



## Ethyl silicate for surface treatment of concrete – Part II: Characteristics and performance

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### ABSTRACT

In this study, several aspects of the surface treatment of concrete with ethyl silicate were investigated. After the treatment, two different types of concrete ( $w/c$  0.45 and 0.65) showed a dramatic decrease in capillary suction, chloride diffusion coefficient and carbonation depth. A penetration depth into the concrete of about 3–5 mm was assessed. Microstructural investigations showed evidence of a chemical interaction of the amorphous silica gel within the concrete forming calcium silica gel and a selective distribution of silica gel, which was mainly deposited in the small pores. In addition, the brightness and colour changes of the concrete surfaces after the treatment were analyzed quantitatively, finding limited differences, further decreasing over time. This last aspect is relevant for architectural applications of concrete, for example façades of residential buildings, where the architects may be interested in using the texture of concrete but are concerned by possible colour changes related to surface treatments application.

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## 1. Introduction

### 1.1. Surface treatment of concrete

In reinforced concrete, the penetration of aggressive agents through the cover zone is critical for the reinforcement corrosion and hence for the durability of the concrete structure.

In old structures, the problem of durability used to be totally underestimated during construction, due to the scarce awareness of the degradation mechanisms and processes affecting reinforced concrete. This often led to unsuitably high water to cement ratio and small cover thickness, presently causing urgent repair works in such old concrete structures. Even in the cases when reinforcement has not started to corrode yet and the cover is still intact, the rebars may be threatened by incipient corrosion due to deep carbonation [1,2].

In new structures, a common strategy to delay carbonation and ingress of chlorides through the cover zone is decreasing the total

porosity of the concrete, for example by decreasing its water to cement ratio. However, this strategy exhibits two main drawbacks: firstly, the protection could be insufficient in highly aggressive service environments (e.g. marine, industrial, etc.) and, secondly, the concrete is typically overdesigned everywhere in the structure, except for the cover zone. The required additional consumption of cement and admixtures could be avoided by selectively improving the quality of the cover zone, e.g. by an appropriate surface treatment. Of course, surface treatments should not be viewed as a basis for reducing cover or for inadequate mix design [1], but as a means for selectively enhancing the concrete properties near the interface with outdoor aggressive environments [3].

Hence, in order to increase the durability of new and existing concrete structures, several kinds of surface treatments have been proposed, mainly classified according to EN 1504-2:2004 [4] as *hydrophobic impregnation* (producing a water-repellent surface, with no pore filling effect), *impregnation* (reducing the surface porosity, with partial or total pore filling effect) and *coatings* (producing a continuous protective layer on the surface of concrete). Hydrophobic impregnation [5–7] is usually performed through silanes or siloxanes application and is aimed to prevent the water ingress into concrete and, hence, to slow down the decay processes, due to the fact that both chemical and physical–mechanical attack is very slow, or even negligible, in dry conditions. Impregnation and coatings are usually performed through organic polymers, aimed at creating a physical barrier against the ingress of aggressive

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agents responsible for the degradation of concrete and/or reinforcement, i.e. chloride and other soluble salts, CO<sub>2</sub>, chemical agents and water itself. In both cases, the use of organic products gives rise to:

- durability concerns, as they quickly lose their initial properties in outdoor environments, due to their sensitivity to UV, oxygen, temperature, humidity, pollutants, etc. For example, reduced efficiency of hydrophobic properties of silanes–siloxanes–polysiloxanes after about 5 years has been reported [8], hence the surface treatment should be repeated quite often;
- compatibility concerns, especially when continuous coatings or pore-blocking sealers are used. In such cases, the transport of water vapour is almost reduced to zero and water trapped behind the protective surface layer may cause its detachment.

In order to increase the durability of surface treatments, the use of inorganic products has been proposed as well. These products are mainly aqueous solutions of sodium silicate, also known as “waterglass”, and, to a much lower extent, potassium silicates and fluosilicates [9]. These silicates, usually considered as pore-blockers, are supposed to react with portlandite to form C–S–H gel and NaOH [9] and to increase the performance of the impregnated layer, in terms of hardness and impermeability. However, the experimental data on the performance of sodium silicates for concrete impregnation are limited [5] and their mechanism in improving the concrete performance is still unclear [9]; moreover the possible increase in the risk of alkali–silica reaction due to the formation of NaOH as a by-product from the reaction with portlandite should be investigated. While some authors [5] report that sodium silicate minimally penetrates into concrete and is quite ineffective in preventing water absorption and chloride penetration, others report that its performance can be significantly improved by post-treatment with cationic surfactants (alkyl quaternary ammonium salts) [10].

In the present paper, the use of ethyl silicate, widely used for the consolidation of weathered stones in architectural restoration [11], for the surface treatment of concrete is investigated.

### 1.2. Ethyl silicate as a consolidant for stone

Ethyl silicate (ethyl ester of silicic acid – Si(OC<sub>2</sub>H<sub>5</sub>)<sub>4</sub> – often referred to as TEOS – tetraethylorthosilicate) is an alkoxy silane compound which is usually applied onto the stone surface by brushing or spraying, in solution with low viscosity organic solvents. Once penetrated into the pores, it undergoes a two-stage curing process: firstly hydrolysis, giving silanol and ethanol, and secondly dehydration/condensation of silanol, leading to the precipitation of amorphous silica gel inside the stone pores [12]. In silicate-rich stones, e.g. sandstones, silanol binds to the hydroxyl groups present in the silicate phases, thus leading to an appreciable increase in cohesion and mechanical strength [13]. On the contrary, in carbonate stones, e.g. marble, the ethyl silicate hardening simply results in a pore-filling effect, with limited re-adhesion and consolidation effects [14].

The reasons for the wide use of ethyl silicate for stone consolidation are mainly its small monomer size and low viscosity, leading to deep penetration into the stone, and its hardening by-products (ethanol and water), which are volatile and do not damage the stone. Moreover, the final reaction product of TEOS is silica gel, which exhibits good compatibility with stone and good durability, unlike many polymeric consolidants [8]. The incomplete reduction of open porosity (which allows transport of water vapour) of stone and the absence of an abrupt interruption between the impregnated and the untreated zones [13] are further advantages of this consolidant.

### 1.3. Applications of TEOS to concrete

The application of TEOS to concrete is expected to take advantage of the same features largely exploited for stone consolidation, i.e. good penetration depth, good chemical–physical–mechanical compatibility, high durability of the final product (silica) and absence of the pore-blocking effect. This last aspect is very important for a satisfactory durability of the surface treatment, because the possible presence of water trapped behind the consolidated layer (e.g. from infiltration) might lead to its detachment, especially in case of freeze–thaw cycles. Moreover, the concrete impregnation with TEOS is expected to take advantage of the pozzolanic behaviour of ethyl silicate, which is based on the production of amorphous silica during the TEOS sol–gel process and which was assessed in the first part of the paper [15]; the formation of C–S–H is expected to improve the compatibility of the treatment with concrete and its final performance.

## 2. Materials

### 2.1. Concrete

Two concrete mixtures based on ordinary Portland cement (CEM I 42.5N), with water to cement ratio (w/c) 0.45 and 0.65 respectively, were prepared (Table 1) and concrete cubes of size 150 × 150 × 150 mm<sup>3</sup> were manufactured. The samples were cured for 4 months at  $T = 20 \pm 1$  °C and 90 ± 5% RH.

### 2.2. Ethyl silicate

The characteristics of the ethyl silicate (Estel 1000, CTS, Italy) used for surface treatment of concrete are reported in Table 2. The solvent, white spirit, is a mixture of saturated aliphatic and alicyclic hydrocarbons denatured with dichloropropane.

### 2.3. Samples

After the 4 months curing, three 150 × 150 × 50 mm<sup>3</sup> prisms were obtained from each cube by cutting: one external prism was treated with TEOS on one face according to the procedure described in Section 2.4, the other prism was left untreated for comparison and the inner prism (certainly not carbonated) was cut into 20 × 20 × 20 mm<sup>3</sup> cubic samples for microstructure characterization.

### 2.4. Surface treatment and curing

In order to ensure constant moisture content throughout the sample and a good penetration of ethyl silicate into concrete, a pre-conditioning of the samples was carried out. As a matter of fact, materials in equilibrium with an environmental relative humidity and temperature around 40% and 20 °C, respectively, are recommended for a better outcome of the treatment [16]. Thus, the following procedure was adopted: the samples were dried at 50 °C for 48 h and then stored in a climatic chamber at  $T = 20 \pm 1$  °C

**Table 1**  
Mix design and properties of concrete.

	w/c 0.45	w/c 0.65
w/c	0.45	0.65
Cement content (kg/m <sup>3</sup> )	330	275
Maximum aggregate size (mm)	32	32
Bulk density (kg/m <sup>3</sup> )	2430	2362
Air content (vol.%)	2.0	2.5
Compressive strength at 28 d (MPa)	52.5	31.0

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