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CONCRETE SURFACE TREATMENT: EFFECT OF EXPOSURE TEMPERATURE ON CHLORIDE DIFFUSION RESISTANCE

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ABSTRACT

An investigation is reported on the influence of exposure temperature on the chloride diffusion properties of 7 widely used surface treatment systems for concrete. The variation in dry film thickness, based on the manufacturers recommended application rate, is measured. Chloride diffusion is determined for both treated concrete and standardised sintered glass substrates. It is shown that whilst all surface treatment systems perform best at low and medium exposure temperatures, the chloride flux through treatments applied to concrete and sintered glass substrates differs greatly. Based on this study, a tentative classification of the surface treatment systems for different exposure temperatures has been developed.

Introduction

Chlorides and carbon dioxide can enter concrete and ultimately induce reinforcement corrosion leading to the degradation of the concrete itself and eventually affecting the structures' ability to carry its design load. Surface treatment systems in the form of polymeric materials can limit the rate of ingress of water into the concrete (1-3) and so improve the long term durability of reinforced concrete by reducing chloride penetration (4-6). However, what is less certain is how effective these surface treatment systems are when subjected to the effects of typical environmental conditions, and in particular exposure temperature. Thermal energy has an important influence on the state of flux of the ionic movement itself and also on the structural and chemical nature of the polymer treatment.

Diffusion is the main process governing the transportation of chloride ions through semi-permeable materials and the fundamental parameter describing this process is the diffusion coefficient, D, of the material, for the diffusing ion at a given temperature. However, the critical temperature aspect is often not fully taken into account in assessing the comparative performance of surface treatment systems. The investigation reported here compares the chloride diffusion characteristics of 7 different commonly used surface treatment systems for concrete, tested under a range of exposure temperatures.

Materials

Concrete Substrate

A concrete grade of 35 N/mm^2 was selected as it represents the lower end of the typically used strengths for structures exposed to chloride containing environments. A normal Portland cement (NPC) of grade 42.5N to BS 12 (1991) was used together with a 20mm maximum sized gravel aggregate corresponding to BS 812 (1992). The test specimens were 25mm thick discs cut from cylinders of 100mm diameter by 300mm and were standard water cured at 20°C for 28 days, prior to preparation for surface treatment application. Details of the mix proportions used are given in Table 1.

Standard (Sintered Glass) Substrate

Due to the chemical and physical variability of concrete, a standardised substrate was also tested, using sintered borosilicate glass, which had a consistent and inert pore structure. The nominal diameter and thickness of the disc was 65mm and 5mm respectively. The pore size was between 10 and 16μm, corresponding to ISO 4793, grade P16 (7).

Surface Treatment Systems

The 7 surface treatment systems selected, and 1 primer required by two of the coating materials, consisted of four different generic types, the main characteristics of which are given in Table 2. These materials were chosen based on an extensive literature review of surface treatment systems carried out prior to this work and were considered to be the most effective types for reducing chloride ingress.

The epoxies, methacrylate and acrylic are all designed to be applied to a substrate where they cure and are expected to form uniform defect-free surface coatings which physically prevents chloride ingress by blocking liquid water movement. The silane/siloxane blend, on the other hand, is designed to penetrate the capillaries of the substrate where it lines the pores and, through solvent loss, forms a surface which is hydrophobic. The passage of water in its liquid form is, therefore, impeded together with any dissolved chlorides.

Specimen Preparation

The sintered glass discs were all brought to a standard condition by oven drying at 105°C for 24 hours. Prior to coating, these discs were stored in the laboratory at 20°C and 55% RH for 7 days.

TABLE 1.
Concrete Mix Proportions.

DESIGN STRENGTH, N/mm^2	CONCRETE MIX PROPORTIONS, kg/m^3					W/C	SLUMP, mm
	Water	NPC	Sand	10mm	20mm		
35	185	285	730	400	800	0.65	75

The concrete substrate test specimens were obtained from 100mm diameter x 300mm cylinders after 28 days standard water curing at 20°C. The top 25mm from the as-cast surface was discarded to minimize the variability arising from the surface concrete. Thereafter, discs of 25mm thickness were cut from the cylinder using a diamond saw and lightly lapped with silicon carbide paste. The specimens were then cleaned with warm water to remove any surface grit which might affect the bonding of the surface treatment system to the concrete substrate.

Both the sintered glass and concrete substrate surfaces were de-greased with acetone (GPR propanone 99%) The specimens were then placed in an air curing room at 20°C, 55% RH for 7 days, at which point all had achieved a constant weight.

This substrate condition is likely to be considerably drier than that of a substrate in-situ, particularly in chloride bearing environments. However, this dry condition provides an optimum for surface treatment and for the ultimate potential for chloride diffusion resistance of each material to be established. Furthermore, at this early stage of the work it was felt more important to provide consistent substrate moisture conditions. The study is being extended to establish the effect of the variation in substrate characteristics, including that of moisture condition, on surface treatment performance.

The 7 surface treatment systems were applied onto the substrate with a nylon brush using the manufacturers recommended coverage rate given in Table 2. Each layer was cured at 20°C, 55% RH for 24 hours before applying the next layer. The two pack materials were mixed carefully in the proportions specified immediately prior to application. The dry coating was inspected with a x10 hand held lens and any visible pin holes sealed by local reapplication. The thickness of the composite system was measured before and after each coating layer using a micrometer (0.01mm accuracy) following the procedure defined in ASTM D1005 (8). Dry thickness was calculated as the mean of five equally spaced radial points taken at mid-radius.

For the silane/siloxane system it was not possible to measure the depth of penetration into the substrate and consequently this was calculated assuming a uniform application as;

$$\textit{Mean Penetrant Depth, cm} = \frac{\dfrac{\textit{Dry Weight, g}}{\textit{Wet Weight, g}} \; x \; \textit{Volume Applied, cm}^3}{\textit{Area, cm}^2} \qquad (1)$$

After treatment, all specimens were stored at 20°C, 55% RH for 7 days prior to testing.

The temperature and relative humidity (RH) chosen for treatment curing represents mean laboratory conditions. In practice, these conditions are unlikely to simulate actual conditions in typical chloride environments and variations in the atmospheric temperature and RH during the period of curing will affect the treatments performance characteristics. Further research is underway to investigate the effects of such ambient temperature and RH variations on a variety of performance criteria for surface treatments.

Measurement of Chloride Diffusion

Series I Tests: Sintered Glass Substrate

For the sintered glass specimens, chloride diffusion was measured using a non-perturbative concentration difference (CD) type test, details of which have been given previously (6). The test

TABLE 2.

Main Characteristics of Test Surface Treatment Systems.

SURFACE TREATMENT SYSTEM	GENERIC TYPE	SOLID FORM	COVERAGE RATE	REC. MIN. THICKNESS PER COAT	POT LIFE	SOLIDS CONTENT	METHOD OF CURE	CURING TIME (at 20°C)
P-S/Sx-1	Silane/Siloxane, blend	Solvented	0.2 l/m^2	2 coats[1]	N/A	20%	Solvent loss + reaction	Min. 2 Hrs.
CS-E-1	Epoxy, High Build	Catalysed, Solventless	0.2 l/m^2	200µm wet (2 coats)[2]	40 mins.	100%	Chemical	7 days
CS-E-2	Epoxy, pitch	Catalysed, Solventless	0.25 l/m^2	250µm[3]	60 mins.	100%	Chemical	7 days
CS-E-3	Epoxy, pigmented	Catalysed, Solventless	0.2 l/m^2	200µm (2 coats)[3]	40 mins.	100%	Chemical	7 days
CS-E-4	Epoxy, pitch	Catalysed, Solvented	0.25 l/m^2	250µm wet 175µm dry (2 coats)[4]	4 Hrs.	70%	Solvent loss + chemical	7 days
CS-M-1	Methacrylate, pigmented	Solvented	0.175 l/m^2	75µm dry 175µm wet (2 coats)[5]	N/A	43%	Solvent loss	6 Hrs.
CS-A-1	Acrylic, pigmented	Aqueous dispersion	0.18 l/m^2	75µm dry 180µm wet (2 coats)[5]	N/A	41%	Drying	16 Hrs.
Primer PM-S/Sx/A-1*	*Silane/Siloxane Acrylic Primer*	*Not Known*	*0.4 l/m^2*	*Multiple flood coats*[1]	*N/A*	*Not Known*	*Not Known*	*Min. 2 Hrs.*

* *Used with CS-M-1 and CS-A-1.* [1] *Low pressure spray.* [2] *Brush or airless spray* [3] *Stiff brush.* [4] *Brush or spray.* [5] *Brush or roller.*

specimens were sealed in a diffusion cell containing de-ionised water and placed in a 20°C immersion tank containing a 5M sodium chloride solution. For the untreated control disc, a 1M sodium chloride solution was used on the upstream side as is was found that with higher upstream concentrations, diffusion through the specimen was too rapid for accurate measurement to be made. The increase in concentration of chlorides on the downstream side was measured daily using an Ion Selective Electrode (ISE). Steady state diffusion conditions were reached between 7 and 14 days depending on the surface treatment system being tested. The diffusion coefficient of the composite system was calculated using the following equation derived from Dhir et al (9);

$$\ln (C_1 - C_2) = -\frac{D_{comp}A}{VL}(t_1 - t_2) + \ln C_1 \qquad (2)$$

Where, C_1 = Upstream chloride concentration (ppm)
C_2 = Downstream chloride concentration (ppm)
D_{comp} = Coefficient of chloride diffusion for the composite specimen (cm^2/s)
A = Specimen test area (cm^2)
V = Volume of downstream cell reservoir (cm^3)
L = Composite specimen thickness (cm)
t_1, t_2 = Time difference in between C_2 concentration readings

The coefficient of chloride diffusion for the surface treatment system alone was then calculated using the following equation;

$$D_c = L_2 \times \frac{1}{\left(\frac{L}{D_{comp}}\right) - \left(\frac{L_1}{D_1}\right)} \qquad (3)$$

Where, D_c = Coefficient of chloride diffusion for surface treatment alone (cm^2/sec)
D_1 = Coefficient of chloride diffusion for untreated substrate (cm^2/sec)
L_1 = Substrate thickness (cm)
L_2 = Coating thickness (cm)

Series II Tests: Concrete Substrate

Due to the higher degree of resistance to chloride transmission with the concrete substrate, a rapid chloride diffusion test (PD test) was used. This utilized a perturbative application of a small potential difference (7.5V DC) at a low current (250mA) across the concrete composite specimen in a diffusion cell. The increase in concentration of chloride on the downstream side was measured daily using the ISE as in the CD test above. The results were obtained between 16 and 28 days depending on which surface treatment system was being tested. For the PD test, the following equation derived by Dhir et al (10) and based on Einstein's relationship between drift velocity and the diffusion coefficient was used to calculate the chloride ion diffusion coefficient of the composite specimen.

$$D_{comp} = \frac{J\,K\,T}{C_1\, e_o \left(\frac{V}{d}\right)} \qquad (4)$$

Where, K = Boltzman's constant, (1.38×10^{-23}J/K)
T = Absolute temperature (°K)

e_0 = Electron charge, (1.602×10^{-19}C)
V = Applied electrical potential difference, (7.5V)
d = Distance between anode and cathode, (5.5cm)
J = Flux (ppm/cm/sec)

and

$$J = \left(\frac{V}{A}\right)\frac{dC_2}{dt} \tag{5}$$

Where, dC_2= Rate of diffusion of chloride ions into the cell reservoir, C_2.
dt = Time difference in seconds between C_2 concentration readings.

The diffusion coefficient for the surface treatment system alone was then calculated using Eqt. 3.

The treated test specimens were sealed in the uPVC diffusion cells and placed in the immersion tanks at 5 different temperatures, -10, 10, 20, 35 and 45°C, representing exposure environments varying from winter highway to desert conditions. For the sub-zero temperatures it was necessary to add 30% by volume of ethylene glycol to the tank and cell solutions to prevent freezing. The chloride concentration of the upstream solution was therefore reduced to 4M for the -10°C exposure temperature. The presence of the ethylene glycol was found not to affect the measurement of chloride transmission in solution using the ion selective electrode. Due to the complexity and long test periods singulate tests were used which had previously been established as adequate for determining D (9).

A good correlation (r = 0.98) between the calculated D values of specimens tested under both CD and PD conditions has been established (9) so that direct comparison between calculated diffusion coefficients from both the Series I and Series II tests could be made.

Results and Discussion

Variation of Dry Film Thickness

A series of 5 repeatability tests with the glass sintered and concrete substrates were carried out to assess the variation of dry film thickness development for each of the 7 surface treatment systems. Table 3 compares the variation in measured dry film thicknesses with the manufacturers recommended values.

The recommended thickness was not achieved for any of the treatments applied onto the glass sintered discs. It was found that with the concrete substrate only three coating types achieved the recommended coating thickness, the acrylic (CS-A-1), and the two coal-tar based epoxies (CS-E-2 and CS-E-4). Comparing the glass sintered and concrete substrates together, it can be seen (Table 3) that the mean thicknesses achieved were less with the sintered glass (with the exception of the methacrylate). This suggests that there was a greater penetration into the sintered disc than into the concrete, most probably due to a larger pore volume and size.

For all materials tested the results suggest that the recommended coverage rate should be increased. Based on this small sample, a multiplication factor of 1.2 - 1.3 would be appropriate to achieve the manufacturers recommended dry thickness on concrete. Factors such as the near surface porosity of the concrete and non-volatile content of the surface treatment systems may also have to be taken into account. Lower grade and poorly cured concrete with a higher near surface porosity would probably reduce the coating thickness due to treatment penetration. This may not be detrimental provided that subsequent additional applications of the surface treatment system result in the minimum dry thickness being achieved. However, these results do have major implications for the way in which surface treatment systems are specified. The variability of both the concrete substrate and surface treatment systems emphasises the need for in-situ performance tests to be based on a minimum dry thickness rather than specifying a wet application rate.

TABLE 3.

Variation in Dry Film Thickness.

SURFACE TREATMENT SYSTEM	DRY FILM THICKNESS, µm					*Min. Thickness* / *Rec. Thickness*	
	Manufacturers Recommended	Mean		Range			
		Concrete	S.G.	Concrete	S.G.	Concrete	S.G.
CS-E-1	400	342	306	285-373	220-440	71%	55%
CS-E-2	250	272	152	199-330	120-220	80%	48%
CS-E-3	400	372	208	288-452	180-240	72%	45%
CS-E-4	350	370	178	290-450	180-360	83%	51%
CS-M-1	150	128	132	104-144	110-160	70%	74%
CS-A-1	150	190	148	114-324	130-170	76%	87%
P-S/Sx-1	N/A	163*	140*	N/A	N/A	N/A	N/A

** Calculated from Eqt 1*

Series I Tests - Sintered Glass Substrate

Table 4 gives the diffusion coefficients of the glass sintered substrate composites and also that calculated for the surface treatments alone. It can be seen that all the surface treatment systems were effective in increasing resistance to chloride diffusion by up to nearly 7000 times. However, in

TABLE 4.

Coefficients of Chloride Diffusion - Sintered Glass Substrate.

SURFACE TREATMENT SYSTEM	COEFFICIENT OF CHLORIDE DIFFUSION		PROTECTION FACTOR w.r.t Control
	D_{comp}, $\mathrm{cm^2/s} \times 10^{-9}$	D_c, $\mathrm{cm^2/s} \times 10^{-9}$	
Control*	2,200	-	-
CS-E-1	0.76	0.05	2,900
CS-E-2	0.71	0.01	3,100
CS-E-3	3.95	0.22	600
CS-E-4	0.42	0.19	5,300
CS-M-1	0.70	0.02	3,100
CS-A-1	74.70	2.90	30
P-S/Sx-1	0.33	0.01	6,600

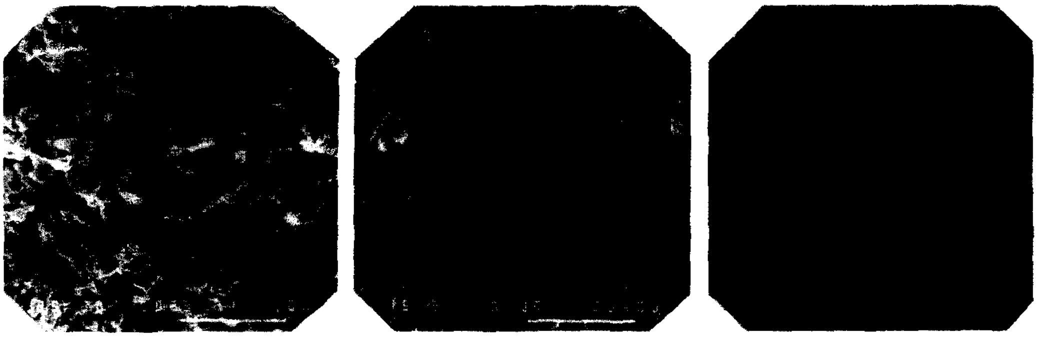
** 1M NaCl upstream concentration* D_{comp} *= Composite specimen* D_c *= Treatment system alone* comparison to the other treatments, the improvement due to the acrylic (CS-A-1) treatment was far more limited.

The general order of effectiveness of the surface treatments against chloride diffusion was:

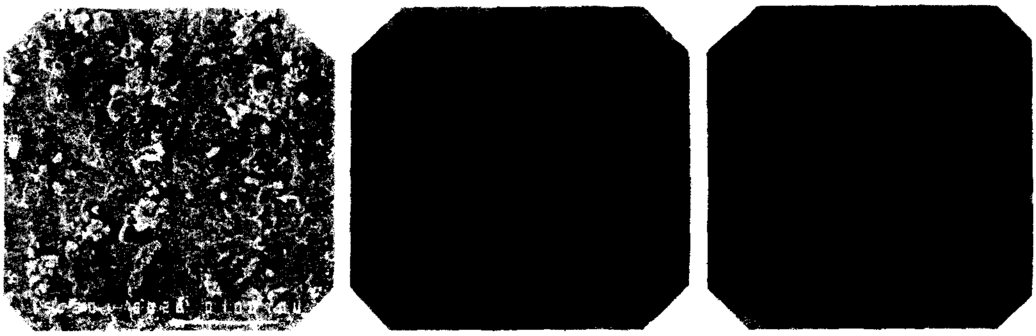
Silane/Siloxane > Pitch Epoxy > MMA > Non-pitch Epoxy > Acrylic

Photomicrographs of a selected series of the surface treatments applied to the sintered glass substrate are shown in Figure 1. These directly support the chloride diffusion test results as it can be seen that the physical barriers formed by each of the surface treatments show the presence of defects.

The acrylic shows a thin and poorly developed coating whereas the methacrylate was well formed but showed a series of small surface cracks. It was not clear what the significance of these were or how far they penetrated into the coating. In contrast, the epoxy provides a well formed barrier which did not crack but showed a number of holiday type perforations of the coating. The silane/siloxane blend, being a pore liner, does not have these problems and may explain why it performed well.



a. Untreated SG Substrate *b. CS-E-1 (Epoxy)* *c. CS-M-1 (Methacrylate)*



d. CS-A-1 (Acrylic) *e. P-S/Sx-1 (Silane/Siloxane blend)* *f. P-S/Sx-1 @ x5400 mag. (Silane/Siloxane blend)*

Figure 1. Comparison of Surface Treatment Systems on Sintered Glass Substrate (at x300 mag.)

The coal-tar based pitch epoxies provided an effective barrier against chloride diffusion but they were found to emit noxious fumes during application. This factor together with their black colour makes them unacceptable for anything other than hidden applications.

Series II Tests: Concrete Substrate

Table 5 gives the measured chloride diffusion coefficients for the concrete composite specimens. The diffusion coefficients for the surface treatment systems alone were calculated using Eqt. 3 and the results are shown in Figure 2.

At the end of the accelerated exposure test period, Table 5 shows that for a number of surface treatment systems no chloride was detected as passing through the test specimen under the sub-zero exposure conditions. However, at a 10°C chloride exposure temperature only the silane/siloxane blend was wholly resistant to chloride diffusion.

It can be seen from Table 5 that for the control specimen, the chloride diffusion coefficient increases with increasing exposure temperature up to 45°C. Above 35°C exposure temperature this trend is less marked, suggesting that the ion diffusion velocity approaches a maximum value. This trend is probably due to the higher mobility of the chloride ion at elevated temperatures and similar trends have been noted elsewhere (10,11).

The diffusion values for the treated specimens follow a broadly similar trend although there are some exceptions. The CS-E-1 coating performed badly at the sub-zero temperature and on removing the specimen from the test cell it was found that there was extensive cracking across the surface of the coating. This suggests that this particular epoxy is not resistant to frost attack. Indeed, it was found that far from enhancing the performance of the concrete specimen at -10°C, the application of the coating actually rendered the substrate more susceptible to chloride diffusion. This was due to deterioration of the surface of the concrete substrate, possibly caused by stresses induced by the cracking of the epoxy. However, at the higher temperatures epoxy CS-E-1 performed comparatively well, again following the established trend.

The CS-E-4 coating would seem to perform well at the medium range temperatures but relatively poorly at high (>35°C) and sub-zero temperatures rendering it impractical for middle eastern and winter highway conditions. However, the other coal-tar based epoxy, CS-E-2, performed well at the lower end of the temperature scale. This suggests that even small changes in the epoxy composition can greatly affect performance under different environmental conditions. From the available information it may be deduced that for epoxy coatings at least, a reduction in percentage of solids content (from 100% for CS-E-2 to 70% for CS-E-4) leads to a reduction in chloride protection at extreme temperatures.

Of the other surface treatments the silane/siloxane (P-S/Sx-1) performed well at all temperatures except for the 45°C exposure environment. This may be due to the hydrophobic pore-lining nature of the material which deteriorates at high temperatures leading to a reduction in water-repellant properties.

The methacrylate coating (CS-M-1) performed well at all exposure temperatures, especially below 35°C and would seem suitable for use in a wide range of climatic conditions. The acrylic coating (CS-A-1) was the least effective of the materials tested, particularly at the higher exposure temperatures. Despite this, the acrylic coating still improved the resistance to chloride diffusion of the composite specimen by a factor of at least 2.5.

TABLE 5.

Coefficients of Chloride Diffusion - Concrete Substrate.

SURFACE TREATMENT SYSTEM	COEFFICIENT OF CHLORIDE DIFFUSION, D_{comp}, $cm^2/sec \times 10^{-9}$				
	Exposure Temperature				
	-10°C	10°C	20°C	35°C	45°C
Control	1.48	6.67	25.94	37.79	39.78
CS-E-1	a	0.14	0.16	0.21	0.98
CS-E-2	*	0.24	0.31	0.76	1.42
CS-E-3	*	0.21	0.29	0.72	1.18
CS-E-4	0.87	0.41	0.43	0.47	3.54
CS-M-1	0.06	0.08	0.09	0.28	2.54
CS-A-1	0.08	2.57	4.20	7.04	10.30
P-S/Sx-1	*	*	0.07	2.17	7.10

No chloride diffusion was detected *[a] Coating fractured*

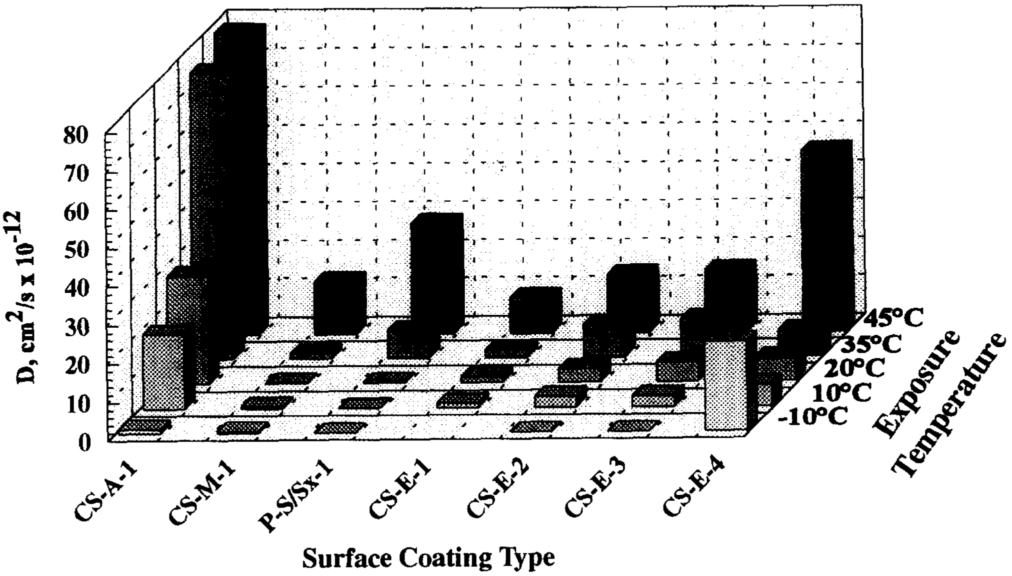


Figure 2. Variation of D Values for Surface Treatment Systems with Exposure Temperature.

When the results from the sintered glass substrate test at 20°C are compared with the results of the concrete substrate test at 20°C (Figure 3), it can be seen that the materials performance as measured by its chloride diffusion coefficient dramatically improves when applied onto a concrete substrate.

On the concrete substrate, D values for the surface treatment systems alone were reduced by around 75% for CS-E-2 to around 98% for CS-M-1 and CS-A-1 compared with the same materials applied to the glass sintered substrate. This would seem to suggest that these surface treatment systems, at least in part, derive their protective characteristics from the physical and chemical interaction with the substrate and that the nature of the substrate can significantly affect their performance. It was

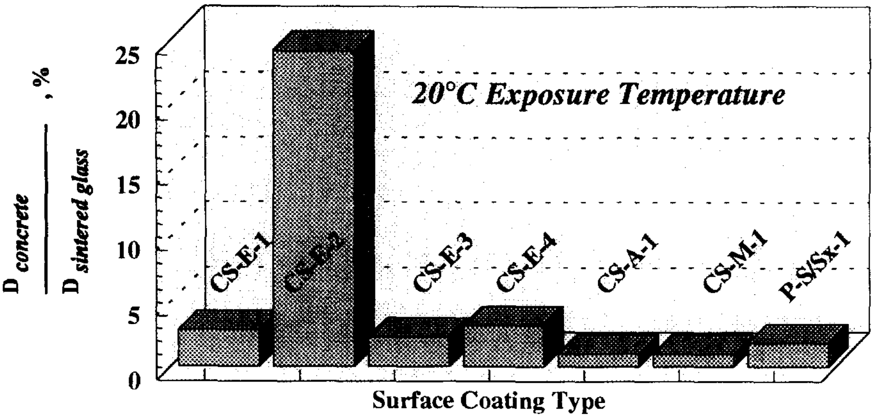


Figure 3. Comparison of Treatment Performance on Glass Sintered and Concrete Substrates.

not clear why this performance difference occurred for different substrates, but it would appear that factors such as pore fluid chemistry, size and distribution of pores and micro-surface roughness and structure play an important role.

A tentative, qualitative classification based on the composite diffusion coefficient, D_{comp}, is given in Table 6. This shows the degree of protection afforded against chloride diffusion by the surface treatment systems tested, over the range of exposure temperatures and when applied to a concrete substrate. At this stage it is not possible to establish the likely service life or how D_{comp} is affected by variations in the substrate characteristics or by weathering and ageing processes.

TABLE 6.

Effectiveness of Surface Treatment Systems.

SURFACE TREATMENT SYSTEM	EXPOSURE TEMPERATURE		
	Low (-10 to 10°C)	Medium (10 to 35°C)	High (35 to 45°C)
CS-E-1	Poor	Good	Good
CS-E-2	Good	Good	Good
CS-E-3	Good	Good	Good
CS-E-4	Good	Good	Poor
CS-M-1	**Excellent**	**Excellent**	Good
CS-A-1	Good	Poor	Poor
P-S/Sx-1	**Excellent**	**Excellent**	Poor

Excellent = $D_{comp} < 0.1 \times 10^{-9}\ cm^2/s$ *Good* = $D_{comp}\ 0.1\text{-}1.5 \times 10^{-9}\ cm^2/s$ *Poor* = $D_{comp} > 1.5 \times 10^{-9}\ cm^2/s$

However, it is important to begin to classify surface treatment systems due to their highly variable performance, even when, as was the case in this study, the application conditions are closely controlled. It is hoped that the proposed tentative classification can be further developed to allow the real potential of surface treatment systems to be understood.

Conclusions

1. All surface treatment systems, applied using the recommended coverage rate and method of application to a mature dry concrete surface, resulted in dry film thicknesses below that suggested as being a minimum by the manufacturer. It would therefore appear that typical application rates suggested by manufacturers are inadequate and multiple coats are required.

2. These trends suggest that, to ensure adequate performance, surface coating systems should be specified in terms of dry thickness. However, as yet no reliable test method is available for the in-situ measurement of this parameter.

3. All treatment systems provided a high degree of protection against chloride ingress and this followed the established trend of increasing diffusion rate with exposure temperature. There were a couple of exceptions to this for the epoxies at the sub-zero exposure temperature. Protection factors of up to 400 were measured compared with the untreated control.

4. Those materials that performed particularly well were the silane/siloxane hydrophobic pore liner, epoxy and methacrylate coating systems. However, even for these treatments the protection factors were greatly reduced at the 45°C exposure temperature.

5. The chemical and physical composition of the substrate would seem to have a significant effect on the intrinsic chloride diffusion properties of the material being applied. Chloride diffusion through materials applied to the glass sintered substrate was substantially higher than for the same materials applied to the concrete substrate.

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