

Comparative evaluation method of polymer surface treatments applied on high performance concrete

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An investigation on the protection characteristics of three different polymer surface treatments applied on concrete was carried out by means of a large number of tests. Two of the polymers used, i.e., silane and siloxane, are commercial products, while the third, a fluorinated polymer, is still being studied. These treatments were applied on high performance concretes (0.33 water/cement ratio) with two different curing times. Their protection efficiencies were evaluated by testing water absorption (by immersion and by capillarity) and evaporation, chlorides penetration and artificial ageing; two other tests were carried out to verify concrete surface colour changes caused by applied treatments and by external exposure. To evaluate the overall behaviour of each product, the results obtained were translated into a parametric scale and reported using comparative graphics.

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1. Introduction

Long-term performance of concrete structures is one of the most important problems in the construction field. Surface application of waterproofing polymer coatings is an effective method to improve concrete durability. Indeed much of the deterioration of concrete can only occur in the presence of water since aggressive agents penetrate concrete and react harmfully with the cement paste only when dissolved in water [1]. Deterioration can be caused both by chemical and by physical attack. External chemical attack occurs mainly through the action of aggressive ions, such as chlorides and sulfates, or of carbon dioxide, as well as by many natural or industrial liquids and gases. Physical causes of damage include alternating freezing and thawing of concrete and the associated action of de-icing salts [2].

Deterioration and long-term poor performance led to accelerated research into the microstructure of cements and concretes; as a result improved concretes have been developed. These materials, called High Performance Concrete (HPC), have high strength and low permeability due to a low water/cement (w/c) ratio, superplasticizer admixtures and, in some cases, silica fume or fly ash mineral additions [3]. However, if the HPC reduced permeability is not sufficient to avoid deterioration, then a surface protection should be considered.

Today a lot of polymer surface treatments exist to protect concrete, such as fluorinated, silane and siloxane

polymers, acrylic or epoxy resins, and polyurethane. One way of classifying these treatments is by the manner in which the protection or benefit is achieved. There are three main classes of treatments: (i) *coatings and sealers*, which form a pinhole-free film of finite thickness over the concrete surface that acts as a barrier to the passage of water; in this class there are polyurethane, acrylic and epoxy resins. (ii) *Pore-lining treatments* involve hydrophobic materials that line concrete surface pores and repel moisture; the most important materials in this class are the silicone compounds, such as silane, siloxane and fluorinated polymers. (iii) *Pore-blocking treatments* make use of a family of products which are claimed to penetrate concrete and block pores; the most common examples in this group are liquid silicate and liquid silicofluoride applications [4].

Whatever the type of treatment is, a product should fulfil some fundamental requirements, such as impermeability to liquid water, good permeability towards water vapour and chemical and photochemical stability. In some cases a further requirement is a reduced colour change.

In order to evaluate if a treatment fulfils these requirements, the Italian Committee for Stone Material Normalisation introduced some recommendations (called NORMAL) about test methods on treated stones. In this research NORMAL recommendations were used to test surface treated concrete because international

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standards (ASTM, UNI) concern plain concrete only.

In recent years some researches were carried out on concrete surface treatments. Marusin [5] tested NaCl water solution absorption and desorption on a large number of surface treatments; Pasetti *et al.* [6] tested water penetration, water vapour permeability and abrasion resistance of some fluorinated treatments. Colleparidi and Coppola [7] tested water absorption, chloride penetration, sulphate penetration, carbon dioxide penetration, freezing-thawing cycles and calcium chloride aggression on concrete treated with silane polymer and acrylic paint. Borsje [8] tested water absorption, resistance to heat treatment and evaporation rate on a concrete treated with a hydrophobic paste. Bush [9] tested water absorption and chloride penetration on a concrete treated with silane polymer. Swamy [10] tested chloride penetration and carbonation depth on concrete treated with an acrylic resin.

The aim of this article is to suggest an evaluation methodology for concrete surface treatments selection based on a large number of tests concerning waterproofing, chloride penetration and colour change. Tests were carried out on high performance concretes (0.33 w/c, with and without silica fume) at two different curing times and treated with three different coatings: siloxane, silane and fluorinated polymers. In this way it was possible not only to compare the efficacy of different treatments but also to assess the influence of curing time: usually treatments are applied as soon as concrete structures are built up, but it could happen that an existing building should be treated.

In this paper only test results concerning concrete mixes without silica fume are reported in detail, while those concerning concrete mixes with silica fume are used as a comparison.

2. Experimental

2.1. Materials

Two concrete mixes were manufactured to produce high performance concretes and each one studied at two different curing times (Old corresponds to 16 months of curing, New to 2 months). For both mixes the w/c ratio is 0.33, but one (concrete S) has a 15% wt addition of silica fume and the other (concrete C) has no silica fume addition. Concrete properties are reported in Table I.

Three different superficial treatments were used to protect concrete: siloxane, silane and fluorinated polymers. Siloxane polymer is a transparent liquid based

TABLE I Concrete mix proportions and compressive strength

	CO	CN	SO	SN
Curing time (months)	16	2	16	2
Aggregates (maximum diameter 25 mm) (kg/m ³)	1574	1580	1574	1500
Cement II/A-L 42.5 (kg/m ³)	630	630	630	630
Silica fume (kg/m ³)	—	—	94.5	94.5
Superplasticizer (% by weight of cement)	1.5	1.0	1.5	1.0
Total water (kg/m ³)	208	208	208	208
W/C ratio	0.33	0.33	0.33	0.33
Density (kg/m ³)	2378	2407	2356	2372
28 days compressive strength (N/mm ²)	66.0	65.5	82.5	76.5

on silicon resins in water; solid residual is 5% wt and density is 1020 ± 20 kg/m³. Silane polymer is a pure isobuthyltriethoxysilane with a density of 890 kg/m³. The third polymer is an ionomeric polyurethane based on perfluorinated macromonomer, dispersed in water (solid residual 10% wt) with a density of 1100 kg/m³.

2.2. Sample preparation

Two types of concrete mix specimens were used: cubes of 50 mm side and cubes of 100 mm side. For each concrete, a block of 100 × 100 × 400 mm was cut by a circular diamond blade saw to obtain 24 specimens 50 × 50 × 50 mm and 4 slabs 100 × 100 × 25 mm. Samples 100 × 100 × 100 mm were directly cast in cubic forms.

Each specimen was brushed and then subjected to a compressed air jet to remove superficial impurities. After that, specimens 50 × 50 × 50 mm were dried in an oven at a temperature of 100 ± 5°C until constant mass was reached: cubes were weighed at intervals of 24 hours until the difference between values obtained from two successive values of mass was less of 0.1% of initial sample mass. Specimens 100 × 100 × 100 mm were dried in an oven at a temperature of 100 ± 5°C for 7 days.

The application of polymer treatments was carried out according to the advice of the suppliers. Siloxane polymer was applied by paint-brush; silane polymer was applied with three saturating flows, pouring it directly on the concrete surface. Fluorinated polymer was applied in two coats by paint-brush. After application, all the specimens were dried at room temperature: 50 × 50 × 50 mm specimens for 72 h, 100 × 100 × 100 mm specimens for 7 days. Testing procedure for 50 × 50 × 50 mm specimens is reported in Fig. 1.

2.3. Water absorption

Water absorption was carried out according to Normal 7/81 protocol [11] using 50 × 50 × 50 mm specimens. Three specimens of each concrete were used for every

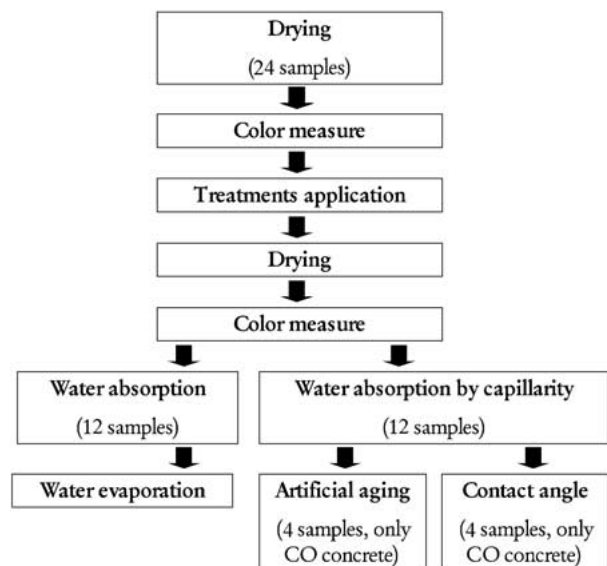


Figure 1 Schematic diagram of tests carried out on CO and CN samples (50 × 50 × 50 mm).

treatment and three untreated specimens were retained. After treatment application and the 72-hour-desiccation at room temperature, all the specimens were placed in an oven at $60 \pm 5^\circ\text{C}$ to reach constant weight. Then, after cooling in a dessiccator, the mass was determined. Subsequently, the specimens were immersed in deionized water at 20°C , on a plastic grille support to guarantee the largest surface exposure. The water level was approximately 20 mm over the top surface of the specimens. Samples were weighed after 1, 2, 4, 8, 24 hours and then every 24 hours of immersion. Before weighing, the adhering water was removed with a damp cloth. The water absorption was calculated from the difference between two consecutive mass determinations, as a percentage of the initial mass. The test was stopped when the difference between two consecutive mass determinations was less of 0.1% of the initial mass.

2.4. Water evaporation

Tests were carried out, according to Normal 29/88 protocol [12], on saturated specimens previously subjected to the water absorption test. The adhering water was removed with a damp cloth and specimens weighed, then they were placed, supported by a metallic grille, in desiccators containing silica gel with blue indicator. The test was carried out at $20 \pm 1^\circ\text{C}$. Mass was determined after 30 minutes, 1, 2, 4, 8, 24 hours and then every 24 hours. The water amount contained inside the specimens at time t_i was calculated with the following formula:

$$Q_i = \frac{w_i - w_0}{w_0} \cdot 100$$

where Q_i is water content expressed as a percentage of the dry mass (%); w_i is mass of the specimen at the time t_i (g); w_0 is mass of the dry specimen (g).

The test was stopped upon reaching the following condition:

$$0.90 < \frac{w_0 - w_{i-1}}{w_0 - w_i} < 1.00$$

where w_0 is mass of the specimen at the beginning of the test (g); w_{i-1} is mass of the specimen at the time t_{i-1} (g); w_i is mass of the specimen at the time t_i (g); $t_i - t_{i-1}$ is 30 min, 1 h, 2 h, 4 h, 16 h the first day, then 24 h.

2.5. Water absorption by capillarity

Water absorption was carried out according to Normal 11/85 protocol [13] using $50 \times 50 \times 50$ mm specimens. Three specimens for each treatment and three untreated specimens of each concrete were used for a total of twelve samples tested. After treatment application and 72 hours-desiccation at room temperature, all the specimens were placed in an oven at $60 \pm 5^\circ\text{C}$ to reach constant weight. Then, after desiccator-cooling, the mass was determined. After weighing, each specimen was laid on a filter paper pad about 10 mm thick, partially immersed in deionized water with the formwork side in contact with the pad. The amount of water absorbed by capillarity was determined by weighing the specimens

after 5, 10, 15, 30, 45 minutes and 1, 2, 4, 8, 24 hours and then every 24 hours. The amount of absorbed water M_i at the time t_i per surface unit was calculated as follows:

$$M_i = \frac{w_i - w_0}{S}$$

where w_i is mass of the specimen at the time t_i (g); w_0 is mass of the dry specimen (g); S is contact surface area (m^2).

The test must be stopped when:

$$\Delta M = \frac{M_i - M_{i-1}}{M_i} \cdot 100 \leq 1\%$$

where M_i and M_{i-1} is the amount of absorbed water at the time t_i and t_{i-1} per surface unit.

If after 11 days this condition is not yet fulfilled, the test must be stopped anyway to avoid mould formation on the filter paper. In our case, all tests were stopped after 11 days.

2.6. Artificial ageing

This test was carried out on samples $50 \times 50 \times 50$ mm, previously subjected to the water absorption by capillarity test, using a specimen for each treatment and an untreated specimen, for CO concrete only, for a total of four samples tested. Samples were placed for 300 hours in a Erichsen Solarbox 3000e apparatus for the artificial ageing. Internal conditions were: constant temperature of 65°C , Xenon lamp radiation with UV filter, cycles of 18 minutes of water-saturated atmosphere and 101 minutes of dry atmosphere. Then, samples were dried in an oven at $60 \pm 5^\circ\text{C}$ to reach constant mass and then subjected again to the absorption by capillarity test.

2.7. Chlorides penetration

This test was carried out on $100 \times 100 \times 100$ mm samples of CN concrete only. A specimen for each treatment and an untreated specimen were immersed in a 15% NaCl water solution for 21 days, according to NCHRP Report n. 244 [14]. After 21 days of soaking, the cubes were exposed to room conditions for 14 days to allow them to dry. To determine the chloride ion distribution, holes were drilled through the centre of each cube face, on two sides of the cube, to obtain powder samples at different depth intervals. These intervals were 0 to 12 mm, 12 to 24 mm, 24 to 36 mm. The total chloride ion content was determined by chemical analysis (Volhard method).

2.8. Colorimetric analyses

The evaluation of colour change on polymer-coated specimens was carried out by means of a Minolta Chroma Meter 300 colorimeter; the determinations were carried out according to the Normal 43/93 protocol [15]. The colour changes were evaluated by the L^* , a^* , b^* system (ASTM D-1925, CIE 1976).

Twenty-five colour determinations on different spots were carried out, before and after the treatment, on a formwork side of each specimen. The average of the twenty-five spots was calculated to evaluate the colour change.

2.9. Colour variation due to external exposition

The test was carried out on $100 \times 100 \times 100$ mm samples of CN and SN concrete, using a specimen for each treatment and an untreated specimen. After preparation and colorimetric analysis, specimens were exposed to the external climate of Milan for about 3 weeks and then subjected to a new colorimetric analysis.

3. Results and discussion

3.1. Water absorption and evaporation

Results are reported considering that, for the water absorption test, a treatment has a good behaviour if treated concrete absorbs little water, while, for the evaporation test, a treated concrete has a good behaviour if a lot of water is lost.

The CO untreated specimens absorbed water very quickly (Fig. 2a) reaching a value of 4.17% wt after only 8 hours and a final absorption value of 4.51%. During the evaporation period these specimens produced 1.67% wt of water. CN untreated specimens reached (Fig. 2b) a value of 5.62% after 8 hours of immersion and 5.94% at the end of the absorption and produced 3.09% of water during the evaporation test. Therefore the test was able to point out a difference of water ab-

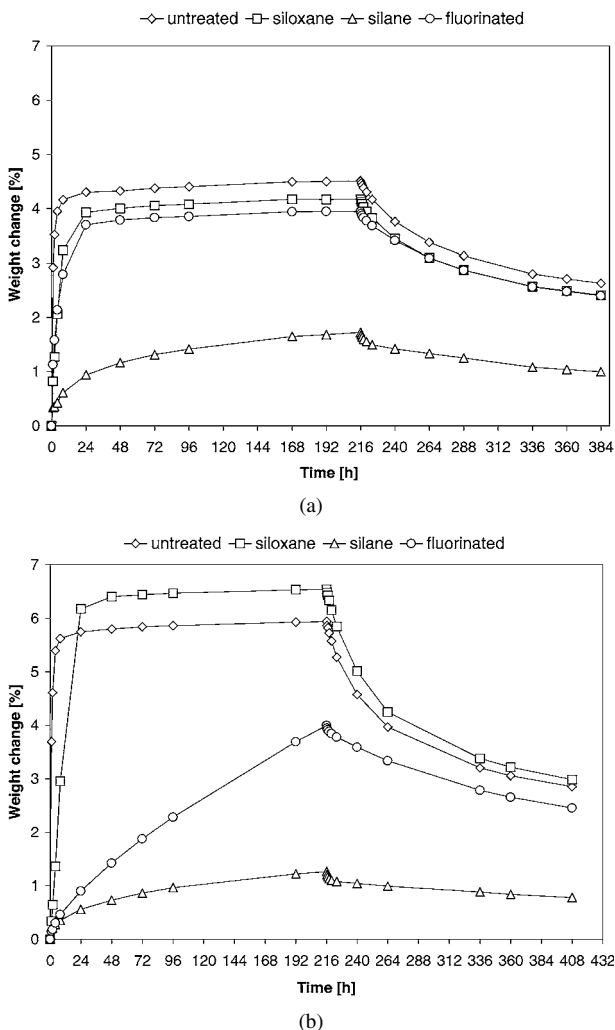


Figure 2 Water absorption and water evaporation tests on (a) CO and (b) CN concrete specimens: Each curve results from the average on three samples.

TABLE II Concrete porosity properties by mercury intrusion porosimetry (MIP)

	CO concrete	CN concrete
Total pore cumulative volume (mm^3/g)	100.35	183.18
Specific surface area (m^2/g)	1.52	2.07
Total sample porosity (%)	14.24	25.46

TABLE III Results relative to young untreated concrete: Comparison between C and S

Test	Parameter	Concrete	
		CN	SN
Water absorption	Absorbed water after 1 hour of immersion (% wt)	3.69	3.32
	Absorbed water after 216 hours of immersion (% wt)	5.94	6.48
Water evaporation	Issued water (% wt)	3.09	1.98
Water absorption by capillarity	Absorbed water after 1 hour (kg/m^2)	0.78	0.49
	Absorbed water after 264 hour (kg/m^2)	7.71	5.03
External exposure	ΔE : Horizontal surface	11.54	0.62
	ΔE : Vertical surface	9.96	0.54

sorption and evaporation between two concretes with the same mix but a different curing time: in particular, the more mature concrete absorbed and evaporated less water than the newer one. This result agrees with mercury intrusion porosimetry analysis: CO concrete results, actually, (Table II) less porous than CN concrete.

Specimens treated with siloxane polymer have a starting water absorption lower than untreated concrete in both cases (CO and CN concrete), but after 24 hours they have already a behaviour similar to untreated concrete. Siloxane treatment does not seem to obstruct water evaporation: indeed, results of the evaporation test are similar to untreated concrete.

Silane-treated specimens have absorbed water values much lower than the untreated ones in both CO and CN concrete.

Specimens treated with fluorinated polymer have a different behaviour depending on the concrete type: with CO they have a starting water absorption lower than untreated concrete but beginning from 24 hours of immersion they have a behaviour similar to untreated concrete; with CN concrete they have a starting water absorption and final values of absorbed water much lower than untreated concrete. Both with CO and CN concrete, these specimens have a water evaporation behaviour worse than untreated concrete.

This test was also carried out on concrete SO and SN, but only results relative to untreated SN are reported (Table III). In the first hour of water absorption, C and S concrete have a similar behaviour, but at the end of immersion samples S have absorbed more water. During the evaporation test S concrete produced less water than C concrete. The same remarks hold true for SN and SO treated samples.

3.2. Water absorption by capillarity

The CO untreated specimens absorbed less water than CN untreated specimens (Fig. 3a and b): indeed the

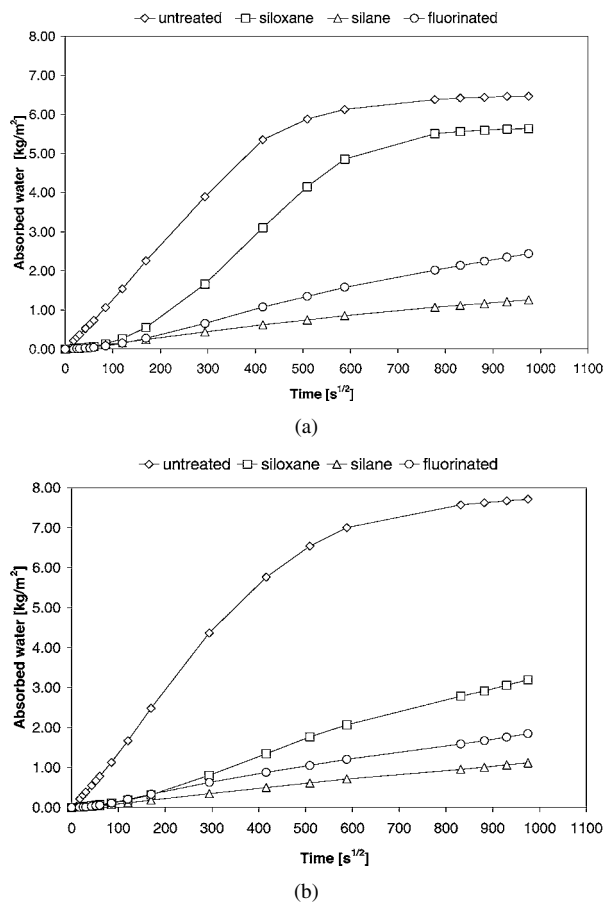


Figure 3 Water absorption by capillarity test on (a) CO and (b) CN concrete specimens: each curve results from the average on three samples.

final amounts of absorbed water were 6.46 kg/m² and 7.71 kg/m², respectively. In this case the test results are also sensitive to a difference of curing.

Concrete treated with siloxane polymer obtained starting values of absorbed water much lower than untreated concrete both with CO and CN samples. Final values are similar to untreated in the case of CO, while they are lower than untreated in the case of CN.

Both concrete samples (CO and CN) treated with silane polymer have absorbed water values much lower than the corresponding untreated samples with an almost linear uptake with (time)^{1/2}.

Specimens treated with fluorinated coating have absorbed water values much lower than untreated ones for both curing times, though CN concrete has a slightly better behaviour.

It appears from Table III, that SN untreated concrete has a better behaviour than CN concrete throughout the test. The same remarks apply when comparing SO and CO untreated concrete. Treated specimens of both SN and SO concrete show similar results to the corresponding C-specimens, irrespective of superficial treatment.

3.3. Artificial ageing

To compare specimens behaviour before and after artificial ageing, independently from the amount of water absorbed by untreated specimens, the capillarity curves are reported as “% reduction of absorbed water” called “effective protection.” As an example, an effective protection of 100% corresponds to a total protection

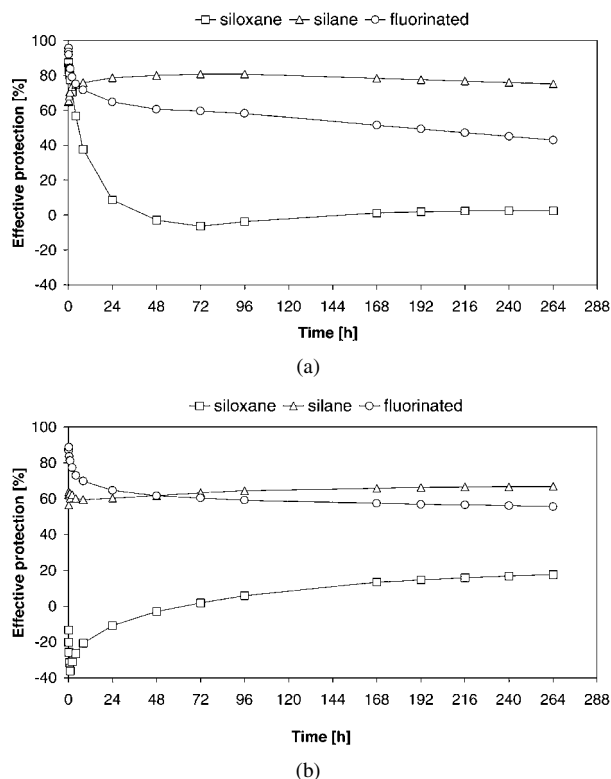


Figure 4 Water absorption by capillarity test on CO concrete specimens (a) before and (b) after artificial ageing.

against water (i.e., no absorbed water), while an effective protection of 0% indicates a behaviour identical to untreated concrete.

Concrete treated with siloxane (Fig. 4a and b), after artificial ageing, has a very bad behaviour in the first 24 hours, while it slightly improves at the end of the test.

Specimens treated with silane coating are slightly worse after artificial ageing than before.

After artificial ageing, concrete treated with fluorinated polymer has a poor behaviour at the beginning, while it improves slightly at the end of the test.

3.4. Chlorides penetration

After 21 days of immersion under a 15% wt NaCl aqueous solution, untreated CN specimen showed the amount of chloride ion penetrated in the first depth (12 mm) of 0.49% wt, from 12 to 24 mm of 0.19% and from 24 to 36 mm of 0.03%.

For treated samples it was decided to analyse only the first depth: specimens treated with siloxane showed 0.05% of penetrated ions, those treated with silane 0.00% and those treated with a fluorinated coating 0.37%. Then siloxane and silane seem to offer a very good protection against chlorides penetration, while the fluorinated coating seems to assure a very poor protection.

3.5. Colorimetric analyses

Results reported in Table IV show colour changes caused by applied treatments: parameter L^* represents the surface lightness and its values vary from 0 (corresponding to black) to 100 (corresponding to white);

TABLE IV Specimen surface colour variation after treatment with coating polymers: Average on 6 specimens for each treatment, 25 spots for each specimen

Treatment	CO concrete				CN concrete			
	ΔL^*	Δa^*	Δb^*	ΔE	ΔL^*	Δa^*	Δb^*	ΔE
Siloxane	-1.27	0.16	0.83	1.54	-0.69	-0.01	0.17	0.72
Silane	-1.75	0.21	0.15	1.78	-0.91	-0.01	-0.23	0.94
Fluorinated	-2.59	0.28	1.32	2.92	-2.58	0.07	0.89	2.73

TABLE V CN concrete specimens surface colour variation after 3 weeks of external exposure: Average on 25 spots for each specimen

Treatment	Horizontal surface				Vertical surface			
	ΔL^*	Δa^*	Δb^*	ΔE	ΔL^*	Δa^*	Δb^*	ΔE
Untreated	-11.50	0.22	-0.93	11.54	-9.81	0.39	1.65	9.96
Siloxane	-0.78	0.04	-0.62	1.00	0.05	0.05	-0.38	0.39
Silane	9.73	-0.36	-0.96	9.78	1.10	0.01	-0.26	1.13
Fluorinated	-3.15	0.39	0.81	3.27	-1.26	0.23	0.61	1.43

parameter a^* indicates a coordinate of chromaticity from green (-60) to red (+60) and parameter b^* a coordinate from blue (-60) to yellow (+60). Parameter ΔE represents a "total colour variation" and it is calculated as follows:

$$\Delta E = \sqrt{\Delta a^{*2} + \Delta b^{*2} + \Delta L^{*2}}$$

Siloxane applied on CO concrete produces a darkening of the specimens since ΔL^* is negative. Chromatic variations Δa^* and Δb^* are smaller than the lightness variation and ΔE , being 1.54, is not too high. When siloxane is applied on CN concrete darkening is smaller and ΔE is about one half of the value observed in CO concrete.

Samples treated with silane show a surface darkening, too; moreover, values for CO are larger than those for CN. Compared to siloxane, total colour variations are slightly larger.

The most important darkening is produced by fluorinated treatment: its ΔL^* values are about -2.60 independent of concrete type. In this case it is possible to observe a slight displacement towards yellow in the CO sample. Colour changes were visible with the naked eye: ΔE values are about 3.

3.6. Colour variation due to external exposition

Untreated specimens, after external exposure, show (Table V) a remarkable darkening, surely due to the humidity absorbed during the test.

Colour variations produced by external exposure on specimens treated with siloxane were negligible, while they were remarkable on specimens treated with silane; in particular, ΔL^* values were positive, i.e., there was a lightening up of the specimens. Samples with fluorinated polymer showed a limited surface darkening.

Comparing CN and SN untreated concrete (Table III), results show that SN has a total colour change remarkably lower than CN. The treated specimens behaviour depends on the coating: siloxane treated SN samples have a colour change greater than that of the CN samples; on the contrary, silane and

fluorinated treated SN samples have a better behaviour than CN samples.

4. Tests evaluation analysis

To evaluate the overall behaviour of each product from the obtained results, it seemed appropriate to translate each outcome onto a parametric scale and to report them in a radar graphic. Considered outcomes were relative to both CO and CN concretes: when tests were carried out on both concretes, the average score was used. Results in a parametric form were calculated assigning score 0 to the worst result among untreated and treated specimens, and score 5 to the best. For example, in the chloride penetration test, chloride penetration into untreated concrete specimens was 0.49% wt, inside fluorinated treated specimens 0.37%, inside siloxane treated specimens 0.05% and into silane treated specimens 0.00%. So, untreated concrete score was 0, fluorinated 1.2, siloxane 4.5 and silane 5. Graphics obtained in this way are reported in Fig. 5.

From the diagram relating to the siloxane it can be observed that this treatment has a good behaviour in the first hour of water absorption and water absorption by capillarity tests, but it grows worse at the end of these tests. On the other hand, it is excellent in the water evaporation test and very good against chloride penetration. After artificial ageing, its performances are not so good. In colour tests, siloxane obtained the lowest colour changes.

Silane was demonstrated to have an excellent behaviour in water absorption, water absorption by capillarity, chloride penetration and artificial ageing tests. In the water evaporation test it was not so good because it held down more water than untreated samples. Silane application on untreated concrete produce a very low colour change (indeed it has a very good score in this test), but colour change after external exposure is remarkably greater.

Fluorinated treated concrete obtained a fair result in the water absorption test, poor in chloride penetration and in water evaporation. On the other hand, it was very good in both water absorption by capillarity and artificial ageing tests. Fluorinated application on concrete

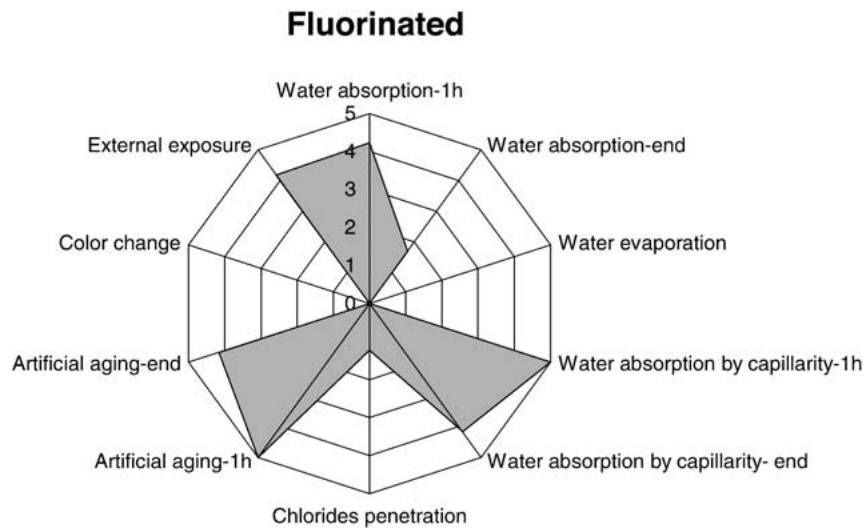
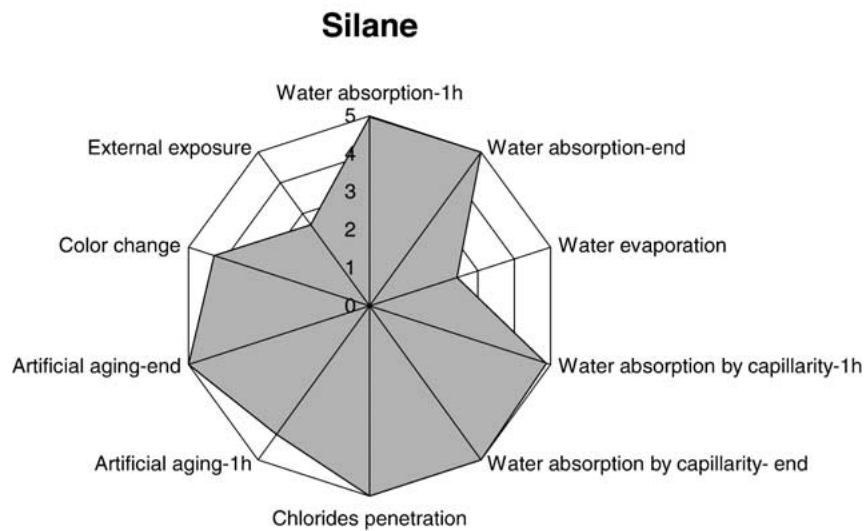
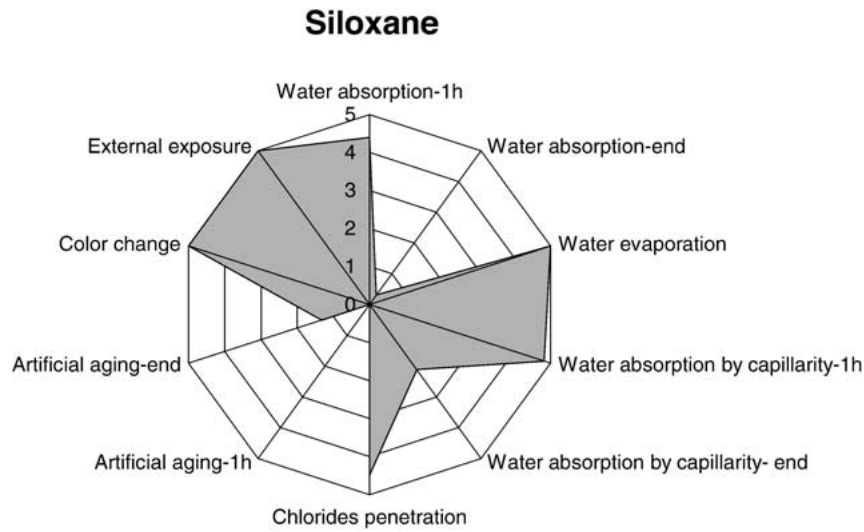


Figure 5 Treatments evaluation graphics obtained by expressing in a parametric form the results of carried out tests.

causes surface darkening, but in the external exposure test it obtained a good score.

5. Closing remarks

When managing with so many experimental data, the test analysis with radar diagrams here introduced seems an appropriate choice and probably the only effective

one; indeed, one obtains an overall evaluation about a specific treatment and, at the same time, is enabled to select the best product, among many others, with respect to technical requirements.

Moreover, two technical considerations of fundamental relevance emerged during this work. First, the influence of concrete curing time upon superficial

treatment effectiveness is strong: in most cases the results obtained on young concretes are undoubtedly better than those obtained on the old ones. Then, whenever possible, it seems advisable to apply treatments on young concrete. Second, concerning concrete with or without silica fume addition, although few experimental data are discussed in this paper, it seems that this component does not affect results obtained by whatever treatment here considered.

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