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Performance Evaluation of Silane/siloxane Based Penetrating Sealer for Structural Applications in Marine Environment

R.Vedalakshmi S.Sathyanarayanan, S.Sreejakumari and T.Bharathidasan

Corrosion and Materials Protection Division, CSIR-Central Electrochemical Research Institute, Karaikudi-630 003, Tamilnadu.

ABSTRACT

Concrete cracking is considered as the most common type of distress observed throughout the structure and occurs from the stage of hardening itself. In a marine environment where bridges are subjected to seawater spray/splash, cracks of width as small as 0.05 mm could be significantly detrimental and they must be sealed to maintain durability. Penetrating sealers when applied on concrete surface, they form chemical bond with the pore walls, thus change the surface tension of the concrete. In turn, treated surface produces water repellancy and retards the permeation of aggressive ions such as chloride and sulphates enter into the concrete.

Silane and siloxane are common reactive sealers with low viscosity tend to penetrate deeper into the concrete. In the present study, a new silane based sealer has been formulated and tested using standard test codes. Various rigorous tests such as depth of penetration, water absorption and repellancy, pore size reduction (using MIP), chloride penetration (Rapid chloride permeability test) and scanning electron microscopic (SEM) studies were carried out.

From the studies, it can be inferred that developed silane/siloxane sealer penetrated up to 50 mm depth. Vapour transmission rate of sealer treated concrete surface was found to be in the range of 8-10 g/m^2 over 24 hrs and indicated that the sealant remains breathable. The charge passed under RCPT test was 130 coulombs which is 10 times lower than that of untreated concrete. SEM studies showed the formation of acicular crystals throughout the concrete surface and confirmed the reaction of silane with $Ca(OH)_2$. The detailed investigation revealed that concrete treated with low viscous silane /siloxane, performed better by a minimum factor of 10 than that of untreated concrete.

Keywords: penetrating sealer,depth of penetration, low viscosity

INTRODUCTION

Concrete cracking is considered the most common type of deck distress observed throughout the structures. Cracks that may be either transverse, longitudinal or random (1). They are formed excessive tensile strength and reduction in fatique strength of concrete and caused by temperature changes, concrete shrinkage, bending from self-weight and traffic loads (2). The presence of these cracks often leads to eventual structural deficiency as they permit the ingress of harmful substances such as chlorides, moisture and sulphates causing corrosion of rebars. American Concrete Institute (ACI) committee 244 (3) limits crack width to 0.15 mm for structures subjected to seawater wetting and drying. In a harsh environment where concrete bridge decks are subject to sea spray/splash, cracks of width as small as 0.05 mm could be significantly detrimental and they must be sealed to maintain durability (4).

Measures to minimize corrosion activity include application of some type of surface treatment such as penetrating sealer, coating on concrete surfaces and steel rebar, corrosion inhibitors etc., to prevent chloride from contacting the rebar. Even in the absence of chloride, bridge decks that initially experienced shrinkage/thermal cracking would continue to deteriorate due to traffic load and evolved loss in their structural integrity (4). Surface treatment sealers are also known as penetrating sealers, which decrease the overall permeability of concrete, and/or to seal/fill the cracks to prevent the direct intrusion of chloride.

The two most common reactive sealers are siloxanes and silanes. Silanes are functional monomeric Si-compounds with four chemical attachments. These tend to have a low viscosity facilitating penetration into the concrete and quicker chemical reactions. Siloxanes are linear Si-O-Si polymers with a wide range of viscosities (ranging from thin to oil like substances) and may be either reactive or not with concrete, depending on the functionality. 90 % of the chemical reaction between the concrete and the silane sealer occurs within one hour because of the high alkalinity environment of the concrete (pH of 13 - 14).

EXPERIMENTAL PROCEDURE

SILANE RESIN PREPARATION

Siloxane resin was prepared by mixing siloxane and silane at the ratio of 1:1 in toluene using magnetic stirrer for 6 hours. Dibutyl dilaurate (DBT) was used as a catalyst. The viscosity of the resin was adjusted to 5 cst for silane system I and II and 200 cst for system III.

CASTING OF CONCRETE SPECIMENS

Mix proportion of 1:3.07:4.5 with w/c ratio of 0.5 as specified in B388 (5) was used to cast the concrete specimens. After 28 days of immersion curing, the specimens were air dried at room temperature for 2 days. Silane was applied on the concrete surface both by spraying and brushing. Cubical concrete specimens of size100 mm were cast to test the depth of penetration. Whereas concrete discs of size 100 mm diameter and 50 mm height as specified in ASTM C1202 (6) were cast to assess the penetration rate of chloride qualitatively in a rapid way. Breathable nature of sealer was ascertained by measuring vapour transmission rate as per BT 001 (7) on 100 mm size cubical concrete specimen over a period of 10 days.

Sorptivity is the rate of absorption of water as a function of time and done as per ASTM C1585 (8). Cylindrical concrete specimens of size 50 mm height and 100 mm diameter were used to conduct this test. Alkaline resistance of the sealer is important in the case when it applied on the new structure. Cubical concrete specimens of size 100 mm were used for this purpose and test was conducted as per BT 002 (9) in 0.1N KOH for 14 days. Water absorption test was conducted as per ASTM C 642 (10) using cylindrical specimen of size 100 mm diameter and 50 mm depth.

A small piece of concrete cut from the inside of the concrete both treated and untreated concrete (around 10-15 mm depth) was examined by scanning electron microscope. Cubes after conducting depth of penetration test were used for this purpose. Reduction in pore size if any on silane treated concrete was done using mercury intrusion porosimeter (MIP).

RESULTS

The depth of penetration test was conducted after 30 days of application of sealer on concrete surface and the results are given in Table.1.From the table, it can be seen that the silane system II penetrated to a maximum depth of 50 mm but doesn't show any hydrophopbic property as given in Fig.1. The concrete which absorbed sealant was impermeable to the water and its color remained unchanged. But silane system I and III showed hydrophobicity but penetrated up to the depth of 10 and 6 mm respectively. It seems siloxane contributed to the hydrophobic nature; the silane is responsible for the penetration.

Breathability of the sealer is to allow water vapour to migrate up through the concrete surface and evaporate,while preventing penetration of water in the liquid form. If the sealer fails to breathe then it will loose adhesion from the substrate after forming blisters. It can be quantitatively estimated by measuring moisture vapour transmission rate (MVTR). As per BT 001, the minimum requirements for moisture vapour transmission rate shall be 70-85 % that of untreated concrete. From table 2, it could be inferred that the concrete treated with system ! and III showed lower vapour transmission rate which is 85 % and 80 % of untreated concrete respectively and satisfy the requirement as given in BT 001. Surface treated concrete with silane system II is having a MVTR value of 10.83 gm/m^2/24 hour which I almost equal to the untreated concrete. This result shows the developed sealant systems are breathable.

Sorptivity is a measure of absorption of water by the concrete through capillarity as a function of time when one face is exposed to the water. It depends upon the mix proportion of the concrete, type of admixtures added, age and degree of hydration and presence of micro cracks. Fig.2 compares the rate of infiltration of water through silane treated concrete with the untreated concrete. It clearly indicates it is higher in untreated concrete when compared to silane treated concrete and the slope of the curve indicates the sorptivity. From Table 2, it is seen that system 1 recorded the lowest sorptivity value of 0.34 x10^{-3} mm/s$^{0.5}$ which is 10 times lower than that of control concrete.

One of the most important property of the concrete is the low permeability so that it can resist the ingress of water and thus reduces the corrosion distress in structures. Estimating % of water absorption is a good indicator to assess the permeability characteristics of concrete. As recommended by BT001, it shall be maximum of 1% after 48 hours. From table 2, it is understood that silane system III shows lowest water absorption of 0.93 % of the three systems tested.

Alkaline resistance is important in the case of the penetrating sealer applied on the newly built concrete structures. It has less significance in the case of carbonated and older concrete. Reduction in weight in presence of KOH ions is measured as alkaline resistance. As per BT 002, it should be less than 3 %. From Table 2 , it is observed that concrete treated with sliane system I ,II and III show weight-loss of 1.11% ,1.03% and 0.10 % respectively whereas untreated concrete shows 4.29 %. Results showed System III has 10 times higher alkaline resistance than that of system I and II.

Penetrated chloride value is an indicator of corrosion resistance of any type of sealer. If the pH of the concrete is above 11.5 and there is absence of the chloride ion, the steel can sustain its passivity. But, if there is the presence of the chloride ion, the mild steel lost its passivity even at pH above11.5 and leads to the corrosion. It depends on the Cl^-/OH^- ratio. There is a threshold limit for the chloride (water soluble) content around the steel, when it became greater than this limit, the steel became vulnerable to the corrosion. As per ACI committee 201, this limit is 0.10% of water soluble chloride content by weight of the cement. As the chloride content increase around the steel above this limit, the rate of corrosion also increases. Hence resistance to chloride ion penetration is an important parameter for any treated concrete.

Under RCPT test, the electrical conductance of the concrete is related to the penetration of chloride ions and it is an indirect way to find the rate of diffusion of chloride through the concrete. As per ASTM C1202 if the conductance of the concrete is greater than 4000 coulombs, the chloride ion penetration is high whereas if it is in the range between 100-1000 coulombs then the penetration is low. Accordingly, the electrical conductance of treated concrete with System III shows the lowest value of 130 coulombs which is 11 times lower chloride penetration rate than that of untreated concrete.

From the systematic investigations it is understood that silane system III performed better than that of other two systems in all the durability tests conducted even though the depth of penetration is only 6 mm. To understand the reason behind this, MIP and SEM studies were carried out and the results are given in Fig.3 and 4 respectively. From Fig.3 it is clearly seen under MIP test, at each pore size, the intruded volume of mercury on treated concrete using the system III is less than that of other systems tested. In the case of untreated concrete it is higher than that of silane treated concretes.

SEM images of untreated concrete as given in Fig.4, scattered needle like structure which are ettringite and sheet (bundle) like structure (C-S-H gel) and scattered large number of $Ca(OH)_2$ crystals were observed. In concrete treated with system I, silicon crystals are formed and uniformly distributed assures the reaction between $Ca(OH)_2$ and silicon resin penetrated. In system II, small number acicular form of crystals were observed here and there and indicates the partial reaction of silane with $Ca(OH)_2$. Micrograph of system III clearly shows the large number of acicular form of crystals throughout the surface of the microstructure and clearly indicates the formation of silicon crystals. These crystals filled the pores thus densifies the microstructure and made impermeable cover concrete. So it is evident that the system III performed better than system I and II. Thus microstructural studies confirmed the formation of silicon crystals which are capable to repel the water and also reduce the surface tension of pore walls thus make the surface more hydrophobhic.

CONCLUSIONS

1. Silane molecules are smaller in size and capable to penetrate deeper into concrete
2. Hyrophobicity of the sealer played a greater role than viscosity in resisting penetration of chloride and water.

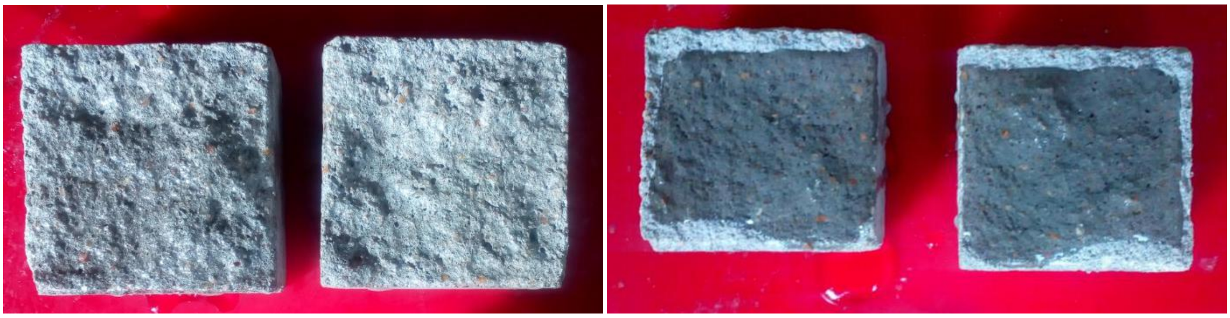
3. Organo siloxane molecules are contributing much in hydrophobicity than the ethyl ester of silicic acid .Octyl silane is not effective as compared to ethyl ester of silicic acid in resisting chloride penetration through the concrete.
4. Selection of suitable catalyst and it's concentration is contributing towards the hydrophobicity of silane system.

Table 1: Depth of penetration

system	Viscosiy (cst)	Depth of penetration (mm)	Depth of hydrophobicity (mm)
I	5	10	10
II	5	50	Hydrophobicity not exist
III	200	6	Hydrophobicity exist only upto 3mm

Table 2: Performance of sealer systems under various test

system	RCPT (Coulombs)	Vapour Tranmission Rate (g/m^2/24 hours)	Sorptivity ($x10^{-3}$) $mm/s^{0.5}$	Water absorption (%)	Alkaline resistivity test (%)
I	200	9.66	0.34	1.69	1.11
II	482	10.83	0.95	1.10	1.03
III	130	8.67	0.58	0.93	0.10
Control concrete	1511	10.66	3.53	2.69	4.29



Full depth of penetration (System II) Upto 6 mm (systemIII)

Fig.1 Depth of penetration of sealer treated concrete

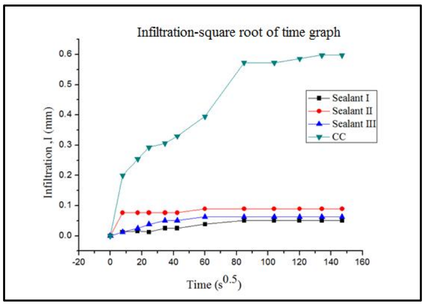


Fig.2. Infiltration *Vs* square root of time

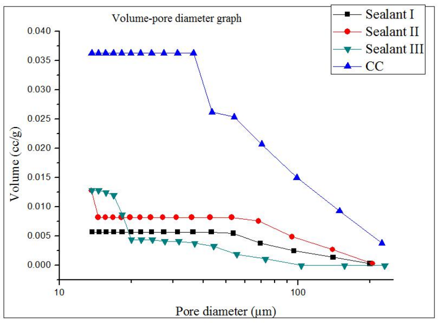
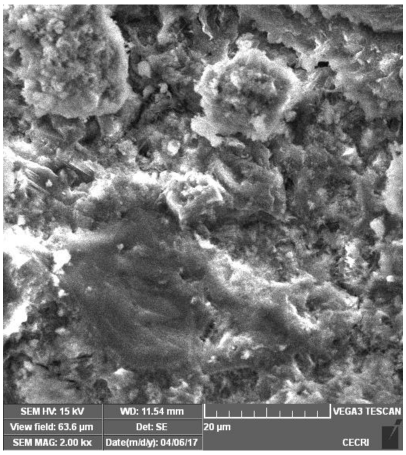
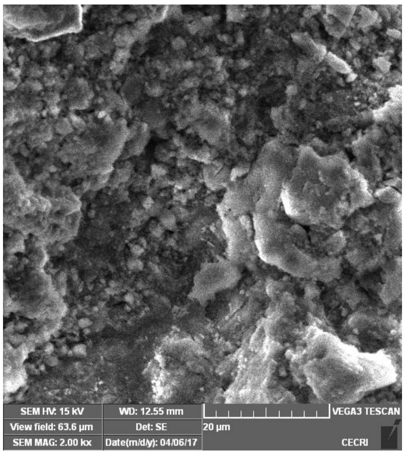


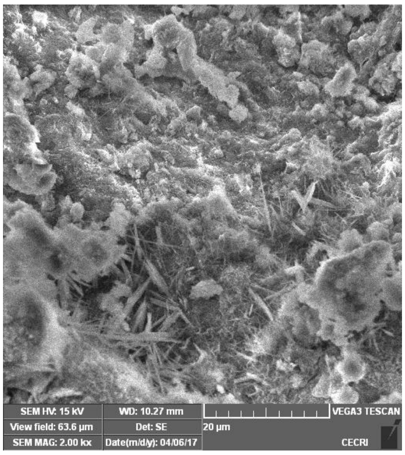
Fig.3. MIP :Volume *Vs* pore diameter



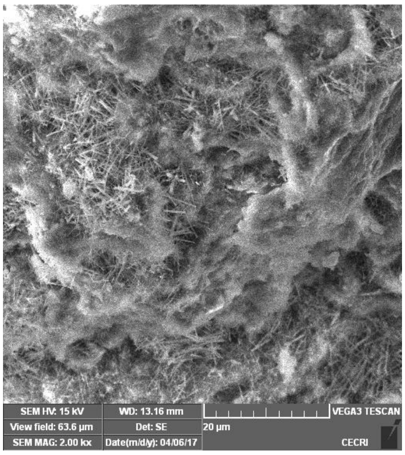
untreated concrete



concrete treated with sealant system I



concrete treated with sealant system II



concrete treated with sealant system III

Fig.4: SEM micrographs of untreated/ silane treated concrete

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