

Research Paper

EVALUATION OF TEST METHODS FOR IMPREGNANT MATERIALS IN CONCRETE

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The corrosion of steel reinforcement in concrete is a major problem for the construction industry. Corrosion affects the durability of reinforced concrete structures which will lead to their failure. One of the main causes of concrete corrosion is chloride ions from deicing salt. The aim of this research was to evaluate the effectiveness of different test methods for measuring chloride penetration in concrete. Also investigate how the chloride penetration changed with different impregnate coatings on the concrete was examined. All the experiments were carried out on concrete cubes. Sorptivity test was carried out to examine the water uptake of the concrete cubes by considering uncoated as well as coated with impregnate materials in tap and salt water. The results showed that more water ingress into the concrete cubes immersed in the tap water. Salt ponding experiment were carried out on treated and untreated specimens and, chemical analysis method, Voltard's, was used to measure the depth of penetration of chloride ions into concrete samples. The Salt ponding tests on the cubes immersed in the salt water showed that the highest chloride concentration were in the increment of 5 mm and the concentration decreased with depth. The chloride ions concentration was similar for all the cubes at 20 mm depth. This suggests that deeper coring is required to determine the depth to which the chloride ions reach. The untreated concrete cube had higher chloride content than the treated cubes

Keywords: Sorption test, Salt ponding test, Impregnate materials, Chloride ingress, Corrosion, Volhard's methods

INTRODUCTION

It is a well-known fact that buildings, bridges, aqueducts, monuments, statues, carved facades and other structures which constitute Europe's cultural heritage are subject to varying degrees of cosmetic damage as a result of the combined effect of natural

weathering and deposition of industrial pollutants. Ironically, pollution is a product of industrial growth, a sign of prosperity in industrialized countries. Some of the structures often become damaged to the extent that there is loss of strength and danger to the public. The general problem of weathering has been

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recognized for many years. For example, the Building Research Establishment first published 'The Weathering of Natural Building Stones' in 1931 which was reprinted in 1972 (Schaffer, 1972). Application of surface treatments has become recognized as one of the most practical methods of conserving historic structures. Various types of protective treatments have been developed over the years, varying from natural materials such as lime washes to polymeric coatings. Heritage organizations responsible for the maintenance of structures tend to prefer the former but these have the disadvantage of coloring (whitening) the surface of the stone, and require to be reapplied at regular intervals. Many types of sophisticated polymers and protective coatings are now commonly used in many countries to counter the problem of stonework degradation due to natural weathering or to prevent premature corrosion of steel reinforcement in highway concrete bridges. Most have the advantage of not changing the original color of the surface. However, these new treatment materials have finite lives and therefore need to be renewed after approximately 15 years due mostly to degradation caused by the effects of ultra-violet radiation (Pfeifer and Scali, 1971). There has been considerable interest in developing methods for assessing the performance of surface treatments which can enable owners of structures to appreciate when treatment has been properly applied or when the need arises to retreat an already treated but subsequently weathered surface. For instance, Whiting *et al.* (1992) investigated methods of evaluating the effectiveness of penetrating sealers as part of an SHRP program. The National Bureau of

Standards (1997), Heritage Institutions and the European Commission (Van Hees *et al.*, 1995) have all sponsored investigations involving methods of assessing the efficacy of coatings on porous building materials in recent years. Other examples include the EC programs on the 'effects of air pollution on listed buildings' (1986-1990), the STEP program on the 'Protection and Conservation of the European Cultural Heritage' (1989-1992), EC environmental program on the 'Environmental Protection and Conservation of Europe's Cultural Program' (1991-1994). Further investigations have also been carried out on the efficacy of surface treatments on different historic structures in England (Butlin *et al.*, 1991), and other European countries (Ross *et al.*, 1990). Literature on this subject reveal an array of previous research and published works focused mostly on laboratory-based and destructive site methods (Gerdes, 1995; Bunty and Rostasy, 1985; Wendler *et al.*, 1993). A few standard recommendations exist for non-destructive site assessment of surface-treated structures. The objective of this investigation was to evaluate two different test methods currently used to measure chloride penetration in concrete by comparing the ingress of chloride ions into concrete, and examine the water sorption of concrete cube samples treated with different impregnate materials. In turn to determine whether current test methods are adequate for the measurement of chloride ingress into concrete

LITERATURE REVIEW

The durability of concrete plays an important role in the surface life of a structure. The

durability of the reinforced structure cannot be measured directly, however, it is measured in terms permeability that measures the resistance against penetration of various harmful substance into the concrete. Three mechanisms permeability, diffusion and sorption are responsible for the movement of the fluids (gases and liquids) into the concrete. Permeability is the flow under pressure, in diffusion, the flow takes place due to the difference in concentration and the sorption phenomenon is also a process of diffusion in which main mechanism is capillary suction. The cover of the reinforced concrete structures is the first barrier that comes in contact with the aggressive substances. Hence, the quality of the cover concrete must be evaluated in terms of permeability in order to rate the durability of the structural element. In countries, like Japan due to the long coast line most of the structures are exposed to the marine environment. In such environments the main durability problem is chloride induced reinforcement corrosion. Diffusion is considered one of the main mechanisms for the transportation of the chloride ions into the cover concrete. Water ingress into the concrete accelerates the process of transportation. The covercrete may have poor resistance against the permeation of aggressive substances, chemical, abrasion and frost action due to the following factor such as Poor curing, Segregation, Inadequate compaction, Bleeding, and Micro-cracking. The poor quality leads to deterioration of concrete in terms of reinforcement corrosion that may result in Spalling of cover concrete or cracking in concrete due to corrosion, Alkali Silica Reaction (ASR) and freeze thaw action. In the

past, generally, it was believed that by increasing the strength of concrete durability can be improved. However, from the much early deteriorations of concrete structures it was realized that not only the strength but also the resistance against permeations must be ensured.

The Durability of hydraulic-cement concrete is defined as its ability to resist weathering action, chemical attack, abrasion, or any other process of deterioration. Durable concrete will retain its original form, quality, and serviceability when exposed to its environment (ACI Committee 201). Nevertheless, until recently, developments in cement and concrete technology have concentrated on higher and higher strengths. There was an assumption that 'stronger concrete is durable concrete'. It is now known that, for many conditions of exposure of concrete structures, both strength and durability have to be considered explicitly at the design stage (Neville, 1995). There is no generally accepted method to characterize the pore structure of concrete and to relate it to its durability. However, several investigations have indicated that concrete permeability both with respect to air and to water, is an excellent measure for the resistance of concrete against the ingress of aggressive media in the gaseous or in the liquid state and thus is a measure of the potential durability of a particular concrete (CEB-FIP Model Code 1990). Durability of structural members mainly depends on the penetration resistance of a very thin layer of their surfaces generally known as cover concrete. This thin surface layer was termed as "covercrete" first time by Dewar (1985). Covercrete acts as first barrier against the

ingress of aggressive substances like chloride ions, carbon dioxide from the environment or against the resistance to chemical, frost or abrasion attack. Covercrete normally has different composition, microstructure and properties as compared with the core concrete, its vital role in the durability performance recently been recognized (Dewar, 1985; Kreijger, 1984; Mayer, 1987; Newman, 1987). Main causes for this difference includes segregation, improper placement of concrete, inadequate compaction, type of finishing and most importantly due to poor curing. Presence of micro-cracks also increases its vulnerability towards deterioration. A brief description of these factors is included here (Torrent and Fernández, 2007). In fact there are so many tests methods that are currently being used to measure the permeation resistance of concretes, will be included. Each test method works on a certain principle, however all the tests face some problem due to specific properties of concrete such as ageing of concrete due to on-going hydration, Reactivity of concrete with penetrating substances studied, for instance water, carbon dioxide, chloride ions, etc. Variability of concrete properties with moisture content of concrete, Sensitivity of concrete pore structure to preconditioning, e.g., micro cracking upon drying, Pore water composition, its effect on, and interaction with, transport processes.

EXPERIMENTAL WORK

The present research will focus on an experimental work such as Salt ponding test, Sorption, and Dye diffusion test. In which casted 15 concrete cubes of size about 100

mm with Type C as specified in EN: 1766 (2000). The used mix is represented as in Table 1 and is a representative of concrete that is typically used on highway bridges. The aggregate was prepared and the concrete cast in accordance with standard TRL (Transport Research Laboratory) procedures. The aggregate and water were first mixed for 4 minutes in EL 34 3540 /01 ELE Concrete mixer. The cementitious materials were added and mixing for more 3 minutes was carried out. The concrete was then placed in the moulds

Materials	Fraction	Saturated Surface Dry weights(kg/m ³)
Cement	30.96	
Laleham	Sand	55.64
Laleham	5-10mm	32.85
Laleham	10-20mm	65.7
Free Water		13.95
w/c ratio		0.45

in two layers and each layer was vibrated until fully compacted. The specimens were cured under damp hessian for 24 hours, demoulded and cured under damp hessian for a further 6 days. The cubes were then kept for a further 18 days to complete the curing of the concrete. (Total 28 days from totally casting).

Four types of protective coating were used to coat the concrete cubes for the chloride ingress test and Table 2 lists the type of product coatings.

Before the concrete cubes were coated with impregnate materials they were all

Table 2: Types of Coating Product			
Product Type	Base	Colour	Curing Time
A	Silane	Colourless	7 Days
B	Water Based Silane	End colour depends on initial colour of surface	7 Days
C	Water Based Silane	White	7 Days
D	Water Based	Cream/ Greyish	7 Days

cleaned of dust and loose material by blowing the surfaces with a compressed air. The amount of impregnate material on all the cubes was calculated by weighting the cubes before coating and after coating. The manufacturer’s recommendations on coating amounts were followed applied as shown in Figure 1. Impregnate material D was a two part coating system applied over two days while A, B and C were applied in one coat. In which prepare



the cubes for sorption test, salt ponding test by immersion and dye penetration test. Ten cubes were taken, two were kept untreated, as controls, and the other eight were coated on all faces with products A-D. Two cubes were treated with each product. The cubes were cured for seven days.

Five cubes were taken, one cube was left

untreated and the other four had one surface coated with one of the impregnate products in case of salt ponding test. All the coated cubes



had timber frames glued to the treated surfaces as show below in Figure 2. The depth of the frame was 10 mm. The untreated cube had a frame attached in the same way as for the treated frames, for use as a control. The frame was required to allow salt ponding of the concrete surface.

The Sorption test was carried out to examine the moisture absorption into the impregnate coated and untreated cubes. Ten cubes were tested. The test method being used is given in TRL. The test was carried out in two parts. The 10 concrete cubes were weighed and then placed in a container of tap water. The weight gain or loss of treated and untreated cubes were monitored. The weight gain of the samples was monitored after 1, 4, and 24 hours and then periodically up to eight

days. Before weighting, the samples surface were dried with a paper towel. After eight days the experiment was repeated with the cubes being immersed in water containing 5 g/l of NaCl. Two different tests were carried out to examine the sodium chloride ingress into concrete cubes. One test used fully coated cubes and the other using the salt ponding cubes, these had only one surface coated with an impregnate material and a wooden frame glued onto that surface. The ten cubes used in the sorption test were used for this test in case of Salt ponding of fully coated cubes. They were fully immersed in a container filled with tap water containing sodium chloride. The concentration of the salt was 5 g/l as before. The samples were left for three months. The Ponding and Chloride profile stages for salt ponding test that has been carried out in accordance to BS EN 14269:2007 as shown in Figure 3. To measure the chloride ingress into the concrete, samples of concrete at different depths in the cube were taken. A diamond drill which allowed dry drilling was used to take powder samples at different depths, 5, 10, 15 and 20 mm. A minimum size of approximately 1 g was required.

The four cubes with the impregnate coating and the untreated cube which had the wooden

frames glued. A sodium chloride solution of concentration 5 g/l was poured into the frame. The frames were topped up with salt solution every 14 days for three months in case of Salt ponding experiment for one surface coated cubes. The cubes were then used in a dye penetration test. The dye penetration test method was followed from Chamberlain (2004). The test samples were all broken in two using Contest Instrument Type GD10A compression testing machine. The samples base were immersed to a level of 10 mm in a solution of fluorescein dye (0.5 g/l) for about 120 h. The cubes tested were supported in a tray on polymer sheet to ensure their bases had the dye around them. The samples were kept in the dye for 5 days. They were then removed and the water level in the concrete noted. The samples were then air dried for two days. The level of flures in dye in the samples was measured using a UV lamp. The samples tested by this method were salt ponded cubes which had only one face coated by the impregnate materials and five cubes which had been fully coated and salt ponded by immersion in a container of salt water.

DISCUSSION ABOUT RESULTS

Sorption Test

The results show that all the concrete cube

Figure 3: Dust Sample Collection and Diamond Drill used for Chloride Profiling



gained weight over a period of eight days. Some of the samples lost weight over the first few hours of the test but then gained weight

steadily. The weight gain was greater for all the samples in the tap water. This suggests that the salt in the water may reduce the ingress

Table 3: Shows the Amounts of Consumption of Impregnate Material

Cube ID	Product	Mass (g)	Mass+Pro (g)	Mass of coating (g)
A1	A	2371.1	2379.6	8.5
A2	A	2370.1	2377.2	7.1
B1	B	2431.7	2439.6	7.9
B2	B	2356.5	2360.6	4.1
C1	C	2382.8	2386.3	3.5
C2	C	2387.4	2391.1	3.7
D1	D	2303.6	2313.3	9.7
D2	D	2356.7	2368.8	12.1
	area =	0.01m ²		

Table 4: Weight Gain of Concrete Cubes (Dipped in Tap Water for 8 Days)

Product	Time								
	Cube ID	1 h	4 h	1 day	2 day	3 day	4 day	7 day	8 day
	Weight gain (g/m ²)								
A	A1 (F)	1.9	2.7	4.0	4.6	5.2	5.8	5.9	4.1
	A2 (J)	1.1	1.7	2.2	3.2	3.4	3.7	4.3	4.5
B	B1 (D)	3.0	5.5	12	14.9	17.6	21.8	22.8	23.7
	B2 (G)	1.4	3.2	8.9	13.6	16.2	19.1	24.4	24.9
C	C1 (H)	0.7	1.0	1.6	2.7	3.3	3.8	4.4	4.7
	C2 (K)	0.7	1.2	2.5	4.1	4.6	5.4	6.6	6.7
D	D1 (I)	2.8	8.2	17.5	20.4	24.9	29.2	33.9	34.3
	D2 (L)	6.1	13.4	14.5	18.1	19.4	20.5	22.1	22.0
Untreated	M	22.9	27.7	32.5	34.4	35.0	35.7	36.3	37.0
Untreated	N	24.5	29.2	34.1	36.0	36.7	37.3	38.5	38.2

Table 5: Weight Gain of Concrete Cubes (Dipped in Salt Water for 8 Days)

Product	Cube ID	Time							
		1 h	4 h	1 day	2 day	3 day	4 day	7 day	8 day
A	A1 (F)	0.4	0.6	0.7	0.6	0.3	0.1	0.0	0.0
	A2 (J)	0.5	0.1	0.5	0.7	1.0	1.5	1.6	1.7
B	B1 (D)	0.2	0.2	0.4	0.9	2.0	2.4	2.6	2.6
	B2 (G)	-0.7	-0.5	0.7	1.7	2.5	4.5	4.9	5.4
C	C1 (H)	0.5	0.7	0.9	1.4	1.5	2.0	2.3	2.5
	C2 (K)	0.6	0.9	1	1.2	1.5	2.2	2.3	2.5
D	D1 (I)	1.7	1.2	3.0	4.0	4.7	6.3	6.5	6.9
	D2 (L)	3.0	3.8	5.5	6.2	6.7	7.7	8.1	8.2
Untreated	M	4.7	5.2	6.5	7.2	7.6	8.4	8.5	8.8
Untreated	N	7.4	7.8	9.2	9.8	10.1	11.0	11.1	11.3

of water into the untreated and impregnate coated concrete cubes (Tables 4-5). There is some experimental variation results between

samples coated with the same material this may be due to the differences in the amount of coating on the cubes (Table 3).

Figure 4: Performance of Product a in the Sorptivity Test

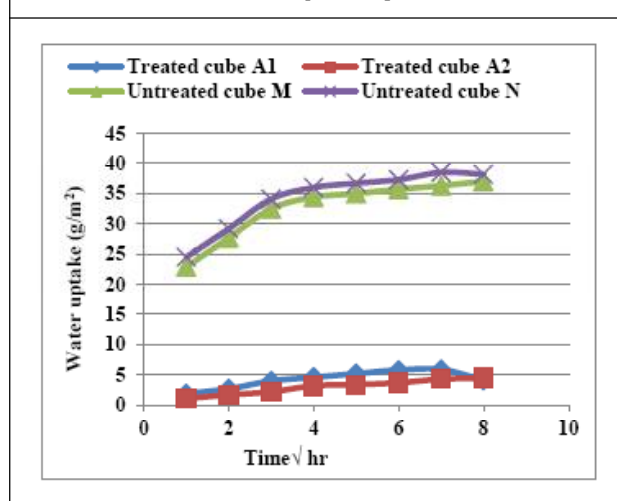


Figure 5: Performance of Product B in the Sorptivity Test

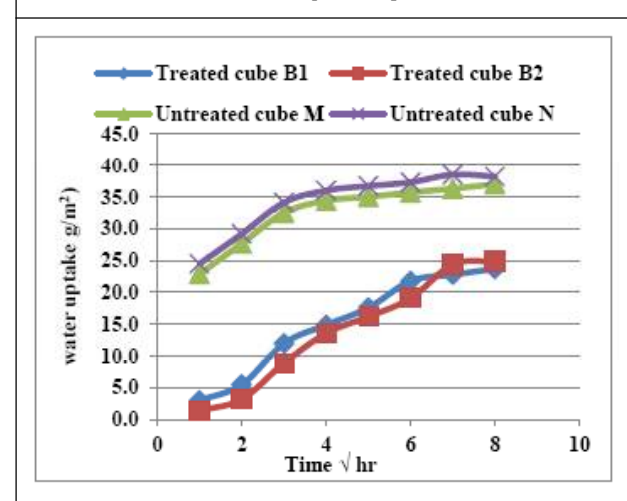


Figure 6: Performance of Product C in the Sorptivity Test

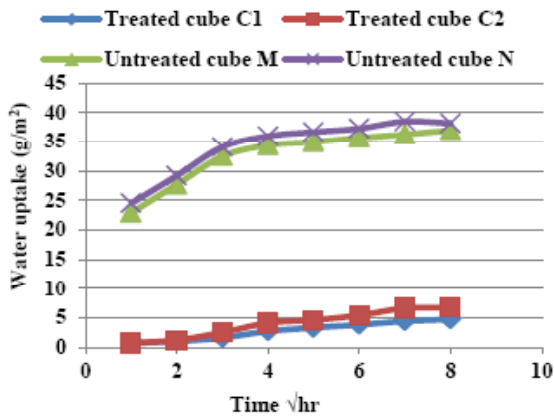


Figure 7: Performance of Product D in the Sorptivity Test

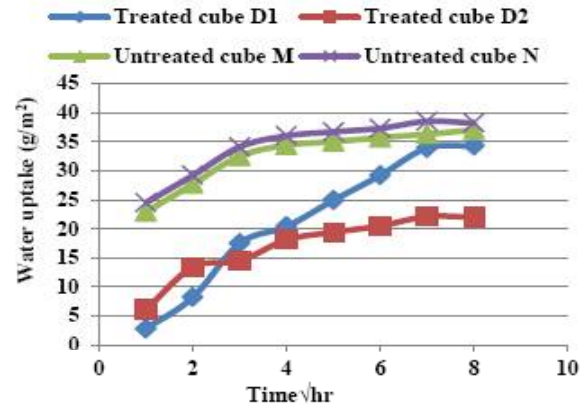


Figure 8: Performance of Product A in the Sorptivity Test

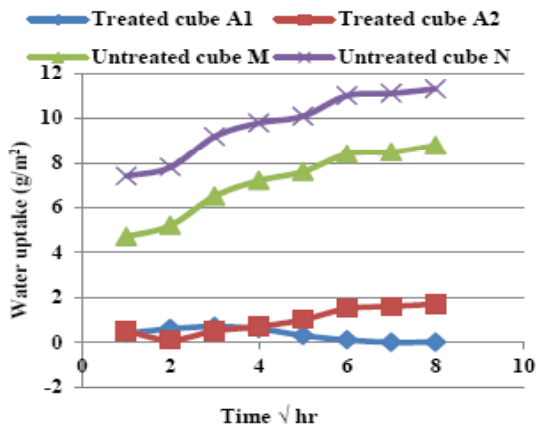


Figure 9: Performance of Product B in the Sorptivity Test

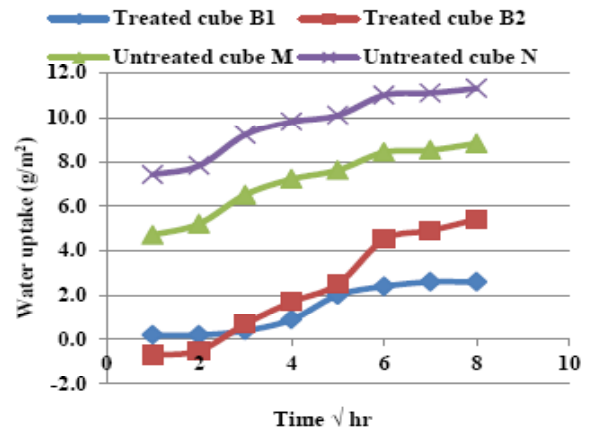


Figure 10: Performance of Product C in the Sorptivity Test

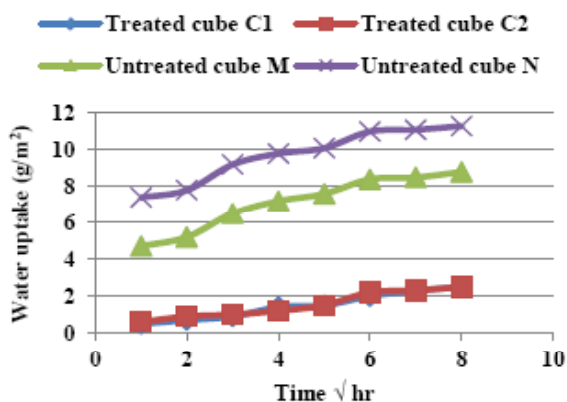
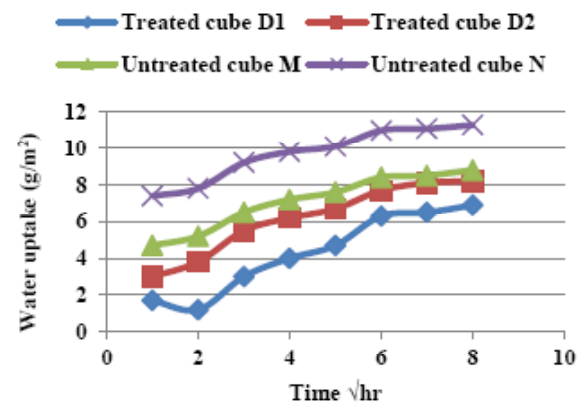
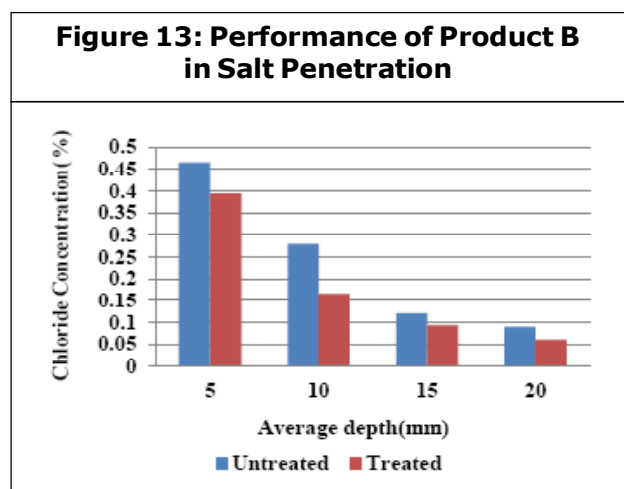
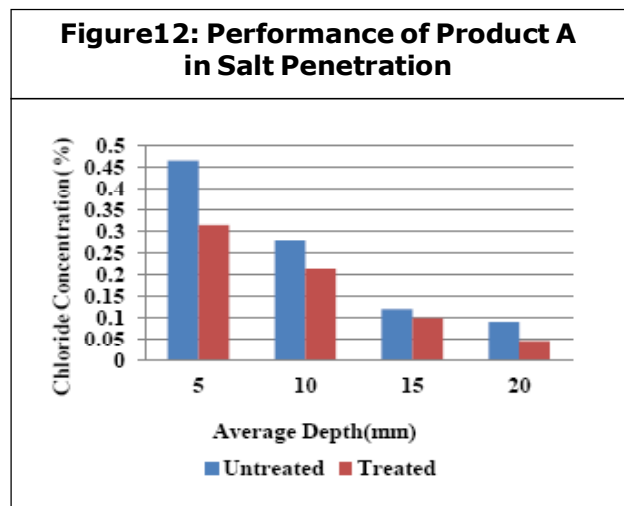


Figure 11: Performance of Product D in the Sorptivity Test



The result obtained during this two sets of test (immersed in Tap and Salt water) to measure the water uptake into concrete cubes shows that the cubes that coated with Silane water base impregnate materials has steadily and lower rate of Water absorption that has been travelled into concrete when compared to other two product which are water base impregnate materials. The variation in two cubes with the same coat in the same water, particularly cube D2 which has been coated with product D as shown in Table 4, the rate of water uptake between 2.8 g/m² to 34.3 g/m² compare to cube D1 which has been coated with the same product. However the same product and when dipped in salt solution for same period has gained water in range between 1.7 g/m² and 8.2 g/m². Similarly in Sorption test the performance of different Impregnate products (A, B, C, and D) were represented in Figures 4-7 and Figures 8-11 (immersed in Tap and Salt water). The performance (Tap water) of products (A, B, C, and D) in the Sorption test was depicted in Figures 4-7. In which Water uptake (22.50 g/m²) was lower at initial time duration as when compared to longer time duration (36.50 g/m²). Thus there is an increase in (all products A, B, C, and D) Water uptake in longer time duration (38.35%) as when compared to initial time duration in all Untreated concrete cubes. Similarly the Water uptake in products was varied in the range of about A (1-5 g/m²), B (3-25 g/m²), C (1-6 g/m²), and D (5-27.50 g/m²) in all Treated concrete cubes. The performance (Salt water) of products (A, B, C, and D) in the Sorption test was depicted in Figures 8-11. In which Water uptake (4-8 g/m²) was lower at initial time duration as when

compared to longer time duration (8-12 g/m²). Thus there is an increase in (all products A, B, C, and D) Water uptake in longer time duration (60%) as when compared to initial time duration in all Untreated concrete cubes. Similarly the Water uptake in products was varied in the range of about A (0.5-1.70 g/m²), B (-0.70-5.40 g/m²), C (0.60-2.50 g/m²), and D (1.70-6.90 g/m²) in all Treated concrete cubes. Thus there is an increase in (all products A, B, C, and D) Water uptake in longer time duration (70.50%), (87.03%), (76%), and (208%) as when compared to initial time duration in all Treated concrete cubes.



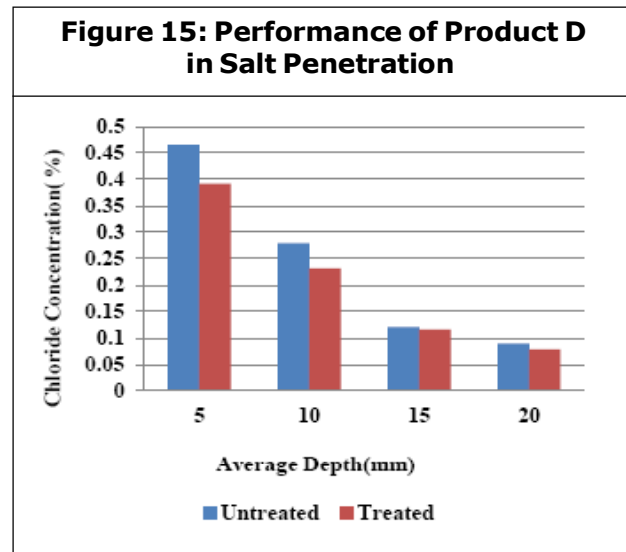
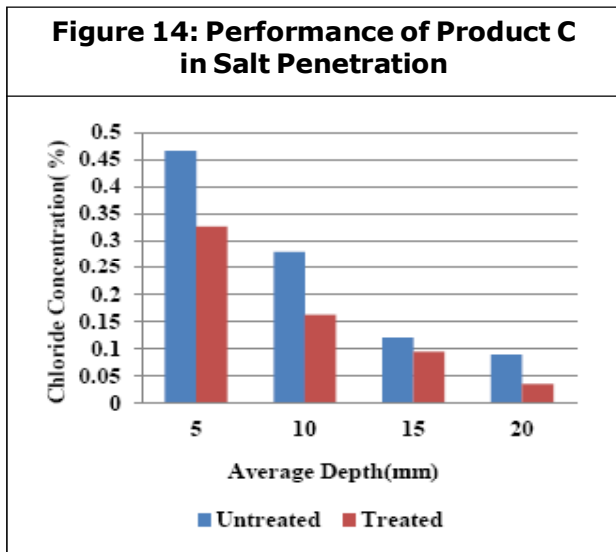


Table 6: Chloride Concentration Obtained During Titration Test by Using Volhard’s Methods

Product	Total Chloride Ion Concentration(%)			
	Depth Range (mm)			
	0-5	5-10	10-15	15-20
A	0.3143	0.2138	0.0995	0.0465
B	0.3941	0.1672	0.0933	0.0603
C	0.3265	0.1626	0.0954	0.0363
D	0.3917	0.2311	0.1165	0.079
		Untreated Cubes		
Control 1	0.4653	0.2796	0.1207	0.0899

Salt Ponding Test

The results for the fully coated concrete cubes which had been immersed in a NaCl solution for about 3 months and the chloride levels at different depths were measured by Volard’s method are given in Table 6. The results for each Coated concrete cube is compared with the result for the Untreated concrete cubes as represented in Figures 12-15.

Dye Penetration Profile

The depth of Fluorescein dye in the concrete cubes was examined using a UV fluorescent lamp. The average penetration of the water and dye were calculated, it can be observed that, there is a lot of variation in penetration, because the concrete cubes have been split and the surface is uneven, and also the dye could travelled through the porous concrete and around the aggregates. In which two sets

Table 7: Dye Depth With Respect to Water Level for One Surface Treated Concrete Cube

Product/Picture Code	Dye Average Depth for One Surface Treatment	Water Average Level for One Surfaces Treatment
AA	16.84	25.43
B/B	11.392	27.06
C/C	13.90	30.55
D/E	14.38	27.63
Untreated/O	26.75	31.11

Figure 16: Dye Depth for one Surface Treated Concrete Cube in Products A,B,C and D Respectively



Figure 17: Dye Depth for all surface Treated Concrete Cube in Products F,G,H, L and N Respectively



of results of one face coated and fully coated concrete cubes as shown below in Figures 16-17 and Tables 7-8 showed Dye depth compare to Water level for one and all surface treated concrete cubes.

The results show that have been obtained throughout this test (Figures 16-17) shown

clearly that the untreated cubes, O and N have a higher level of water penetration while the dye penetration is similar to the coated cubes. The exception being the cube A/F where water had ingresses to a very high level but the dye penetration was similar to the other coatings. The water was in the centre of the cube and

Table 8: Dye Depth With Respect to Water Level for all Surface Treated Concrete Cube

Product/Picture Code	Dye Average Depth for all Surfaces Treatment	Water Average Level for One Surfaces Treatment
AF	14.77	78.20
B/G	10.03	26.80
C/H	13.69	30.08
D/L	9.214	16.29
Untreated/N	10.17	43.33

not around the edges in this cube. The results for coatings B and C show very little difference for both tests for the dye penetration. This may also related to the amount of coating that has been used for each which is vary in a range between 4.7 to 12.7 g/m² as shown in (Table 7), Product B despite its having lower rate of coating (4.7 g/m²), but Still keep a smaller amount of salt penetrated, this may due to the fact that this product is Silane water base.

CONCLUSION

- The results show that in the case of Sorption test that, the concrete cube gained weight over a period of eight days. Some of the samples lost weight over the first few hours of the test but then gained weight steadily. The weight gain was greater for all the samples in the tap water. This suggests that the salt in the water may reduce the ingress of water into the untreated and impregnated concrete cubes. There is some variation in experimental results between samples coated with the same material this may be due to the differences in the amount of coating on the concrete cubes.
- The Salt ponding tests on the cubes

immersed in the salt water for 3 months showed that the highest chloride concentration were in the increment of 5 mm and the concentration decreased with depth. The chloride ions concentration was similar for all the cubes at 20 mm depth. This suggests that deeper coring is required to determine the depth to which the chloride ions reach. The untreated concrete cube had higher chloride content than the treated cubes.

- In Dye penetration test, the dye penetration was of the order of 15 ml for all the concrete cubes, and when compared with the Salt ponding test suggest that, the Fluorescein dye is not travelling into the concrete to the same level as the chloride ions. However the water level also has been observed and it's clear that the water has been penetrated in larger amount compared to the dye level. The reasons for this, the Fluorescein dye molecular size is larger than the chloride ion and (Molecular weight 36.5) and water (18). In addition to that, UV lamp used was not possess sufficient frequency for detecting low dye concentration levels. In case of Dye penetration test, more number and different

sized specimens, type of dye and lamps with specific range need to be tested to increase the accuracy of the results. Some of the variation in the results particularly in case of Dye penetration test are because of the dye movement around aggregate particles in the cement matrix.

- The measurement of chloride ions at deeper depths is required as the results show that there was chloride concentration present at 20 mm in the samples tested.

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