

Research Paper

EFFECTS OF INTERNAL MOISTURE ON THE PERFORMANCE OF IMPREGNATES

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The durability of reinforced concrete structures is greatly affected by corrosion of steel reinforcement through chloride attack. The use of impregnates is the most widely employed method to tackle chloride attack for structures in the UK. Impregnate manufacturers and DMRB standards stresses the use of impregnates on dry surfaces, achieving a dry surface is easy however moisture below the surface exists and this can affect the performance of the impregnates. The Concrete cubes were cast and cured, before being submerged in a water bath to determine their moisture content at various times. The cubes were surface dried, water and silane based impregnates were applied and then submerged in salt solution. The cubes were dry drilled at various depths of 5-20 mm for dust samples, which were used to obtain chloride concentrations by the method of Volhards titration. The performance of impregnates is dependent on the quality of concrete, as it directly influences the pore structure which in turn determines the rate of chloride ingress. Impregnate application on dry surface showed a chloride concentration of 0.029% at 20 mm. However impregnates applied to a dry surface, but with a moisture content of 1.88% showed a chloride concentration of 0.053%. This indicates the performance of impregnates is effected by moisture. Although solvent based impregnate performed better than the water based in dry condition, their performance are similar when applied in the moist concrete.

Keywords: Impregnate, Pore structure, Chloride concentration, Silane based, Water based, Performance

INTRODUCTION

The need to protect concrete structures against moisture and chemicals has always been necessary as it affects the service life of the structure. Reinforced concrete structures are all around us and play a vital part in society.

One of the many factors that can substantially reduce the design life of reinforced concrete structure is chemical attack. Marine structures, tunnels, retaining walls, concrete pipes and bridges are susceptible to chemical attacks from the ocean, harmful exhaust fumes, acid

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rain and most commonly from de-icing salts.

The De-icing salts, mainly sodium chloride are being used in increasing amounts, as the road network increase in size and winter periods become harsher. The spreading of de-icing salts although has a significant beneficial effect on keeping the roads safe during icy conditions, they pose a great threat to highway structures, due to the chloride ions from the salt penetrating through the pores of the concrete structure (Al-Zahrani *et al.*, 2002). This mechanism involves chloride ions from the de-icing salt penetrating through the pores in the concrete bridge piers and other structures, which over time cause the steel reinforcement to start corroding and the concrete spalling/bursting.

The use of superficial protection for concrete is a possible way of increasing the service life of reinforced concrete structures exposed to harsh environments. This type of protection inhibits the penetration of aggressive agents by diffusion and capillary absorption. The surface protection materials for concrete can be classified into three groups: Pore liners (make the concrete water-repellent), pore blockers (react with some of the soluble concrete constituents and form insoluble products) and coatings (form continuous film on the concrete surface).

The coatings and sealers present the advantage of providing a physical barrier on the concrete surface, which isolates it from the aggressive agents of the external environment. These coatings require a homogeneous and smooth substrate with pores of 0.1 mm width at most (Jacob and Hermann, 1998). This means that they are capable of covering a 0.1

mm wide crack. However, the film breaks if the concrete structure cracks after painting. Moreover, coatings do not allow the concrete to dry if it is wet. This can cause the deterioration of the film, causing the formation of bubbles due to the vapor pressure of the internal humidity. From the architectural point of view, this group of surface treatment modifies the aesthetics of the structure adding brightness or color to the concrete surface, which is sometimes desired (Jacob and Hermann, 1998).

Coating and sealers for surface treatment have been intensely studied in the last fifteen years. Research carried out by (Jones, 2002) studied the importance of the parameters modulus of the material and viscosity in the crack-bridging ability of the coating. Uemoto *et al.* (2001) showed a correlation between the paint pigment volume content and water permeability. Al-Zahrani *et al.* (2002) showed that the accelerated corrosion performance of the four coating systems studied correlates well with the performance results obtained from the physical properties, in particular, water absorption, water permeability, and chloride penetration. Researchers (Medeiros *et al.*, 2009) suggested that the determination of the chloride diffusion coefficient allows a quantitative comparison of the protection systems and, therefore, needs to be made possible in migration tests. This kind of protection is probably the most used by the construction industry and that would be the reason why they are widely studied.

RESEARCH SCOPE AND OBJECTIVE

Until, recently in the UK, hydrophobic

impregnation using high active content silanes and siloxanes materials was the most commonly specified protective provision for reinforced concrete (Vries and Polder, 1997). However, as part of the ground swell movement away from the use of solvent based materials, the past decade as seen increasing use of water based silanes and other materials founded on dissimilar chemistry. Compliance of such alternatives with EN1504-2 has acted as a driver in this. Recently, however, there have been growing concern regarding the onsite performance of all hydrophobic impregnation materials, traditional and alternative (Jacob and Hermann, 1998). It seems that there is marked discrepancy between outcomes of laboratory testing and apparent defence of actual treated structures. It is recommended that the application of impregnate should take place on a "dry surface" to allow correct penetration of the product and hence, maximum effectiveness. Having a dry surface does not necessarily mean the complete structure is dry. It is important that effectiveness of the impregnate can be determined depending on the moisture content within the concrete itself. It is probable that climatic conditions and moisture content prevailing at material application time are extremely influential in this. They bear directly on the achievable dosage with protection materials and thus the starting level of production provided. These influences are evaluated in this study.

In this paper, results from a comprehensive laboratory investigation on the effect of internal moisture on the performance of impermanent are presented. Two impregnates, silane and water based, were evaluated in terms of their

efficiency in retarding water absorption and chloride penetration. The results are compared with the same mixture moisture content mix without any protection applied during the testing.

BRIEF OVERVIEW OF CONCRETE IMPREGNATION

Water repellents or hydrophobic agents are produced from silicon resins that are chemically bound to the concrete base. Currently, the most commonly used agents are silanes, siloxanes oligomeric and a mixture of these two components. Chemically, silanes are formed of small molecules that have one silicon atom and siloxanes are short chains of a few silicon atoms in which the molecules have alkoxy groups (organics) connected to the silicon atom. Silanes and siloxanes react with the silicate of the concrete, forming a stable bonding (Vries and Polder, 1997).

Impregnation makes the surface of the concrete element water repellent but vapor permeable. This helps to protect the concrete from chloride ion penetration, hence reducing the risk of corrosion to the reinforcement (Calder *et al.*, 2006). Impregnates are frequently applied directly to the surface of the element via low pressure sprays which once dry do not affect the visual appearance of the structure and also allow ease of application (Delucchi *et al.*, 1998). Previous research showed that the penetration of the hydrophobic agent is better in finished faces than in formwork faces, due to the higher permeability of the second (Vries and Polder, 1997).

An alternative method reported by Peng Zhang *et al.* (2011) which are to add silane emulsion into fresh concrete or mortar to

produce integral water repellent materials (adding 1%, 2%, 3%, 4%, and 6% of silane emulsion). The influence of silane emulsion on the compressive strength, porosity, and pore size distribution, water capillary suction and chloride penetration have been investigated. The results indicate that addition of silane emulsion moderately reduced compressive strength of concrete.

With 3% of silane emulsion, the reduction is about 10%. The addition of silane emulsion hardly has influence on pore size distribution. Silane does not block the capillary pores, but only forms a hydrophobic film on the walls of capillary pores. Addition of silane emulsion reduces water capillary suction significantly. The reduction rate was more than 89%. Even the surface of internal water repellent concrete is abraded off 7 mm; the material still demonstrates high water repellences because the entire volume is hydrophobic. It is a well-known fact that, concrete will lose strength after exposure to elevated temperature. In this case, the damaged concrete is extremely vulnerable with respect to ingress of water and aggressive compounds. Therefore, the potential for the protection of concrete from excessive ingress of water after exposure to high temperature (accidental fire) has been investigated by (Peng-gang *et al.*, 2011).

Surface impregnation with silane was also applied on concrete exposed to elevated temperature. The efficiency of surface impregnation with respect to absorption of water and salt solutions by concrete with different levels of damage induced by elevated temperature has been investigated in particular. Results indicate that the increased water absorption of damaged concrete can be

reduced significantly by surface impregnation. A reduction of more than 90% can be achieved. The effective chloride barrier established by surface impregnation can help to extend the service life of fire exposed concrete structures. Thus the present investigation will look into the performance of concrete impregnation at varying internal moisture content levels within the concrete specimens to determine how internal moisture affects the applied impregnate.

The use of silane based impregnate have been widely used in the UK and have been shown to be effective through various tests, however, they are classed as a toxic material (Calder *et al.*, 2006). Silane is known to be irritants to the skin and eyes and during use correct safety equipment must be worn (Calder *et al.*, 2006). Silane is also known to be dangerous to the environment and can cause aquatic toxicity (DMRB HD43/03, 2003). As the construction industry is changing towards improved health and safety and environmentally friendly construction, the methods and materials used within it need to adapt as well. This has led to the development of water based impregnates which are claimed to be just as effective but have the added benefit of being non-toxic, rendering them less harmful to the environment and easier to apply. DMRB HD43/03 has included tests which ensure new impregnation products meet the minimum performance threshold before being used on any highways structures (FASTGLOBE, 2012).

EXPERIMENTATIONS

Specifications

36 cubes were produced, of which 12 of them

were impregnated with SB impregnate applied to them, another 12 have been WB impregnate applied and the remaining 12 were untreated to test as a control specimens. The cubes were chosen to be of C30 strength with water cement ratio of 0.60. The cubes were cured in a large tank for 28 days. After 28 days of curing the cubes were removed from the water bath and put in an oven to dry. The cubes were allowed to dry in the oven for 3 days at 85°C and were moved around to ensure equal circulation of warm air to all surfaces. Three cubes out of the 36 were also regularly weighed through the oven drying process to determine when the cubes were dry as their mass would become constant due to all water evaporating away. All the cubes were then removed and set aside to allow them to cool and absorb moisture from the surrounding atmosphere. Two days after allowing the cubes to cool and settle to atmospheric conditions, the cubes were weighted.

For consistency, samples were produced using same mixtures, were kept in the same conditions and treated in the same manner. Two types of impregnates were used. The silane based product, referred as SB, consisted of a colorless silane with an active content greater than 80%. According to manufacturer technical data sheet, the penetration depth greater than 10 mm can be achieved. The water based product, referred as WB, was water borne acrylic co-polymer based impregnate, which is friendly to the environment and less hazardous. It is silicone and solvent free and achieves a penetration of less than 10 mm.

Experimental Procedures

The experimental works were divided into

three main phases:

Phase 1: The absorption of water in cube specimens over a set period of time to determine moisture contents at different saturation level.

Procedure: Once the dry mass of the cubes was determined they were then submerged into a tank of water for 1.5, 3, 6, 9, 24, 27, 30, 33, and 48 h. At each time interval three's specimens were taken from the water tank, surface dried using paper towels and then weighted. The weight of each of the cube was recorded and then moisture content was calculated after being submerged to represent moisture content at that stipulated.

Phase 2: Performance against chloride penetration of impregnated concrete specimens applied at different moisture levels.

Procedure: All cubes were weighted, and oven dried at 105°C until the cubes were fully dried and their mass's had stabilized in the oven, they were allowed to cool as in phase 1 and random cubes were chosen to be reweighed to confirm they had a similar mass as in phase 1 before being submerged in water. At this point the cubes were also given a unique identification number from which it was easy to determine when the impregnate was applied and the type of impregnate used. It was decided that the cubes would be used in batches of six, so two cubes would have no protection, two cubes would have the WB impregnate and two cubes would have the SB impregnate for better representation of specific moisture content.

The first batch was impregnated after at fully dried condition as they would have been

impregnated in accordance to the recommended conditions. From at dry condition, cubes 1 and 2 had no impregnate applied and acted as a control to show chloride ingress with no protection. Cubes 3 and 4 had a SB impregnate applied and cubes 5 and 6 had WB impregnate applied. The second batch was removed from the water tank after 1.5 h from initial submerging in water and surface dried. From them cubes 7 and 8 had no impregnate applied, cubes 9 and 10

had SB impregnate and cubes 11 and 12 had WB impregnate applied. The sequence of impregnation application and identification of the cubes are shown in Table 1.

The required quantify of impregnate was weighted in a high accuracy as per manufacturer recommendation and applied using a brush carefully to all surfaces of the cubes. The products were measured out in separate cups and different brushes were used to ensure that no cross contamination of

Table 1: Cube Identification Numbers, Time and Type of Applied Product

Cube No	Cube ID	Time for specimen submerged in water prior to application of impregnate (h)	Type No	Cube No.	Cube ID	Time for specimen submerged in water prior to application of impregnate (h)	Impregnate Applied
1	0NA	0	NA	19	24NA	24	NA
2	0NA	0	NA	20	24NA	24	NA
3	0SB	0	SB	21	24SB	24	SB
4	0SB	0	SB	22	24SB	24	SB
5	0WB	0	WB	23	24WB	24	WB
6	0WB	0	WB	24	24WB	24	WB
7	1.5NA	1.5	NA	25	27NA	27	NA
8	1.5NA	1.5	NA	26	27NA	27	NA
9	1.5SB	1.5	SB	27	27SB	27	SB
10	1.5SB	1.5	SB	28	27SB	27	SB
11	1.5WB	1.5	WB	29	27WB	27	WB
12	1.5WB	1.5	WB	30	27WB	27	WB
13	3NA	3	NA	31	48NA	48	NA
14	3NA	3	NA	32	48NA	48	NA
15	3SB	3	SB	33	48SB	48	SB
16	3SB	3	SB	34	48SB	48	SB
17	3WB	3	WB	35	48WB	48	WB
18	3WB	3	WB	36	48WB	48	WB

the product occurred. The product was applied until the surfaces were saturated and the cubes were reweighted after application to ensure that there was approximately a 10 g increase in weight. The cubes were then put aside to dry; they were propped up to allow air to circulate underneath the cube allowing the bottom surface to dry off as well. The cubes were left for approximately 4 h to dry before being placed into a sodium chloride bath.

It was noted that the longer the cubes had been submerged in water the harder it was to ensure 10 g of impregnate was applied to the cubes. All effort was made to make sure that all surfaces were coated equally with the impregnate. After the impregnate had dried, the cubes were then placed in a tank with full of sodium chloride solution. The salt source for making the sodium chloride was rock salt which was used in this research.

The specimens were submerged in water for approximately 60 days. After that period, they were removed and dried naturally. The cubes were then dry drilled to provide dust samples and chemical analysis of the samples was carried out to determine the chloride content at various depths. All the cubes were carefully removed from the sodium chloride solution and then as before they were placed in an oven to allow them to dry. The oven was set at 105°C the cubes allowed to dry, after which they were left to cool.

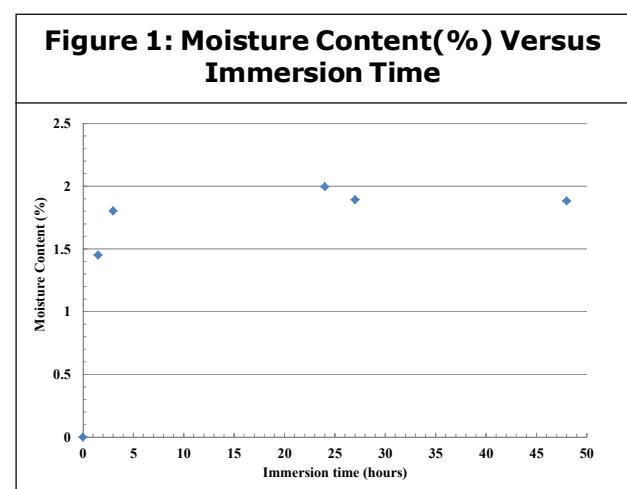
Dust samples were collected at 5, 10, 15 and 20 mm depth. Once all the dust samples had been collected at the various depths, a chemical analysis as per (BS EN 14629, 2007)

was conducted to determine the chloride content in hardened concrete in which Volhard's method was used.

RESULTS AND ANALYSIS

Phase 1: Increase in moisture content of concrete cubes at different period submerged in water.

The moisture content with respect to porosity at dry stage is plotted against submerged period and is shown in Figure 1. It can be seen that, after 1.5 h of being submerged in water the first six cubes had an average moisture content of 1.45%. After 3 h from initial exposure to water the next six cubes had a greater increase in moisture content of 1.80%. Between 1.5 h and 3 h the moisture content intake by the cubes was 0.35%. When the next six cubes were weighted after 24 h, as expected the rate of water ingress decreased and the difference between moisture content at 24 h and 3 h was only 0.19%. The graph trend then appears to be in equilibrium condition. The reason behind the sharp increase in moisture content for the first



3 h is due to the surface pores being dry. When the cubes were placed in the water tank a concentration gradient existed within the cube resulting in a rapid increase in moisture content. However as the surface pores became saturated the rate of increase of moisture content decreased.

Phase 2-Chloride Concentration

The effect of moisture content on the performance of WB and SB impregnates in reducing chloride ingress was evaluated in this section. Two cubes were used for each condition and the average chloride contents (%) at varying depths (mm) are plotted in Figures 2a-2f.

In which, Figure 2a shows the chloride content for the specimens subjected impregnates applied at dry condition. As

expected, the SB impregnate was found effective only having a chloride content of 0.046% at 5 mm which is further decreased by 0.029% at 20 mm depth. WB impregnate had a much higher chloride concentration at 5 mm similar to the unprotected cubes. However at 10 mm, the WB cubes had a drop in chloride content of 0.038%, whereas the unprotected cube had a reduction in chloride at a linear rate. So when the cubes had impregnate applied in the recommended conditions SB performed the best having a huge impact on chloride ingress, this was also the case for WB as it appears to be most effective at a depth of 10 mm.

On the other hand, when impregnate was applied at 1.5, 3, 24, 27 and 48 h after being submerged in water (Figures 2b-2f), irrespective of impregnate type, all cubes appear to start off with a relatively high chloride

Figure 2a: Chloride Content for Dry Cubes

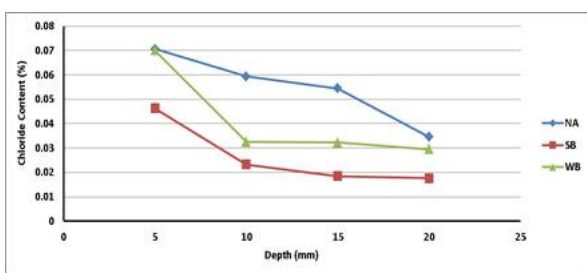


Figure 2b: Chloride Content for Cubes Submerged 1.5 h

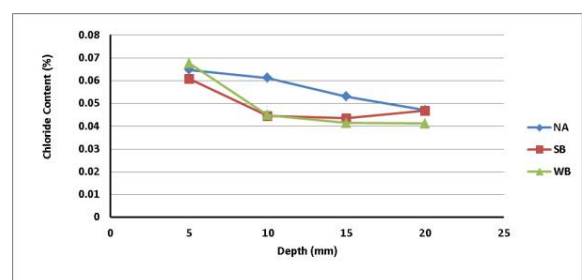


Figure 2c: Chloride Content Submerged 3 h

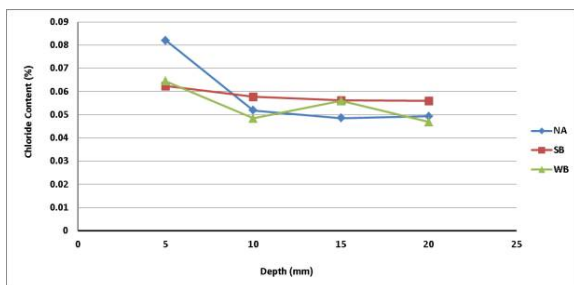


Figure 2d: Chloride content for cubes submerged 24 h

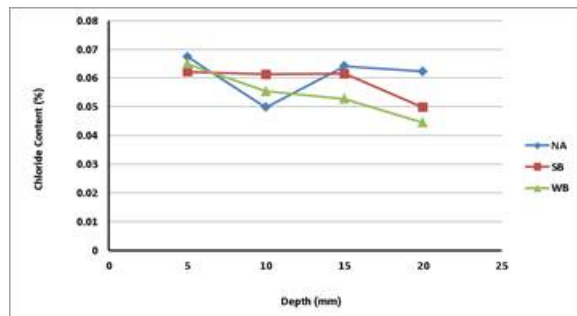


Figure 2e: Chloride Content for Cubes Submerged at 27 h

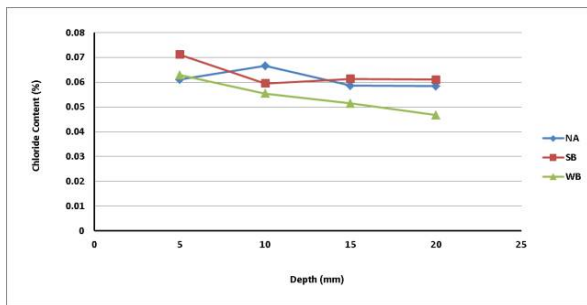
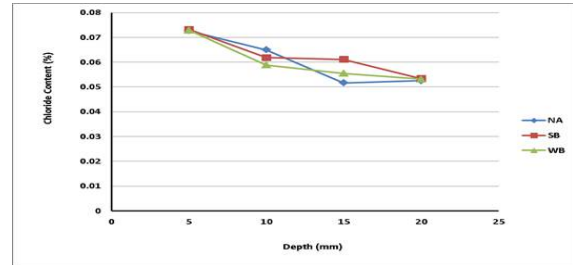


Figure 2f: Chloride Content for Cubes Submerged 48 h



concentration at 5 mm, but no significant advantages was noticed between protected and unprotected concrete specimen. There was also very little difference in performance between SB and WB type impregnate.

In addition to that, graphs of moisture content against chloride concentration at varying depths were plotted in Figures 3a-3d. It can be seen that in dry condition (0% moisture), like demonstrated in previous section, solvent based protection performed

Figure 3a: Cl- Conc. Cubes V/s Moisture Content at 5 mm

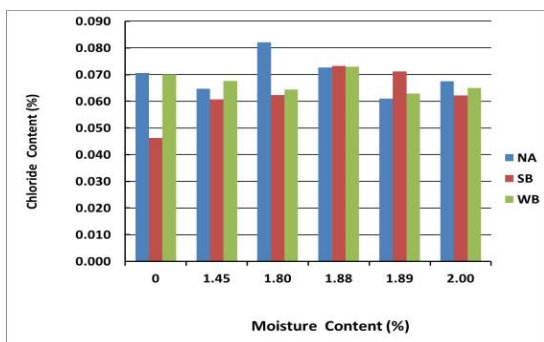


Figure 3b: Cl- Conc. Cubes V/s Moisture Content at 10 mm

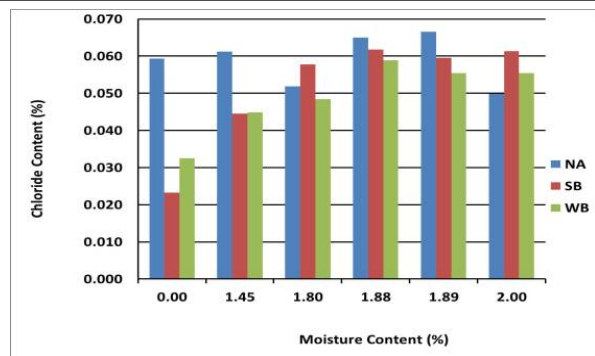


Figure 3c: Cl- Conc. Cubes V/s Moisture Content at 15 mm

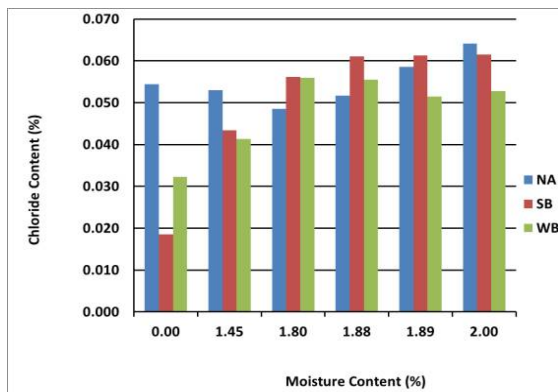
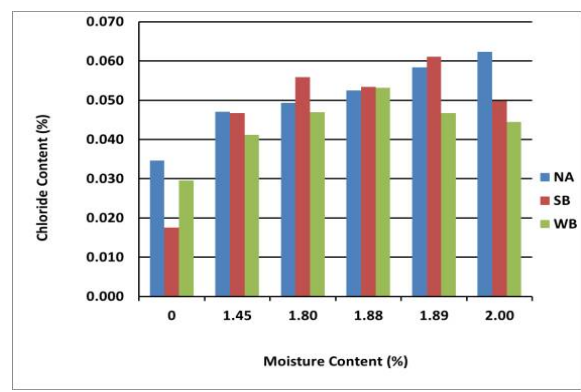


Figure 3d: Cl- Conc. Cubes V/s Moisture Content at 20 mm



better in comparison to water based protection. On the other hand, when moisture content increased to 1.0% or more, the rate of penetration is quite similar in all conditions, indicating no significant benefits are achieved with different types of impregnation.

The percentage reduction of chloride contents with respect to depth and internal

moisture was also studied. It can be seen in Table 2 that, in general, irrespective of impregnate type, the reduction was over 50% in dry condition (51.13% NA, 61.68% SB and 58.57% WB), but the reduction is significantly reduced to average 23.75% (max 39.87%, min 4.91% and standard deviation 0.103) at moisture content 1.45% to 2.00%, reinforcing the adverse effect of internal moisture on the

Table 2: Percentage Reduction of Chloride Concentration at Different Depths at Different Moisture Level

Immersion Period Prior to impregnation (h)	Moisture Content (%)	Impregnate Type	% of reduction from 5 mm to 20 mm
0	0	NA	51.13%
		SB	61.68%
		WB	58.57%
1.5	1.45%	NA	26.56%
		SB	23.30%
		WB	38.80%
3	1.80%	NA	39.87%
		SB	11.70%
		WB	28.12%
24	1.88%	NA	7.46%
		SB	20.96%
		WB	31.25%
27	1.89%	NA	4.91%
		WB	14.08%
		SB	25.80%
48	2.00%	NA	27.70%
		WB	27.39%
		SB	27.63%

performance of impregnate.

CONCLUSION

- The fact known from the research that, as the concrete cubes are left immersed in water for longer periods of time, their moisture content would increase rapidly in initial hours (1.45% within first 1.5 h) and then slow down as the pores became saturated with reduced concentration gradient (1.88% after 48 h immersion).
- This investigation also established an extended relationship between moisture content and performance of impregnates. In order to achieve the best performance, impregnate must be applied to only dry surfaces to allow successful bonding and penetration of the concrete pores. In dry condition, solvent base performs better than the water based impregnate in suppressing chloride. On the other hand, no added benefit was noticed between two impregnate types when they were applied in the partially or fully saturated specimens.
- The percentage reduction of chloride contents with respect to the depth and internal moisture content showed that, solvent based impregnate is more effective when applied in dry condition (reduction 51.13% NA, 61.68% SB and 58.57% WB), but the reduction is significantly reduced to average 23.75% (max 39.87%, min 4.91% and standard deviation 0.103) at moisture content 1.45% to 2.00%. This shows that internal moisture has significant effect on the performance of impregnate. This concentration of the moisture is probably due to the dilation of salt with the trapped

moisture. However, further investigation is necessary to evaluate the concentration when the cubes are eventually become fully dry.

REFERENCES

1. Al-Zahrani M M, Al-Dulaijan S U, Ibrahim M, Saricimen H and Sharif F M (2002), "Effect of waterproofing coatings on steel reinforcement corrosion and physical properties of concrete", *Cem. Concr. Comp.*, Vol. 24, pp. 127-137.
2. Calder A, Anderson N and McKenzie M (2006), *Survey of Impregnated Structures*, 3/359, UK, TRL Limited.
3. Delucchi M, Barbucci A and Cerisola G (1998), *Crack-bridging ability and liquid water permeability of protective coatings for concrete Progress in Organic Coatings*, Vol. 33, p. 76.
4. DMRB HD43/03 (2003), The Impregnation of Reinforced and Prestressed Concrete Highway Structures Using Hydrophobic Pore-Lining Impregnates, SI: Highways Agency.
5. FASTGLOBE (2012), *Silane*, [Online] available at <http://www.fastglobe.net/silane.htm>, Accessed 17 August 2012.
6. Helene P (2000), *Handbook diagnosis and intervention of reinforced concrete structures*, p. 87.
7. Jacob T and Hermann K (1998), *Protection of concrete surfaces: Hydrophobic impregnations, Construction y Tecnología*, March, pp. 18-23.

8. Jones J W (2002), *Method of Hardening and Polishing concrete floors, walls, and the Like*, US Patents, Patent number: US 6, 454, 632 B1, Sep. 24, 2002.
9. Keer J G (1992), *Steel Corrosion in Concrete—Fundamentals and Civil Engineering Practice*, E&FN SPON, London, p. 150.
10. Medeiros M H F and Helene P (2009), “Surface Treatment of Reinforced Concrete in Marine Environment: Influence on Chloride Diffusion Coefficient and Capillary Water Absorption”, *Construction and Building Materials*, Vol. 23, No. 3, pp. 1476-1484.
11. Peng Zhang, Tiejun Zhao, Wittmann F H and Shaochun Li (2011), *Preparation and Characteristics of Integral Water repellent Cement based materials*, Materials Science Forum, Vol. 675-677, pp. 1189-1192.
12. Peng-Gang Wang, Peng Zhang and Tiejun Zhao (2011), *Surface Impregnation of Concrete Damaged by Elevated Temperature*, Material Science Forum, pp. 567-570.
13. Roberge P R (2008), *Corrosion Engineering: Principles and Practice*, 1st Edition, McGraw-Hill Professional, New York.
14. SIDS O (2004), *Silane Initial Assessment Report*. [Online] Available at: <http://www.inchem.org/documents/sids/sids/2530838.pdf>, Accessed 17 August 2012.
15. Uemoto K L, Agopyan V and Vittorino F (2001), “Concrete protection using acrylic latex paints: Effect of the pigment volume content on water permeability”, *Mater. Struct.*, Vol. 34, April, pp. 172-177.
16. Vries J and Polder R B (1997), “Hydrophobic Treatment of Concrete”, *Construction and Building Materials*, Vol. 11, No. 4, pp. 259-265.