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Carbonation Proofing Mechanism of Silicate-Based Surface Impregnations

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Abstract

Silicate-based surface impregnations have received much attention in the protection of concrete against carbonation. However, the understanding of their behavior is still not widely understood. In the present study, protective performance and mechanism of the silicate-based surface impregnations against carbonation were experimentally investigated through the carbon dioxide absorption and the scanning electron microscopy (SEM) observations. Results obtained lead to an exploration hypothesis that the silicate-based surface impregnation products were effective in enhancing the resistance to carbonation due to the synergy effect of pores-blocking effect and the carbon dioxide absorption through the chemical reaction.

1. Introduction

The durability of reinforced concrete structures has received much attention over the last several decades since its deterioration has become a critical issue in most industrial countries. Most of them can be functional well even after a long service in the normal environment. However, for reinforced concrete structure exposed to the aggressive environment, its durability may be affected by many aggressive agents, resulting in the various degradation processes due to deterioration of the concrete itself or due to corrosion of the steel reinforcement inside the concrete. Among them, carbonation is considered as one of the primary causes of the corrosion of steel reinforcement rebar, consequently leading to deterioration of reinforced concrete structures. Carbonation is a natural chemical process that occurs when the carbon dioxide penetrates from the atmosphere into the concrete pores, transforms into carbonic acid in the presence of water then reacts with the calcium hydroxide, forming calcium carbonate (Richardson 1988). The outcome of carbonation process is the pH drops of carbonated concrete from typical values in a range of 12.5 - 13.5 to about 8.3, resulting in the destruction of the passive film which covers the reinforcing steel and prevents them against corrosion (Saetta et al. 1993).

In recent years, impregnations are known to be an effective method for preventing the penetration of aggressive substances in both new and existing concrete structures (Dai et al. 2010). Among these, silicate-based (also known as "waterglass", e.g., sodium silicate, potassium silicate) is the most commonly-used impregnation treatments (Thompson et al. 1997; Baltazar et al. 2014). Despite the fact that the performance of silicate-based surface impregnations has often been studied, the understanding of their behavior, particularly the precise mechanisms which prevent/delay the ingress of aggressive agents (such as water, carbon dioxide) remains unclear.

Many researchers have always seen silicate-based surface impregnations as sealers that penetrate into the concrete, reacts with Portlandite to form C-S-H gel which can fill entirely or partially the concrete pores, improve its surface properties, therefore, protects concrete against the ingress of some deterioration factors (Thompson et al. 1997; Baltazar et al. 2014). One of the first studies that reported the mechanism of silicate sealers was the one by McGettigan et al. (1992). The authors state that silicates can fill the pore structures of concrete by precipitation of SiO₂. The second theory is that the silicates react with calcium hydroxide in concrete to form insoluble calcium silicates (Higgins 1985). The third argument is that the expansive gel formed by silicates, which is similar to the production of alkali-silica reactions swells in the pores (Thompson et al. 1997). Besides, a study conducted by Thompson et al. (1997) suggests that silicates perform as sealers through a combination of mechanisms. Apparently, the above literature review presents that the number of studies was carried out to investigate the mechanisms of silicate-based surface impregnation in enhancing concrete durability is still rather limited. Although the precise mechanisms of this type of impregnation in protecting concrete against the penetration of water were explored (Song et al. 2016), other aggressive agents, such as carbon dioxide, sulfate attack, etc. are not yet addressed, according to the best of authors' knowledge. Therefore,
to maximize the efficacy of these products for the protection of concrete, the knowledge about their mechanisms towards each agent should be individually investigated.

The present paper is a part of a study being carried out focusing on the precise mechanisms of silicate-based surface impregnations in the protection of concrete against carbonation, with the goal of providing the understanding about this type of products in improving the concrete durability. Firstly, the accelerated carbonation test was conducted on concrete treated with several types of inorganic silicate-based surface impregnations that have been used more frequently in Japan, and then the effect of these products was evaluated. Subsequently, as the first step of investigating the carbonation proofing mechanism, the authors decided to examine the characterization of silicate-based surface impregnations separately with concrete, which consisted of product’s carbonation absorption capacity and the crystallization of them under carbon dioxide environment. Based on the experimental results, the new hypothesis of a precise carbonation proofing mechanism of silicate-based surface impregnations is described.

2. Materials

2.1 Silicate-based surface impregnations

For the protection of concrete against carbonation process, six types of inorganic silicate-based surface impregnations were studied: an aqueous sodium silicate solution, SS; an aqueous potassium silicate solution, PS; an aqueous lithium silicate solution, LS; a mixture of sodium silicate and lithium silicate, SS-LS; a mixture of potassium silicate and lithium silicate, PS-LS; a product purchased from the market, which containing sodium, potassium and lithium silicate, SS-PS-LS. These silicate-based surface impregnations were selected to represent two different types of the silicate-based surface impregnations: one is solidifying type which forms insoluble crystal under dry condition to fill pores in concrete; the other is reactive type whose deposition can dissolve in water again and then reacts with the calcium hydroxide in concrete (JSCE 2017; Pan et al. 2010; Baltazar et al. 2014; Franzoni et al. 2013), and it has now been suggested that SS, PS, LS penetrate only a few mm in concrete (JSCE 2017; Pan et al. 2017a, 2017b; EN1504-1 2004). Another reason is SS-PS-LS was purchased from the market while the other impregnations were developed in our laboratory as mentioned above. However, SS-PS-LS might not represent the result of lithium silicate as a solidifying type or sodium silicate and potassium silicate as reactive types but the mixing result of these two types.

2.2 Preparation of specimens

To simulate the low-quality concrete, a base mortar with water: ordinary portland cement: fine aggregate = 0.6: 1: 3 was prepared. The high w/c ratio (0.6) was chosen in order to promote the carbonation process quickly. Besides, general mountain sand is used for fine aggregate. The mortar prisms of size 100 x 100 x 400 mm³ were manufactured and left in the laboratory for 24 hours, then demolded and immersed in water for 6 days. Afterwards, they were cut into cubes of size 100 x 100 x 100 mm³ by a cutter, then kept in a climatic chamber at 20 ± 2°C, RH 60 ± 5% for 28 days.

2.3 Application of impregnations and curing after treatment

The silicate-based surface impregnations were applied on one mold-contact surface of six surfaces of each
mortar cubes by a brush with the quantity recommended by the manufacturer in the instruction for SS-PS-LS (250 g/m²), whereas the remaining five surfaces were coated with an epoxy resin. For accurate evaluation of their effects, the same application procedure and the amount were applied for the other impregnations.

Once the impregnations have been applied, the specimens were cured in a room condition (20 ± 2°C, RH 60 ± 5%) for one month before carrying out the tests. The JSCE Guidelines for application of silicate-based surface impregnations (JSCE 2017, 2009) says that water curing is necessary for reactive type impregnations after being applied to the concrete surface while solidifying type should be only cured in the dry condition. Therefore, a question arises here; what kind of curing should be given to the specimens for promoting efficiency of both types of impregnations? In the present study, we adopted the air curing method which was carried out in the room condition with the appropriate moisture content. Even in this condition, enough amount of water would be available for the impregnations and unhydrated cement in concrete, as the new concrete has excess water in it. The same curing condition for all the products was chosen for better comparing their effects.

3. Performance of silicate-based surface impregnations

3.1 Carbonation acceleration test

In order to investigate the effectiveness of the treatments in increasing the carbonation resistance of concrete, the accelerated carbonation test was carried out according to JSCE-K 572 (JSCE 2017), under an environment with CO₂ concentration of 5%, a temperature of 20°C, and a humidity of 60%. After 28 days, all the specimens were split and the carbonation depth was measured by immediately spraying phenolphthalein solution onto the fracture surfaces. The carbonation depth was evaluated by the average of six data points from two fracture surface of each specimen. The reported value was calculated by the average of three specimens. Besides, the improvement percentage of carbonation resistance by applying the silicate-based surface impregnation (f(%) was also calculated as follows.

\[ f(\%) = \left(1 - \frac{\text{Carbonation depth of treated specimen}}{\text{Carbonation depth of untreated specimen}}\right) \times 100 \] (1)

3.2 Product’s penetration depth

The authors decided to employ the method described in JSCE Guidelines (JSCE 2017) that uses Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) to measure the penetration depth through a chemical analysis on the specimen, as the silicate-based surface impregnation has been reported that has no hydrophobic property itself (Dai et al. 2010). The test procedure was conducted as follows (see Fig. 1). First, the powder samples used for the test are taken by using a micro drill (diameter \( \varphi \): 0.38 mm) drilled into the concrete surface for every 2 mm-deep. The sampling position was set to 6 places or more, and its depth was measured using a caliper deviating bar. Next, mixed 0.300 ± 0.001 g of powder sample in 30.000 ± 0.002 g of water at 20 ± 2°C in a flat-bottom flask, stirred the solution by a stirrer for 10 minutes then kept it in a room at 20 ± 2°C for 24 hours. Finally, the solution was filtered, and the resultant aqueous solution is subsequently introduced into ICP-AES for detecting the concentration of lithium ion, sodium ion and potassium ion contained. Then the alkali metal ion concentration was calculated by the following equation.

\[ R = \frac{A}{a} + \frac{B}{b} + \frac{C}{c} \] (2)

where \( R \) is alkali metal ion concentration (mmol/L), A, B...
and C are the amount of lithium ion, sodium ion, and potassium ion measured respectively, a, b, and c are relative atomic mass of lithium, sodium and potassium.

The depth at which the alkali metal ion concentration in treated surface is increased more than 30% compared with the alkali metal ion concentration contained in the untreated surface is determined as the penetration depth.

### 3.3. Results

#### (1) Carbonation depth

The effect of silicate-based surface impregnation products performed on the mortar samples after 28 days of accelerated carbonation is reported in Fig. 2. Additionally, the improvement percentage of the carbonation resistance by applying impregnation treatment calculated by following Eq (1) was illustrated in Fig. 3.

The results of the accelerated carbonation test show that all of the impregnation products can limit the carbon dioxide ingress thus the carbonation progress is retarded. Among them, SS-PS-LS provided the best performance whereas LS exhibited only moderately effective for 28 days of exposure in the carbonation chamber. The carbonation depth reduced by the application of the silicate-based surface impregnation was up to 90% for the SS-PS-LS and only 10% for the LS (Fig. 3). The other four products (SS, PS and SS-LS, PS-LS) presented the significant reduction in the carbonation depth, particularly in the specimens treated with SS and PS. According to Fig. 3, the carbonation inhibiting effect was in the order of SS-PS-LS > PS > SS > SS-LS > PS-LS > LS. The results confirm the previous findings (Franzoni et al. 2013; Pan et al. 2017) that the sodium silicate impregnation is effective in preventing concrete against carbonation, and additionally show that the other silicate-based surface impregnation products are also effective in protecting concrete against carbonation either.

#### (2) Product’s penetration depth

The concentration of Na, K, Li measured by the method described in JSCE Guidelines (JSCE 2017) and the alkali metal ion concentration were presented in Table 2. As referred above, the product’s penetration depth is determined by the depth, in which the alkali metal ion concentration in treated surface is increased more than 30% compared with the alkali metal ion concentration contained in the untreated surface.

The method described in JSCE Guidelines (JSCE 2017) was successful in investigating the penetration depth of impregnation. As shown in Table 2, the alkali metal ion concentration in treated surface at a depth of 2 mm was much higher (157%), while the alkali metal ion concentration at the others depth were much lower than in untreated surface. Therefore, the penetration depth of...
2 mm was concluded in the specimens of w/c 0.6. The result agrees to the report by Baltazar et al. (2014) in which the penetration depth was measured in the more porous concrete of w/c 0.7, and the quantity applied was 110 – 215 g/m$^2$, which was much lower than the present study (250 g/m$^2$). On the other hand, the value measured was not as high as the minimum penetration depth of 5.0 mm suggested by EN1504-2 (2004). It is very probable that only a small amount of silicate-based surface impregnations can penetrate inside the concrete while the rest is likely to precipitate on the concrete surface.

4. Experimental exploration of the precise carbonation proofing mechanism

In the verification of carbonation inhibiting effect by silicate-based surface impregnations prescribed in JSCE Guidelines (JSCE 2017), we adopted air curing after application, in which the specimens were cured at 20 ± 2°C, RH 60 ± 5% for only one month (JSCE 2017). Then, the sample was exposed in the condition of CO$_2$ concentration of 5%, a temperature of 20°C, and a humidity of 60% for 28 days to promote carbonation before measuring the carbonation depth by colorimetric test using phenolphthalein. The results show that the carbonation depth of specimen treated with impregnation product is lower than the carbonation depth of untreated specimen. The possible explanation for this outcome is that when the silicate-based surface impregnation is applied, it penetrates into the concrete surface, reacts with calcium hydroxide to form C-S-H which has the pore-blocking ability, therefore enhancing the protection of concrete against carbonation (Dai et al. 2010; Thompson et al. 1997; Baltazar et al. 2014; Song et al. 2016; Franzoni et al. 2013; Pan et al. 2017a). However, previous studies have indicated that the precise mechanism should be more complicated because the impregnation may react with substrate, resulting in changing the porosity, pH, and the boundary resistance (Pan et al. 2017b). Furthermore, there is a possibility of a neutralization reaction between silicate-based surface impregnation and carbon dioxide on the concrete surface as the impregnation and carbon dioxide have the alkalinity and acidity respectively. In this section, as the first step of clarifying the carbonation proofing mechanism of silicate-based surface impregnation, the authors decided to investigate the characterization of this type of impregnation separately with concrete, which consisted of carbon dioxide absorption test and the crystallization observation under carbon dioxide environment through scanning electron microscopy (SEM). In terms of carbon dioxide absorption test, since carbonic acid is a weak acid, it is very likely that the carbon dioxide reaction/absorption may not be quantitatively measured. For this reason, an extra test, in which hydrochloric acid was added to the impregnation products was conducted, then the absorption amount of acid was measured to evaluate the carbonation absorption capacity of impregnation products.

4.1 Carbon dioxide absorption

Carbon dioxide absorption test was conducted by letting carbon dioxide gas flow through the bottle containing 250 g silicate-based surface impregnations solutions for from 2 to 5 minutes. Then, the change of appearance was observed, and the pH of formed solution was measured. The test method is shown in Fig. 4.

Furthermore, as it is difficult to quantitatively measure the amount of carbonic acid absorbed by silicate-based surface impregnation, the authors decided to use hydrochloric acid in the second test as an alternative acid to assess the carbon dioxide absorption capacity of each product by measuring the hydrochloric acid amount reacted at the gelation time. The test was conducted as follow. 250 g of silicate-based surface impregnation solutions were placed in a beaker. The solution was stirred with stirrer bar while adding 10% HCl solution to the beaker slowly and carefully. Then, the pH and the added amount of HCl when the gel is formed were measured (see Fig. 5).

4.2 Crystallization of impregnation under carbon dioxide environment

Silicate-based surface impregnation were painted on the surface of a glass petri dish before placing for 14 days in the desiccator which has been filled with carbon dioxide gas concentration of 20%. The impregnation products under carbon dioxide environment were observed using scanning electron microscopy (JCM-6000 Neoscope). For comparison sake, the impregnations were painted on the surface of a glass petri dish in the desiccator which has been filled with normal air, and were also observed by scanning electron microscopy (JCM-6000 Neoscope) after being left there for 14 days (see Fig. 6).
4.3 Results
(1) Carbon dioxide absorption
The results of pH changing before and after the absorption of carbon dioxide are illustrated in Fig. 7. As shown in Fig. 7, regardless of the type of silicate-based surface impregnation products, the pH drops from values in a range of 10.8 ~ 11.4 to values in a range of 10.6 ~ 11.2 when carbon dioxide is absorbed. Evidently, the impregnation has reacted with carbon dioxide in the presence of water (see Fig. 8). The chemical reactions can be described as follow: firstly, carbon dioxide dissolved water to form carbonic acid; then the next phase is the neutralization reaction between silicate-based surface impregnation and carbonic acid.

Carbon dioxide dissolves in water to form carbonic acid:

\[
\text{CO}_2 + \text{H}_2\text{O} \rightarrow \text{H}_2\text{CO}_3
\]  
(3)

Neutralization reaction between carbonic acid and silicate-based surface impregnations:

\[
\text{Na}_2\text{O} \cdot n\text{SiO}_2 + \text{H}_2\text{CO}_3 + (m-1)\text{H}_2\text{O} \rightarrow n\text{SiO}_2 \cdot m\text{H}_2\text{O} \cdot \text{Na}_2\text{CO}_3
\]  
(4)

\[
\text{K}_2\text{O} \cdot n\text{SiO}_2 + \text{H}_2\text{CO}_3 + (m-1)\text{H}_2\text{O} \rightarrow n\text{SiO}_2 \cdot m\text{H}_2\text{O} \cdot \text{K}_2\text{CO}_3
\]  
(5)

\[
\text{Li}_2\text{O} \cdot n\text{SiO}_2 + \text{H}_2\text{CO}_3 + (m-1)\text{H}_2\text{O} \rightarrow n\text{SiO}_2 \cdot m\text{H}_2\text{O} \cdot \text{Li}_2\text{CO}_3
\]  
(6)

The production of the above chemical reaction is the formation of silicate gel which is white, similar to a jelly, cling to the bottle (see Fig. 8). A reasonable explanation for this result is when acidic carbon dioxide reacted with alkaline silicate-based surface impregnation, the pH decreased due to the neutralization reaction, while the polymerization of silicic acid ions progressed, the viscosity increased, then the gel is formed.

In the method of using hydrochloric acid instead of carbonic acid to assess the carbon dioxide absorption capacity of each silicate-based surface impregnation, 10% HCl solution was dropped in the beaker which contained 250 g impregnation product until the gel was formed. The pH at gelation time is shown in Fig. 9 while the amount of hydrochloric acid added (ml) at gelation time is presented in Fig. 10.

As expected, the pH of silicate-based surface impregnation dropped from the values of higher alkalinity...
to the values of lower alkalinity at the time that silicate gel appeared (Fig. 9). Apparently, it proves that the neutralization reaction has occurred between the impregnation and hydrochloric acid. From Fig. 10, the amount of hydrochloric acid added until the gel appeared was highest for SS-LS-PS (50 ml) and lowest for LS (30 ml). Also, in case of SS, PS, SS-LS, PS-LS, the added hydrochloric acid amount was in the range of 43 ml to 47 ml. The acid needed to react with impregnation vary by the type of impregnation. The result of hydrochloric acid added can be summarized in this order: SS-PS-LS > PS > SS > SS-LS > PS-LS > LS.

(2) Crystallization of silicate-based surface impregnation under carbon dioxide environment

The SEM images of silicate-based surface impregnation products precipitated by being dried under carbon dioxide environment and normal air can be observed in Figs. 11 and 12 respectively. The images are taken on the same scale as much as possible for each impregnation products for better comparison.

The SEM analysis of the crystallization of silicate-based surface impregnation products showed that regardless of drying environment, sodium silicate (SS), potassium silicate (PS), lithium silicate (LS) were respectively precipitated into three different types of crystals like needle shape, broken rectangle shape, irregular shape. Throughout this paper, we use the terms ‘needle-shaped crystals’, ‘rectangle-broken crystals’, and ‘irregular crystals’. Besides, in the case of the mixture of several silicate-based surface impregnations (SS-LS, PS-LS, SS-PS-LS), crystals of various shapes were mixed and precipitated.

5. Discussion

The results of the accelerated carbonation test (Figs. 2 and 3) show that all of the silicate-based surface impregnation products could reduce the carbonation rate of concrete. Among them, SS-PS-LS exhibited the best performance while LS provides only moderate performance. The improvement percentage of carbonation resistance by applying the silicate-based surface impregnation was in the order of SS-PS-LS > PS > SS >
SS-LS > PS-LS > LS. The results obtained also suggest that the reactive type or the mixture of several impregnations (SS, PS and their combinations) which contains the reactive type is more efficient than the solidifying type (LS).

As previously mentioned, carbonation is commonly known as the chemical reaction between dissolved calcium hydroxide (portlandite) and carbon dioxide to form calcium carbonate (Richardson 1988; Saetta et al. 1993; Pan et al. 2017b). Past studies have stated that one of the main factors that control the carbonation process is the diffusivity of CO₂, which depends on the pore structure of concrete and the exposure condition (Pan et al. 2017b; Jiang et al. 2000). As mentioned in the introduction, when silicate-based surface impregnations penetrate into the concrete surface, then the impregnation, particularly the reactive types such as sodium silicate, potassium silicate, theoretically react with the hydration products of concrete to form calcium silicate hydrates (C-S-H gels). As same with reactive types, solidifying type (lithium silicate) in solution status also reacts with the hydration products of concrete to form C-S-H gels at the initial stage of the impregnation process. However, solidifying type becomes solidified materials after drying, which is very difficult to be dissolved, then these solidified materials fill the voids of the concrete, resulting in improving the concrete durability (JSCE 2017).

C-S-H gel formation reaction:

\[
\begin{align*}
M_2SiO_3 + yH_2O + xCa(OH)_2 & \rightarrow \\
xCaO \cdot SiO_2 \cdot yH_2O + 2M(OH) & \quad (M: Na, K, Li)
\end{align*}
\]

As a result, the surface of concrete has increased the impermeability, therefore improves resistance to the penetration of carbon dioxide, resulting in reducing the carbonation rate of concrete (Dai et al. 2010; Thompson et al. 1997; Baltazar et al. 2014; Song et al. 2016; JSCE 2017).

On the other hand, silicate-based impregnations are evidently proved to be reactive with carbon dioxide to form silicate gel as shown in formulas (4), (5) and (6). Here, we assumed that silicate-based surface impregnation could reduce the carbonation of concrete because of the carbon dioxide absorption due to the chemical reaction; therefore, the amount of carbon dioxide that penetrates into the concrete and reacts with calcium hydroxide would be lesser than the beginning. Besides, as for the test with alternative acid (hydrochloric acid) to investigate the absorption amount of carbon dioxide into the impregnations, the results reveal that the added amount of hydrochloric acid can be presented as follows: SS-PS-LS > PS > SS > SS-LS > PS-LS > LS (see Fig. 10). By replacing the added amount of hydrochloric acid into the absorption amount of carbon dioxide, it is possible to consider that the carbon dioxide absorption level of impregnations is in the same order. This order shows almost the same tendency as the improvement percentage of carbonation resistance by applying impregnations shown in Fig. 3; therefore, the carbonation reduction may depend on the carbon dioxide absorption level of silicate-based surface impregnations. These evidences support the hypothesis that silicate-based surface impregnations not only react with calcium hydroxide in the concrete to form the C-S-H gel hence delay the penetration of carbon dioxide by filling the micropores; but also react with carbon dioxide (as shown in the above reaction) then form the silicate gel which can absorb and immobilize the carbon dioxide on the concrete surface layer. In particular, both of C-S-H gel and carbonate-silicate gel may coexist, consequently improve the
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