# A-1-2 Performance of surface protection for concrete after applying electrochemical repair method

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ABSTRACT: Desalination and realkalisation are electrochemical repair methods for reinforced concrete structures deteriorated by steel corrosion in concrete due to chloride attack or carbonation. After the electrochemical treatment, surface protection materials should be applied for achieving longer repair effect. However, it has been reported that such materials are often deteriorated at an early stage due to the chemical contact with the highly alkaline concrete pore solution after completing the electrochemical treatment. In this research, experimental investigation was carried out aiming to grasp the performance such as bond strength and water permeability of surface protection materials applied to the concrete after electrochemical treatment. The water-repellent effect of silane-siloxane type impregnation applied after realkalisation was lower than the case of desalination specimens regardless of the water content of concrete at the time of application of surface impregnation. The reason of this is considered that the content of alkali ions around the concrete surface after realkalisation was higher than the case of desalination, which would hinder the formation of impregnated concrete at 14 % of water content was lower than the case of 8 % of water content, although all case of bond strength exceeded 1.0 N/mm<sup>2</sup> which is generally required in Japan.

*KEY-WORDS: Silane-based surface impregnation, polymer cement mortal, epoxy resin, electrochemical repair method, bond strength; water permeability.* 

# **INTRODUCTION**

The steel corrosion in concrete caused by the carbonation or chloride attack is a major deterioration of reinforced concrete (RC) structure. For rehabilitating the deteriorated RC structure effectively, realkalisation and desalination have been developed [1]. These electrochemical repair methods are conducted by applying an electrical field and a direct current between an externally installed anode surrounded in the electrolyte and the steel in concrete, without the removing the concrete like a patch repair method [2]. The cases using these electrochemical methods have been increased gradually [3, 4]. However, the long term repair effect of these electrochemical treatments has not been clarified. Recent investigations reported that the steel corrosion in concrete had been suppressed for approximately 20 years after applying realkalisation and desalination [5]. In order to prevent the penetration of the deterioration factors like chloride ion (Cl<sup>-</sup>) or carbon dioxide (CO<sub>2</sub>) from concrete surface for achieving longer protection effect against steel corrosion after the electrochemical treatment, surface protection methods are sometimes applied to the concrete after the removal of the anode system of the electrochemical treatment.

Meanwhile, with the application of direct electric current, the high alkaline electrolyte solution ( $K_2CO_3$ ,  $Li_3BO_3$ , etc.) penetrates from concrete surface by electric-osmosis. Also, hydroxyl ions (OH<sup>-</sup>) generate around the steel by the cathodic reaction [6]. As a consequence of these phenomenons, alkalinity and moisture content in concrete after electrochemical treatment will increase compared with the case of non-treated concrete. Therefore, it was pointed out that the surface protection materials applied after the electrochemical treatment were sometimes

deteriorated at an early stage.

From this background, this study conducted an experimental investigation to grasp the performance such as bond strength and water permeability of surface protection materials applied to the concrete after electrochemical treatment.

#### EXPERIMENTAL Materials and preparation of specimen Table 1 Mix proportion of concrete

Table 1. Witz proportion of concrete.										
Types of the electrochemical treatment	W/C (%)	s/a (%)	G <sub>max</sub> (mm)	Content (kg/m <sup>3</sup> )						
				С	W	S	G	WRA	AEA	NaCl
Realkalisation	60	48	15	300	180	829	902	0.6	0.005	3.3
Desalination						819			0.002	13.2



Exposed surface where surface protection applied.

The specimens prepared for this study were RC prisms ( $50 \times 100 \times 200 \text{ mm}$ ) with a deformed steel bar D 10 SD 295 A (JIS number) was located at 20 mm depth from the exposed surface as shown in Fig. 1. The mix proportion of the concrete used in this study is shown in Table 1. The water cement ratio (W/C) of concrete was 60 %. Ordinary portland cement (density:  $3.16 \text{ g/cm}^3$ ), fine aggregates (density:  $2.56 \text{ g/cm}^3$ , F.M.: 2.63) and the coarse aggregate (Gmax: 15 mm, density:  $2.55 \text{ g/cm}^3$ ) were used respectively. An amount of premixed Cl<sup>-</sup> for realkalisation specimens was 2.0 kg/m<sup>3</sup> which would be around the critical Cl<sup>-</sup> content for the initiation of steel corrosion. On the other hand, the specimens for desalination premixed Cl<sup>-</sup> of 8.0 kg/m<sup>3</sup> which would cause the severe chloride-induced corrosion of steel. The premixed NaCl was dissolved in the mixing water. All specimens were cured for 28 days in the moist condition at the temperature of 20 °C. After curing, five faces of specimen for applying electrochemical treatment were insulated with epoxy resin coating, excepting one exposed surface (100 × 200 mm) as shown in Fig. 1. On the other hand, two opposite faces (100 × 200 mm) of a specimen for immersing in the electrolyte solution was exposed and other four faces were insulted with epoxy resin coating.

After insulating the side faces of specimen, a part of the specimens for realkalisation were kept in accelerated carbonation environment (20  $^{\circ}$ C, 60% R.H., 5% CO<sub>2</sub>) for 9 months, and the carbonation depth after accelerated carbonation for 9 months was approximately 37 mm, measured according to JIS A 1152. In addition, non-carbonated control specimens for realkalisation were prepared.

# Electrochemical treatment and immersion in electrolyte solution

Fig. 2 shows the experimental flowchart. The electrochemical treatment was applied to the specimens, the direct electric current was applied between the titanium mesh and the steel in concrete. The electric current density was constantly  $1.0 \text{ A/m}^2$  to the concrete surface. The treated period was 2 weeks for realkalisation and 8 weeks for desalination. As the electrolyte solution,  $1.5 \text{ N K}_2\text{CO}_3$  solution for realkalisation and  $0.1 \text{ N Li}_3\text{BO}_3$  solution for desalination were selected respectively. In addition, in order to simulate simply the concrete applied electrochemical treatment, the specimens were immersed in each electrolyte solution used for the electrochemical treatment in a vacuum chamber until the weight of specimen became constant. Furthermore, non-treated control specimens in each case were also prepared.

Fig.1. Outline of concrete specimen.



Fig. 2. Experimental flow.

# Application of surface protection

Surface water content of the specimens for immersion in the electrolyte solution was controlled to around 8 % or 14% in a room of 60 % R.H. and 20  $^{\circ}$ C before applying surface protection to the exposed surface. On the other hand, surface water content of the specimens applied the electrochemical treatment was controlled to around 14 % in the same condition before applying surface protection. Three kinds of surface protection such as epoxy resin coating, PCM coating and silane-based impregnation were selected. Details of surface protection are shown in Table 2.

After applying the surface protection, the specimens were kept in a room of 60 % R.H. and 20  $^{\circ}$ C for 28 days. In addition, after applying the surface protection, a part of the immersed specimen which controlled surface water content as 14 % kept in each electrolyte solution for 28 days as alkali supply promotion test (refer to Fig. 3.). This accelerated deterioration test assumed that the alkaline solution in concrete will be supplied to the interface between surface coating materials and concrete after the electrochemical treatment. The list of specimens used in this research is shown in Table 3. As the legend of specimens, K means the specimen immersed K<sub>2</sub>CO<sub>3</sub> solution, R means the specimen applied realkalisation and RC means the specimen applied realkalisation after carbonation. Furthermore, L means the specimen immersed Li<sub>3</sub>BO<sub>3</sub> solution, D means the specimen applied desalination. The numbers behind the initials denote the surface water content of concrete when the surface protection applied to the specimen. In addition, A means the specimen conducted the alkali supply promotion test after applying surface protection Moreover, the initials behind '-' denote the kind of surface protection materials. Namely, P means PCM, E means epoxy resin coating, and S means silane-based impregnation. On the other hand, only S or P or E are the controlled specimens.

	or y minus of surface protection.				
Types of surface protection	Application rate (g/m <sup>2</sup> )				
Epoxy resin with wet wear resistance	800				
Polymer-cement mortar with acrylic resin (PCM)	PCM (1600) Primer (200)				
Silane-Siloxane type impregnation	200				

Table 2. Specification of 3 kinds of surface protection.



Fig. 3. Alkali supply promotion test.

Name	Method for supplying electrolyte solution	Water Content (%)	Surface Treatment	Alkali supply promotion test	Name	Method for supplying electrolyte solution	Water Content (%)	Surface Treatment	Alkali supply promotion test
R14-S		• 14	Silane	-	D14-S		14	Silane	-
R14-P	Realkalisation		PCM	-	D14-P	Desalination		PCM	-
R14-E			Epoxy-resin	-	D14-E			Epoxy-resin	-
RC14-S	Realkalisation		Silane	-	L8-S	Immersion to Li3BO3	8	Silane	-
RC14-P	(applied after		PCM	-	L8-P			PCM	-
RC14-E	carbonation)		Epoxy-resin	-	L8-E			Epoxy-resin	-
K8-S	Immersion to K <sub>2</sub> CO <sub>3</sub>	8	Silane	-	L14-S		14	Silane	-
K8-P			PCM	-	L14-P			PCM	-
К8-Е			Epoxy-resin	-	L14-E			Epoxy-resin	-
K14-S		14	Silane	-	L14A-S			Silane	0
K14-P			PCM	-	L14A-P			PCM	0
К14-Е			Epoxy-resin	-	L14A-E			Epoxy-resin	0
K14A-S			Silane	0	S	Non-supply	8	Silane	-
K14A-P			PCM	0	Р			PCM	-
К14А-Е			Epoxy-resin	0	Е			Epoxy-resin	-

Table 3. List of concrete specimens.

#### Chemical analysis of concrete after electrochemical treatment

Distribution of ion content (Na<sup>+</sup>, K<sup>+</sup>) and pH in the concrete were measured immediately after the electrochemical treatment using powder samples grinded from concrete plates cut out from a specimen as shown in Fig. 4. The Na<sup>+</sup> and K<sup>+</sup> contents were measured by means of atomic absorption spectrometry using a crushed fine powder sample dissolved by HNO<sub>3</sub> solution. For measuring pH values, sample solution was prepared by mixing 12.5 g of crushed fine powder and 25 ml of pure water, and a pH electrode was used for the measurement.



Fig. 4. Cutting of concrete specimen for chemical analysis.

### **RESULTS AND DISCUSSION**

#### Distribution of ions in specimens after electrochemical treatment

The distribution of  $R_2O$  as total amount of alkali and pH values in the concrete immediately after the electrochemical treatment are shown in Fig. 5. The amount of  $R_2O$  is calculated by Eq. (1).

$$R_2O = Na_2O + 0.658 K_2O (kg/m^3)$$
(1)

The origin of horizontal axis in Fig. 5 corresponds to the exposed surface and the steel bar is located at the center (20 mm cm from the exposed surface) of each specimens.



Fig.5. The distribution of  $R_2O$  and pH in the concrete just after completing the electrochemical treatment.

According to Fig. 5, in the case of specimens applied realkalisation, the  $R_2O$  content around the concrete surface becomes highest in the distribution of  $R_2O$ , because of the K<sup>+</sup> ions introduced from the electrolyte (K<sub>2</sub>CO<sub>3</sub> solution) by electro-osmosis with realkalisation. However, the specimen applied realkalisation after carbonation shows a little higher  $R_2O$  content in the cover concrete than the case of non-carbonated. The reason of this would be that the concrete after carbonation was dried under the accelerated carbonation environment compared with the case of non-carbonated specimen, which promoted the penetration of  $K_2CO_3$  solution due to realkalisation.

The non-carbonated specimen maintained high pH level after realkalisation, while in the carbonated specimen, the pH value at the carbonated concrete surface shows around 11 even after the realkalisation. The specimen subjected to desalination show high pH level, and the peak of pH level and the  $R_2O$  content can be observed around the steel bar, because of migration of  $Na^+$  ions in the concrete due to the electrophoresis with desalination.

It is considered that the high alkali condition at the concrete surface after the electrochemical treatment could cause the early deterioration of surface protection materials applied after the electrochemical treatment.

## The performance of silane-based impregnation applied after supplying electrolyte solution

The variation curves of the contact angle of a water drop on the concrete surface applied silane-based impregnation after supplying electrolyte solution are shown in Fig. 6. From Fig. 6, in the cases of specimen for desalination (L and D series), the water contact angles maintain higher than 90° that indicates surface of specimen have sound hydrophobic during the accelerated deterioration period while a little lower than the specimen which not applied electrochemical treatment (S) regardless of the surface water content. It indicates that the surface water-repellent effect by silane-based impregnation was maintained after desalination regardless of the surface water content.



Fig.6. The variation curves of the contact angle of a water drop on the concrete surface applying silane-based impregnation after supplying electrolyte solution.

However, the specimen for realkalisation (K and R series) are lower than the L and D series regardless of the surface water content. The reason of this would be that the formation of the water repellent layer by the silane-based impregnation was inhibited by the alkali ions ( $K^+$ ) accumulated at the concrete surface in the realkalisation specimen as shown in Fig. 5. Furthermore, the values of the specimen for realkalisation applied silane-based impregnation when 14 % of the surface water content (K14-S, K14A-S, R14-S and RC14-S) is lower than the case

of the specimen applied when 8 % of surface water content (K8-S). As reason of this, it is considered that the formation of the water repellent layer was suppressed in the high moisture content.



Fig.7. The water permeability of the specimen applied silane-based impregnation after supplying electrolyte solution.

Fig. 7 shows the water permeability ratio of the specimen applied silane-impregnation after supplying electrolyte solution. The water permeability ratio was calculated by Eq. (2).

Water permeability ratio  $\left(\frac{\text{ml}}{\text{ml}}\right) = \frac{\text{Measured water permeation (ml)}}{\text{Water permeation of control specimen (S) (ml)}}$ . (2)

From Fig. 7, the tendency is in accordance with the results of the contact angle shown in Fig. 6. Namely, the water permeability ratio of L and D series was lower than K and R series regardless of the surface water content. Furthermore, the water permeability ratio of specimen applied silane-based impregnation when the 8 % of surface water content after immersed in  $Li_3BO_3$  solution (L8-S) shows approximately 1.0. It means that when silane-based impregnation was applied under 8 % water content after supplying  $Li_3BO_3$  solution, the water-repellent effect didn't decrease with the supply of the electrolyte solution.

However, the water permeability ratios of the specimen for desalination controlled 14 % of surface water content (L14-S, D14-S and L14A-S) are larger than L8-S, although the contact angle of a water drop of L and D series is approximately same value regardless of the surface water content. The reason would be that the penetration of silane-based impregnation of L14-S, L14A-S and D14-S was suppressed due to high moisture content compared with the case of L8-S. Actually, the average of the measured penetration depth of L14-S, L14A-S and D14-S was approximately 1.3 mm, which was smaller than that of L8-S measured as 3.2 mm.

#### The Bond strength of PCM and epoxy resin applied after supplying electrolyte solution

Fig. 8 shows the bond strength of PCM and epoxy resin coating applied after supplying electrolyte solution. As shown in Fig. 8(a), the bond strength of PCM on specimen for realkalisation (R and K series) was smaller than that of specimen for desalination (L and D series). In addition, the fracture mode of specimens for realkalisation was interface fracture, although that of specimens for desalination was combination of interface fracture and base material fracture. The reason of this would be that the high alkaline condition at the concrete surface after realkalisation (refer to Fig.5.) could cause the alkaline hydrolysis of PCM, and it decrease the bond strength between concrete and PCM compared with the case of desalination. However, the bond strength of L14-P was lower than the other cases. Therefore, the bond strength of PCM applied after desalination could decrease when the alkali solution will supply the interface between PCM and concrete.

According to Fig. 8(b), the tendency of the bond strength of epoxy resin coating is in accordance with the results of PCM. The fracture mode of epoxy resin coating is base material fracture in all specimens. In addition, all results of bond strength measured in this study exceed 1.0 N/mm<sup>2</sup> which is generally required in Japan and significant change of the appearance of surface coatings was not observed.



Fig. 8. The bond strength of surface protection materials applied after supplying electrolyte solution. (a) The bond strength of PCM. (b) The bond strength of epoxy resin coating.

# CONCLUSIONS

The following conclusions can be drawn from this study:

- 1.  $R_2O$  content around the concrete surface increased with the application of realkalisation, because of the supply of K<sup>+</sup> ions from the electrolyte (K<sub>2</sub>CO<sub>3</sub> solution). On the other hand, the application of desalination resulted in the accumulation of R<sub>2</sub>O content around steel bar due to the electrochemical migration of Na<sup>+</sup> ions in the concrete due to the application of desalination.
- 2. The previous application of realkalisation decreased the contact angle of a water drop, which is the index of the water-repellent effect, on the surface of specimens applied silane-based impregnation, while the previous application of desalination didn't decrease the contact angle value. Moreover, when the surface water content of the specimen applied silane-based impregnation was high (14%), the larger decreased the water-repellent effect was observed.
- 3. The previous application of realkalisation decreased the bond strength of epoxy resin and PCM coating more than the case of desalination. Moreover, when the surface water content of specimen applied these surface coating was high (14%), or when the alkali solution was supplied from the backside of the coating, the larger decrease of the bond strength was observed, although all the measured bond strength exceeded 1.0 N/mm<sup>2</sup> which is generally required in Japan.

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