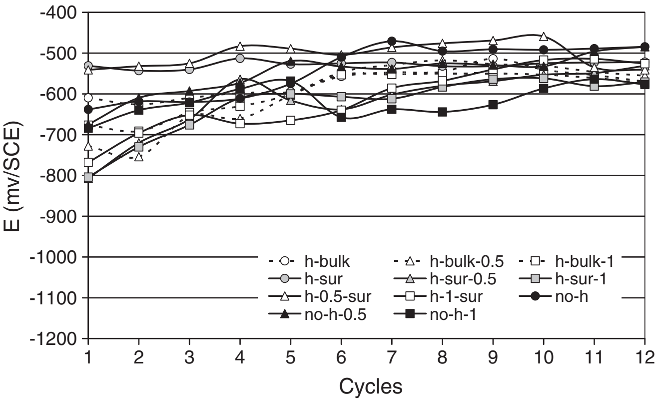

Fig. 6. Corrosion potential of the galvanized steel plates embedded in concrete specimens with w/c = 0.45, as a function of the test time.

Concerning the kinetics of the corrosion process, in the absence of concrete cracks, the polarization resistance (Fig. 5) of the steel reinforcement in bulk hydrophobic concrete with w/c = 0.75 can be four times higher with respect to that in non-hydrophobic concrete, indicating the good efficiency of the hydrophobic admixture in decreasing the corrosion rate; moreover, this value seems to further increase during the test time indicating that zinc passivation increases progressively. In the presence of concrete cracks, the difference between bulk hydrophobic and non-hydrophobic concrete is moderate, but the polarization resistance of steel reinforcement in hydrophobic concrete remains at least three times higher with

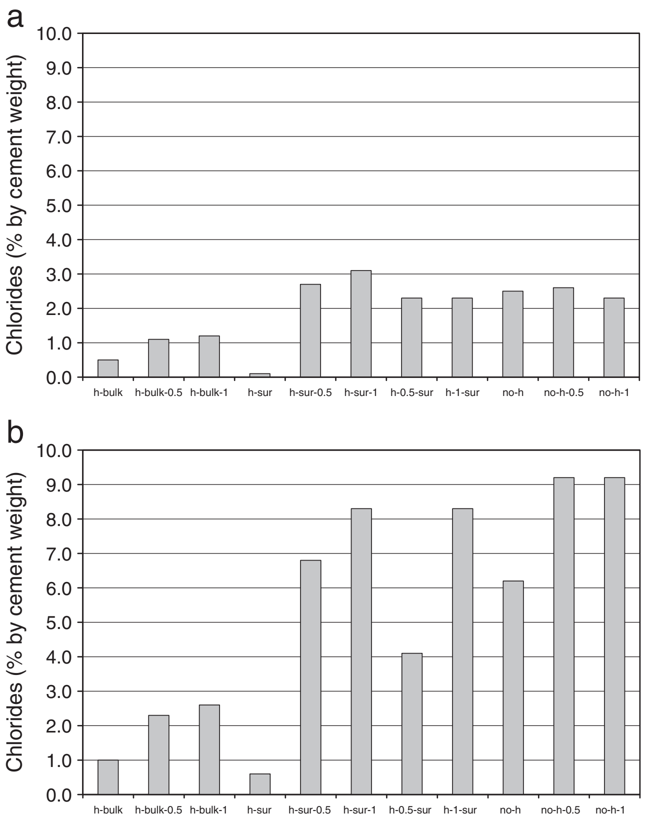

Fig. 8. a. Free chloride ion concentration (% by cement mass) in concrete specimens with w/c = 0.45. b. Free chloride ion concentration (% by cement mass) in concrete specimens with w/c = 0.75.

respect to that of non-hydrophobic concrete. Moreover, in this case, cracked hydrophobic concrete behaves even better than sound non-hydrophobic concrete, whatever the crack width; therefore, bulk hydrophobic treatment seems to cancel, from a corrosion point of view, the detrimental effect of concrete cover cracking.

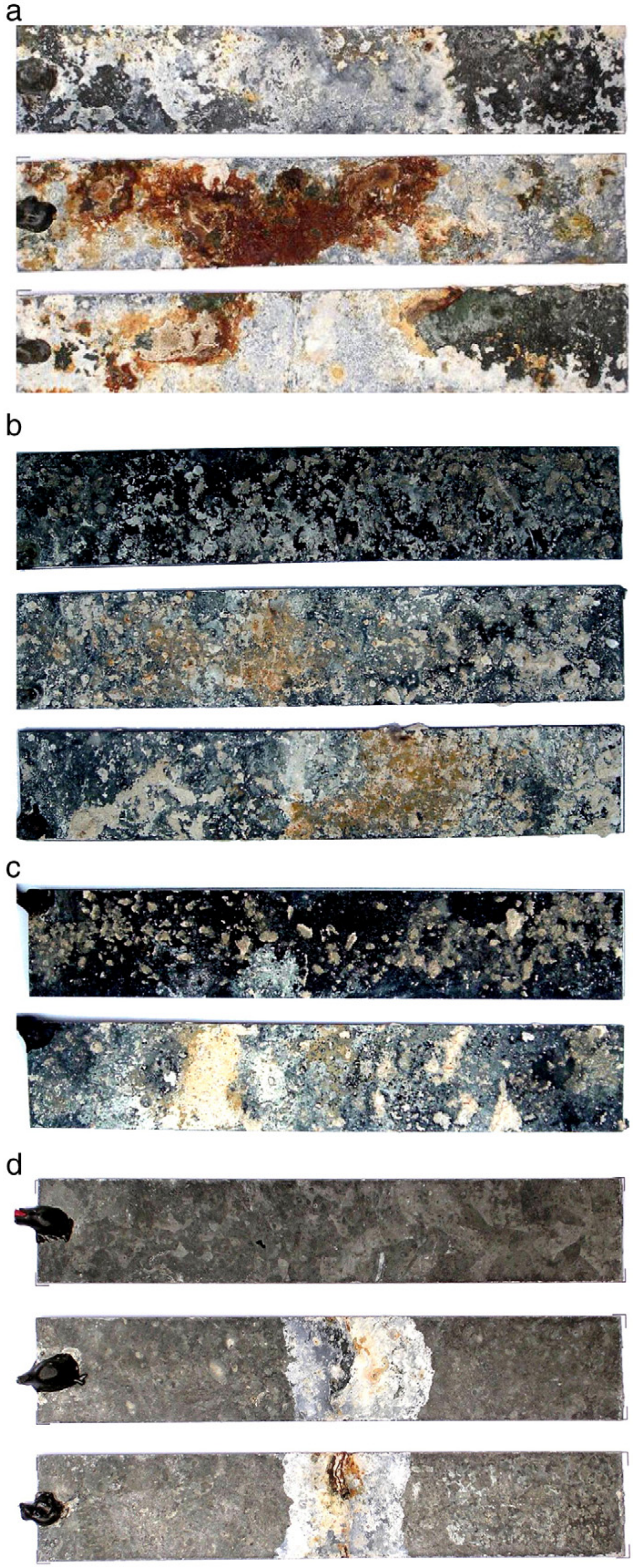

Fig. 9. a. Visual observation of the galvanized steel plate embedded in concrete specimens with w/c = 0.75 in the absence of the hydrophobic admixture (up: no crack; middle: 0.5 mm crack; down: 1 mm crack). b. Visual observation of the galvanized steel plate embedded in concrete specimens with w/c = 0.75 in before cracking surface hydrophobic concrete (up: no crack; middle: 0.5 mm crack; down: 1 mm crack). c. Visual observation of the galvanized steel plate embedded in concrete specimens with w/c = 0.75 in after cracking surface hydrophobic concrete (up: 0.5 mm crack; down: 1 mm crack). d. Visual observation of the galvanized steel plate embedded in concrete specimens with w/c = 0.75 in the presence of the hydrophobic admixture (up: no crack; middle: 0.5 mm crack; down: 1 mm crack).

The two different crack widths do not affect the corrosion results in non-hydrophobic concrete specimens with w/c = 0.75, due to the high porosity of the cement matrix, while, in the hydrophobic ones, a slightly higher corrosion rate is detected in the presence of the wider crack. When hydrophobic surface treatment is adopted, initially a better corrosion behavior is detected just in the absence of concrete cracks or in the presence of 0.5 mm wide cracks covered by the surface treatment. However, after 7 cycles surface hydrophobic concrete behaves almost like non-hydrophobic concrete whatever the surface treatment.

A good quality concrete matrix with a w/c as low as 0.45 seems to partially hide the beneficial effect of the hydrophobic treatment. The corrosion risk described by the free corrosion potential measurements (Fig. 6) is about the same regardless of the cement matrix, especially at the end of the test. However, the polarization resistance of steel reinforcement (Fig. 7) is always higher in the bulk hydrophobic concrete specimens with respect to that of the corresponding non-hydrophobic ones, even if the difference is not as marked as observed in the more porous cement matrix. In particular, in the absence of concrete cracks, bulk hydrophobic concrete shows a polarization resistance that is approximately twice that of other specimens. When concrete cover is cracked the difference is not so evident. However, only bulk hydrophobization and surface hydrophobization applied as a restorative method seem to give a certain increase in polarization resistance. Moreover, due to the low porosity of the cement matrix, the crack width influenced the test results both in the presence and in the absence of silane with corrosion risks and corrosion rates which generally increase with the crack width.

Finally, by comparing the results obtained in concrete specimens with w/c = 0.75 (Figs. 4 and 5) and w/c = 0.45, (Figs. 6 and 7) it is evident that the hydrophobic admixture, even in the presence of concrete cracks, decreases the corrosion rate monitored in very porous concrete, manufactured with w/c = 0.75, to values comparable with those obtained in good quality concrete, manufactured with w/c = 0.45. In other words, silane cancels the detrimental effect, at least from the corrosion point of view, of a large porosity of the cement matrix.

3.3. Chloride content and visual evaluation

The chloride content values reported in the graphs are averaged among the values obtained by the analysis carried out on three specimens of each concrete type.

In concretes with w/c = 0.45 (Fig. 8a) the chloride values ranged from 0.2% by cement weight in sound hydrophobic concretes (regardless the type of treatment) to 3% by cement weight in non-hydrophobic concretes or cracked concretes with the hydrophobic surface treatment. In concretes with w/c = 0.75 (Fig. 8b), the chloride values generally ranged from 0.7% by cement weight in sound hydrophobic concrete (regardless the type of treatment) to 9.0% by cement weight in non-hydrophobic concretes or cracked concretes with the hydrophobic surface treatment. In particular, in the presence of concrete cracks only bulk hydrophobic treatment is always able to reduce chloride penetration (Fig. 8a and b) significantly by at least

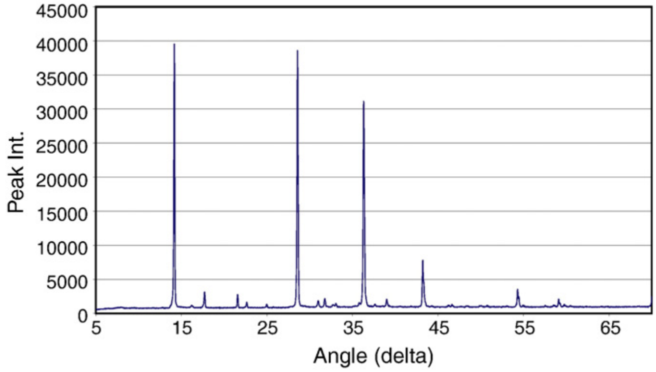

Fig. 10. X-ray Diffraction analysis of white zinc corrosion products.

about 50% and 70% when w/c = 0.45 (Fig. 8a) and w/c = 0.75 (Fig. 8b) are used, respectively.

However, when concrete with high w/c ratio is used (w/c = 0.75) also the surface treatment seems effective in reducing chloride penetration when used as a restorative method in 0.5 mm concrete cover crack width (Fig. 8b).

The corrosion attack evaluated by the visual observations carried out on the galvanized steel plates removed from the specimens after the exposure to the chloride environment were in quite good agreement with the conclusions obtained by the water absorption test (Section 3.1), the electrochemical measurement (Section 3.2) and the chloride content analysis.

The galvanized steel plates extracted from non-hydrophobic concrete (Fig. 9a) showed a very deep general corrosive attack with red rust appearing on the surface indicating that total consumption of the protective zinc layer had occurred.

Concerning with the hydrophobic surface treatment, when applied as a restorative method (Fig. 9b and c), dark Fe–Zn alloys appeared on the plate, with zinc grains no longer visible on the surface, meaning the total consumption of the pure zinc layer, both in sound and in 0.5 mm wide cracked concrete. In the other cases, light red rust appeared also on the surface probably due to the corrosion of Fe from alloying sublayers since Fe–Zn alloys are not stable in presence of chlorides [7,12].

On the other hand, zinc grains were still visible in steel reinforcement embedded in bulk hydrophobic concrete (Fig. 9c) and close to the crack apex a white surface deposit of adherent and compact zinc corrosion products, later identified by X-ray diffraction as zinc oxides, hydroxides and calcium hydroxyzincate (Fig. 10), appeared. In particular calcium hydroxyzincate is a well known passivating zinc corrosion product that, once formed, protects the underlying pure zinc layer from further corrosion [4–11].

For brevity, only photos of the galvanized steel plates embedded in concretes with w/c = 0.75 are reported. However a very similar trend was detected from the visual evaluation of the galvanized steel reinforcements removed from the specimens with w/c = 0.45.

4. Conclusions

The effectiveness of bulk hydrophobic treatment against chloride induced corrosion of galvanized steel reinforcement in concrete specimens with w/c = 0.45 and w/c = 0.75 was compared with that of the surface hydrophobic treatment, even in the presence of cracks 0.5 and 1 mm wide. The surface hydrophobic treatment was applied both before and after cracking as a preventive and a restorative method against reinforced concrete deterioration, respectively. The obtained results showed that bulk hydrophobic treatment is the most effective treatment to improve the corrosion resistance of galvanized steel reinforcements in concrete, also in the presence of cracks. Especially when high w/c are adopted, surface hydrophobic treatment is effective in preventing water penetration just for short period (few days) of contact with water. Moreover, the hydrophobic surface treatment is able to improve significantly the corrosion behavior of galvanized reinforcements in concrete only in the first few exposure cycles and when used as a restorative method in the presence of 0.5 mm wide cracks.

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