



CIVIL ENGINEERING

Quantitative estimation on delaying of onset of corrosion of rebar in surface treated concrete using sealers

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Abstract Surface treatment on the concrete surface using sealers reduces the rate of permeability of chloride and moisture through the concrete. The delaying of onset of corrosion is evaluated for surface treated and untreated concrete using electrochemical techniques. After conducting rapid chloride permeability test (RCPT), using Nernst–Plank equation, the diffusion coefficient of chloride (D_{eff}) is calculated. Substituting threshold chloride concentration of rebar (C_{th}) from cyclic polarization test in the Fick's second law, the time to initiation of corrosion (T_i) is arrived. From the results it is found that the treated concrete with alkyltrialkoxysilane sealer delays the onset of corrosion by four times than that of untreated concrete.

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1. Introduction

Reinforced concrete is an economical, versatile and highly successful construction material used to build concrete structures. The concrete structures such as bridges, offshore platforms, tunnels, high pressure reaction vessel in the nuclear industry are constructed to perform in hostile environments. All of

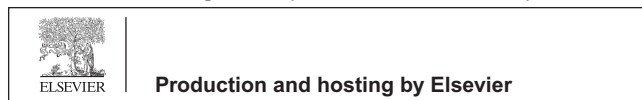
them are subjected to severe environmental conditions throughout its service life for which they are intended. In such situations, structures need to satisfy two basic requirements such as strength and durability. These two may not always be obtained with the techniques and materials adopted for producing conventional concrete. Aggressive ions such as chloride, sulphate, carbon-di-oxide and other chemicals present in the industrial environment permeate through the concrete and corrodes the rebar as well as deteriorates the concrete simultaneously [1–6]. Because of higher shrinkage and heat of hydration the permeability of concrete is not completely ceased even in high strength concrete but its rate gets reduced. The durability of the concrete can be enhanced by making the surface comparatively impervious than bulk.

Addition of mineral admixtures such as fly ash, silicafume, slag in concrete reduces the permeability of bulk concrete by pore densification after forming additional cement hydrates through pozzolanic reaction [7–9]. For reducing the permeabil-

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ity, sealers are widely used on the concrete surface [10–14]. The purpose of concrete sealing is to slow down the rate of chloride and water intrusion into the concrete, providing better corrosion protection for the steel reinforcement. It also provides an additional protection for the portions of bridge deck with increased permeability due to shrinkage cracking (or) increased w/c ratio. The sealer is also expected to provide resistance to gasoline, diesel, ultraviolet light, mild chemical exposures and so on [15]. These sealers are either oil or silane–siloxane based and reduce the permeability up to the depth of 5–10 mm from the top surface. Oil based sealers block the pores of the concrete (e.g.) linseed oil, water/solvent based epoxy. These pore blockers provide little penetration and form a thin film on the concrete surface. The drawbacks of linseed oil are poor frictional properties and longer curing time. It is recommended to reduce the scaling resistance of new constructions. On the other hand, silane based sealers are also called as penetrating sealers chemically react with the concrete surface i.e., polymerize and form a hydrophobic layer under the concrete surface that repels the water and chloride ions (e.g.) silanes and siloxanes, silicones and soluble reactive silicates. The essential requirements of good sealers are short curing time, must allow the concrete to breathe, the color of the concrete should not change after drying and easy to apply. National Cooperative Highway Research Program (NCHRP) study [16] found that concrete coated with silane had a water absorption value that was 30% of untreated concrete. Aitken and Litvan [17] evaluated 57 types of sealers of 12 generic groups on eight types of concrete surface. They found that most of the sealers did not penetrate the substrate to any measurable extent and significant penetration was obtained only with sealers of alkoxysilanes and oligomeric alkyl alkoxy siloxanes. Thompson et al. [18] reported that sealing concrete surface with soluble sodium silicate improved the surface properties such as hardness, permeability, chemical durability and abrasion resistance. Hagen [19] studied the field performance of 16 types of concrete sealers on actual concrete bridge decks. After 3 years of field test, he found that water based silane, solvent based silane and siloxane performed better than the others. After conducting accelerated corrosion test, Ibrahim et al. [20] found that silane, silane–siloxane with top coat and acrylic coating were effective in reducing the rate of reinforcement corrosion. Four types of penetrating sealers of three generic groups were tested against linseed oil to rate their effectiveness by conducting accelerated tests (90-days ponding test, rapid chloride permeability test) in the laboratory [21]. The studies concluded that solvent based silane and water soluble acrylic latex performed better whereas soluble reactive silicates performed worst. The reported investigations on the performance of water based silane–siloxane penetration sealers on the concrete surface is less.

The main objective of the present study is to estimate the delaying of onset of corrosion of rebar embedded in surface treated concrete quantitatively. Three types of sealers were evaluated and their effectiveness were compared with the untreated concrete. Sealers which can be either added during the casting of concrete or applied on concrete surface after hardening of concrete were taken for evaluation studies.

2. Materials and methods

2.1. Cement and aggregates

Ordinary Portland cement conforming to IS 8112 was used [22]. River sand and crushed blue granite of high quality were chosen as fine and coarse aggregates respectively. The different size fractions of coarse aggregate (16 mm downgraded and 12.5 mm downgraded) were taken and mixed to get a specified grading. The fine aggregate was sieved through a 4.75 mm sieve. The fineness modulus of coarse aggregate and fine aggregate was 7.78 and 2.72 respectively. High yield strength thermo mechanically treated (TMT) bars of Fe 415 grade conforming to IS: 1786 [23] was used as rebar. Table 1 shows the chemical composition of the steel rebar used.

2.2. Preparation of concrete specimen and method of measurement

2.2.1. Rapid chloride permeability test (RCPT)

Concrete mix proportion of 1:1.871:3.292 with w/c ratio of 0.5 was used for casting the concrete specimen. The characteristic compressive strength of concrete is 29 MPa after 28 days of curing. Concrete disk of size 100 mm diameter with 50 mm thick was cast to conduct this test. The specimens were cured in potable water for 28 days. After curing, the specimens were dried at room temperature for 7 days. After drying, the surface was cleaned using emery sheet to remove the dust. The circumferential area of the disk was sealed using epoxy resin and thus allows the diffusion of chloride through one direction only. Sealer type I was added during the casting of concrete whereas sealer types II and III was applied on the one side of the disk by brush as per manufacturer's recommendation as given in Table 2. The second coat was applied in 24 h interval. Then the specimens were allowed to cure at ambient temperature for 7 days which allows polymerization to take place on concrete surface. The RCP test was carried out as per the procedure mentioned in ASTM C1202-10 [24]. The disk was fixed between upstream and downstream chamber as shown in Fig. 1 in such a way that the surface treated with sealer was fixed in the upstream chamber containing chloride whereas the untreated surface was fixed with the downstream chamber without chloride. A DC potential of 60 V was applied between the cells for a duration of 6 h. Metal–metal oxide coated titanium was used as electrodes for impressing the voltage. The chloride ion permeated from the upstream chamber to downstream through the concrete was calculated in coulombs as per the following equation:

$$Q = 900(I_0 + 2I_{30} + 2I_{60} + \dots + 2I_{300} + 2I_{330} + I_{360}) \quad (1)$$

where Q charge passed C, I_0 Initial current after voltage is applied A and I_t current at time min after voltage is applied A .

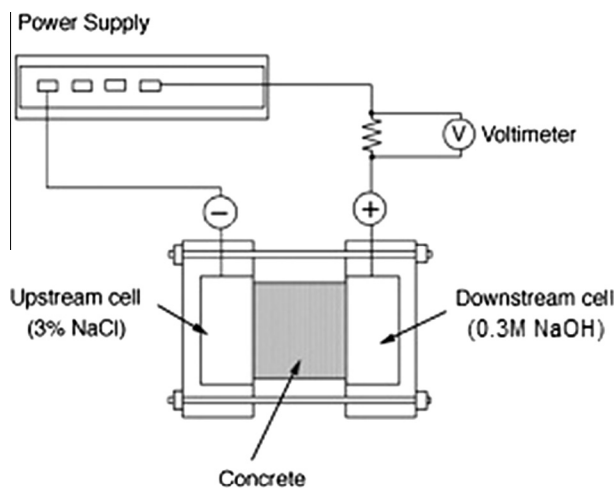
The specimen diameter used in this study is 100 mm which is other than 95 as recommended in C 1202 and hence the above value has been corrected using the following formulae.

Table 1 Chemical composition of steel rebar.

Elements	C	Si	Mn	P	S	Cr	Cu	Fe
Value	0.205	0.193	0.540	0.056	0.024	0.080	0.125	98.60

Table 2 Specification of three types of sealers.

Properties	Sealer type I	Sealer type II	Sealer type III
Commercial name	Water proofing admixture	Aqueous silane siloxane based emulsion	Thixotropic silane and siloxane water repellent cream
Generic type	Silicone ester	Silicone ester	Isobutyltriethoxysilane
Appearance	Milky white liquid	Milky white liquid	White cream
Odour	Slight alcoholic	–	–
pH	7	6.5–7	7–8
Solubility in water	Miscible	Miscible	Miscible
Density	0.95	0.95	0.91
Dosage	125 ml for 50 kg of cement	200 ml/m ²	200 ml/m ²
Reduced water absorption, %	90	95	98.3
Method of application	Added to the concrete	Coated on concrete surface	Coated on concrete surface

**Figure 1** Rapid chloride penetration test setup.

$$Q_s = Q_x \times \left(\frac{3.75}{x}\right)^2 \quad (2)$$

where Q_s – charge passed (coulombs) through 3.75 in. diameter concrete disk, Q_x – charge passed (coulombs) through 4.13 in. diameter concrete disk, x –diameter in mm of the non-standard specimen. After calculating the charge passed, the values were correlated to chloride ion permeability as given in ASTM C1202-10 which is reproduced in Table 3.

2.2.2. Calculation of diffusion coefficient (D_{eff})

At the end of RCP test, the specimens were split into two pieces and then chloride penetration depth was measured by

Table 3 Chloride-ion permeability test as per ASTM.

Charge passed (Coulombs)	Chloride ion penetrability
> 4000	High
2000–4000	Moderate
1000–2000	Low
100–1000	Very low
< 100	Negligible

the colorimetric method. After spraying 0.1 N silver chloride solution ($c_d \sim 0.07$ N) across the depth of concrete, the depth up to which change in color was measured and that value was taken as x_d . As the experiment was conducted only 6 h the binding of chloride is not possible. Hence the non-steady state migration diffusion (D_{nssm}) coefficient was calculated using the modified Nernst–Plank equation [25].

$$D_{nssm} = \frac{RTL}{zFU} \frac{x_d - \alpha\sqrt{x_d}}{t_d} \quad (3)$$

where D_{nssm} is the non-steady state migration diffusion coefficients m²/s, x_d the penetration depth of chloride ions m, t_d the test duration, and α is the test constant calculated by the following equation.

$$\alpha = 2\sqrt{\frac{RTL}{zFU}} \operatorname{erf}^{-1} \left(1 - \frac{2c_d}{c_0}\right) \quad (4)$$

where R is the gas constant 8.314 J/K mol, T the temperature of the upstream test solution K, L the specimen thickness m, z the ionic valence of chloride ions, F the Faraday constant 96,500 C/mol, U the applied potential V and c_0 and c_d are the chloride concentration in the upstream solution and at the chloride penetration front respectively.

The D_{nssm} as calculated from Eq. (3) cannot be directly used to calculate the effective diffusivity, therefore it need to be converted. According to Tang [26], it can be obtained from the following equation.

$$D_{eff} = D_{nssm}(\Phi + K_b W_{hyd}) \quad (5)$$

where Φ is the total porosity per total volume of concrete, K_b is the chloride binding constant and W_{hyd} is the weight of hydration products per unit volume of concrete kg/m³. The value of K_b is assumed based on the experimental data reported by Tang [27] and Delagrave et al. [28]. Similarly the value of W_{hyd} has been assumed as 364 kg/m³ and calculated from the stoichiometry of the cement hydration reactions [29]. The total porosity of the concrete was estimated using oven-drying method as described in Ref. [30].

2.2.3. Determination of chloride tolerable limit

The tolerable limit of chloride is an important parameter to estimate the delaying of onset of corrosion quantitatively. The TMT rebars used in the present study may have higher

tolerable limit and the limit of 0.4% by weight of cement as specified in CEB, ACI [31,32] cannot be used. To estimate this, cyclic polarization was carried out. For this, cylindrical concrete specimens having a size of 66 mm dia \times 140 mm height were cast. At the center of the specimen, 16 mm diameter TMT rebar having a length of 8 cm was embedded. The clear cover of the rebar is 25 mm. Before embedding the rebar, the initial rust was removed by acid pickling. At one of the ends, copper wire was soldered to give electrical connection and that was sealed with heat shrinkable sleeves. Both top and bottom ends were sealed using epoxy resin by leaving an exposed area of 7 cm. During the casting of concrete 0.4%, 0.5%, 0.6% and 1.0% chloride by weight of cement was added. After demoulding, the specimens were cured in potable water containing the same amount of chloride which was added inside the concrete to avoid leaching of added chloride during curing. After 3 days of curing, the cyclic polarization was carried out, in the potential range of -100 mV cathodic to 750 mV anodic from OCP at the scan rate of 120 mV/min. IR compensation was done using the AC resistance method at the frequency of 1000 Hz with an AC amplitude of 20 mV. The experiment was conducted using ACM-GILL AC (Advanced Corrosion Measurements, UK) that was operated by the ACM sequencer/core running/analysis V3 combined software. During the measurement to avoid leaching of OH^- and Cl^- ions from the concrete it was kept immersed in the solution containing 0.04 N NaOH with the same amount of chloride which was added inside the concrete. Platinized titanium cylindrical mesh of size 66 dia \times 70 mm height was used as a counter electrode and saturated calomel electrode was used as a reference electrode. The potential at which current tends to increase has been taken as E_{pit} and experiment was continued up to one decade of current to flow and then reverse polarization was carried out. After conducting the experiment, the concrete specimen was broken open immediately and visually examined for any pits on the rebar. The above experiment was conducted on rebar embedded in untreated concrete only.

2.2.4. Impressed voltage test

Time to cracking of rebar in surface treated/untreated was determined by conducting 3 V impressed voltage test. It is an accelerated electrochemical corrosion test. Concrete specimens were prepared as described in Section 2.2.3 without adding any chloride. The curing was done for a period of 28 days. The specimens were kept immersed in 3.5% chloride solution and a DC potential of 3 V was applied as shown in Fig. 2a and b. Perforated stainless steel electrode was used as a counter electrode. The current was measured periodically and the test was continued till the cracks appeared on the concrete surface. During the test, the concrete specimens were examined periodically for cracks on the outer surface using a magnifying lens.

2.2.5. Corrosion rate

The average current measured during the experimental period for each system was converted into corrosion rate using Faraday's law as below;

$$\text{Loss in weight, } g = \frac{MIt}{zF} \quad (6)$$

where M is the mass of iron g/mol, I is the current A, T is the time days, Z is number of ions involve in the reaction and F is the Faraday's constant $96,500$ C/mol.

$$\text{Corrosion rate, mmpy} = \frac{\text{weight loss} \times 365 \times 10}{\text{Area} \times \text{Density} \times \text{Days}} \quad (7)$$

2.2.6. Determination of time to initiation of corrosion (T_i)

Using Fick's second law, T_i was estimated as below

$$T_i = \frac{x^2}{4D_{\text{eff}} \text{erfc} \left(1 - \frac{c_x}{c_s}\right)^2} \quad (8)$$

where T_i time for chloride to reach $C_x(x, t)$ at cover depth x secs, x cover of concrete cm, D_{eff} diffusion coefficient of chloride m^2/s , C_s surface chloride concentration percentage by weight of cement, C_x threshold chloride concentration at which corrosion initiates on the rebar, percentage by weight of cement.

3. Results and discussion

3.1. Rapid chloride permeability test (RCPT)

Fig. 3 compares the current vs. time plot of untreated concrete and surface treated concrete. The magnitude of current indirectly indicates the amount of chloride passed through the concrete from the upstream to downstream chamber. It is clearly evident that the flow of current is maximum in the range of 0.16 – 0.20 mA in untreated concrete, whereas for sealer type III the current is minimum which is in the range of 0.02 – 0.05 mA. It implies that, the current flow is 8 – 10 times lower than that of untreated concrete. Sealers repel both water and chloride which reduces the current abruptly. Fig. 4 compares the coulombs obtained for untreated/treated concrete. As per ASTM C1202, the untreated comes under the category of high chloride ion permeability; sealer types I and II comes under moderate chloride ion permeability; sealer type III comes under "very low chloride ion permeability" respectively. Sealer type III performed better than the other two types of sealer.

3.2. Chloride tolerable limit

Figs. 5 and 6 compare the cyclic polarization curve of TMT rebar embedded in uncontaminated concrete with 0.4% , 0.6% and 1% chloride contaminated concrete. It is shown that an extended passive region exists on the anodic polarization part for rebar in uncontaminated concrete and this region is started from -250 mV to 550 mV. E_{pit} is observed at 575 mV and this value is in close agreement with the value reported by Saricime and Mahallati [33]. In the presence of 0.6% and 1% chloride contaminated concrete, this passive region gets decreased to 0 – 550 mV and 0 – 450 mV respectively, E_{pit} is same both 0% and 0.4% chloride added concrete. But there is a decrease in pitting potential (E_{pit}) from 575 mV to 500 mV when the chloride concentration level is increased from 0% to 0.6% (Table 4). The level of chloride at 0.6% , $E_{\text{pit}} - E_{\text{corr}}$ also get decreased to 810 mV from the value of 985 mV and confirms that breakdown of the passive film is occurred and that level has been taken as threshold concentration. At 1% chloride contaminated concrete, E_{pit} is decreased further and reached to the value of 450 mV. After broke open the specimen pits were observed on the rebar at 0.6% chloride added level on the other hand no pits were observed at 0.4% . The presence of ferrite–martensite structure on the outside radius of the TMT rebar and higher Cr (0.080%) than cold twisted rebar

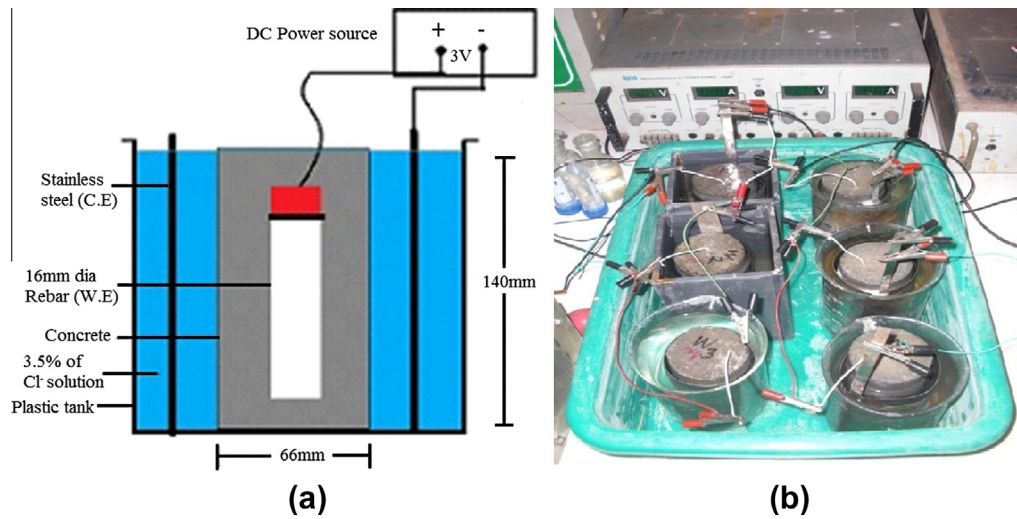


Figure 2 Impressed voltage test setup. (a) Schematic diagram. (b) Impressed voltage test setup.

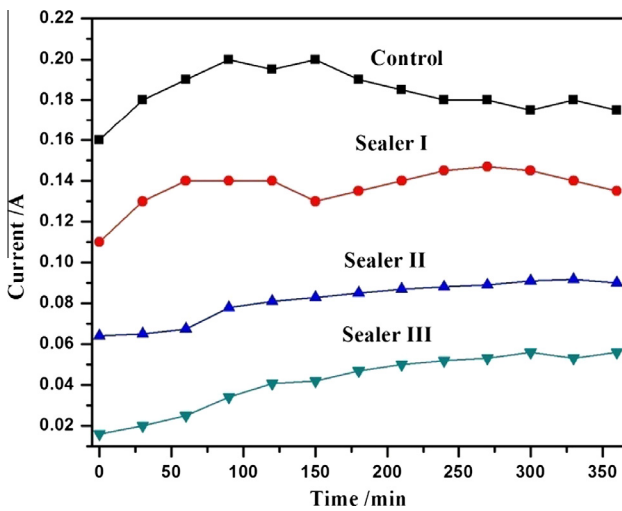


Figure 3 Evolution of current values over time of three sealers during RCPT.

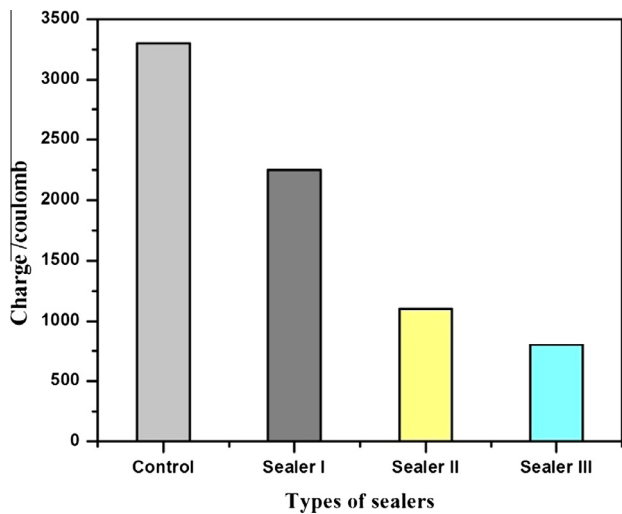


Figure 4 Comparison of coulombs in untreated/treated concrete.

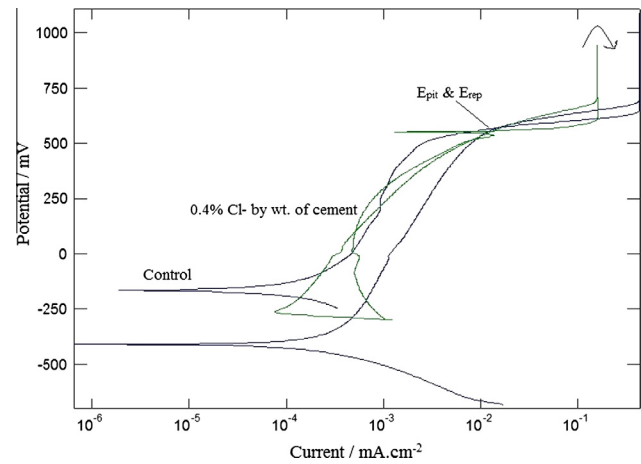


Figure 5 Cyclic polarization curves of TMT rebar in 0% and 0.4% Cl⁻ added concrete.

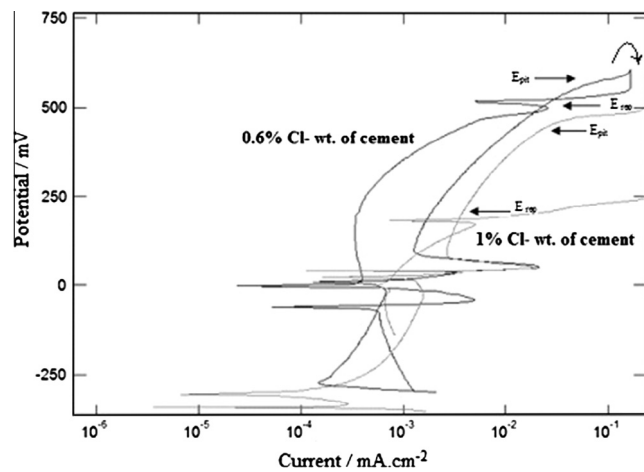


Figure 6 Cyclic polarization curves of TMT rebar in 0.6% and 1.0% Cl⁻ added concrete.

Table 4 E_{corr} and E_{pit} obtained from the cyclic polarization test.

Cl^- wt of cement	E_{corr} , (mV vs. SCE)	E_{pit} , (mV vs. SCE)	$E_{\text{pit}} - E_{\text{corr}}$, (mV vs. SCE)	Visual observation
Control	-410	575	985	No pit observed
0.4	-260	575	835	No pit observed
0.6	-260	550	810	Pits observed
1.0	-320	450	770	Pits observed

(0.04%) increases the tolerable limit of chloride from 0.4 (cold twisted deformed bar) to 0.6% chloride by weight of cement [34].

3.3. Time to initiation of corrosion

The diffusion coefficient of chloride of untreated/treated concrete and as a consequence time to initiation of corrosion of rebar embedded in them is compared in Fig. 7. It shows that sealer type III has very low diffusion rate of chloride 6.8942×10^{-12} and delays the onset of corrosion by 4.5 times compared to that of untreated concrete. It seems that because of higher hydrophobicity created by sealer type III than others, the rate of ingress of chloride is very much reduced.

3.4. Impressed voltage test

The corrosion resistance of rebar in untreated/treated concrete is evaluated in the presence of chloride and moisture. Fig. 8 compares the current flow during the test. The current flow is higher both in untreated concrete and treated concrete with sealer type I which is in the range of 0.5–6.5 mA. In sealer type II and III, the current flow is reduced to the range of 0.5–1.5 mA which is four times lower than that of untreated concrete. Higher the current flow indicates the more amounts of water and chloride permeated through the concrete. It occurs in untreated concrete whereas in treated concrete, the ingress of chloride and moisture is hindered because of its repulsive action.

When the rebar is embedded in concrete, the OH^- ions present inside the concrete forms a passive film $\text{Fe}(\text{OH})$ immediately on the surface as given in Eqs. (11) and (12). When current flows during the test, the negatively charged chloride ions moved towards the positively charged rebar at a faster rate

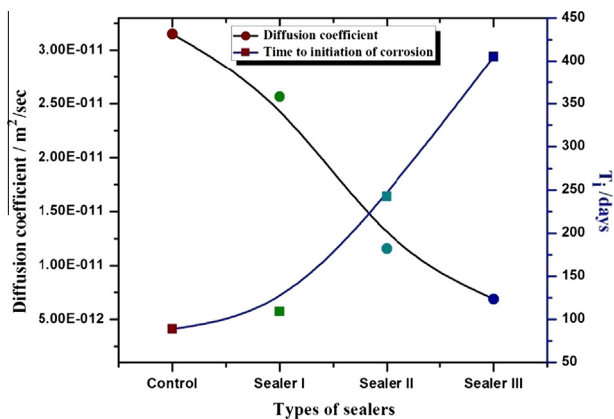
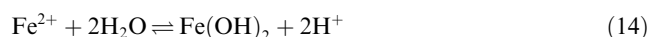
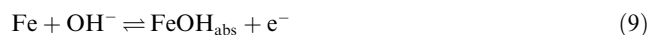


Figure 7 D_{eff} and T_i : comparison between treated with untreated concrete.

along with the water. When the chloride reaches the rebar, it loses one electron and critically forms iron hydroxyl chloride and finally forms ferrous chloride (FeCl_2) as given in Eqs. (11) and (12). In the presence of water, Fe get oxidized to ferrous and ferric hydroxide (FeII and FeIII) complex as given in Eqs. (13)–(15). $\text{Fe}(\text{OH})_2$ is further oxidized to Fe_2O_3 as given in Eq. (16) which is responsible for cracking of concrete because its volume is four times more than that of the rebar



Untreated concrete allows more amount of chloride than treated concrete which increases the dissolution rate of rebar and as a consequence the concrete cracks very earlier i.e., at 21 days. Fig. 9 clearly shows that after 21 days of testing, rust is observed only on untreated concrete surface. The reduced rate of water and chloride ingress in treated concrete reduces the dissolution rate of rebar and takes more time i.e., 60–86 days to crack the concrete. The results conveyed that

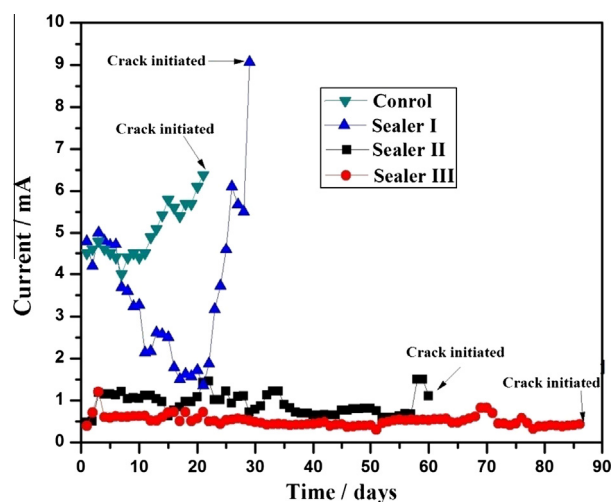


Figure 8 Comparison of current values during impressed voltage test.

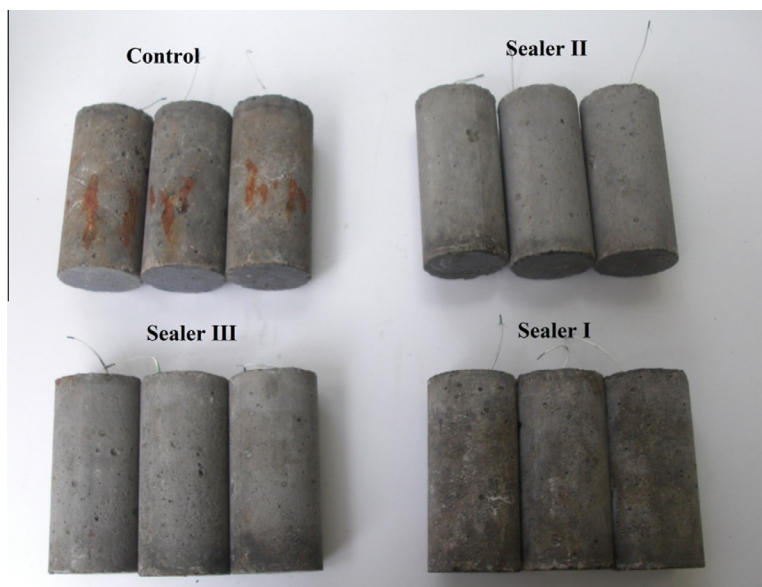


Figure 9 Rusting on concrete surface after conducting 21 days of the impressed voltage test.

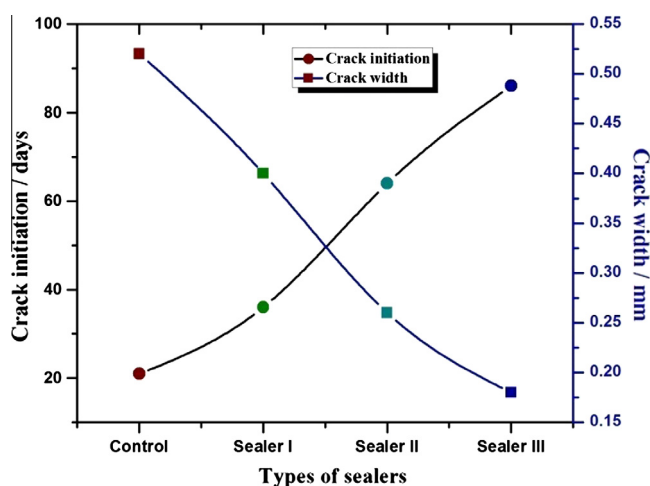


Figure 10 Comparison of time taken to crack in untreated/treated concrete.

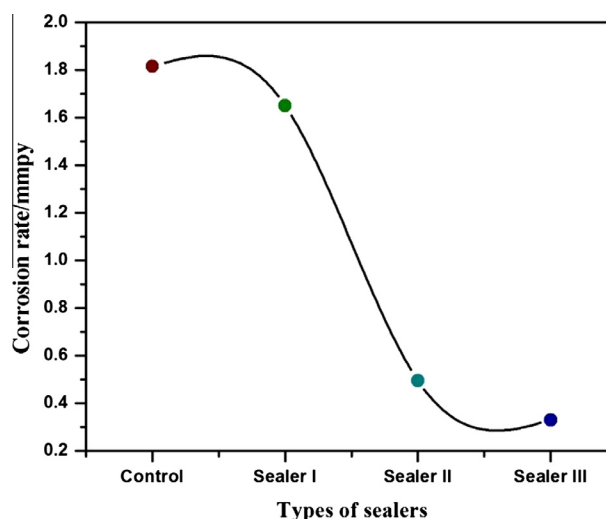
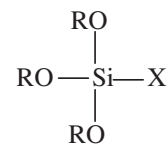


Figure 11 Comparison of corrosion rate of rebar.

the time taken for corrosion induced cracking on surface treated concrete is delayed by four times. Compared to untreated concrete, the crack width also less on treated concrete (Fig. 10). Fig. 11 compares the corrosion rate of rebar in untreated and treated concrete. From the figure, it is understood that rebar in treated concrete with sealer type III is having a lowest corrosion rate of 0.2015 mmpy. From the results it could be concluded that the protection efficiency of the three sealers evaluated is in the order of type III > type II > type I.

From the foregoing discussion, it is found that alkyltri-alkoxy silane performed better than silicone ester group. Silanes are smallest silicone molecules, which can penetrate deep into the concrete surface. The OH^- ions present in the concrete, react with silane and forming a silicone resin network. This reaction makes a strong chemical bond which provided the higher hydrophobicity and get permanently bonded to the substrate

and cannot be washed out. The chemical structure of alkyltri-alkoxy silane is shown below



where -OR is a alkoxy group i.e., methoxy, ethoxy, butoxy, etc., -X is a organic group, such as methyl, ethyl, butyl or octyl, and it provides hydrophobicity. Generally hydrophobicity increase in the order of methyl < ethyl < butyl < octyl [35]. Sealer III is a isobutyltriethoxy silane. The molecular formula is $\text{C}_{10}\text{H}_{24}\text{O}_3\text{Si}$ and molecular weight is very low i.e., 220. The penetration depth in concrete is around 12 mm for sealer type III whereas for sealer type II penetration depth is around

4–6 mm. Sealer types I and II belongs to silicone ester group and having lower hydrophobicity as compared to the alkyltri-alkoxy silane group. Another advantage of water based silane–siloxane is more eco-friendly than solvent based silane–siloxane.

Silanes, siloxanes and silicones produce the same end product, a hydrophobic silica gel. The basic difference between these products lies in the size of the molecules, silanes being the smallest and silicones the largest. The reasons for better performance of sealer type III than type II and I are small size of the silane molecules being able to penetrate deeper into the concrete and higher hydrophobicity due to the presence of alkyl organic group.

4. Conclusion

- i. The presence of the butyl organic group of alkyltri-alkoxy silane based sealer (sealer type III) increase the hydrophobicity thereby repels the chloride and water more effectively than ester group silicone based sealer (sealer type I and II).
- ii. Being the smaller molecular size of silane based sealer able to penetrate deeper into the concrete i.e. up to 12 mm whereas silicone based sealer which can penetrate only up to 4–6 mm.
- iii. Accelerated corrosion tests conducted in 3.5% sodium chloride solution on treated/untreated concrete, the corrosion resistance of the three sealers evaluated is in the order of type III > type II > type I. These water based sealers are environmental friendly than solvent based sealers.
- iv. The reasons as given in (i) and (ii), the surface treated concrete using sealer type III enhances the service life of the concrete by four times than that of untreated concrete during both initiation and propagation period of deterioration process of rebar in concrete structures.

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