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# Study on applicable evaluation of surface impregnant based on drying and wetting test

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### ABSTRACT

Recently, in Japan many concrete structures deteriorated by de-icing salts have been reported where de-icing salts were scattered on a road to avoid a traffic accident due to a road surface freezing. Use of de-icing salts has been increased since a studded tire was prohibited in 1990's. This results in the occurrence of the early steel corrosion in concrete structure. It is needed to establish the economical and proper maintenance method for these deteriorated concrete structures due to de-icing salts. Silane impregnation is expected to be the cost effective method on extending the service life of deteriorated concrete structures. In this study, some silane impregnations were tested to evaluate the basic performance (impregnation depth, water liquid permeability, water vapour permeability and chloride permeation) using dying and wetting test. As the results, it is possible to evaluate the performance of the impregnant for the concrete affected by de-icing.

Keywords. Surface Impregnant, De-icing Salt, Steel Corrosion, Repair, Evaluation Test

### **INTRODUCTION**

The chloride-induced corrosion in concrete structure is a typical deterioration mechanism of a concrete structure since the national land is surrounded and enclosed by sea, in Japan. A lot of concrete structures deteriorated by the steel corrosion have been reported. Therefore, a large number of studies have been done concerning the chloride-induced corrosion. The standard method and strategy for a concrete structure deteriorated by chloride-induced corrosion have been established in Japan.

On the other hands, de-icing salts is scattered on the road to avoid a traffic accident due to a road surface freezing in a mountain area and in a snowy area. Use of de-icing salts has been drastically increased since a studded tire was prohibited in 1990's. The corrosion of steel bar and the delimitation of concrete due to de-icing salts have been frequently and intensively observed in concrete member affected directly by splash water, drainage and leakage with de-icing salts. In these affected parts in concrete structure, a lot of the chloride ion was permeated and accumulated intensively. This phenomenon on the chloride ion permeation is a significantly difference between de-icing salts and seawater. In the case of de-icing salts, a high concentrative NaCl solution attacked directly to a concrete structure and member in a particular area where concrete is intensively affected by splash water, drainage and leakage. A permeation of the chloride ion supplied from de-icing salts is very quick and this results in the occurrence of the early steel corrosion in a concrete structure. Thereby, many early deterioration cases on slabs and concrete members around joint due to de-icing salts have been reported in Japan (Aoyama 2004). The prediction method for the permeation of chloride ion into concrete was established in Japanese standard specification (JSCE 2008). However, this method was not available for the permeation of chloride ion supplied from deicing salts and the proper prediction method for de-icing salts has been not established.

On the other hands, it is needed to establish the economical and the proper maintenance method for these deteriorated concrete structures due to de-icing salts and other deterioration factor. Provisional measures for deteriorated concrete structures are needed depending on an economical limitation under a maintenance finance condition. Silane impregnation has been frequently applied as a cost effective repair and a provisional measures to extending a service life of concrete structure in above cases. Silnae impregnation has the effectiveness on the suppression and the reduction of steel corrosion in concrete resulted in drying concrete surface (Tonooka 2007). In previous studies, the influence of water content of concrete and curing condition on the performance of surface impregnation was investigated in order to extend the service life on a construction and a repair of concrete structure (Konno 2006 and Konno 2007).

In this study, commercial silane impregnants which have been recently improved its performance were used and was compared to these performance of a conventional alcohol solvent silane. The silane systems were tested with wetting and drying test that simulate the chloride ion permeation under the application of de-icing salts. The performance of silane system was evaluated for the purpose of the application for concrete structure affected by de-icing salts.

# **OUTLINE OF EXPERIMENT**

**Concrete.** Ordinary Portland cement was used (density:  $3.16 \text{ g/cm}^3$ ), and the fine aggregate (density:  $2.62 \text{ g/cm}^3$ , absorption: 1.32 %) and the coarse aggregate (density:  $2.60 \text{ g/cm}^3$ , absorption: 1.91 %, Gmax: 20 mm) was used from Tedori River. Mix proportion of concrete is shown in **Table 1**. W/C was 65 % and the water cement ratio was considered as exiting

W/C (%)	s/a (%)	Unit Weight (kg/m <sup>3</sup> )			$/\mathrm{m}^3$ )	AE water	AE	Slump	Air
		W	С	S	G	reducing agent (cc/m <sup>3</sup> )	agent (cc/m <sup>3</sup> )	(cm)	(%)
65	46	172	272	828	968	1020	1088	$10 \pm 2.5$	$5.0 \pm 1.0$

# Table 1. Mix proportion of concrete

concrete surface that have a low quality due to weathering action.

**Surface impregnants.** Surface impregnants used in this study is shown in **Table 2**. Four types of commericial silane (A, B, C and D) were selected. Each concentration of active ingredient of tested silane impregnants (A, B, C and D) is higher than a conventional impregnant of which the concentration range from 20 % to 25 %. Active ingredient of impregnant B is selected as alkoxy silane in order to form a dense and thin impregnated layer adjacent surface concrete. E was 100 % hexyltriethoxy silane (absence of solvent) and E was similar in performance of a conventional alcohol solvent silane.

The dosage of impregnant A, B, C and D was a standard dosage recommended by each manufacture (A, B, C350 and D). On the impregnant C, another dosage that was 50 g/m<sup>2</sup> (C50) was prepared as an impregnation of a low performance. Furthermore, the dosage of impregnant E was 50 g/m<sup>2</sup> (E). In this dosage (50 g/m<sup>2</sup>) of impregnant E, the volume of the active ingredient for imapregnation E was equal to the volume of the active ingredient for a conventional alcohol solvent silane. No treatment was prepared as a control. Type of impregnation is shown in **Table 3**.

**Specimen.** The size of specimen was 100 mm x 100 mm x 50 mm. One day after casting, the mold was removed and kept in a laboratory (20  $^{\circ}$ C, 60  $^{\circ}$  R.H.) during one day. All surface of concrete specimen was treated using a disk sunder for the preparation of the impregnation. The surface impregnation was done with the prescribed dosage of these impregnants.

**Drying and wetting test method.** After fabrication of specimen, drying and wetting test was done. First step (drying step) is drying a concrete specimen under 40 °C, 30 % R.H., 24

Impregnant	А	В	С	D	Е
Appearance	White paste	Clear liquid	Light- gray gel	Clear liquid	Clear liquid
Main ingredient	Octyl- triethoxy silane	Alkoxy silane	Silane- siloxane complex	Alkyl- alkoxy silane	Hexyl- triethoxy silane
Active ingredient	95~99%	≧94%	≧90%	≧98%	100%
Standard dosage $(g/m^2)$	200	150	350	175~264	50
Number of applications	1	1	1	2	1

 Table 2. Properties of impregnants

Table 3.	Type	of impr	egnation
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Impregnation	Α	В	C50	C350	D	Е	Ν
Impregnant	А	В	С	С	D	Е	No treatment
Dosage (g/m <sup>2</sup> )	200	150	50	350	264	50	_

hours and second step (wetting step) is immersing a concrete specimen under the saturated NaCl solution immersion, 0.5 hour. One cycle of test consists of drying and wetting steps. Test was repeated until 14 cycles. In the wetting step, the water level on the top of the specimen was 10 mm under water.

**Measurement**. The mass of specimen was measured using an electrical scale with accuracy 0.01 g. The impregnation depth was measured using a slide calliper with 0.1 mm of accuracy. Hydrophobic part on the split surface of specimen where water mist sprayed on this surface was defined as hydrophobic depth. The chloride permeation depth was measured by the slide calliper with 0.1 mm of accuracy. The colour change part of split surface where 0.1 mol/l silver nitrate solutions were sprayed on this surface after drying and wetting test was defined as the chloride permeation depth. The chloride ion content of concrete was measured based on JCI-SC5 with an automatic titrator. The sample was taken from the centre parts of the split surface until maximum 30 mm in depth and the size of sample was 10 mm in width, 50 mm in length and 10 mm in depth. The sample was ground the powder sample was put through a sieve of 150  $\mu$ m. The powder sample passed through a sieve of 150  $\mu$ m was used for measuring the chloride ion content of concrete.

## **RESULTS AND DISCUSSION**

**Impregnation depth.** The impregnation depth is shown in **Figure 1**. The average impregnation depth for A, C350 and D showed a larger value. These impregnants (A, C350 and D) were improved to obtain a good performance and a large impregnation depth. Consequently, the large impregnation depth was obtained in this study. The average impregnation depth for B was smaller than other commercial impregnants, since this impregnant was designed in order to form the dense and thin impregnated layer. Incidentally, the order of maximum impregnation value among them was similar to the order of the average impregnation value. Also, these trends were observed in relationships between average and minimum impregnation depth.

**Total mass gain in wetting step and total mass reduction in drying step.** The total mass reduction and total mass gain are shown in **Figure 2** and **Figure 3**, respectively. Total mass reduction and total mass gain were accumulated from the mass changes of each step.



Figure 1. Impregnation depth

Total mass gain (water absorption) for A, B, C350 and D was less than that for C50 and E. Total mass gain for B, C50, and E of smaller dosage were widely varied. It is considered that the inadequate impregnated layer is formed in some parts, since these dosages for them are small. Therefore, it is noted that the adequate dosage of impregnant is needed to form the faultless impregnated layer since a quality of a concrete in a surface layer on in-situ structure is widely varied even if the same concrete mixture was used.

The mass reduction for A, C350 and D were somewhat smaller than C50 and E. It is considered that the large impregnated layer prevents slightly water vapour from permeating through this layer. Also, the mass reduction for B was as same as that for C50 and E.

**Mass change.** The mass change (= mass reduction – mass gain) is shown in **Figure 4**. According to the definition of mass change, the mass change of the larger value has the lager hydrophobic effect. The average mass change, for impregnant A, B, C350 and D was larger than that for C50 and E. It is concluded that the effectiveness in drying concrete surface for them was higher than conventional impregnants (C50 and E). On the other hand, mass



Figure 2. Total mass gain



Figure 3. Total mass reduction

change for C50 and E were significantly small since they have some fault-impregnated parts of them due to the inadequate dosage of the impregnant. Therefore, it should be is important that the adequate dosage of the impregnant is needed to form the faultless impregnated layer.

**Chloride penetration depth.** The chloride permeation depth is shown in **Table 4**. The chloride permeation depth for no treatment was very large. On the other hand, the chloride permeation depth for impregnated specimens with the standard dosage (A, B, C350, and D) was not observed by the color change test. It is considered that the chloride ion cannot permeate into concrete over the impregnation depth. For C50 and E, the chloride permeation area was partially observed. It is considered that this chloride permeation was caused by the inadequate impregnation for them.

**Profile of chloride ion content.** Profile of the chloride ion content of concrete is shown in **Figure 5**. In Figure 5, the right side figure is a scaled-up view for the results of their impregnated concrete. The chloride ion content for control was very high at near surface. On the other hand, the chloride ion content for impregnant A, B, C350 and D were 0 kg/m<sup>3</sup> at more than 1 cm of depth. The permeation of chloride ion was drastically reduced by the silane impregnation. For C50 and E, the chloride ion content was observed in more than 1 cm of depth. The difference in the hydrophobic performance of the silane impregnation resulted in the difference in the permeation of chloride ion. As result, it is considered that the effectiveness in preventing chloride ion permeation into concrete for these impregnants (A, B, C350 and D) is higher than conventional impregnants (C50 and E).



Figure 4. Mass change

fable 4. Ch	loride pern	neation depth
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Impregnation	А	В	C50	C350	D	Е	Ν
Average	0.0	0.0	1.0	0.0	0.0	1.9	20.7
Max.	0.0	0.0	13.4*	0.0	0.0	20.7*	26.8

\*: partially permeation part (max.)

The relationship between the total mass gain and the average chloride ion content (corresponding to total permeation of chloride ion) is shown in **Figure 6**. The average chloride ion content was calculated from the accumulation of the chloride ion content at every depth. The average chloride ion content was proportional to the average chloride ion content. The chloride permeability of the impregnated concrete can be evaluated by evaluating the water liquid permeability of the impregnated concrete even in the drying and wetting process.

On the other hand, there was not necessarily observed in correlation between the impregnation depth and the average chloride ion content. It is considered that the performances of impregnated concretes depend on not only impregnated depth but also chemical composition and dosage of impregnant even when the same impregnation depth is obtained. Thereby, the effectiveness in preventing chloride ion from permeation into concrete was different among them.



Figure 5. Profile of the chloride ion content of concrete



Figure 6. Relationship between the total mass gain and the average chloride ion content

#### **CONCLUSION**

The main results in this study are shown as follows;

- (1) Every impregnation with the standard dosage has a good performance. On the other hand, impregnants of the smaller dosage result in forming the inadequate impregnated layer in some parts.
- (2) Therefore, it is noted that the adequate dosage of impregnant is needed to form the faultless impregnated layer in order to keep the good performance after the impregnation.
- (3) The effectiveness in preventing chloride ion permeation into concrete for these impregnants (A, B, C350 and D) is higher than conventional impregnants (C50 and E).
- (4) The chloride permeability of the impregnated concrete can be evaluated by evaluating the water liquid permeability of the impregnated concrete even in the drying and wetting process.

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