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Technical Note

Assessment of surface treatment systems for protecting concrete structure

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ABSTRACT

The BS EN 1504–2:2004 groups surface treatments into three types: impregnations, hydrophobic impregnations, and surface applied corrosion inhibitors. Impregnations reduce surface porosity by filling concrete pores, while hydrophobic impregnations create a water-repellent surface without filling pores. Surface applied corrosion inhibitors form a protective film on the rebar surface. Impregnation strengthens the surface by blocking pores with reaction products that reduce ingress of aggressive agents. Hydrophobic impregnation produces a hydrophobic and water-repellent surface that inhibits water penetration while allowing concrete to breathe. Corrosion inhibitors migrate to the steel surface and form a mono-molecular film, preventing further corrosion. In this study accelerated corrosion test was used for determination of the effectiveness of each category in offering corrosion protection by subjecting concrete specimens coated with these surface treatments to accelerated corrosion conditions that is intended to induce corrosion in the embedded rebars. This paper presents test results for the performance of these three surface treatments on grade G30 and G40 concretes. Results show that hydrophobic agents are more effective than traditional impregnates in reducing water absorption and chloride penetration.

1. Introduction

Steel embedded in concrete is effectively prevented from corroding in most, but not all, circumstances by the formation of a protective passive layer or film on the steel surface. This protective layer is maintained by the highly alkaline concrete environment. However, if the concrete becomes contaminated with chemical species that disrupt the passive layer or reduce the concrete's alkalinity then, providing there are sufficient concentrations of moisture and oxygen present, corrosion of the reinforcement can occur in areas where the passive layer no longer protects the steel (Ahmad, 2003). Loss of steel passivity within concrete structures largely occurs through the action of two processes namely, carbonation and chloride contamination. Corrosion of the steel reinforcement can occur through either process individually or through a combination of both (Page, 2007). Carbonation leads to a reduction in alkalinity of the concrete and ultimately results in the depassivation of the steel reinforcement through general dissolution of the protective passive layer. The contamination of concrete through the ingress of chloride ions, from a variety of sources such as de-icing salts, marine environments and contaminated water results in a more localized breakdown of the passive layer leading to de-passivation of the steel through pitting corrosion. However, for either corrosion processes to take place and be sustained sufficient concentrations of moisture and oxygen are required at the areas where the passive film no longer protects the embedded steel reinforcement. The corrosion process of reinforcement steel can be simplified into a two-stage process namely (Tuutti, 1982), the 'initiation phase' and the 'propagation phase' as shown in the Fig. 1A below.

The first phase corresponds to the initiation time, t_o, taken for chlorides or CO_2, to penetrate the concrete cover in sufficient quantities to destroy the passive film (depassivation).

The second phase covers the period of active corrosion from t_o to the time at which safety or durability of the structure are affected (loss of load bearing capacity, spalling or delamination). The length of this period is determined by the rate of corrosion (governed by the oxygen availability, relative humidity and temperature) and the ability of the concrete cover to withstand internal stresses. In this case, surface treatment and inhibitor could be used to increase the time of both phases by reducing the rate of ingress of chlorides or CO_2 from the environment, increasing the degree of chloride binding, or increasing

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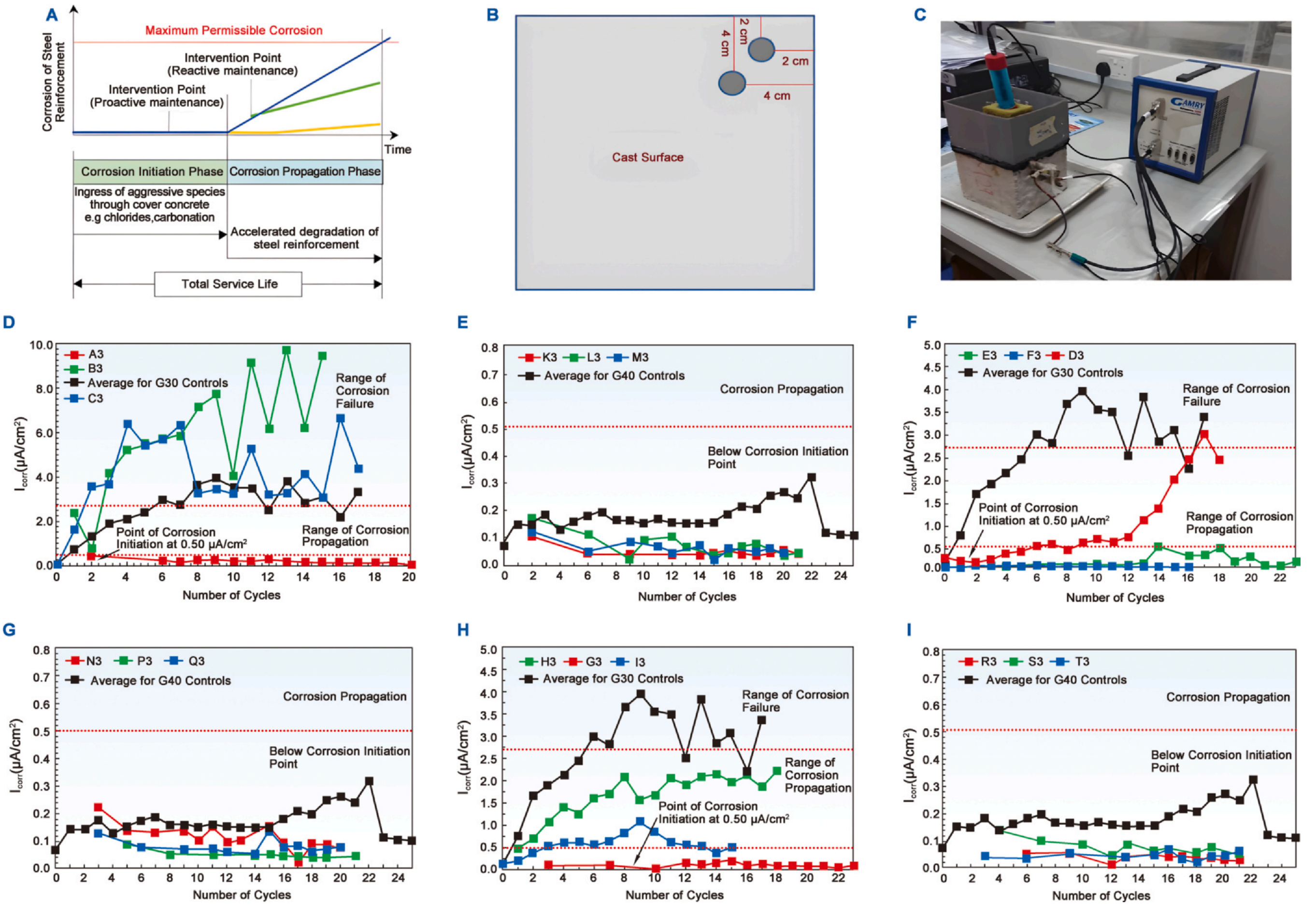

Fig. 1. (A) Simplified corrosion model (after Tuutti); (B) Top view of the concrete specimen during the casting process; (C) Illustration of the testing process with the potentiostat device; (D) G30 Corrosion current density–Category I; (E) G40 Corrosion current density–Category I; (F) G30 Corrosion current density–Category II; (G) G40 Corrosion current density–Category II; (H) G30 Corrosion current density–Category III; (I) G40 Corrosion current density–Category III.

the rate of ingress of dissolved oxygen to sustain the cathodic reaction and the electrical resistance of the concrete, etc.

Surface treatments are grouped into three types according to the BS EN 1504–2: 2004.

(i) Impregnations reduce the surface porosity by filling totally or partially the concrete pores;
(ii) Hydrophobic Impregnations produce a water repellent surface that generally has no pore filling effect;
(iii) Surface Applied Corrosion Inhibitors form a protective film on the rebar surface.

In the first type of impregnations, the reaction product can block the pores, strengthening the concrete surface (Ibrahim, Al-Gahtani, Maslehuddin, & Almusallam, 1997). The underlying basis of the impregnation mechanism is to utilize pore blockers to act as a physical barrier that reduces the ingress of aggressive agents (chloride ions and water/moisture). Molecules of the employed surface treatment chemicals are able to penetrate the surface into the concrete pores and react with the hydration products of concrete, the product of which will fill up the existing voids in the concrete's pore system, thus reducing the surface porosity.

Hydrophobic impregnation mechanism relies on the production of a hydrophobic and water-repellent surface that seeks to minimise the ingress of water (Johansson & Janz, 2009). With such a surface, water molecules that find their way onto the surface will exhibit the characteristic large contact angle it makes with the surface. Silanes, siloxanes and similar substances function by penetrating concrete pores, forming a hydrophobic layer, thus inhibiting penetration by water in liquid form (which may be contaminated with chloride), but allowing water vapour to enter and exit, allowing concrete to "breathe" freely.

Migrating corrosion inhibitors can repair the corroded steel when applied on the concrete surface without damaging the concrete (Mammoliti et al., 1999). The application of corrosion inhibitors onto the concrete surface involves the transport of the corrosion inhibitors to the rebar where it has to reach a sufficiently high concentration to protect steel against corrosion or reduce the rate of the ongoing corrosion. When corrosion inhibitor migrates to the steel surface, a monomolecular film is formed on the steel surface. This additional layer of protection, prevents the steel from further corrosion. In addition to this, the salting-forming component of the corrosion inhibitor can react with calcium hydroxide resulting in a gel layer blocking the pores of the concrete surface. However, currently there is no standard performance test found in literature for Surface Applied Corrosion Inhibitors.

This study focused on accelerated corrosion test used for determination of the effectiveness of each category in offering corrosion protection by subjecting concrete specimens coated with these surface treatments to accelerated corrosion condition that is intended to induce corrosion in the embedded rebars. The performance of each category I, II and III shall be compared to determine the most effective category for resistance against corrosion. The accelerated corrosion test shall be performed on grade G30 and G40 concrete based on LTA mix design. The test specimen shall be subjected to wet and dry cycle until the termination condition is satisfied. Polarization resistance and corrosion current density are determined using linear polarization resistance measurement (ASTM G59–97:2014). Corrosion current density values shall serve as a gauge of the extent of corrosion. Based on the

Table 1
Mix designs for G30 and G40 Concrete Grades.

Type	Cement	Coarse Agg 20 mm	Fine Agg	Water	Admix 1	Admis 2	Slump
G40 PBFC	400	900	890	160	Daratard P88 0.45%	Daracem Super 21 M 1.58%	160 – 210 mm
	Remarks: Actual amount of admixture will be adjusted to meet the slump						
G30 OPC	380	875	945	190	N.A	Daracem super 21 M 0.21$	90 – 100 mm

Remarks: Actual amount of admixture will be adjusted to meet the slump

recommended guidelines from an ACI committee report (ACI 365.1R-00), specific corrosion current density values have been selected to represent different stages of corrosion at 0.5 μA/cm^2 corresponds to initiation stage, between 0.5 and 2.7 μA/cm^2 for propagation stage and above 2.7 μA/cm^2 for failure stage.

2. Materials and methods

2.1. Experimental methodology

For accelerated corrosion tests, corrosion monitoring will proceed for the specimens of G30 and G40 concrete grades with their mix designs displayed in Table 1.

During the casting process, two reinforcement bars will be positioned vertically at a distance of 2 cm and 4 cm away from one of the corners of the concrete cube, such that they will be protruding outwards from the cast surface. The top view of the cast surface shown in Fig. 1B will help to illustrate the rebars' orientation. A square panel was used as a guiding tool to ensure that the positioning of the rebars during the casting process are kept constant.

The rebar used for this setup is 180 mm in total length, with 110 mm of it embedded within the concrete and the remaining 70 mm protruding out from the cast surface. Copper wires are also electrically connected to the exposed portion of the rebar so as to facilitate sufficient electrical connectivity for the electrochemical measurements that will be taken. For the exposed portion of the rebar, a layer of epoxy will be applied to prevent the rebar from reacting with the environment. Care has also been taken to ensure that the epoxy coating will not cause electrically connectivity issues for the connecting wires as they will be soldered directly onto the exposed portion of the rebar.

After the casting process, the positions of the rebars will be fixed according to the orientation mentioned above. The concrete specimens will then undergo the usual 28 days of curing to allow them to attain the desired structural properties. However, during the curing stage, there will be inevitable exposure of the rebars towards water and moisture since the concrete specimens are in a saturated state. Upon the completion of the curing process, the specimens will then undergo 7 days of drying before the products are applied onto the surface above the rebars. The products are also allowed to dry for 7 days before we commence with the wet and dry cycles.

Prior to the start of the wet and dry cycles, the same surface will also be fitted with a salt water reservoir that will be housing 10% NaCl (salt solution) during the wet and dry cycles (as shown in Fig.1C). Each wet and dry cycle will constitute 24 h of a wetting period (saltwater reservoir is filled with saltwater solution) at 50 ˚C in a heating oven, and 24 h of a drying period (salt water reservoir is emptied and no longer contain NaCl solution) at 50 ˚C in a heating oven as well.

2.2. Materials

In general, the following general criteria and principle will be used as a guidance in order to select the suitable types and products from market for this study.

1) The product will need to have a bonding to the concrete surface via some chemical reactions with the cement products in the capillary pores to ensure the product will not delaminate throughout its entire service life. For impregnation product, only those having reaction with the cement products to form the insoluble crystal will be considered in this study.
2) For hydrophobic impregnation and migrating corrosion inhibitors products, depth of penetration that is reported or claimed by the supplier will be considered as one of the criteria. Following the practice adopted by APAS, as guideline, for Silane based hydrophobic impregnation, the minimum depth of penetration will be set at 5 mm and for Siloxane based hydrophobic impregnation at 3 mm. For the migrating corrosion inhibitors, depth of penetration of 65 mm will be used as the minimum by considering the position of 25 mm diameter rebar with 40 mm concrete cover.
3) The product is environmentally friendly, and should not cause any safety and health hazard (including fire hazard) during the application, curing and throughout its entire service life. Water based solution will be preferred or if solvent based, the volatile organic matter must be below acceptable level (below 350 g/L).
4) In terms of flammability, we will use Globally Harmonized System (GHS) of classification and labelling of chemicals liquid flammability criteria as a guideline. The product selected in this project must be at least category 3 and 4, which is not classified as 'dangerous' (extremely or highly flammable).
5) The product should not be sensitive to disturbance (for example, the vibration or the suction wind during the application and curing). In this case, product with fast drying and curing time is preferred. Cement based product like powder-based crystallization additive which requires curing for more than 24 h to ensure good bonding with the substrate is not preferred.
6) Product application method should be suitable for tunnel environment (simple application by brush, roller or low-pressure spray and the product can be used for vertical and overhead application)
7) The product allows simple surface preparation and cleaning (with water spray, brush, etc.). without compromising its performance during its service life.
8) Product that has track record to be used in the tunnel or other infrastructure project with the same objective of reducing the chloride ingress and minimizing the risk of corrosion of reinforcement steel in the concrete will be preferred. It has been found from several literatures that surface applied hydrophobic impregnation is the most common method recommended to reduce the risk of corrosion due to chloride ingress in many infrastructure projects that is exposed to atmospheric agents (bridges, marine structures, etc.).

A total of 9 products in 3 categories were selected for this study as shown in Table 2.

To facilitate easier reference to the specimens under investigation, the identification of each specimen is tabulated and shown in Table 3 below. The specimens will have the wet and dry cycles terminated at the different corrosion milestones whenever possible, else they would be spaced reasonably apart to provide for a meaningful analysis.

3. Results and discussion

3.1. Corrosion current density for concrete coated with category I products

For 3 products of category I, the corrosion current density for product 1 (A3), product 2 (B3) and product 3 (C3) were compared to the average

Table 2
9 Products Selected for the Study.

Category	Product Name	Product Content Information
Impregnation	Product 1	Silicate-Based Impregnator Soluble in water Viscosity: $<$ 100 cps Density: 1.20 – 1.25 g/mL Water penetration: $<$ 6 mm Water absorption: 1.7%
	Product 2	Sodium Silicate [Xi] Soluble in water Density: 1.150 – 1.170 g/mL
	Product 3	Silica particles in alkaline water Chemical composition: SiO_2 in water pH: 7 – 11 Density: 1 g/cc Viscosity: $<$ 50 mPa.s Particle size: 5 nm Molecular Size: 5 nm
Hydrophobic Impregnation	Product 1	Chemical composition: isobutyltriethoxysilane Density: 0.88 g/mL
	Product 2	Chemical composition: silane-siloxane Colour: Pale Yellowish Siloxane Content: 8 % – 10% Density: 1–1.05 g/cm^3
	Product 3	Colour: Yellowish white Density: 0.9 g/cm^3 Solubility: Idrosolubile
Surface applied corrosion inhibitors	Product 1	Chemical composition: alkoxylated, Triethoxyoctylsilane Density: 0.870 g/cm^3 Dynamic Viscosity: 84000 mPa.s
	Product 2	Chemical composition: 2-aminoethanol Colour: Clear, Yellow pH: 10.8 Density: 1.14 g/cm^3 Soluble in water Kinematic Viscosity: $>$ 20.5 mm^2/s
	Product 3	pH: 9.0 – 9.7 Density: 1.03 – 1.05 kg/L

G30 control as shown in Fig.1D. With the exception of A3, the corrosion current density plots of the B2 and C3 show that the rise in corrosion current density is almost at the same rate or even faster than that of the G30 controls, signifying that these products are unable to provide corrosion protection onto concrete surface. As such, only product 1 within this category, is able to withstand the harsh conditions induced by the wet and dry cycles and is effective towards corrosion protection.

To compare all three Category I products performance against the controls, the corrosion current density for product 1 (K3), product 2 (L3) and product 3 (M3) were compared to the average G40 control as shown in Fig. 1E. The corrosion current density for all 3 products is slightly lower than that of the G40 controls, and the generalised findings are that corrosion has not been initiated in G40 concrete specimens in the cases for both with and without products at least when Category I products are concerned. It is difficult to determine if such low corrosion current densities are attributable to the effectiveness of the product or the properties of G40 as a concrete grade.

3.2. Corrosion current density for concrete coated with category II products

Category II products performance against the controls, the corrosion current density for product 1 (D3), product 2 (E3) and product 3 (F3) were compared to the average G30 control as shown in Fig. 1F. It is evident that the working mechanism of the hydrophobic impregnation is able to induce a significant reduction in corrosion current density. Corrosion initiation is not even observed for 2 of the products (E3 and F3) as all 3 products are able to withstand the harsh conditions of the accelerated corrosion setup at least up till the 10th wet and dry cycle. As an overview of the Category, the corrosion current densities do not rise as significantly as they did for Category I products.

For the hydrophobic impregnation products on G40 concrete, there is generally also a slight decrease in corrosion current density as compared to the G40 controls. The corrosion current density for G40 concrete specimen coated with product 1 (N3), product 2 (P3) and product 3 (Q3) were compared to the average G40 control as shown in Fig.1G. Application of the hydrophobic impregnation products may have helped to lower the corrosion current density marginally to be below that of the controls, but the trends exhibited by the G30 specimens coated with products are not observable within G40′s corrosion monitoring.

3.3. Corrosion current density for concrete coated with category III products

For Category III products the performance against the G30 concrete controls, the corrosion current density for mixed mechanism product 1 (hydrophobic & MCI) (G3), MCI product 2 (H3) and MCI product 3 (I3) were compared to the average G30 control as shown in Fig. 1H. Corrosion initiation has been observed for the pure MCI products (H3 and I3) at the 1st and 2nd cycle while the mixed mechanism product (G3) maintains a corrosion current density value below 0.5 μA/cm^2 throughout the cycles up till the 23rd cycle. Further study on the penetration depth of MCI products should be carried out to verify the effectiveness of MCI products.

For category III products on G40 concrete, a slight reduction in corrosion current density is also observable (Fig.1H). However, just like in other categories, quantifying the effectiveness is not meaningful as all of the values are already below 0.5 μA/cm^2. Also, due to the

Table 3
Identification Reference for G30 and G40 Specimens.

G30 Concrete		Accelerated Corrosion Test Stages			G40 Concrete		Accelerated Corrosion Test Stages		
	Products	Corrosion Initiation	Propagation	Failure		Products	Corrosion Initiation	Propagation	Failure
Category (I) Impregnation	1	A1	A2	A3	Category (I) Impregnation	1	K1	K2	K3
	2	B1	B2	B3		2	L1	L2	L3
	3	C1	C2	C3		3	M1	M2	M3
Category (II) Hydrophobic	1	D1	D2	D3	Category (II) Hydrophobic	1	N1	N2	N3
	2	E1	E2	E3		2	P1	P2	P3
	3	F1	F2	F3		3	Q1	Q2	Q3
Category (III) MIC & Combination	1	G1	G2	G3	Category (III) MIC & Combination	1	R1	R2	R3
	2	H1	H2	H3		2	S1	S2	S3
	3	I1	I2	I3		3	T1	T2	T3
Control	-	J1	J2	J3	Control	-	U1	U2	U3

instantaneous nature of corrosion current measurements, slight variations may occur for the recorded readings. Hence the insignificant reduction could also be misleading if we consider the variations in the measured readings.

4. Discussion

For G40 concrete specimens, the recorded corrosion current density values are virtually below.

0.50 $\mu A/cm^2$ with most even registering a very low value that is consistently under 0.10 $\mu A/cm^2$. This was also the case for the G40 controls, barring specimen U3. The issue encountered for G40 specimens is mostly due to the practical difficulties encountered upon the application of the corrosion current density monitoring method for higher resistivity concrete. Some assumptions that are made during corrosion current density measurement include the estimation of the probable Ohmic drop resulting from the relatively high electrical resistivity of concrete as well as an approximation of the area of the rebar that is actually being polarized. To reduce the uncertainties involved in determining a more accurate corrosion current density, an improved or better estimation of I corr from a more accurate measurement of R p taking into consideration the Ohmic drop compensation and the area of rebar actually polarized are required. Thus, for these reasons stated above, namely having adequate compensation for the Ohmic drop as well as a more accurate determination of the area of the rebar that is undergoing polarisation, the corrosion current density monitoring technique may face more obstacles in their measurements when the method has been used for concrete grades with higher concrete resistivity. As an overview however, there's little to distinguish between the various specimens among G40 concrete as the differences between corrosion current density values are way too insignificant to have any conclusive deductions made. Thus, if anything, conclusions are more likely to be drawn from G30 specimens due to the more distinct trend lines that have been obtained from them.

5. Conclusions

Based on the review of the G30 results shown previously, Category I can be considered to be ineffective in corrosion resistance as 2 out of the 3 products are unable to cause a significant reduction as compared to the controls for G30 concrete. In fact, for two out of the three Category I products, they fare very similarly or are even worse than the controls, indicating almost negligible corrosion resisting ability.

Given the dual mechanism nature of product 1 of Category III, it can also be considered to be a part of the products employing hydrophobic impregnation mechanism as well since active reduction of water, oxygen and chloride ions is taking place. Therefore, the findings seem to be suggesting that products which employ the hydrophobic impregnation mechanism (including Category III Product 1) have displayed the best ability to resist corrosion for both G30 and G40 concrete.

Another key finding is a higher grade concrete with higher density and lower porosity is able to delay the onset of corrosion significantly. However, this is not tantamount to saying that G40 concrete does not face the threat of corrosion. Rather it is more appropriate to say that the timeline of the study was not able to adequately capture the initiation of corrosion in G40 concrete. With more wet and dry cycles, it may be that significant corrosion will be observed even if the corrosion current density remains consistently low.

Declaration of interests

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: LU Jinping reports was provided by Hitchins International Pte Ltd. Lu Jinping reports a relationship with American Concrete Institute (Singapore Chapter) that includes: board membership. Lu Jinping has patent pending to Lu Jinping. Sam Leng Choon Kim is employed by Hitchins International Pte Ltd.

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