SHORT AND LONG TERM PERFORMANCE OF SILANE TREATED CONCRETE

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ABSTRACT

This paper presents a laboratory study on the effectiveness of silane when used as a surface treatment for concrete which has been cured in a hot, dry environment. The performance of silane treated concrete is assessed using short and long term tests and compared with untreated concrete. The concrete properties studied are moisture migration, cement hydration, pore structure, permeability, water absorption and chloride ions penetration.

The results of this study indicate that silane treatment marginally influences the rate of water vapour transmission through concrete and therefore it is not effective to retain moisture within the pores of concrete. Lack of moisture stops hydration and as a results the concrete is porous and permeable.

However, silane is an effective water repellent and this property is reflected in the properties of the treated concrete which is far more resistance to water absorption and chloride diffusion. The effectiveness of silane -as a water repellent- deteriorates with time of exposure, thus results in a shorter effective service life.

1 INTRODUCTION

Water plays an important role in the chemical and physical deterioration of concrete, as it is the medium for transport of corrosion-inducing substances. Water is also necessary for chemical reactions in concrete, therefore, producing low permeability concrete improves its durability and ensures long term performance. In practice it is extremely difficult to produce concrete which is fully resistant to penetration of harmful substances and therefore one of the options to improve its performance and durability is to use surface protective treatments.

Silane is an organo-silicon compound which is water repellent and therefore used frequently to protect concrete. Within the organo-silicon compounds it has the smallest molecular size and this advantage allows silane to penetrate concrete to a substantial depth. The mechanism by which silane provides protection to concrete involves chemical reaction firstly, with the moisture within the pores of concrete to form silanol product (Si-OH) groups, and secondly a conversion reaction with the concrete substrate and condensation to form an active substance containing silicon resin (Si-R) groups [1-5]. This active substance which is hydrophobic lines the pores of concrete and increases surface tension resulting in a higher contact angle with water and hence more difficult to wet [2]. Grobe [3] used Infrared Spectroscopy (IR) and Nuclear Magnetic Resonance (NMR) to study the interaction between silane and silica surface substrate. The IR results showed detectable (Si-OH) peaks due to the formation of strong chemical bonds between the substrate and the organo-silicon compound, while NMR indicated broad peaks due to the formation of a protective layer. However, the protective layer was not identified.

Silane treatment does not block the pores of concrete, therefore, it has no potential to resist the diffusion of gases and water vapour, however, its hydrophobic character theoretically reduces the risk of reinforcement corrosion due to chloride contamination [6], and also improves its resistance to alkali silica reaction [7] by keeping the concrete dry. Robinson [8] showed that silane does not improve the carbonation resistance of concrete and has only a little influence on the water vapour diffusivity. However, the performance of silane treated concrete was far better than that of the untreated concrete in resisting the diffusion of chloride ions. The diffusion coefficient was one order of magnitude less for the treated concrete.

The most important advantage of silane as a surface treatment for concrete is its water repellency. Littmann [9] showed practically that the water uptake is a function of the contact angle. However it is important to highlight the fact that the hydrophobic action of a water repellent treatment decreases over a long period of exposure to water, as the contact angle declines due to water absorption [2]. This feature of silane can, in some instances, limit its service life to only 2 years, (Cabrera and Hassan [10]).

This paper presents results of a laboratory study on the short and long term performance of silane treated concrete which was exposed to a hot and dry environment. The influence of silane treatment on the moisture conditions of concrete and its effect on concrete properties such as: cement hydration, pore structure and gas permeability are presented. Also the water repellency of silane treated concrete and its resistance to chloride ions penetration are discussed.

2 MATERIALS AND SAMPLES PREPARATION

2.1 SILANE TREATMENT

The concentration of silane in a particular treatment has a great effect on its protection performance, as increasing the silane concentration results in deeper penetration depth and consequently better waterproofing efficiency [6,11,12]. In this project the silane used was 100% monomeric alkyl isobutyl tri-alkoxy silane.

2.2 PREPARATION OF CONCRETE SPECIMENS

The concrete mix used in the study was composed of a cement: fine aggregate: coarse aggregate ratio of 1: 2.33: 3.5 by weight, the cement content was 325 kg/m^3 and the water/cement ratio was 0.55. The characteristic strength of the concrete mix was 55MPa at 28 days. The cement used was opc complying with the requirements of BS 12 [13]. The fine aggregate was sand conforming to zone M of BS 882 [14]. Gravel was used as a coarse aggregate with a nominal maximum size of 20 mm. Concrete cubes (100 mm) were cast and left over-night covered with wet hessian and polyethylene sheets.

2.3 PREPARATION OF MORTAR SPECIMENS

The mortar mix was the concrete mix without the coarse aggregate, it was prepared and cast into slab moulds (400 mm x 250 mm x 50 mm). The mortar slabs were also left overnight covered with wet hessian and polyethylene sheets.

2.4 CEMENT PASTE SPECIMENS

The cement paste with 0.55 w/c ratio was mixed in a domestic blender and cast into cylinders of 50 mm diameter and 50 mm thickness. The cylinders were sealed and rotated over night in a rotating laboratory cylinder at a speed of 20 rotation/min to avoid segregation.

2.5 CURING REGIME AND SILANE APPLICATION

All specimens were demoulded one day after casting and transferred to a hot dry environmental chamber kept at 35°C, 45% RH. Wind was simulated using a fan which gave a wind velocity of 3 m/s. This environment simulates the average conditions of the Gulf and other Mddle Eastern areas. The specimens were treated with silane after an initial curing period of 14 days. Application of silane was carried out by brushing the surfaces of the specimens with two coats as recommended by the manufacturer. The specimens were then kept in the hot dry environmental chamber until needed for testing.

3 METHODS OF TESTING

3.1 PHYSICAL PROPERTIES AND DEPTH OF PENETRATION

The silane used in this study was characterised by measuring its viscosity, specific gravity, and surface tension. These properties are important with regards to its penetration depth and consequently its performance. The depth of silane penetration into the surfaces of concrete, mortar, and cement paste specimens was determined by measuring the unwetted (hydrophobic) layers of freshly cut specimens splashed with water.

3.2 CEMENT HYDRATION AND THE EFFECT OF SILANE TREATMENT

The hydration products of the treated and untreated cement paste specimens were analysed using Thermogravimetric analysis (TG/DTG), The degree of hydration was calculated from the measured non-evaporable water and also from the total calcium hydroxide content measured. The non-evaporable water is obtained from the weight loss between 100 and 400°C, while the total calcium hydroxide content from the weight loss between 400 and 500°C (dehydration of $Ca(OH)_2$) and the weight loss between 550 and 800°C (decomposition of calcium carbonate $CaCO_3$). Details of this technique and the determination of degree of hydration are given in reference [15].

At the age of 28 days, 5 mm slices were cut from the top surface of the treated and untreated cement paste cylinders. The slices were ground into powders, dried in a microwave oven to constant weight [16], and left to cool to room temperature in a desiccator. The samples were then tested.

3.3 MOISTURE MOVEMENT AND WATER VAPOUR TRANSMISSION

The moisture content of the treated and untreated concrete cubes was determined at 1,3,6, and 12 months. The cubes were weighed after the desired curing period (W_a), dried to constant weight in an oven at 105°C, and weighed again (W_d) after cooling in a desiccator to room temperature. The moisture content (MC) was then calculated using the following formula:

The rate of water vapour transmission was calculated using the wet cup method [17]. After a curing period of 28 days, mortar cores (25 mm) were drilled from the casting faces of the treated and untreated mortar slabs and sliced into 5 mm discs. The mortar discs were then sealed to the top of plastic bottles containing distilled water and left in the hot dry environmental chamber at 35°C, 45% RH and 3 m/s wind velocity. The weight loss of the sealed bottles was recorded up to 90 days.

3.4 PORE STRUCTURE AND OXYGEN PERMEABILITY

The measurements of pore size distribution and oxygen permeability were carried out on treated and untreated specimens at different ages (1 to 12 months). Mercury intrusion porosimetry was employed to measure the pore structure, while the Leeds permeameter cells were used for oxygen permeability [18].

Mortar cores of 20 mm thickness and 20 mm diameter for mercury porosimetry and 25 mm diameter for oxygen permeability were drilled from the casting faces of the slabs. The cores were dried in an oven at 105°C for 24 hours before testing.

3.5 CHLORIDE PERMEABILITY

The Leeds modified voltage driven test was adopted for measuring the chloride permeability under a potential difference of 30 volts. The apparatus is similar to the FHWA [19] with some modification in the area of data measurement, collection and processing. The test was carried out on treated and untreated mortar specimens (100 mm diameter and 25 mm thickness) up to the age of 1 year. The apparatus and the test procedure are given in detail in references [15,20].

3.6 WATER ABSORPTION AND CHLORIDE DIFFUSION

At the age of 28 days treated and untreated concrete cubes were immersed in 15% sodium chloride (NaC1) water solution for a period of 1 year. The weight gain due to water absorption was recorded and used in the calculation of the waterproofing efficiency. At the end of the immersion test, powder samples were obtained by drilling the concrete cubes at various depths and these were used for the determination of chloride concentration profiles according to BS 1881 [21]. The unsteady state chloride diffusion coefficient was then calculated using Fick's second law.

The steady state diffusion coefficient was also determined for the treated and untreated mortar cores using a new accelerated diffusion test [15]. This new accelerated method is based on saturating the specimens with chloride ions before testing them in the static diffusion cell. The effective diffusion coefficient was calculated in this case according to Fick's first law, and compared to the unsteady state diffusion coefficient.

4 PRESENTATION OF RESULTS AND DISCUSSION

4.1 DEPTH OF SILANE PENETRATION

The average depth of silane penetration into the surfaces of concrete, mortar, and cement paste specimens is shown in Figure 1. The variations in penetration depth appear to be a function of the substrate porosity. Silane has relatively small molecules (5-10 A°), similar in size to water molecules [22]. However, it penetrates more than water due to its lower values of viscosity and surface tension as shown in Table 1.

FIG. 1 Depth of silane penetration

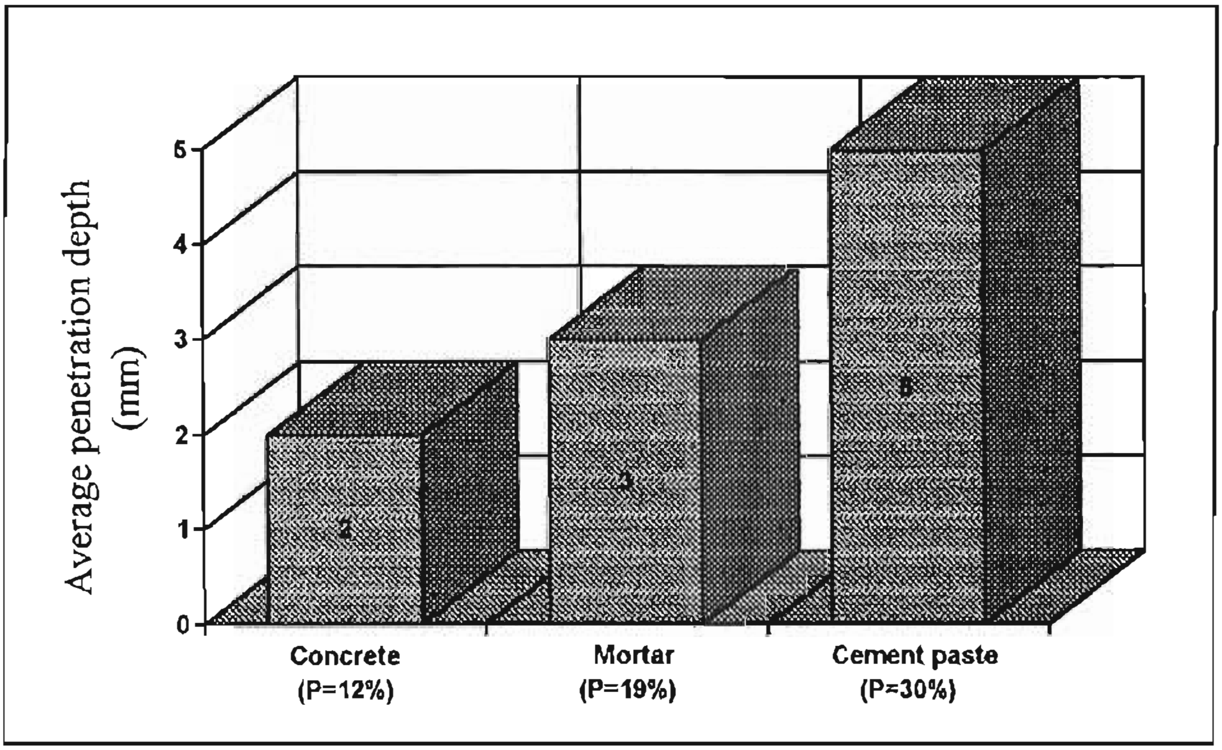


TABLE 1 Physical properties of silane

Property	Silane	Distilled water
Viscosity (poises)	0.79	1
Specific gravity	0.93	1
Surface tension (N/m)	26×10^{-3}	72×10^{-3}

4.2 CEMENT HYDRATION

The thermogravimetry (TG) results of the silane treated and untreated cement paste specimens are shown in Figure 2. Figure 3 shows the differential thermogravimetry (DTG) results, and Table 2 gives the calculated values of non-evaporable water and total calcium hydroxide content together with the degree of hydration. The results show clearly the effect of relative huniidity on cement hydration. Both treated and untreated specimens exhibited low degrees of hydration due to the relatively dry environment where they were cured. Parrott and Killoh have shown that cement hydration practically ceases below 60% RH [23]. The results also reveal that silane treatment to concrete does not improve its carbonation resistance, as indicated from the calcium carbonate peaks (550-800°C) in Figure 3.

FIG. 2 TG results for silane treated and untreated cement paste specimens

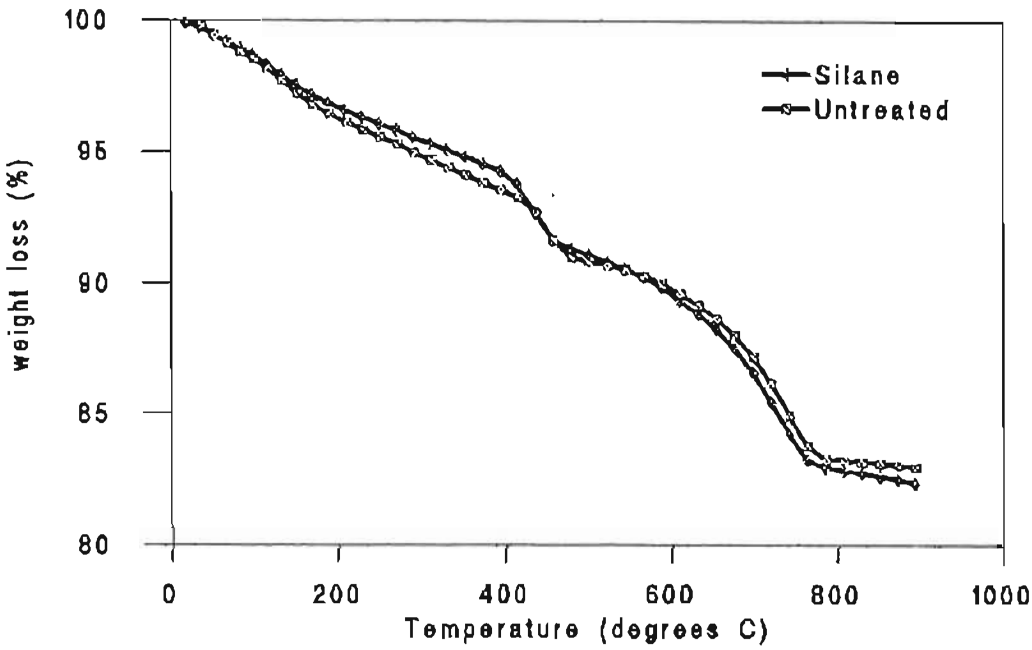


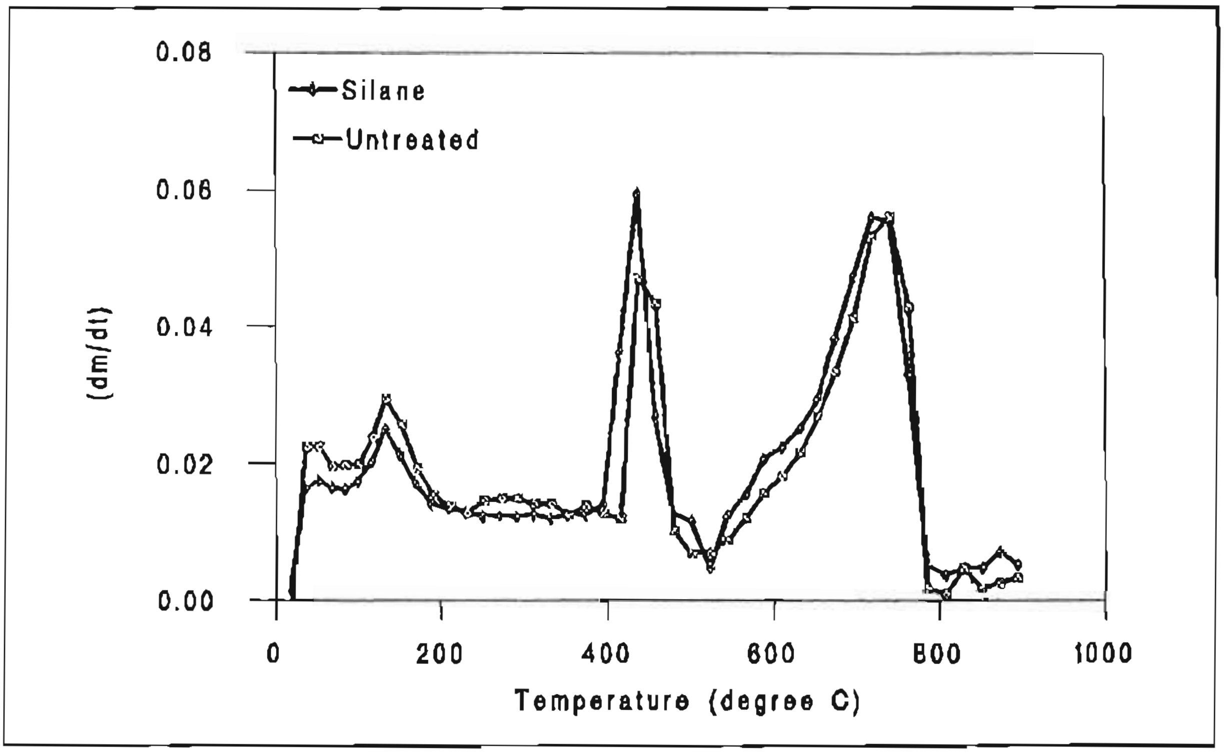
TABLE 2 Hydration of silane treated and untreated cement paste specimens

	Silane treated	Untreated
Non-evaporable water	6.6 (52.4)	7.01 (55.6)
Calcium hydroxide	20.10 (70.5)	18.61 (65.3)

Values in brackets indicate the degree of hydration percentage based on the value for fully hydrated cement

The non-evaporable water content of the silane treated specimens was slightly lower than that of the untreated specimens, while the total calcium hydroxide content was higher (Table 2). As indicated before silane reacts with the moisture available within the cement pores to form silicon resins. Work by Lattey [24] indicated that the active hydrophobic layer is still formed even when silane is applied to the surface of dry cement paste specimens with 0% RH. Thus when silane is applied to a relatively dry specimens it consumes part of the non-evaporable water to form silicon resin, causing a reduction of the calculated non-evaporable water. The water of the silicon resin is probably lost at higher temperature causing an apparent increase in the amount of calcium hydroxide.

FIG. 3 DTG results for silane treated and untreated cement paste specimens



Values in brackets indicate the degree of hydration percentage based on the value for fully hydrated ceinent

4.3 MOISTURE CONTENT AND WATER VAPOUR TRANSMISSION

The amount of evaporable water obtained by drying the concrete specimens at 105°C is shown in Figure 4. The result shows a small difference in moisture content at early age (1 month), however this difference diminishes with time as it is clear from the results of 3-12 months.

The results of water vapour transmission were in agreement with the results of the moisture content determination. Figure 5 shows the weight loss through the silane treated and untreated mortar discs with time. The rate of water vapour transmission was calculated from the measurements of weight loss and found to be 84 ($g/m^2/day$) for the silane treated specimens and 89 ($g/m^2/day$) for the untreated specimens, which indicates that silane affects slightly moisture migration out of concrete.

Fig. 4 Moisture content versus concrete age for silane treated and untreated specimens

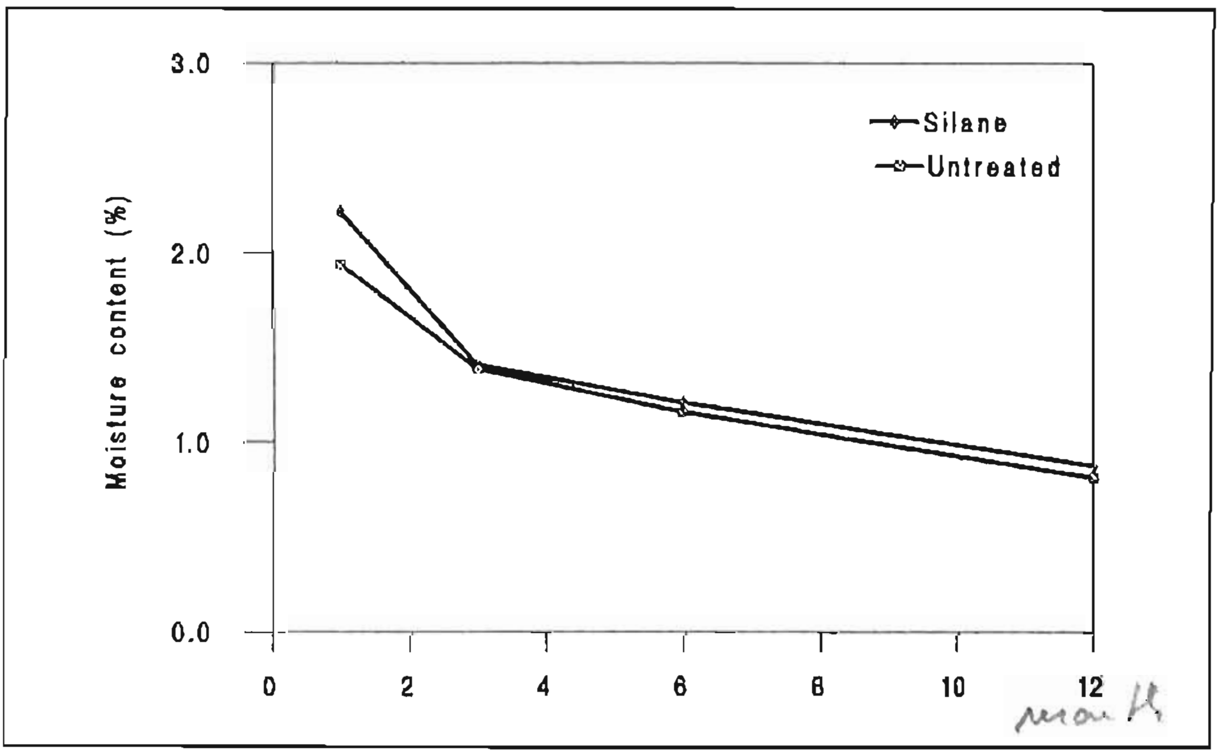
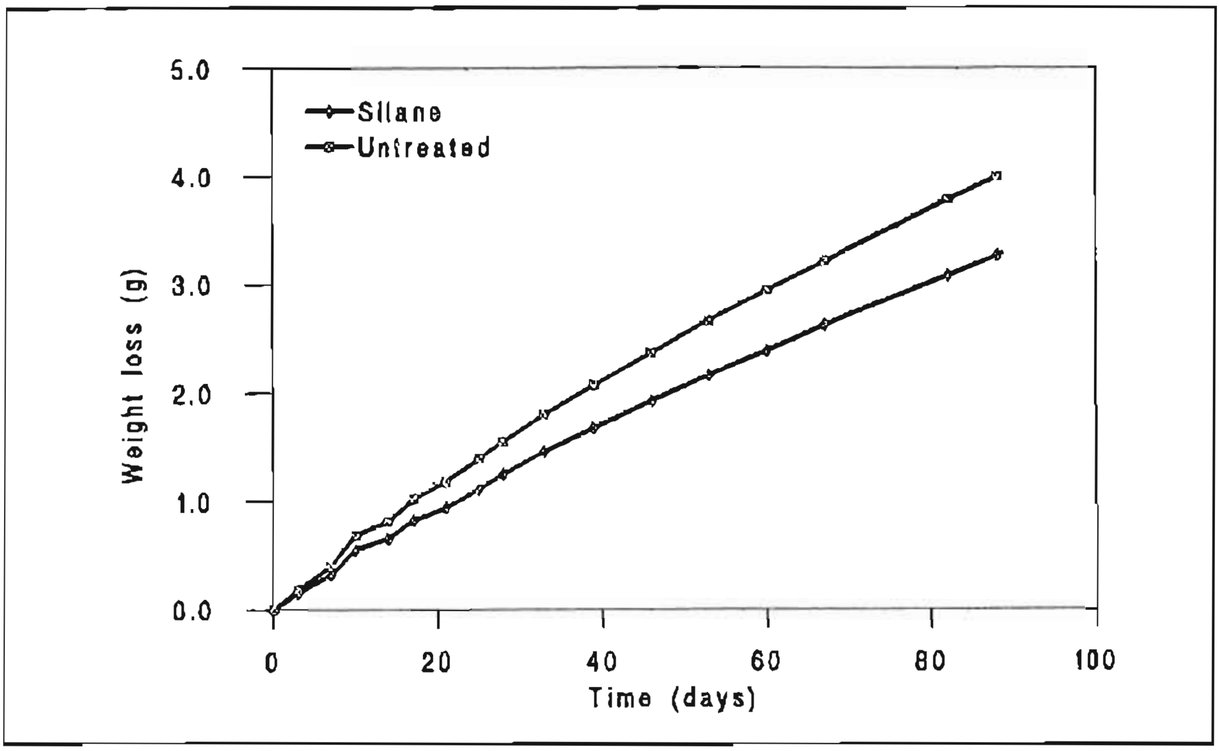


FIG. 5 Water vapour transmission through sitane treated and untreated mortar specimens



4.4 PORE STRUCTURE AND OXYGEN PERMEABILITY

Since silane treatment does not noticeably affect moisture evaporation from concrete, the likelihood of silane treatment improving the pore structure of concrete exposed to dry environments appears slight. Measurements of pore size distribution using mercury microporosimetry were made using treated and untreated specimens. The results are expressed in terms of total capillary pore volume (pore size > $0.01\mu m$) and "apparent average pore diameter" (pore size with maximum occurrence). These are shown in Figure 6 and Figure 7. The silane treated specimens showed slightly higher volume of capillary pores than the untreated specimens for all tested ages (Figure 6) and coarser pore diameters as indicated by the apparent average pore diameter (Figure 7).

FIG. 6 Volume of capillary pores of silane treated and untreated mortars

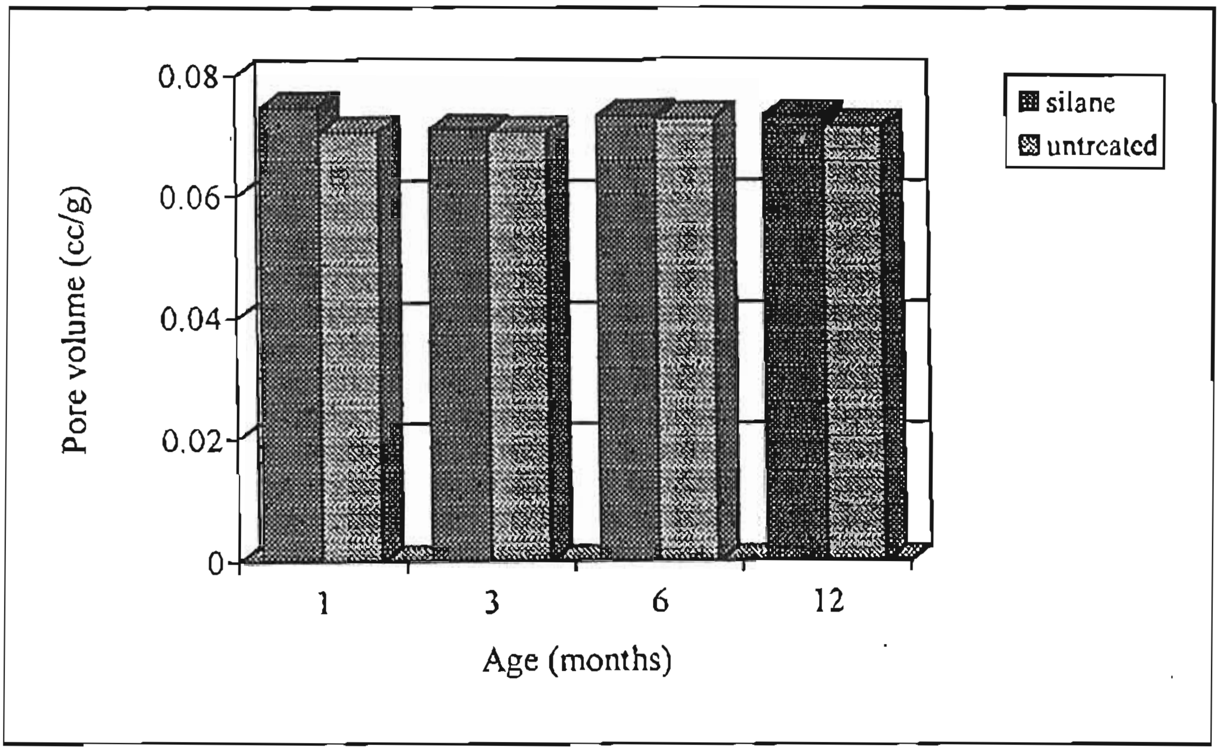
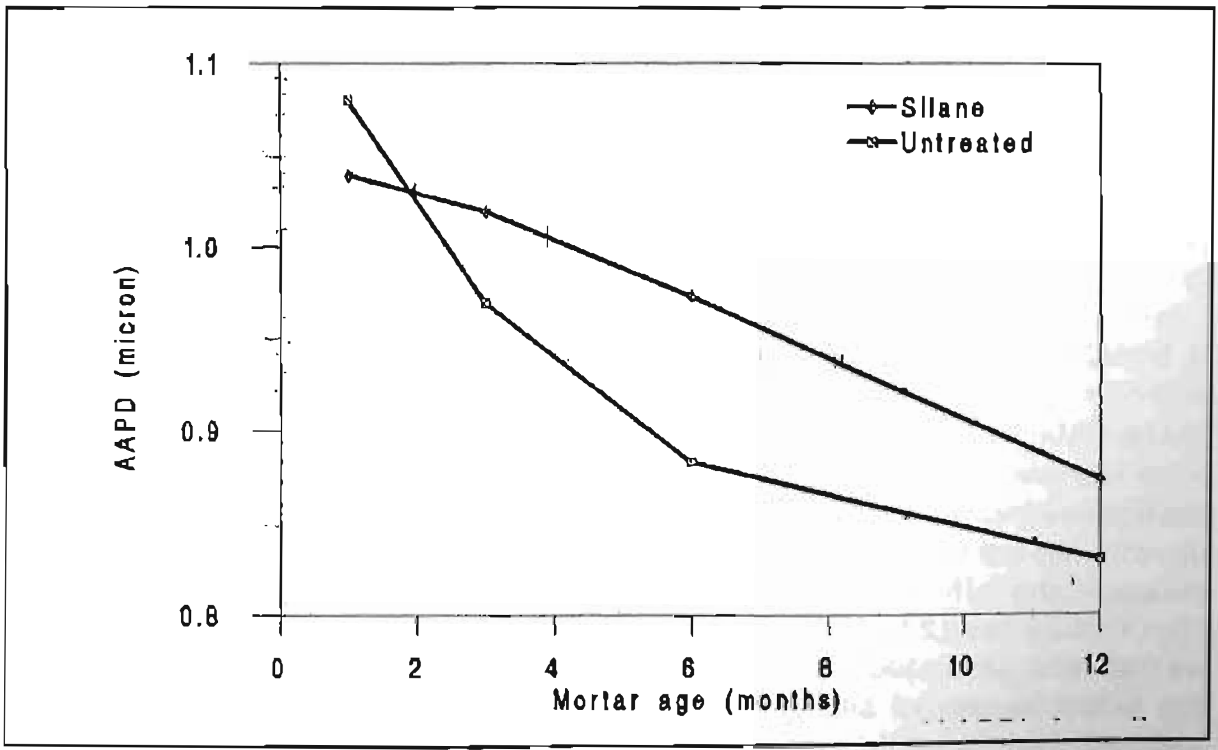
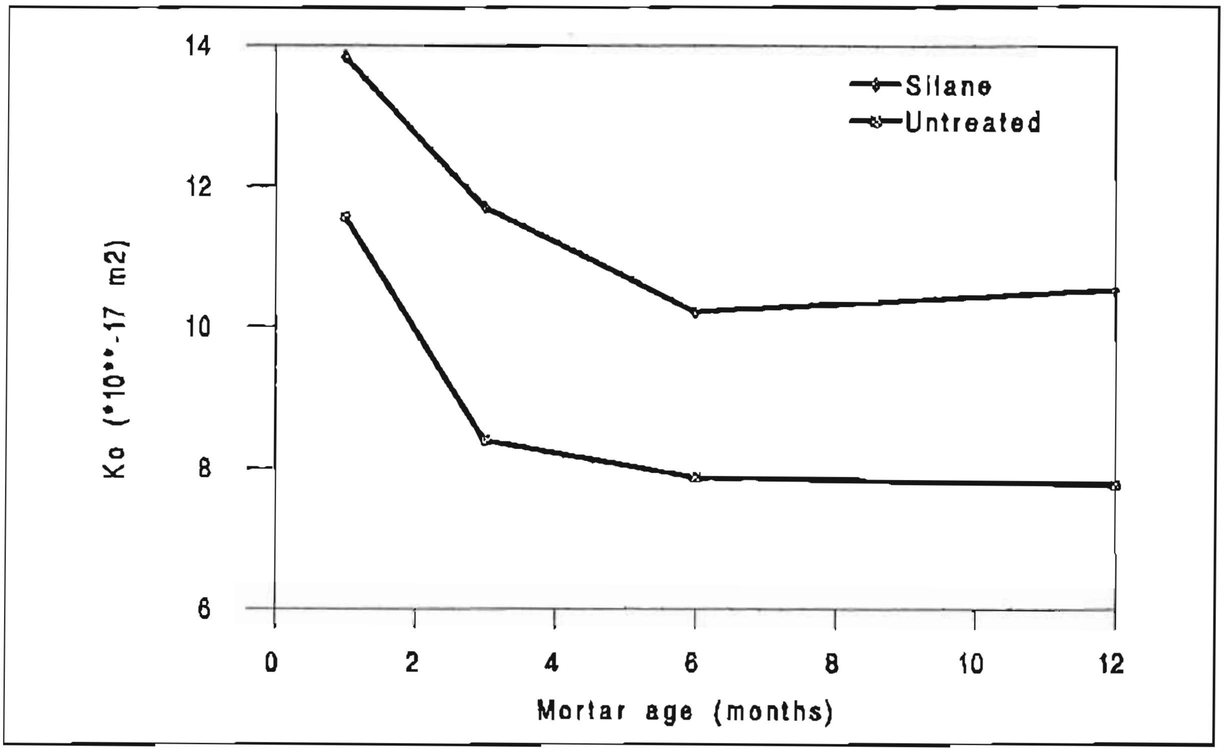


FIG. 7 Apparent average pore diameter (AAPD) for silane treated and untreated mortars at different ages



The permeability of concrete is one of the most important properties for controlling the long term performance and durability of concrete. Cabrera and Hassan [23] showed that the permeability is directly related to the concrete pore structure and can be used to assess the efficiency of surface treatment compounds. The intrinsic permeability values of the silane treated and untreated mortar specimens up to 12 months are shown in Figure 8. As expected no improvement in the oxygen permeability was detected, in fact silane treated specimens show an increase in the value of permeability in relation to non-treated specimens. These results confirm the previous findings [25] regarding the relationship between permeability and AAPD.

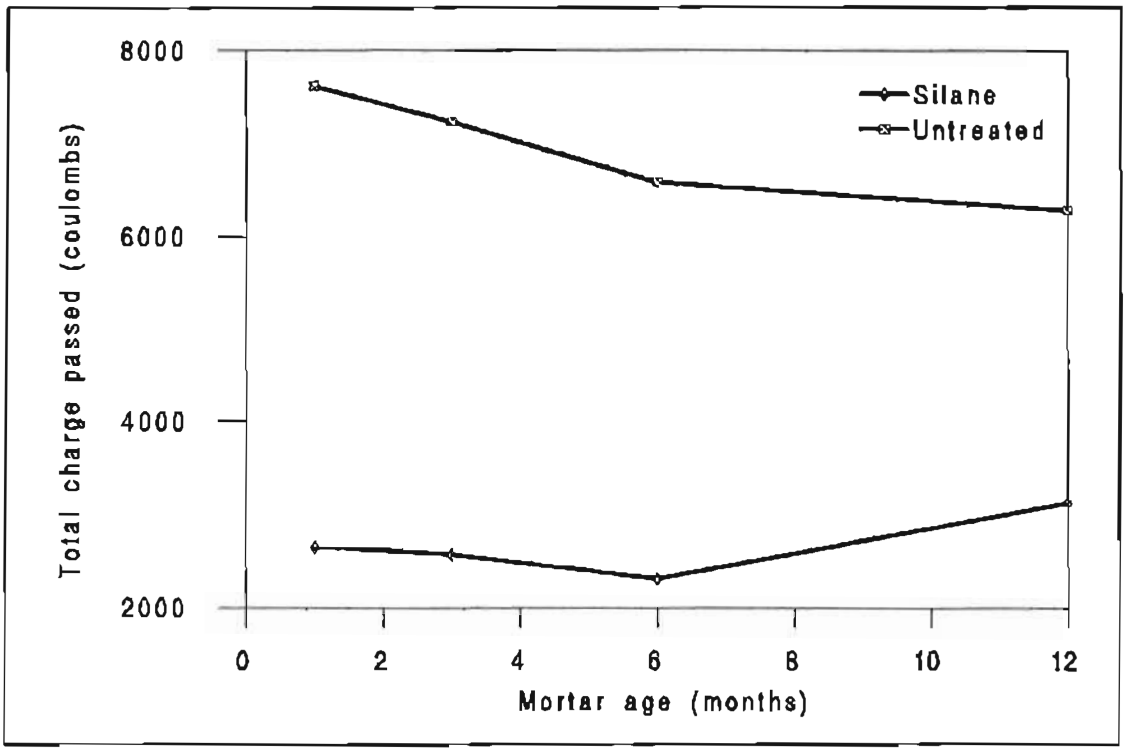
FIG. 8 Oxygen permeability of silane treated and untreated mortars at different ages



4.5 CHLORIDE PERMEABILITY

In the chloride permeability test the total charge passed, which is calculated from the area under the current-versus-time curve expressed in Coulombs, is used to represent the chloride permeability. The value of charge passed is directly related to the chloride diffusion coefficient. It has been used before to measure the efficiency of surface treatments and their equivalent concrete cover thickness [26]. Figure 9 shows the charge passed values of the silane treated and untreated mortar specimens at different ages. The results reflect the effectiveness of silane treatment in improving the chloride permeability. The chloride permeability of the silane treated specimens was about 3 times lower than the untreated specimens at early age. However, this difference is reduced with time and found to be only 2 times after 1 year. This feature is a clear indication that silane looses part of its initial efficiency with time.

FIG. 9 Total charge passed versus age for silane treated and untreated mortar specimens



4.6 WATER ABSORPTION AND CHLORIDE DIFFUSION COEFFICIENT

The water absorption results of the silane treated and untreated concrete cubes due to immersion in 15% NaC1 water solution up to 1 year are presented in Figure 10. The untreated concrete exhibited very high absorption within the first day of immersion in salt water, in fact it became almost saturated. Absorption after one day was negligible. The silane treated concrete showed excellent waterproofing efficiency during early periods of immersion. After about 6 months the rate of water absorption increased noticeably with time, indicating that silane had lost a great part of its water repellency. Simdar work [6] showed that silane had a retarding effect on the water absorption and chloride penetration up to 115 days, after this period the value of C1-diffusion increase rapidly.

Silane treatment is specified by the Department of Transport in the UK for use in all concrete highway structures [27]. The results of this study indicate that silane treatment can provide an effective barrier against ingress of water and chloride ions into concrete for a short period, but does not maintain its water repellency for longer periods and therefore, does not provide protection for periods of 20 to 25 years optimistically specified by the Ministry of Transportin the UK.

FIG. 10 Water absorption versus ponding time for silane treated and untreated concrete specimens

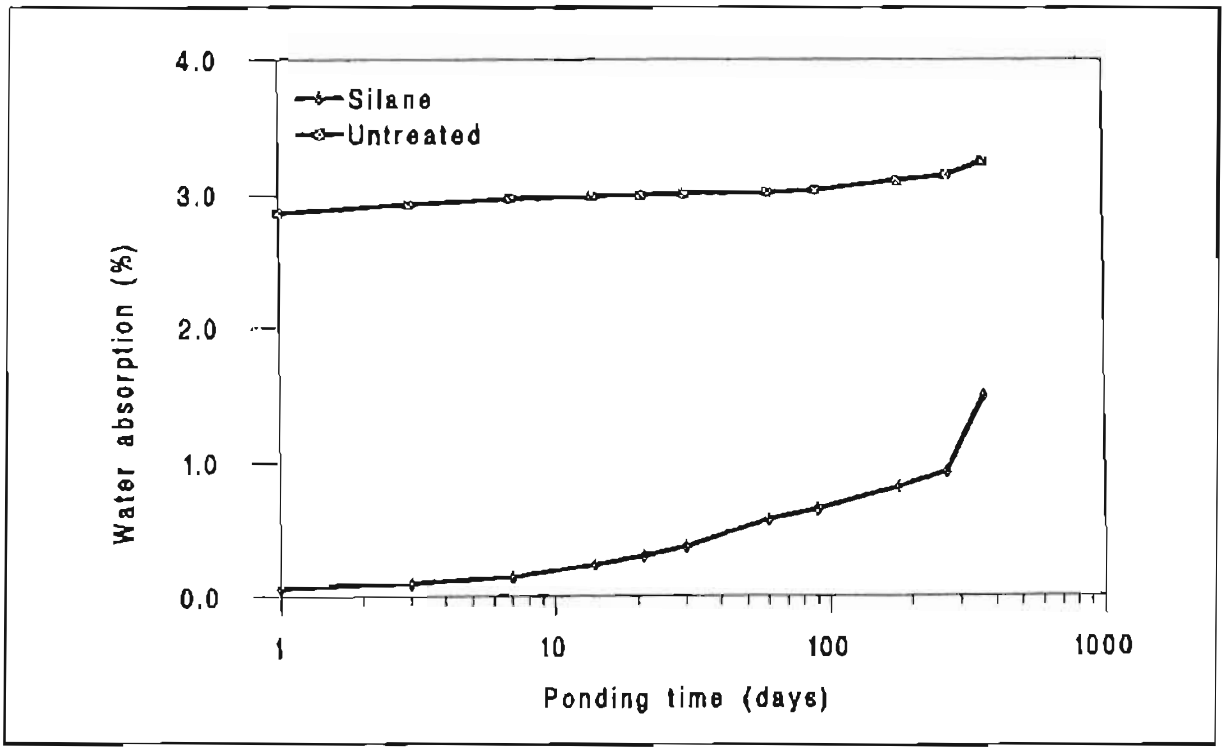
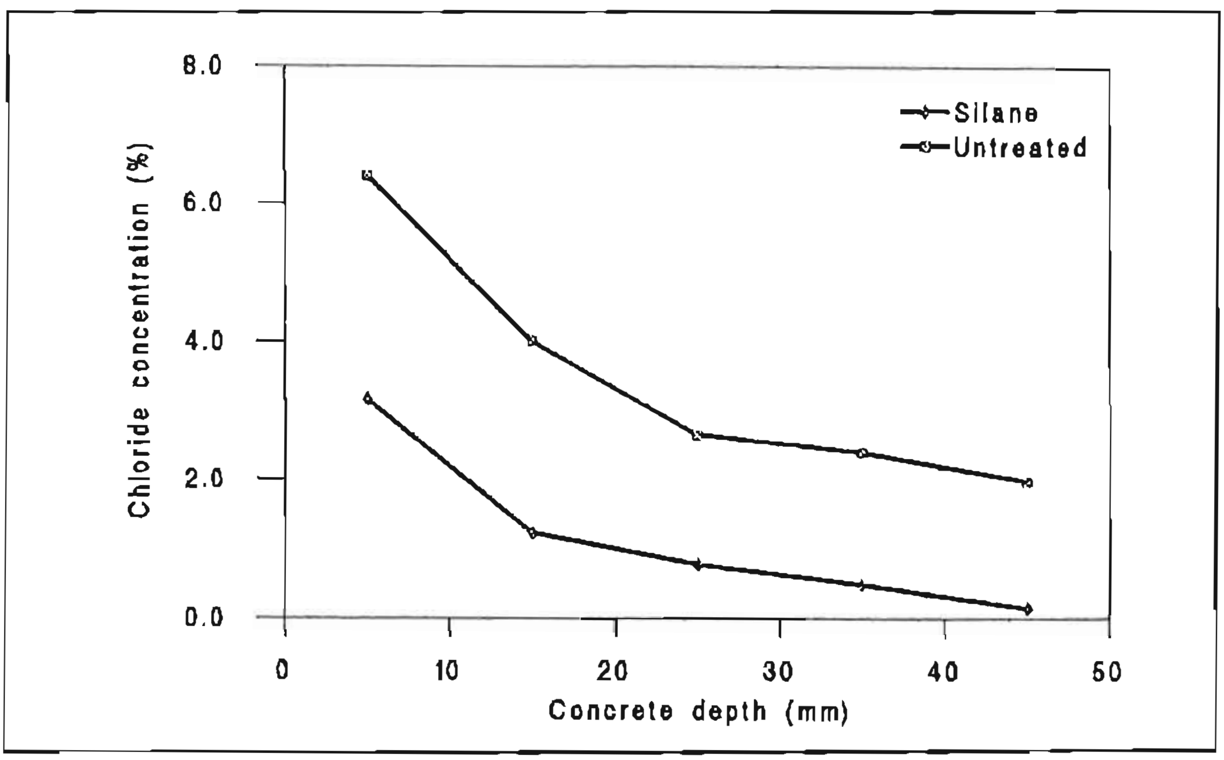


FIG.11 Chloride concentration profiles for silane treated and untreated concrete specimens



The chloride concentration profiles of silane treated and untreated concrete specimens are plotted in Figure 11. The results show the reduction of chloride concentration with depth, and also the influence of silane treatment in reducing the chloride concentration in concrete. The effective chloride diffusion coefficients were calculated from the measurements of chloride concentrations according to Fick's second law of diffusion and are given in Table 3. Table 3 shows also the steady state chloride diffusion coefficients of the silane treated and untreated mortar specimens calculated according to Fick's first law. Both tests show that the effective chloride diffusion coefficient of silane treated specimens is lower than the untreated specimens by almost one order of magnitude. However, there is evidence that this difference diminishes with time indicating again that the water repellency of silane is time dependent.

TABLE 3 The chloride diffusion coefficient of the silane treated and untreated specimens

Test method	Silane treated specimens (cm^2/s)	Untreated specimens (cm^2/s)
Immersion test	5.51×10^{-9}	8.20×10^{-8}
Static diffusion cell	2.48×10^{-9}	2.14×10^{-8}

5 CONCLUSIONS

From the results of this study the following conclusions are offered:

1 The depth of silane penetration is a function of the porosity of the concrete substrate.

2 Silane treatment competes with the cement for water, particularly when there is not enough moisture available within the pores of the hydrating concrete, to form the hydrophobic layer of silicon resin.

3 Silane has a slight effect on the moisture migration and the rate of water vapour transmission out of concrete.

4 Silane does not improve the gas permeability of concrete.

5 Silane is an effective treatment in resisting chloride penetration into concrete. It reduces the chloride diffusion coefficient by one order of magnitude. However, its water repellent efficiency reduces with time.

6 Short term tests are not enough to judge the performance of surface treatments. Long term performance tests should be incorporated in specifications of acceptance of silane or silane containing compounds.

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