Performance evaluation of concrete surface treatment materials

Mohammed Ibrahim

Civil Engineering

June 1996

Abstract

The reduction in the useful service-life of reinforced concrete structures in the Arabian Gulf is of concern to the construction industry. In this region, reinforced concrete structures rarely serve their full design life. In such environments, both concrete and reinforcing steel need to be protected. Among other alternatives, this protection can be achieved by the use of concrete surface treatment materials. While several studies have been conducted on the effectiveness of such coatings in inhibiting reinforcement corrosion, data are lacking on their performance in environments characterized by the presence of chloride and sulfate salts. Furthermore, the effect of thermal and moisture variations, typical of that prevailing in the Arabian Gulf environment, on the performance of concrete surface coatings are not fully elucidated

In this study, the performance of six concrete surface treatment materials were evaluated. The results indicated that silane/siloxane with top coat was the most efficient in delaying the concrete deterioration in most of the exposure conditions. Among all the surface treatment materials used in the this study, silane/siloxane with top coat, silane and acrylic coating were the most effective in providing adequate protection and hence reducing the rate of reinforcement corrosion. Furthermore, these three surface treatment materials were the most effective in reducing reinforcement corrosion in in-service structures. Also, silane/siloxane with top coat was most effective in reducing sulfate attack, as compared to other coatings. The concrete specimens treated with silane, silane/siloxane with top coat and acrylic coating exhibited the least water absorption and reduction in compressive strength as a result of thermal and moisture variations. Silane/siloxane with top coat and acrylic coating were effective in reducing carbonation of concrete.

Performance Evaluation of Concrete Surface Treatment Materials

by

Mohammed Ibrahim

A Thesis Presented to the

FACULTY OF THE COLLEGE OF GRADUATE STUDIES

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DHAHRAN, SAUDI ARABIA

In Partial Fulfillment of the Requirements for the Degree of

MASTER OF SCIENCE

In

CIVIL ENGINEERING

June, 1996

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dedicated to

my beloved parents

whose prayers, inspiration and love led to this accomplishment

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THESIS ABSTRACT

NAME : MOHAMMED IBRAHIM

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SURFACE TREATMENT MATERIALS

MAJOR FIELD : CIVIL ENGINEERING (STRUCTURES)

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The reduction in the useful service-life of reinforced concrete structures in the Arabian Gulf is of concern to the construction industry. In this region, reinforced concrete structures rarely serve their full design life. In such environments, both concrete and reinforcing steel need to be protected. Among other alternatives, this protection can be achieved by the use of concrete surface treatment materials. While several studies have been conducted on the effectiveness of such coatings in inhibiting reinforcement corrosion, data are lacking on their performance in environments characterized by the presence of chloride and sulfate salts. Furthermore, the effect of thermal and moisture variations, typical of that prevailing in the Arabian Gulf environment, on the performance of concrete surface coatings are not fully elucidated.

In this study, the performance of six concrete surface treatment materials were evaluated. The results indicated that silane/siloxane with top coat was the most efficient in delaying the concrete deterioration in most of the exposure conditions. Among all the surface treatment materials used in this study, silane/siloxane with top coat, silane and acrylic coating were the most effective in providing adequate protection and hence reducing the rate of reinforcement corrosion. Furthermore, these three surface treatment materials were the most effective in reducing reinforcement corrosion in in-service structures. Also, silane/siloxane with top coat was most effective in reducing sulfate attack, as compared to other coatings. The concrete specimens treated with silane, silane/siloxane with top coat and acrylic coating exhibited the least water absorption and reduction in compressive strength as a result of thermal and moisture variations. Silane/siloxane with top coat and acrylic coating were effective in reducing carbonation of concrete.

MASTER OF SCIENCE

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ان انخفاض العمر الإفتراضي في المنشآت الخرسانية المسلحة في الخليج العربي يقلق المهتمين في صناعة المنشآت ونادراً ماتخدم هذه المنشآت الخرسانية في هذه المنطقة العمر الإفتراضي الذي صممت من أجله . لذلك فإن كلاً من الخرسانة وحديد التسليح يحتاجان إلى حماية إضافية . وهناك عدة طرق للحماية ، منها إستخدام مواد معالجة الأسطح الخارجية للخرسانة . ولقد أجريت عدة دراسات على فعالية هذه الأنواع من الطلاءات لحماية الخرسانة وحديد التسليح ، إلا أنه لاتوجد دراسات كافية لمستوى أداء هذه الطلاءات في البيئة التي تحتوي على نسبة عالية من أملاح الكلورايد والكبريتات معاً . أيضا لاتوجد دراسات على فعالية هذه المواد تحت تأثير تغير الحرارة والرطوبة والمماثلة لبيئة الخليج العربي .

تم في هذه الدراسة تقييم أداء ست مواد أساسية من مواد معالجة أسطح الخرسانة وأظهرت النتائج أن مادة سيلين / سيلوكسين (Silane siloxane with top coat) مع الطلاء الخارجي الأكثر فعالية في تأخير تدهور الخرسانة لمعظم الحالات .

ولقد أظهرت مادة السيلين / سيلو كسين مع الطلاء الخارجي وطلاء سيلين / اكريليك أفضل فعالية في توفير بعض الحماية ضد صدأ حديد التسليح في الخرسانة . وكذلك فإن هذه المواد أظهرت فعالية في خفض نشاط صدأ حديد التسليح في المنشأت القائمة ، كما أن مادة سيلين / سيلوكسين مع طلاء خارجي كانت الأكثر فعالية في خفض تأثير الكبريتات مقارنة بالمواد الأخرى . وأظهرت العينات الخرسانية التي عولجت بمواد السيلين / سيلوكسين مع طلاء خارجي وطلاء أكريليك أقل إمتصاصاً للماء وأقل فقداناً في قوة الضغط نتيجة تغيير الحرارة والرطوبة ، وكذلك أظهرت هذه المواد فعالية في خفض عملية كربنة الخرسانة (The Carbonation) .

بحث درجة الماجستير في العلوم الهندسية جامعة الملك فهد البترول والمعادن الظهران ـ المملكة العربية السعودية يونيو ١٩٩٦م (صفر ١٤١٧هـ)

CHAPTER 1

INTRODUCTION

1.1 CONCRETE DURABILITY IN THE ARABIAN GULF

The reduction in the useful service-life of reinforced concrete structures in the Arabian Gulf is of concern to the construction industry in this region [1]. In this part of the world, reinforced concrete structures rarely serve their full design life. As a result, considerable resources have to be diverted towards repair and rehabilitation of these deteriorated structures. The deterioration of these structures is mainly attributed to the environmental and geomorphic conditions, which are characterized by severe ground and ambient salinity and high temperature and humidity regimes [2].

The major types of concrete deterioration, in this region, in decreasing order of importance are:

- (i) reinforcement corrosion,
- (ii) sulfate attack,
- (iii) salt weathering, and

(iv) plastic and drying shrinkage cracking.

However, the overwhelming number of cases of concrete failure due to reinforcement corrosion overshadows that due to other causes.

The last decade has witnessed, on a global level, a growing concern for problems of corrosion damage in reinforced concrete structures. Bridge-deck deterioration and corrosion damage to parking garages in the USA, deterioration of coastal structures due to attack by sea water and salt spray, corrosion in many structures due to the use of chloride-based accelerating agents, and highly prevalent and premature corrosion deterioration of concrete construction in the Arabian Gulf states together constitute probably the largest group of reinforced concrete structures undergoing deterioration due to a single causal factor. Field surveys conducted in an area which is typically representative of the conditions along the Arabian Gulf seaboard indicate that the premature deterioration of concrete in the Gulf states can be attributed to a number of factors [3]. These include, poor quality concrete, inadequate cover to reinforcing steel, and a high level of chloride contamination in concrete, inducted mainly through chloride-bearing constituent materials, such as aggregates and mixing and curing water.

Portland cement concrete provides both chemical and physical protection to the reinforcing steel. The chemical protection is provided by the highly alkaline nature of the pore solution (pH > 13). At this high pH, steel is passivated in the presence of oxygen, presumably due to the formation of a submicroscopically thin γ - Fe₂O₃ film [4]. Hime and Erlin [5] suggested that the passivating layers on steel surface could have a composition other than that of γ - Fe₂O₃. According to Page [6], the lime-rich layer, which is observed at the steel-concrete interface, provides further protection to the steel. This was confirmed by Leek and Poole [7] who reported that the interfacial layer consists of an aggregate-free zone of portlandite (Ca(OH)₂) of variable thickness (5 to 15 μm) disrupted by inclusions of calcium silicate hydrate (C-S-H) gel. This layer is thought to screen most of the surface of the steel from the aggressive ions and to act as an alkaline buffer to pH reductions resulting from the hydrolysis of corrosion products [8]. According to Sagoe-Crentsil and Glasser [9], both Ca(OH)2 and C-S-H gel form a buffering pair, and a high pH is as readily maintained by C-S-H gel as by Ca(OH)2. The physical protection to steel is provided by the dense and impermeable structure of concrete which retards the diffusion of the aggressive species, such as chlorides, carbon dioxide, oxygen and moisture, to the steel-concrete interface.

Depassivation of steel occurs by the reduction of the pore solution pH, due to carbonation, or by ingress of chloride ions to the steel-concrete interface. A number of mechanisms by which chlorides break down the passive layer have been proposed, e.g., the chemical dissolution of the film [10], the built up of the metal holes at the film-substrate interface [11], and due to high chloride concentrations at the iron oxide-pore solution interface which leads to local acidification and pitting [12]. Leek and Poole [13], based on SEM-EDS studies of the passive film breakdown on steel in mortar prisms, have shown that chloride ions initiate reinforcement corrosion by breaking the bond between the film and the metal.

Irrespective of the mechanisms controlling the depassivation of steel due to chloride ions, it is clear that these ions play a dominant role in initiating reinforcement corrosion. From this perspective, ACI 318-85 [14] limit, the water soluble chlorides to 0.15% by weight of cement. ACI Committee 224 [15] adopting a more conservative approach has suggested that the acid-soluble chloride concentration should not be more than 0.2% by weight of cement. The British Standards BS 8110 [16] allows a maximum chloride content of 0.4%. The Norwegian Code, NS 3474, allows an acid soluble chloride content of 0.6%, RILEM permits 0.4% and the revised Australian Standard for Concrete Structures, AS 3600, allows an acid soluble chloride content of 0.8 kg/m³ of concrete (0.22%).

by weight of cement for a typical concrete mix). Rasheeduzzafar et al. [17] indicated that the chloride threshold limits for cements with upto 8% C₃A agree very well with the ACI 318 limit of 0.15% water soluble chlorides. Additionally, they reported that ACI, BS and Australian Code limits, however, appear to be conservative for concretes made with high C₃A cements. Lambert et al. [18] suggested that the critical level of chloride below which there was no significant probability of reinforcement corrosion was around 1.5%. They attributed the increased chloride tolerance in their specimens, (compared to BS 8110 limit of 0.4%), to the protective nature of concrete produced under well-controlled laboratory conditions.

Recent research findings, however, have shown that cement alkalinity also significantly influences the chloride-binding and hence the free chlorides [19,20,21,22]. Taking into account the concomitant effect of chloride and alkalinity, Hausmann [23] suggested the critical Cl⁻/OH⁻ ratio to be around 0.6. Gouda [24] using the pH values of the electrolyte representative of concrete pore solution indicated that the threshold Cl⁻/OH⁻ ratio was 0.3. Lambert et al. [25] investigated the relationship between Cl⁻/OH⁻ and corrosion current density in various cements. Their investigations indicated that the passive conditions of steel in concrete, (characterized by corrosion current density (I_{corr}) substantially lower

than 100 nA/cm²) were maintained until a threshold Cl⁻/OH⁻ ratio of approximately 3 was exceeded. There was a considerable scatter in the values of I_{corr} recorded at Cl⁻/OH⁻ ratios in excess of 3 and even at Cl⁻/OH⁻ ratios as high as 15 to 20, there were instances of bars suffering no significant corrosion. Mangat and Guruswamy [26] reported that for steel fibers in concrete under conditions of marine exposure, no visible signs of corrosion were present at Cl-/OH- ratios as high as 320. Mangat and Molloy [27] indicated that a universal threshold Cl⁻/OH⁻ level is not applicable to different cement concretes. In their investigations, reinforcing steel corrosion was observed in the control matrix when the pore fluid Cl⁻/OH⁻ ratio was 13, while at values of 17 and 18, in the silica fume cement concrete, reinforcement corrosion was insignificant. Minimal reinforcement corrosion in the silica fume and blast furnace slag cement mortar specimens placed in the aggressive environment of sabkha, even at Cl⁻/OH⁻ of 3.3 and 6.5, respectively, was reported by Al-Amoudi et al. [28].

Chloride ions are often unintentionally inducted into concrete through the constituent materials like salt contaminated aggregates or water, and some times intentionally in the form of chemicals to accelerate the setting of concrete. Moreover, they may penetrate the hardened concrete if exposed to aggressive environment. Typical cases are structures exposed to marine environments, bridge

decks and parking garages exposed to deicing salts, and structures situated in salt laden environments. In the case of structurally damaged concrete, the diffusion of chlorides is made even more easy.

The harmful effects caused by chlorides are:

- (i) depassivation of the passive film,
- (ii) increase in the electrical conductivity of concrete, thereby facilitating the flow of electric current from the anodic to the cathodic sites and vice-versa,
- (iii) hydration of the calcium silicates, influencing the long-term strength improvement and impermeability.

While the chlorides contributed by the constituent materials can be controlled by strict adherence to improved construction practices, the ingress of chloride ions from the service environment can only be controlled by producing a good quality concrete and/or coating with impermeable membranes.

To circumvent the problems stated above, it is not uncommon to incorporate additional methods of corrosion protection in concrete construction. Many options are available including applying coatings (impermeable membranes) to concrete, using corrosion inhibitors and coated reinforcement.

1.2 NEED FOR THIS RESEARCH

As discussed earlier, deterioration of concrete structures is a widely spread phenomena all over the world. Vast resources are directed every year on repair and rehabilitation of concrete structures. In slabs, beams, foundations, under ground structures, and columns, low quality porous concrete significantly increases the ingress of chlorides, oxygen, moisture, sulfates and carbondioxide to steel-concrete interface. This situation is ideal for the initiation of reinforcement corrosion and subsequently increases rate of deterioration due to corrosion of reinforcement, specially if the cover to the reinforcement is insufficient or due to low quality concrete. In walls and foundations, porous and permeable concrete is extremely vulnerable to degradation due to sulfate attack.

Some parts of the structures, such as piers and footings, frequently come into contact with the sulfate and chloride ions. The chloride ions, when they penetrates concrete and reach the steel-concrete interface, cause reinforcement corrosion, while sulfate ions react with the hydrated cement paste to reduce the strength of concrete. One method to reduce this problem may be to stop the supply of oxygen and moisture to the concrete which are essential for the reinforcement corrosion to occur.

This can be achieved by applying surface treatment materials on the concrete. This method can also be used to rehabilitate corroding structures. In view of these facts, it is very important to investigate the performance and effectiveness of some concrete sealers and coatings in reducing reinforcement corrosion in in-service structures.

Of all the methods of protecting and preserving the existing structures and converting them into serviceable and usable elements, the use of surface treatment materials has the unique advantage that they can be applied to protect new and inservice structures, or as part of a program of refurbishment of damaged and deteriorating structures.

Although, some studies have been conducted to evaluate the performance of penetrating sealers, data on the performance of these materials in chloride and sulfate bearing environments are scarce. Carbonation and corrosion of reinforcing steel are also serious problems, which are not adequately investigated for surface treated concrete. Also, there is hardly any data on the performance of sealers in the heat-cool and wet-dry environments.

Therefore, there is a need for research to evaluate the performance of some surface treatment materials in the Arabian Gulf environment. The aforesaid situation necessitates an evaluation of surface treatment systems in environments contaminated with chlorides and sulfates, and their effectiveness as low-oxygen and low-carbon dioxide permeation materials.

The need for such a protective systems for concrete construction in the Arabian Gulf states is all too obvious in view of the prevalent corrosion deterioration problems in the region.

Furthermore, the other difficulty with penetrating sealers and coatings is the wide range of such materials available in the market, it becomes extremely difficult to choose the right type of material since sealers of similar generic types are known to have considerably different diffusion characteristics. The nature and severity of exposure, due to extreme weather conditions, such as large variations in temperature and humidity, is a major factor in determining the performance characteristics of these materials. Although certain sealers can significantly reduce the intrusion of chloride into concrete, the long-term protection in severe environments, such as in the Arabian Gulf, is still need to be further elucidated.

1.3 RESEARCH OBJECTIVES

The general objectives of this research were to evaluate the performance of some penetrating sealers and coating materials under the conditions simulating those of the Arabian Gulf environment.

The specific objectives are:

- 1. to evaluate the chloride diffusion co-efficients in the concrete specimens coated with sealers and coatings.
- 2. to monitor the performance of concrete specimens coated with sealers and coatings in a sulfate environment,
- 3. to evaluate the corrosion protection offered by concrete specimens coated with sealers and coatings.
- 4. to assess the performance of sealers and coatings when exposed to thermal variations,
- 5. to investigate the effect of wet-dry cycling on the integrity of the sealers and coatings.

- 6. to evaluate the effectiveness of sealers and coatings in preventing carbonation of concrete.
- 7. to assess the usefulness of sealers and coatings in reducing reinforcement corrosion in in-service structures, and
- 8. to provide recommendations for the selection of sealers appropriate for the Arabian Gulf environment.

The experimental variables are shown in Figure 1.1.

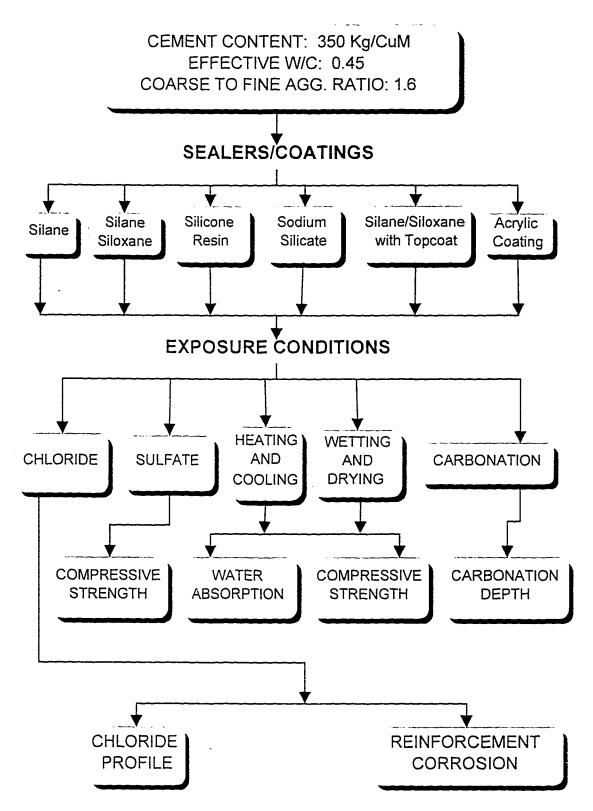


Figure 1.1 Experimental Variables

CHAPTER 2 LITERATURE REVIEW

2.1 PREVENTIVE METHODS

As discussed in the preceding chapter, the environmental and geomorphic conditions in the Arabian Gulf necessitate the production of a very dense and impermeable concrete. In addition to the normal construction practices, both, concrete and the reinforcing steel need to be protected to extend the useful service-life of the reinforced concrete structures. Protection to reinforcing steel is provided by the use of chemical inhibitors or coating the bar itself. Additional protection can be provided by coating the concrete with sealers and/or coatings.

While there is a growing awareness of causes of reinforcement corrosion, however, there is no universal answer to its mechanism. This has led to greater attention being diverted towards the preventive measures. In particular, greater consideration is given to surface treatment materials and/or epoxy coated rebars which can protect reinforced concrete structures from deterioration.

Penetrating sealers are used basically to prevent the corrosion of reinforcing steel caused by aggressive agents such as chlorides and sulfates. The effectiveness of surface treatment materials in preventing chloride and sulfate ions from penetrating the concrete matrix depends on their own ability to penetrate and there by protect the concrete matrix. This is largely dependent on the concrete matrix's void system and quality. If the concrete matrix has large voids then the penetration of sealers will be greater [29].

Surface treatment materials are generally categorized as follows:

2.1.1 Penetrants

These are low viscosity liquids which can penetrate into concrete and line its pores. They have little effect on the appearance of the structure, as, they are colorless. They protect concrete from deterioration by forming a hydrophobic layer and thus repel water, which not only causes corrosion of reinforcing steel, but also provides the medium for the diffusion of chlorides and other aggressive species. The important feature of the penetrating sealers is that, they do not block the pores of concrete and there by allow the evaporation of water vapor and other gases from the concrete mass.

2.1.2 Sealers

Sealers are intermediate between penetrants and coatings. They protect concrete by blocking the pores. They are more viscous than penetrants and generally form a film on the surface of concrete.

2.1.3 Coatings

Coatings are still more viscous than sealers and provide protection to concrete by forming a thick film on the surface. Because of less breathability, these materials do not allow ingress of potentially harmful, water vapor and other gases, and may cause concrete deterioration.

During the past decade, numerous sealers have been used, such as oils, resins, petroleum products, silicones, and other organic materials. However, only few of them were effective in preventing the intrusion of deleterious materials, such as chlorides, sulfates, oxygen, carbondioxide and moisture [30].

A wide range of organic polymers are used as sealers and coatings, while the most widely used penetrating materials tend to be siliceous which line the pores of concrete forming silicone resins providing protection through their water repellent

properties. There is another class of penetrating material whose action is to block the pores of the concrete such as epoxy resins [31]. Although significant variation in their performance exist, there are certain specific formulations of different chemical materials which exhibit very good to excellent performance. These materials are able to reduce the intrusion of chloride by 80 to 99 percent when compared to uncoated concrete [32].

Certain sealers appear to offer added corrosion protection to embedded steel when cracks are present. This observation suggests that cracks in such members could be given multiple coats of these sealers to achieve even better corrosion performance [33].

In concrete structures containing chloride, the use of a vapor permeable surface on its own will be of limited value, although it may give the structure a more uniform appearance and will help shed water [34]. In repairs involving possible chloride contamination and/or permeable concrete, sealing of the concrete adjacent to the repair with a penetrating resin sealer would be of considerable benefit provided it blocks the diffusion of oxygen to the cathodic areas of the reinforcement. The sealer used must soak in to the concrete and not simply penetrate the first few microns. With use of a right type of sealer, significant

penetration in slightly permeable concrete can be achieved, reducing its susceptibility to the ingress of moisture or moisture laden sulfate and chloride salts, specially in the case of foundations. In the formulation of penetrating sealer, both the viscosity and the molecular size of the resin are important, low viscosity on its own does not permit significant penetration of many concrete surfaces which are micro porous in nature. Conventional water based coatings do not penetrate significantly into concrete, but there are now becoming available polymer dispersions in water where the particle size of the solids in the dispersion is approximately 0.01 mm, which will penetrate into porous concrete. Alternatively, resin solutions in volatile solvents can be used effectively to reduce the permeability of concrete. Deep polymer impregnation would involve the removal of evaporable moisture in concrete to a depth which goes as close to the reinforcing steel as the technique permits. This would be followed by soaking of concrete with a catalyzed monomer for a time sufficient to permit penetration to the dried depth and polymerization of the monomer by the application of heat [35].

In the history of surface treatment materials, linseed oil is one of its kind. Usually it is supplied in the market as a solution of 50% boiled linseed oil and 50% mineral spirits or kerosene. Performance of linseed oil depends on several factors such as type of linseed oil, type of solvent and application conditions. But, to

achieve greater penetration, diluted linseed oil is preferred. Because, lime contained in concrete reacts with linseed oil resulting in saponification of oil [36].

Review of literature indicates that epoxies are also used since a long time. They have been routinely used as penetrating sealers and as coating materials. The solids content is generally in the range of 17 to 100%. It is a two component material. These two components react chemically to form a protective film. Two coats are usually recommended to reduce pinholes. A major disadvantage with this material is that, they choke the concrete pores when subjected to ultra-violet radiation and also have limited breathability. As, thermal expansion of epoxies is higher than that of concrete, it causes cracking and subsequently permitting the ingress of deleterious materials. The other disadvantage is that they lose their waterproofing effect after being subjected to abrasion. Also, they are expensive.

Many other varieties of synthetic resins are also used, including acrylics, polyurethanes, and hydrocarbon resins. Acrylic resins are used with different solvents. In this case also two coats are recommended. Acrylic resin is also, one of the most effective sealers. However, because of the less flexibility of the cured film, of acrylic resin, it may crack and thereby provide an access to the deleterious

materials. Therefore, to provide a good bond the concrete surface has to be prepared properly.

Urethanes were first proposed in Japan because of their reportedly excellent resistance to water, weathering, and cracking [37]. They are excellent UV, abrasion and acid resistant than epoxies, but less alkali resistant. The added disadvantage with urethanes are low penetration, adhesion and breathability. More recently, two types of silicone compounds have been used as surface treatments. One type consists of impervious coating which seals the surface so that neither liquid water nor water vapor will penetrate. The other type consists of water repellents which prevent the passage of liquid water but do not stop the water vapor. It has been shown that, although silicones reduce the initial rate of absorption of water, after continued immersion, the total water absorbed in the treated and untreated concrete is similar.

Silane is one of the surface treatment material which is presently under consideration among most of the researchers around the world. It is marketed as a waterproofing material. They exhibit both hydrophilic and hydrophobic properties, as they possess a non-polar as well as polar component. In the silanes used to preserve buildings, vehicle for water repellent action is the non-polar

organic alkyl group. The effectiveness, durability and stability of a water repelling agent depend on the size and nature of this group. The remainder of the four possible bonds of the central silicon atom are linked to alcohol groups. They are hydrophilic and react with water in a reaction called hydrolysis, thereby splitting of the alcohol. The silanol created in this way can be formally regarded as an alkyl silica. The speed at which this occurs is governed by the surrounding pH. A chemical compound of this type combines the properties of the organic alkyl group and those of the inorganic silicate. This alkyl silica is highly reactive. With the neighboring molecules it forms compounds with low-degree of polymerization but is also able to react with the inorganic surface of the building material. The surface of chemically bonded alkyl groups formed in this way makes the substrate water repellent. A frequent argument against the use of monomeric silane is their volatility. Up till now there are no confirming results about this problem. Therefore, it is necessary to test this material comprehensively.

Siloxanes are the recent development in the family of penetrating sealers. Very limited data is available on the performance of this material. It is claimed by most of the marketing agencies that they are penetrating and surface reactive, but, as they are slightly big, they are less volatile. Therefore, they reside longer on the substrate and allow greater time for penetration and reaction. However, it is

claimed that, despite being larger in size than the monomeric silanes, siloxanes can still penetrate deeply.

Numerous laboratory studies [38,39,40] have been conducted to evaluate the effectiveness of sealers in protecting concrete surfaces against the penetration of moisture and chloride solution. However, no standard test method has been developed to evaluate water absorption, water vapor transmission and chloride ion intrusion and also there is no standard specimen size for these tests.

Cabrera and Hassan [41] have indicated that the use of an "effective treatment" does not only protect concrete against penetration of harmful substances, but also improves the performance properties of badly cured concrete by allowing the redistribution of its internal moisture. They used ponding test to determine the chloride diffusion coefficients. Treated and untreated specimens were immersed in 15% NaCl solution for one year. Chloride profiles were drawn and diffusion coefficients were calculated. The effectiveness of the surface treatments were studied by monitoring water absorption upto one year immersion.

Chloride ion penetration is the major cause of the corrosion of the reinforcing steel in concrete. Ordinary portland cement concrete even at high cement content

and low water cement ratio does not constitute an effective barrier against chloride penetration and therefore, using surface treatment is a necessary requirement to protect concrete structures exposed to chloride ladden environments [42].

Pfeifer and Scali [43] presented data on tests made with 21 treatment compounds which were used to protect concrete against chloride penetration. Treated concrete cubes were immersed in 15% NaCl solution for 21 days. These results indicated that epoxies, methyl methacrylates, urethanes and silane provide an excellent barrier against the ingress of chloride ions into concrete.

Work by Swamy and Tanikawa [44] indicated that even in the case of chloride contaminated concrete, surface treatments can still play a significant role in protecting steel by preventing the ingress of oxygen, water and chlorides in concrete exposed to sea water.

Sealers were found to be more effective on good concrete than on inferior concrete. Substantial differences were found in laboratory tests on the effectiveness of various sealers. Some sealers decreased water absorption of concrete to approximately one third of that of untreated concrete, while others had

little effect or even an increase in the water uptake by as much as 30 % was noted [45].

Fluckiger et al. [46] studied the effect of organic coatings on water and chloride transport in reinforced concrete. They concluded that the surface coatings strongly reduced the water and chloride uptake of concrete.

Porter [47] indicated that some surface treatment materials increase the durability of concrete. The materials used in his study were, linseed oil, flourosilicates, epoxies, latex paints, synthetic rubber and neoprene. Freezing and thawing while submerged in water, freezing by refrigerated air and thawing by water immersion, freezing in air while partially submerged and weathering were among the test variables. 3" by 6", concrete cylinders and 3" by 3" by 16.25" prisms were used to develop data on the objectives mentioned above. Cylindrical specimens were examined and weighed periodically, and were considered to have failed when 25% of their original weight had been lost due to crumbling or scaling. Prism specimens were tested periodically to determine their fundamental transverse frequency and the number of cycles of freezing and thawing which caused 40% reduction in the square of the frequency, was used as a failure criterion.

Jones et al. [48] studied the effect of exposure temperature on chloride diffusion resistance of seven widely used surface treatment materials. Silane/siloxane, epoxies and methacrylates were among them. In this study, only diffusion coefficients and film thickness were measured. It was noted in this study that, as the temperature increased, the diffusion resistance decreased. Among all the surface treatment systems, silane/siloxane performed satisfactorily at all the temperatures, except 45° C.

Sealers can be sprayed, roller applied or brush applied. The material cost for these excellent performance materials may range from about \$0.15 to \$0.5 per sq ft. Application costs may vary significantly depending on the shape of the structure. However, they are solvent based materials and require safety precautions. These materials generally cause a color darkening of concrete. One of the best performing materials is a true penetrant that does not cause any color change.

Ohama et al. [49] studied the water tightness and resistance to chloride ion penetration of concrete treated by silane. They used one component and two component type alkyl alkoxy silane. In one component type alkyl alkoxy silane.

to 5.3. In two component type they used alkyl alkoxy silane as an under coat and acrylic resin and polymethyl methacrylate as top coat. To measure the penetration depth of sealer, the cured cylindrical specimens were split and the cross-sections were sprayed with a small amount of water. The depth of unwet rim of each cross-section measured was taken as sealer penetration depth. Similarly, to determine the chloride ion penetration depth, the specimens immersed in NaCl solution, were split and the cross-sections were sprayed with 0.1% fluorecein sodium and 0.1N AgNo3 solution. The depth of rim of cross-section that changed to white color was measured and taken as chloride ion penetration.

CHAPTER 3

EXPERIMENTAL PROGRAM

The experimental program was planned to develop data on following parameters:

- 1. Effectiveness of sealers and coatings in reducing the water absorption by concrete.
- 2. Performance of coated and uncoated concrete specimens in Wet-Dry environment.
- 3. Performance evaluation of surface-coated concrete specimens as compared to uncoated concrete specimens subjected to Heat-Cool cycles.
- 4. Protection to steel reinforcement from corrosion provided by sealers and coatings.

- 5. Performance of surface-coated concrete specimens relative to uncoated specimens against sulfate attack.
- 6. Evaluation of sealers and coatings in CO₂ environment.
- 7. Performance of concrete specimens coated with sealers and coatings relative to uncoated concrete specimens in inhibiting chloride diffusion.
- 8. Effectiveness of sealers and coatings in reducing reinforcement corrosion in inservice structures.

The methodology used to attain the objectives of this study are discussed in the following sections.

3.1 MATERIALS

3.1.1 Cement

ASTM C150 Type I Portland Cement was used to make the concrete specimens.

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3.1.2 Aggregate

The coarse aggregate used in this study was crushed limestone procured from

Abu-Hadriah. The specific gravity and absorption of the coarse and fine

aggregate were determined according to ASTM C127 and are shown in Tables

3.1 and 3.2, respectively. The grading of coarse aggregate selected confirmed

to ASTM C33 and is shown in Table 3.3.

3.1.3 Surface Treatment Materials

The sealers and coatings investigated in this study are:

1. Sodium Silicate (S1)

2. Silicon Resin Solution (S2)

3. Silane/Siloxane (S3)

4. Reactive Silane/Siloxane with Top Coat (S4)

5. Alkyl alkoxy silane (S5)

6. Two Component Acrylic Coating (S6)

3.2 CONCRETE MIX DESIGN

The following parameters were kept invariant in all the mixes.

Cement Content:

 350 kg/m^3

Effective W/C ratio:

0.45

Coarse / Total Aggregate ratio:

0.63

3.3 SPECIMENS

For corrosion studies, 3"×6" height cylindrical reinforced concrete specimens with a 12 mm diameter steel bar were used, while for other tests unreinforced concrete specimens of similar dimensions were utilized.

3.4 SPECIMEN PREPARATION

3.4.1 Mixing of Concrete and Specimen Casting

The concrete constituents were mixed in a revolving drum type mixer for approximately 3 to 5 minutes to obtain uniform consistency. The concrete was filled in the molds in three layers and vibrated for consolidation.

3.4.2 Curing

After casting, the specimens were covered with a wet burlap, as shown in Plate 3.1, followed by a plastic sheet. They were demolded after 24 hours of casting and curing was continued for two weeks. The burlaps were wetted from time to time. Following the curing period, specimens were kept in the oven for

24 hours at 70° C to expel the moisture. Further, the specimens were air dried at room temperature for 24 hours prior to coating them with sealers.

3.4.3 Surface Preparation

Preparation of the substrate prior to the application of a surface coating significantly influences the effeciency of a sealer/coating. Failure of the surface coatings in many instances was attributed to the improper surface preparation [50]. All concrete surfaces, whether new or existing, should be cleaned to remove laitance, oil, grease, or surface contamination. The specimens were cleaned as per the manufacturer's specifications prior to the application of the sealer/coating.

3. 4. 4 Application of Sealers/Coatings

After surface preparation, the sealers/coatings were applied with the brush as per the manufacturer's specifications. Two coats of sealers/coatings were applied on the concrete specimens allowing a drying time of 2 hours between each. In some cases, to achieve the required application rate, recommended by the supplier, more than two coats were necessary. Table 3.4 shows the rate of application of the sealers and coatings used in this study.

3.5 EXPERIMENTAL TECHNIQUES

3.5.1 Chloride Profile

The primary aim of exposing the specimens to the chloride solution was to determine the chloride diffusion coefficients in the coated and uncoated concrete specimens. To achieve this, the concrete specimens were kept in a 5% NaCl solution, as shown in Plate 3.2, for three months at 40 °C. Unidirectional diffusion of chloride ions was ensured by applying the sealer on one surface and the other surfaces were coated with wax. The solution concentration was periodically checked and maintained at the required level.

After one and two months of exposure, the specimens were retrieved, washed, and dried to remove the surface moisture and salt, and thin slices of concrete were obtained at 5, 15, 30, 50, 100 and 150 mm intervals by dry cutting. After slicing, the specimens were crushed and ground to a fine powder passing ASTM No.100 sieve. Three grams of the powder was taken and washed into a beaker with 10 ml of hot distilled water, to which 3 ml of concentrated nitric acid was added. The mixture was then shaken for thorough mixing. The volume of the solution was made to 50 ml by adding distilled water and was left to boil for one

minute. After the digestion process, the solution was filtered and the filtrate was made up to 100 ml with distilled water.

Chloride concentration was determined using a chloride ion selective electrode in conjunction with a double junction reference electrode. This was done using a microprocessor based ionalyzer. The chloride profile so obtained was used to evaluate the diffusion coefficients solving the Fick's second law of diffusion with appropriate boundary conditions, using an algorithm developed at KFUPM [51].

3.5.2 Sulfate Resistance

After applying the sealers, half of the specimens were kept in a 3.1% Na₂SO₄ solution and a the other half were placed in the potable water. After 2,3,4 and 6 months of exposure, three specimens from the sulfate solution and the potable water were retrieved, washed to remove the salt and tested to determine the compressive strength. The sulfate resistance was evaluated by determining the reduction in the compressive strength, using the following formula:

$$Rc = \frac{(CW-CS)}{CW}$$

Where

Rc = Reduction in compressive strength, %

CW = Compressive strength of specimen placed in water

CS = Compressive strength of specimen exposed to the sulfate solution

3.5.3 Reinforcement Corrosion

For reinforcement corrosion studies, two groups of concrete specimens measuring 3"x6" with a 12 mm diameter steel bars were cast. After two weeks of curing, the specimens were coated with different surface treatment materials. The first group of specimens were kept in 5% NaCl solution and reinforcement corrosion, was evaluated by monitoring corrosion potentials and measuring corrosion current density at regular intervals.

The corrosion potentials were measured using a high impedance voltmeter and a saturated Calomel electrode (SCE). Corrosion current density was

determined by linear polarization resistance method (LPRM) using a Potentiostat/Galvonostat. Figure 3.5 shows the schematic diagram of experimental set-up to measure the corrosion current density. The resistance to polarization (Rp) was determined by conducting a linear polarization scan in the range of \pm 10 mV of the corrosion potential. A scan rate of 0.1 mV per second was used. The corrosion current density was determined using Stern - Geary formula [52].

$$I_{corr} = B / Rp$$

where: $I_{corr} = corrosion current density, \mu A/cm^2$

Rp = Polarization Resistance, Ohm. cm²

B =
$$(\beta a. \beta c) / 2.3 (\beta a + \beta c)$$

 βa and βc are the anodic and cathodic Tafel constants, respectively.

Anodic and cathodic Tafel constants of 120 mV were used in this investigation.

Lambert et al. [18] have indicated a good correlation between corrosion current density measured using LPRM and gravimetric methods, using these values.

In the second group of specimens, reinforcement corrosion was accelerated by impressing an anodic potential of 2 Volts. For this purpose, the bars in the concrete specimens were connected in series to a DC power source. Stainless steel plates were used as cathodes. The current supplied to each of the specimen, due to the application of a fixed potential of + 2 V, was monitored at

30 minutes interval by measuring the potential drop over a 1 Ohm resistor. The two leads of the resistor were connected to a data acquisition system for monitoring the current. The current supplied to each specimen was plotted against time. The time-current curves were used to evaluate the time to cracking of concrete due to reinforcement corrosion.

3.5.4 Heat - Cool Treatment

To evaluate the performance of sealers and coatings under the hot weather conditions, the concrete specimens were subjected to heat - cool treatment. After coating them with sealers and coatings, the concrete specimens were placed in the oven, as shown in Plate 3.3, which was maintained at 70° C for 8 hours and then at room temperature (23° C) for 16 hours to complete one heat-cool cycle. At the end of 30,60,90 and 120 cycles the water absorption and the compressive strength of the coated and uncoated concrete specimens was determined according to ASTM C 642 and ASTM C 39, respectively.

3.5.5 Wetting and Drying

To evaluate the performance of sealers/coatings in the splash zone of a marine environment, the coated and uncoated concrete specimens were exposed to wetdry environment. The wet - dry cycles were simulated by wetting the

specimens for one day and drying them for two days. The effect of wet-dry cycles on the coated and uncoated concrete specimens was evaluated after 10,20,30 and 40 cycles by measuring water absorption and compressive strength.

3.5.7 Carbonation

In certain areas of the world, carbonation is the major cause of reinforcement corrosion. In the presence of moisture, carbondioxide forms carbonic acid which then reacts with the hydration products of cement, particularly calcium hydroxide, to form calcium carbonate.

$$CO_2 + H_2O \rightarrow H_2CO_3$$
 (3.1)

$$Ca(OH)_2 + H_2CO_3 \rightarrow CaCO_3 + 2H_2O$$
 (3.2)

Because the conversion of Ca(OH)₂ to CaCO₃ decreases the alkalinity, reinforcement is susceptable to corrosion. The experimental set up used to evaluate the effectiveness of sealers/coatings in preventing carbonation of concrete is shown in Plate 3.4. To distribute CO₂ uniformly, it was passed through 3" deep water and specimens were kept 1" above the water level. After continuous storage in the carbonation chamber for 1,2.3,4 and 5 weeks, the specimens were retrieved

from the carbonation chamber and cut at four levels. The carbonation depth was measured by spraying phenolphthalein on a freshly broken surface. The phenolphthalein indicator gradually changes from colorless to pink at broken surfaces with a pH of greater than 9 - 9.5, while the carbonated surface remains colorless. The specimens were cut at four levels, and atleast 10 readings spread all over each section, i. e; 80 points on each specimen, at each test period, were measured and average values were recorded.

3.5.8 Rehabilitation

Figure 3.6 shows the schematic diagram of the concrete specimens used to evaluate the performance of concrete sealers and coatings in reducing reinforcement corrosion in in-service structures. 2.5" × 4" × 12" prism concrete specimens were used to simulate the actual partially immersed column structures. Before the application of sealers and coatings, the steel in the reinforced concrete specimens, was corroded by impressing an anodic potential of 4 V. After the reinforcement corrosion became well established, the corrosion potentials and corrosion current density was measured, as discussed in the section 3.6.3. Surface treatment materials were then applied on the top 8" of the concrete specimens. leaving bottom 4" portion uncoated.

After the application of surface treatment materials the concrete specimens were exposed to 5% NaCl solution and the effectiveness of sealers and coatings was monitored by measuring corrosion potentials and corrosion current density at regular intervals.

Table 3.1: Absorption and Specific Gravity of Coarse Aggregates

Size	Absorption (%)	Bulk Specific Gravity
3/32"	1.712	2.609
3/16"	1.621	2.607
3/8"	1.477	2.605
1/2"	1.034	2.643

Table 3.2: Grading of Coarse Aggregate

Size	Wt. Retained	Cum.Weightet Rained, %	% Passing	ASTM C33 No. 7
3/4"	0	0	100	100
1/2"	10	10	90	90-100
3/8"	45	55	45	40-70
3/16"	40	95	5	0-15
3/32"	5	100	0	0-5

Table 3.3 : Absorption and Specific Gravity of Fine Aggregate

Absorption %	Bulk Specific Gravity
1.562	2.55



Plate 3.1: Curing of Concrete Specimens

Table 3.4: Manufacturer's Coverage Rates of Sealers and Coatings

Sealers/Coatings	Coverage Rate
Sodium Silicate	0.55 L/m ²
Silicone Resin Solution	0.5 L/m ²
Silane/Siloxane	0.4 L/m ²
Silane/Siloxane with Top Coat	0.15 L/m ²
Silane	0.25 L/m ²
Two Component Acrylic Coating	2.5 Kg/m ²

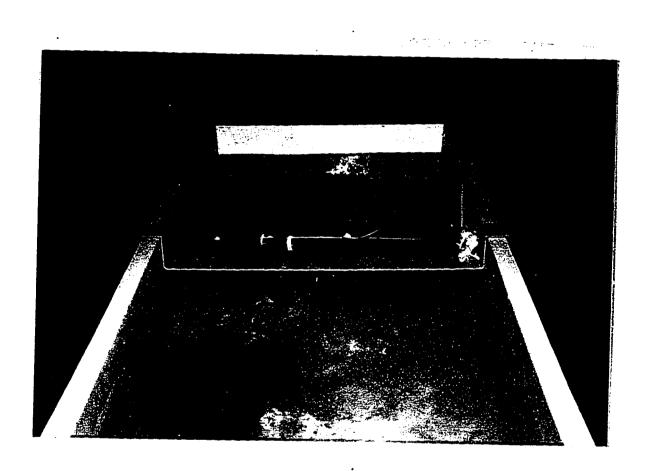
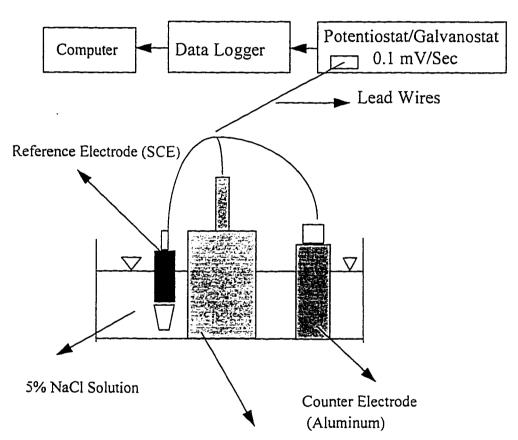


Plate 3.2: Heaters used to Maintain the Temperature of the Chloride Soluiton



Concrete Specimen with Embedded Steel bar

Figure 3.5: Schematic Diagram of Experimental Set-Up to Measure the Corrosion Current Density

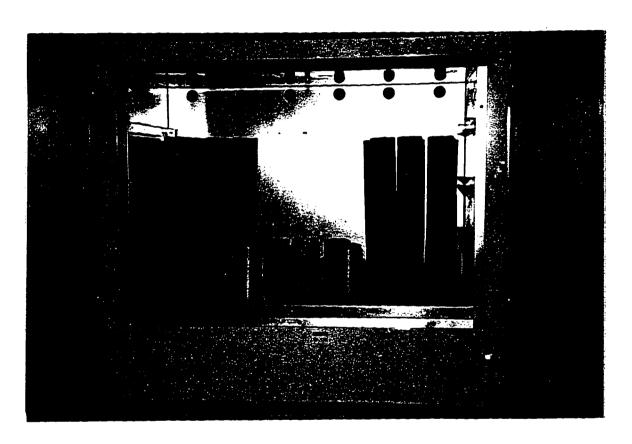


Plate 3.3: Specimens Exposed to Heat-Cool Treatment

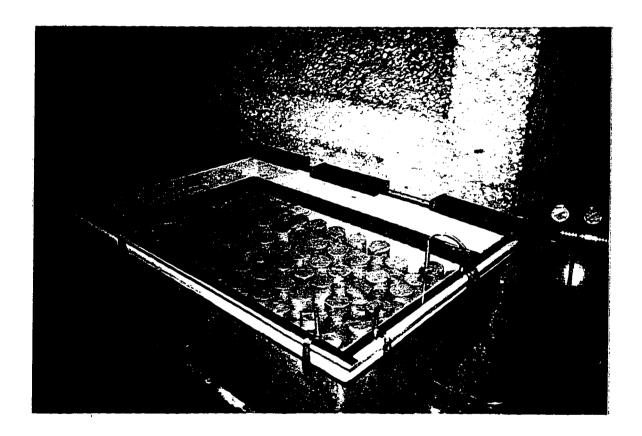


Plate 3.4: Carbonation Chamber with the Specimens

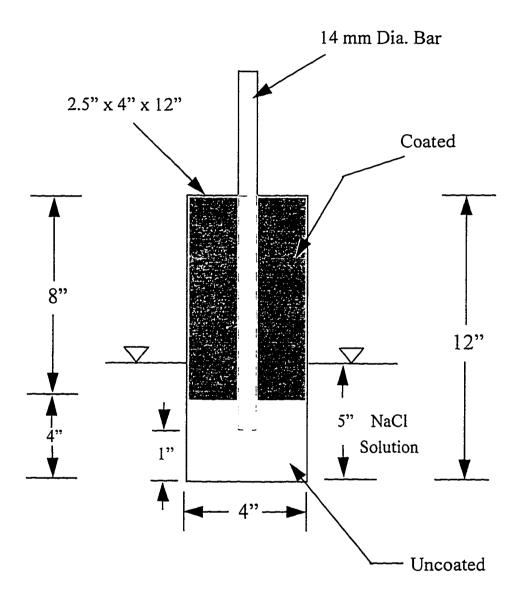


Figure 3.6: Schematic Diagram of Concrete Specimen used to Evaluate the Performance of Sealers and Coatings in in-Service Structures

CHAPTER 4

RESULTS

4.1 ABSORPTION

The 48-hour absorption in the uncoated and coated concrete specimens, cured for 14 days under burlap, are shown in Figure 4.1.1. Figure 4.1.2, shows the compressive strength of the coated and uncoated concrete specimens before exposing to heat-cool and wet-dry environments. Figures 4.1.3 and 4.1.4 show the rate of water absorption and rate of moisture loss in the coated and uncoated concrete specimens, respectively. The water absorption in the uncoated concrete specimens was around 3.94%, while it was in the range of 0.29 to 4.38% in the coated concrete specimens. The absorption in the concrete specimens coated with acrylic coating, silane and silane/siloxane with top coat was 0.29, 0.4 and 0.67%, respectively. The water absorption in the concrete specimens coated with sodium silicate increased by 10% and that in the concrete specimens coated with two component acrylic coating it decreased by 93% compared to the uncoated concrete specimens. The increase in water absorption in the concrete specimens coated

with sodium silicate, as compared to the uncoated concrete specimens, may be attributed to the fact that, sodium silicate is soluble in water. The absorption in the concrete specimens coated with silane/siloxane with top coat was less than those coated with silane/siloxane only. The compressive strength in all the coated and uncoated concrete specimens, before exposing to heat-cool and wet-dry environments, was more or less similar to each other (Figure 4.1.2). In fact the compressive strength in the concrete specimens coated with silicone resin solution and sodium silicate was slightly higher than those coated with other coatings and sealers.

Although the performance of surface treatment materials, cannot be evaluated by measuring the water absorption only, a performance grading system can be suggested, based on this test to aid in the selection of sealers.

The suggested performance rating are shown in Table 4.1.1

Table 4.1.1: Suggested Performance Grading for Sealers and Coatings

GRADE	REDUCTION IN WATER ABSORPTION
A	90-100%
В	80-90%
С	70-80%

According to the above rating, silane and two component acrylic coating fall under grade A and silane/siloxane with top coat can be classified under Grade B. Silicone resin solution and silane/siloxane did not perform satisfactorily as the reduction in the water absorption was less than 75%. The water absorption in the concrete specimens coated with sodium silicate was more than that in the uncoated concrete specimens.

4.2 WET - DRY TREATMENT

The effect of wet-dry cycling on the water absorption in the coated and uncoated concrete specimens is plotted in Figure 4.2.1. A decrease in the water absorption was observed in all the coated and uncoated concrete specimens (Figure 4.2.1).

after 10 wet-dry cycles. This reduction in the water absorption can be attributed to the densification of concrete due to hydration of cement. However, after 10 wet-dry cycles, there was a steady increase in the water absorption in all the coated and uncoated concrete specimens, because of formation of micro cracks within the concrete due to moisture variation.

The water absorption in the concrete specimens coated with acrylic coating and silane/siloxane with top coat, decreased by 93 and 86%, respectively, compared to the uncoated concrete specimens, after 40 wet-dry cycles. In the concrete specimens coated with silane, silane/siloxane with top coat and two component acrylic coating there was no significant increase in the water absorption after 20 wet-dry cycles. The water absorption in the concrete specimens coated with sodium silicate was higher than that in the uncoated concrete specimens.

The variation in the compressive strength of concrete specimens subjected to wet-dry treatment is plotted in Figure 4.2.2. No significant reduction in the compressive strength was observed in the coated and uncoated concrete specimens, upto 10 wet-dry cycles (Figure 4.2.2). Thereafter, a continuous reduction in the compressive strength was recorded with increasing wet-dry cycles. In the uncoated concrete specimens and those coated with sodium silicate there

was 12.36 and 12.78%, reduction in the compressive strength, after 40 wet-dry cycles. In the concrete specimens coated with other coatings an average reduction of 6.8% in the compressive strength was noticed, except in the specimens coated with silicone resin solution, in which the reduction was 10.13%. These data suggest, that silane/siloxane, silane/siloxane with top coat, silane and two component acrylic coating were effective in reducing the damage due to wet-dry cycling.

4.3 HEAT-COOL TREATMENT

The water absorption in the coated and uncoated concrete specimens, exposed to 30, 60, 90 and 120 heat/cool cycles are shown in Figure 4.3.1. These values increased with the number of heat-cool cycles in all the concrete specimens, except those coated with silicone resin and silane/siloxane. This increase in the water absorption, with increasing heat-cool cycles, may again be attributed to the formation of micro cracks within the concrete due to thermal variation. The water absorption in the concrete specimens coated with silane/siloxane, silane/siloxane with top coat and silane was more or less similar, with an average value of 1.85% after 120 heat-cool cycles. The water absorption in the concrete specimens coated

with sodium silicate was higher than that in the uncoated concrete specimens. The least water absorption, after 120 heat-cool cycles, was indicated in the concrete specimens coated with the acrylic coating. The water absorption in these specimens were one-fifth of those in the uncoated concrete specimens.

Figure 4.3.2 shows the effect of heat-cool cycles on the reduction in the compressive strength, in the coated and uncoated concrete specimens. A steady increase in these values was indicated in all the specimens due to heat-cool treatment. The highest reduction in the compressive strength was indicated in the concrete specimens coated with sodium silicate and silicone resin solution. The reduction in the compressive strength in uncoated concrete specimens and those coated with sodium silicate and silicone resin solution was 17.63, 16.36 and 13.76%, respectively, after 120 heat-cool cycles. An average strength reduction of 7.8% was observed in the concrete specimens coated with other coatings. Among the coatings investigated, silane/siloxane, silane/siloxane with top coat, silane and two component acrylic coating were effective in minimizing the damage due to heat-cool treatment.

4.4 ACCELERATED REINFORCEMENT CORROSION

BY IMPRESSED POTENTIAL

As discussed in chapter 3, reinforcement corrosion was accelerated by impressing an anodic potential of 2 Volts and the resulting current was plotted against time. The time-current plots for the uncoated concrete specimens and those coated with sodium silicate, silicone resin solution, and silane/siloxane are shown in Figures 4.4.1 through 4.4.4, respectively. These curves were analyzed to evaluate the time to cracking of concrete due to reinforcement corrosion. Plates 4.4.6 through 4.4.12 show the cracked and uncracked concrete specimens. The time to cracking of concrete, due to reinforcement corrosion, was taken as the point at which a significant increase in current or a change in the slope of time-current curve occurred.

The time-current curve for the uncoated concrete specimens is shown in Figure 4.4.1. The current in the uncoated concrete specimens was high, right from the initial stages, indicating low resistance. The current in all the three uncoated concrete specimens, increased to a maximum value after about 144 hours of impressed potential. At the same time, corrosion products and hair line cracks were observed on the surface, as shown in Plate 4.4.12. Thereafter, the current

decreased continuously, starting from the point at which the first crack appeared, upto 648 hours, after this time a sudden increase in the current was again noticed. This decrease in the current, after first point of cracking is mainly because of filling of cracks with the rust products.

Among the coated concrete specimens, those treated with sodium silicate cracked first. The time-current curves for these concrete specimens, as shown in Figure 4.4.2, indicate continuous increase in the current upto 176 hours of impressed potential and at this exposure period all the three specimens cracked. The concrete specimens coated with silicone resin solution cracked when the current was 0.7 mA corresponding to 685 hours (Figure 4.4.2). The behavior of these concrete specimens was similar to that of uncoated concrete specimens after first point of cracking. The concrete specimens treated with silicone resin solution performed slightly better than those coated with sodium silicate.

The concrete specimens coated with silane/siloxane performed better than those coated with the above mentioned two sealers. In these specimens current was very low, initially. But a steady increase in the current, because of reduction in the resistance, was noted upto 2125 hours of impressed potential, after which hair line

cracks and corrosion products were observed on the surface of these specimens (Plate 4.4.8).

The specimens coated with silane/siloxane with acrylic top coat, silane and two component acrylic coating did not crack at all, as shown in Plates 4.4.9 through 4.4.11, and the current was very low even after 4350 hours (180 days) of impressed potential. Among these, the concrete specimens coated with silane/siloxane with acrylic top coat performed better than those coated with silane and two component acrylic coating, as the current required to maintain a potential of +2V in these specimens was the least, as shown in Figure 4.4.5.

4.5 REINFORCEMENT CORROSION

The effectiveness of surface coatings in reducing reinforcement corrosion, in normal chloride-environment, was evaluated by measuring the corrosion potentials and the corrosion current density at periodic intervals.

4.5.1 Corrosion Potentials

The corrosion potential curves for the uncoated concrete specimens and those coated with silicone resin solution and sodium silicate, are shown in Figure 4.5.1.1. The corrosion potential curves for the concrete specimens coated with silane/siloxane, silane/siloxane with top coat, silane and two component acrylic coating are shown in Figure 4.5.1.2. Each point in these figures is the mean of readings taken on triplicate specimens with similar treatment and exposure conditions. These curves were used to evaluate the time to initiation of reinforcement corrosion based on the ASTM C 876 criterion. According to this criterion, if the potentials are numerically less than -270 mV SCE, there is a 90% probability of reinforcement corrosion.

Table 4.5.1.1 shows the time to initiation of reinforcement corrosion, based on ASTM C 876 threshold potential of -270 mV. The time to initiation of reinforcement corrosion in the uncoated concrete specimens and those coated with sodium silicate was more or less similar, being 10 and 12 days, respectively.

Among the coated concrete specimens, those treated with sodium silicate. silicone resin solution and silane/siloxane were not able to reduce reinforcement corrosion for a long time. The concrete specimens coated with silane/siloxane

performed slightly better than other coated specimens and could resist reinforcement corrosion only upto 55 days of exposure, after which the corrosion potentials were less than -270 mV SCE. The corrosion potentials on steel in the concrete specimens coated with silicone resin solution were very high initially and increased with time. After about 40 days of exposure the corrosion potentials reached -270 mV SCE indicating the initiation of reinforcement corrosion.

The corrosion potentials on steel in the concrete specimens coated with silane/siloxane with top coat and silane were more than -270 mV SCE upto more than 300 days of exposure. However, the time to initiation of reinforcement corrosion in the concrete specimens coated with silane/siloxane with top coat and silane was indicated after 320 and 340 days of exposure to 5% NaCl solution (Table 4.5.1.1).

4.5.2. Corrosion Current Density

The variation of corrosion current density (I_{COTT}) on steel, with time, in the coated and uncoated concrete specimens exposed to 5% NaCl solution is shown in Figure 4.5.2.1. These curves for the uncoated concrete specimens and those coated with sodium silicate, silicone resin solution and silane/siloxane are shown in Figure 4.5.2.2. Figure 4.5.2.3 shows the I_{COTT} on steel in the concrete specimens coated with acrylic coating, silane and silane/siloxane with top coat.

The I_{COTT} on steel in all the coated and uncoated concrete specimens initially was less than 0.02 μ A/cm² (Fig. 4.5.2.1). However, it increased with time in the uncoated concrete specimens and those coated with silicone resin solution, sodium silicate and silane/siloxane. The I_{COTT} in these concrete specimens, after 360 days of exposure, was 0.328, 0.31, 0.286 and 0.193 μ A/cm², respectively.

Silane/siloxane with top coat, silane and two component acrylic coating were very effective in reducing the rate of reinforcement corrosion. The I_{corr} on steel in the concrete specimens coated with these coatings was very low throughout the testing period. The I_{corr} on steel in the concrete specimens coated with silane/siloxane with top coat and silane was 0.0058 and 0.0061 $\mu A/cm^2$.

respectively. It should be noted that these values were less than $0.01~\mu\text{A/cm}^2$ which is considered as the maximum value for long-term maintenance-free performance [53]. However, the corrosion current density on steel in the concrete specimens coated with acrylic coating was $0.0108~\mu\text{A/cm}^2$, after about 360 days of exposure to 5% NaCl solution.

4.6 SULFATE ATTACK

Figure 4.6.1 shows the reduction in the compressive strength in the uncoated concrete specimens and those coated with sodium silicate, silicone resin solution, silane/siloxane, silane/siloxane with top coat, silane and two component acrylic coating. These specimens were exposed to sulfate solution for a period of 12 months. Plates 4.6.2 through 4. 6. 8 shows the appearance of coated and uncoated concrete specimens immersed in the sulfate solution, compared to those cured in the water for 330 days.

After two months of immersion in the sulfate solution, the reduction in the compressive strength was very low and there were no signs of sulfate attack in all the coated and uncoated concrete specimens. However, after three months of exposure, softening of the cement paste around the aggregates was observed in the

uncoated concrete specimens and those coated with sodium silicate and silicone resin solution. After six months of exposure, sulfate attack was predominant in the uncoated concrete specimens and those coated with sodium silicate and silicone resin solution.

A steady reduction in the compressive strength was measured in all the coated and uncoated concrete specimens (Figure 4.6.1). After about 330 days of exposure, a reduction of 41, 39 and 36% was noted in the uncoated concrete specimens and those coated with sodium silicate and silicone resin solution, respectively, compared to similar specimens cured in water.

The deterioration of concrete due to sulfate attack was observed in the specimens coated with silane/siloxane after 6 months of exposure, and the reduction in compressive strength was 26.8% after about 300 days of immersion in the sulfate solution.

There were no signs of sulfate attack, upto eight months of exposure, in the concrete specimens coated with silane. However, after this period aggregates were exposed because of softening of cement paste. The behavior of specimens coated with two component acrylic coating was similar to that of specimens coated with

observed within the coating. This coating was most effective in minimizing the damage due to sulfate attack, among all the surface treatment systems used in this study, upto 6 months of exposure. A reduction of 1.95% in the compressive strength was noted after about 180 days of exposure to the sulfate solution, which increased to 19.63% after eleven months of exposure.

Among all the surface treatment systems, silane/siloxane with top coat was effective in minimizing the damage due to sulfate attack. There was only 0.3% reduction in the compressive strength after about 2 months of exposure and it reached to 8.3% at the end of the test, corresponding to 330 days of immersion in the sulfate solution. No signs of cracking of the coating or softening of cement paste around the aggregates was observed.

4.7 CARBONATION

The carbonation depth and weight gain curves for coated and uncoated cylindrical concrete specimens are shown in Figures 4.7.1 and 4.7.2, respectively. After continuous storage of the coated and uncoated concrete specimens in the carbonation chamber for 1, 2, 3, 4 and 5 weeks, the depth of carbonation was

measured by spraying phenolphthalein on the cut cross-sections of the specimens. Plates 4.7.3 and 4.7.4 show the phenolphthalein sprayed cross-section of specimens coated with silane/siloxane with top coat and two component acrylic coating respectively, after 180 days of continuous storage in the carbonation chamber.

In the uncoated concrete specimens and those coated with sodium silicate, silicone resin solution carbonation started within seven days of exposure. The carbonation depth and weight gain in the uncoated specimens, after about five weeks of continuous storage in the carbonation chamber, was 28.3 mm and 84.8 g.. respectively. The concrete specimens treated with sodium silicate performed slightly better than those treated with silicone resin solution, silane/siloxane and silane, the depth of carbonation being 13.1 mm and the weight gain was 65.8 g.. after about five weeks of exposure. Among all the coated specimens those coated with silicone resin solution, silane/siloxane and silane did not perform well and were not effective in preventing carbonation. The depth of carbonation in these specimens was 24.8, 23.9 and 25.1 mm, respectively after five weeks of exposure. Initially the carbonation process in the specimens coated with silane was very slow, but it accelerated after three weeks of exposure.

Plates 4.7.5 through 4.7.8 show the phenolphthalein sprayed cross-sections of specimens coated with sodium silicate, silicone resin solution, silane/siloxane and silane, after about 14 days of exposure to accelerated carbonation environment.

Plate 4.7.9 shows the extent of carbonation in the uncoated concrete specimens after two weeks of exposure.

4.8 CHLORIDE PROFILE

Variation of chloride concentration with depth in the uncoated concrete specimens and those coated with sodium silicate, silicone resin solution, silane/siloxane, silane/siloxane with top coat, silane and two component acrylic coating, for 30 and 90 days of exposure, is shown in Figures 4.8.1 through 4.8.7. Figures 4.8.8 and 4.8.9 show the chloride profiles in the coated and uncoated concrete specimens, exposed to 5% NaCl solution maintained at 45° C, for 30 and 90 days, respectively. Table 4.8.1 shows the chloride diffusion coefficients for these concrete specimens.

A significant reduction in the chloride concentration with depth was observed in all the coated and uncoated concrete specimens, upto a depth of 50 mm. Beyond this depth, the chloride concentration remained more or less unchanged.

The chloride concentration, at all depths, in the uncoated concrete specimens and those coated with sodium silicate, was more than any other coated concrete specimens. There was considerable reduction in chloride concentration, in the concrete specimens coated with silane, silane/siloxane with top coat and acrylic coating, as compared to the uncoated concrete specimens. The chloride concentration in these specimens did not change much, as the exposure period increased from 30 to 90 days (Figures 4.8.4, 4.8.5 and 4.8.6). As expected, the chloride concentration, in the other coated and uncoated concrete specimens. increased gradually, with the period of exposure. The penetration of chloride ions was found to be influenced by the type of coating.

As stated in Chapter 3, the solution to Fick's second law of diffusion, as shown in equation 4.1, was used to calculate the chloride diffusion coefficients. The solution contains the following parameters, the chloride concentration C_X , at any depth x, the chloride concentration at the surface of the concrete specimens C_S and the time t at which the chloride concentration was measured. The surface

concentration was calculated by regression analysis of the experimentally calculated chloride profile.

Table 4.8.1: Chloride Diffusion Coefficients in the Coated and Uncoated Concrete Specimens

Sealer/Coating	Diffusion Coefficient, cm ² /sec
Sodium Silicate	20.06 x 10 ⁻⁸
Silicone Resin Solution	16.86 x 10 ⁻⁸
Silane/Siloxane	14.83 x 10 ⁻⁸
Silane/Siloxane with Topcoat	7.83 x 10 ⁻⁸
Silane	9.86 x 10 ⁻⁸
Acrylic Coating	8.18 x 10 ⁻⁸
Uncoated Concrete	21.83 x 10 ⁻⁸

$$\frac{Cx}{Cs} = 1 - \frac{x}{2(D_e t)^{1/2}}$$
 (4.1)

The diffusion coeffecients for the uncoated concrete specimens and those coated with sodium silicate were much higher than those coated with sealers and coatings. The diffusion coefficient for the concrete specimens coated with silane/siloxane with top coat was 7.83 x 10⁻⁸ cm²/sec, whereas it was 21.83 x 10⁻⁸ cm²/sec in the uncoated concrete specimens. Silane and two component acrylic coating were also effective in reducing the diffusion of chloride ions.

4.9 REHABILITATION

Figure 4.91 and 4.9.2 shows the variation of corrosion potentials and corrosion current density in the coated and uncoated concrete specimens before and after the application of surface treatment materials. Each point in these figures is the mean of readings taken on triplicate specimens with similar treatment and exposure conditions.

4.9.1 Corrosion Potentials

The corrosion potentials of steel in all the coated concrete specimens increased after the application of surface treatment materials except those coated with sodium silicate. The corrosion potentials in the uncoated concrete specimens and those coated with sodium silicate were almost unaffected by the application of surface treatment materials. Highest increase in the corrosion potentials was indicated in the concrete specimens coated with silane. The corrosion potentials in the concrete specimens coated with silane before the application was -597 mV. and it increased to -318 mV after the application. This increase in corrosion potentials in the most of the coated concrete specimens was mainly due to the reduced supply of oxygen. Furthermore, sodium silicate was not effective in reducing the supply of oxygen to the concrete.

4.9.2 Corrosion Current Density

There was decrease in the corrosion current density in all the coated concrete specimens, after the application of surface treatment materials, except those coated with sodium silicate. The corrosion current density in the uncoated concrete specimens and those coated with sodium silicate continued to increase throughout the exposure period.

The largest reduction in the corrosion current density was observed in the concrete specimens coated with silane. The corrosion current density in the concrete specimens was $0.315~\mu\text{A/cm}^2$ before the application of surface treatment material and it reduced to $0.141~\mu\text{A/cm}^2$ after the concrete specimens were treated with silane.

The performance of concrete specimens coated with silane/siloxane and silicone resin solution was not better than those coated with silane/siloxane with top coat and acrylic coating.

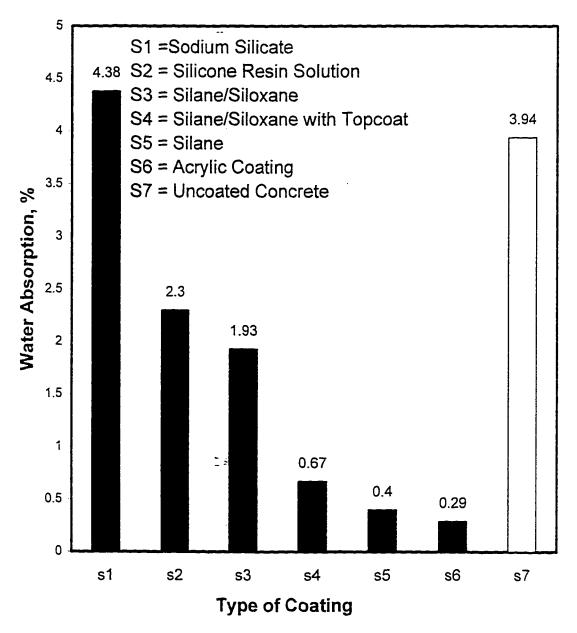


Figure 4.1.1: Water Absorption in the Coated and Uncoated Concrete Specimens

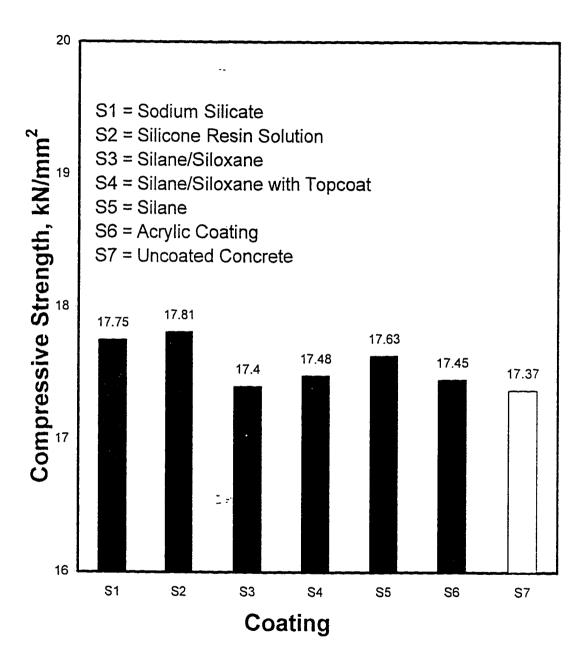


Figure 4.1.2: Compressive Strength in the Coated and Uncoated Concrete Specimens before Exposing to Heat-Cool and Wet-Dry Environments

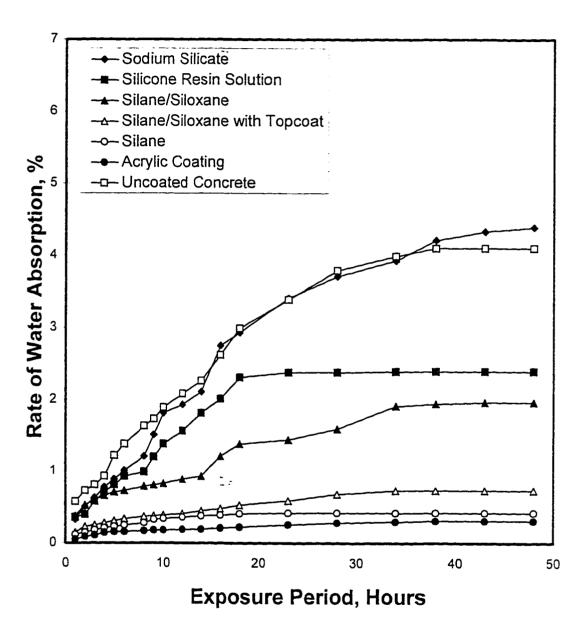


Figure 4.1.3 : Rate of Water Absorption in the Coated and Uncoated Concrete Specimens

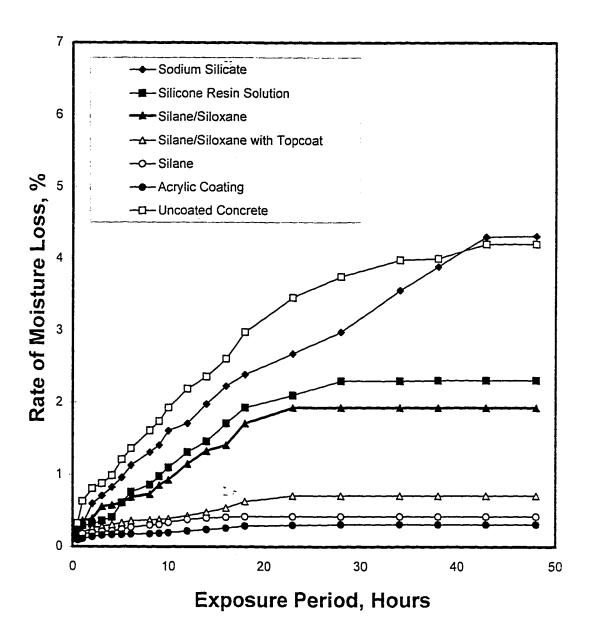


Figure 4.1.4 : Rate of Moisture Loss in the Coated and Uncoated Concrete Specimens

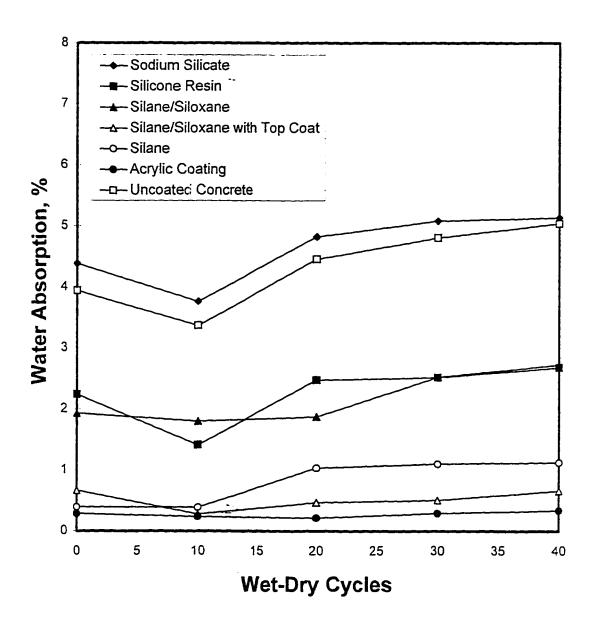


Figure 4.2.1 : Effect of Wet-Dry Cycling on Water Absorption.in the Coated and Uncoated Concrete Specimens

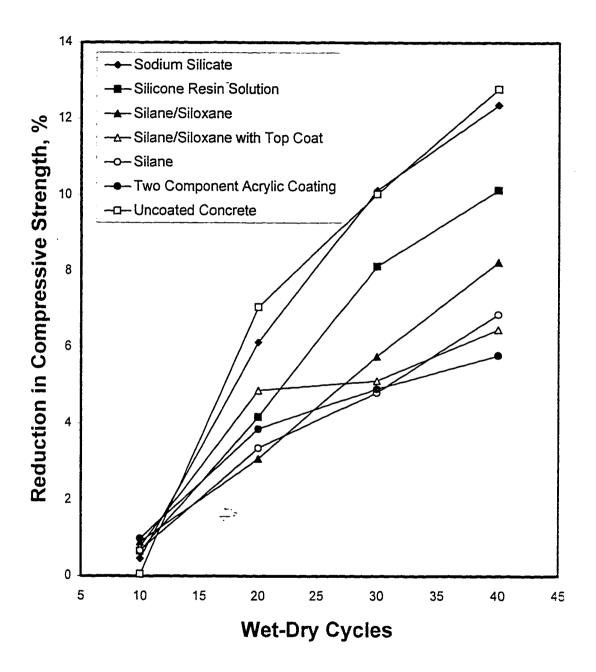


Figure 4.2.2 : Effect of Wet-Dry Cycling on the Compressive Strength in the Coated and Uncoated Concrete Specimens

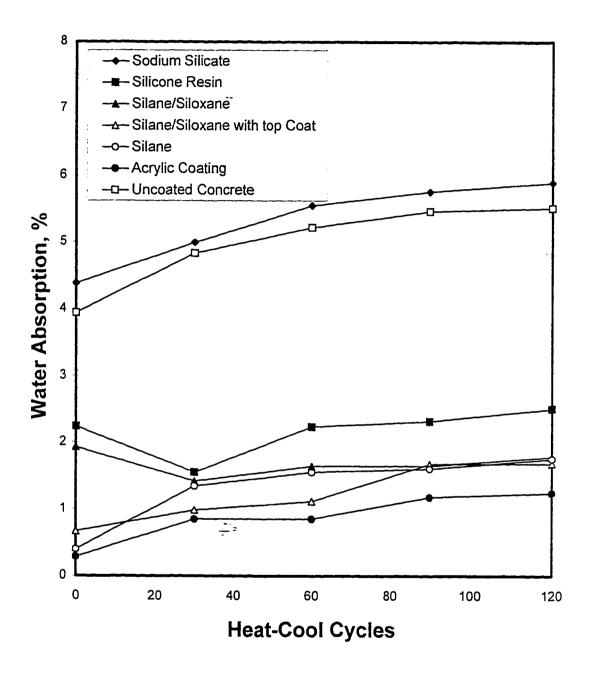


Figure 4.3.1: Effect of Heat-Cool Cycling on Water Absorption in the Coated and Uncoated Concrete Specimens

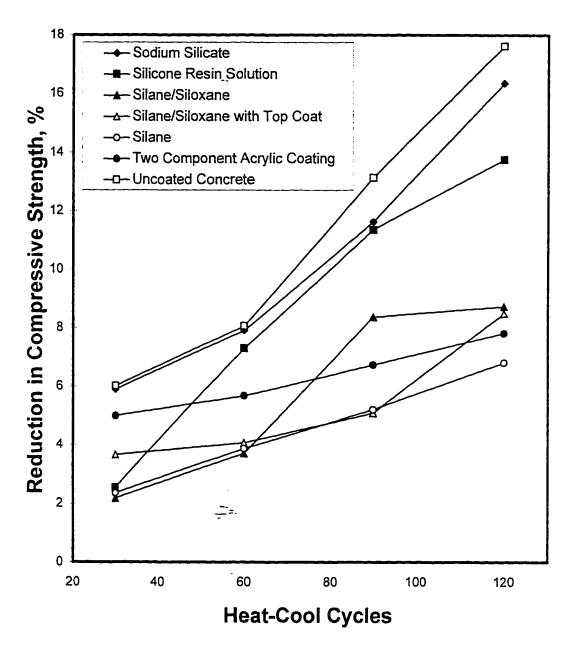


Figure 4.3.2: Reduction in Compressive Strength in the Coated and Uncoated Concrete Specimens Subjected to Heat-Cool Treatment

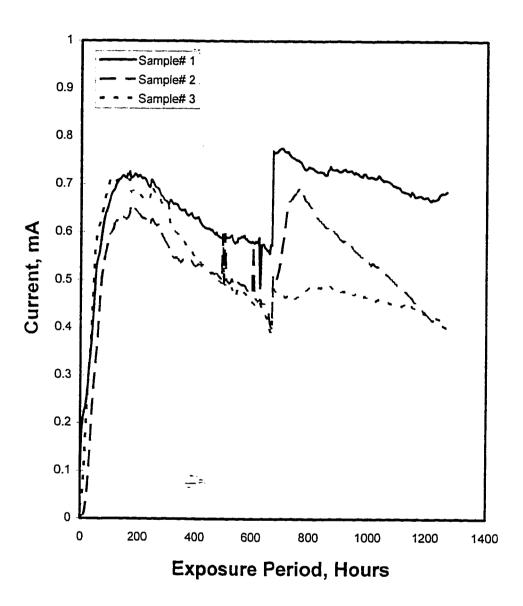


Figure 4.4.1: Variation of Current in the Concrete Specimens Coated with Sodium Silicate

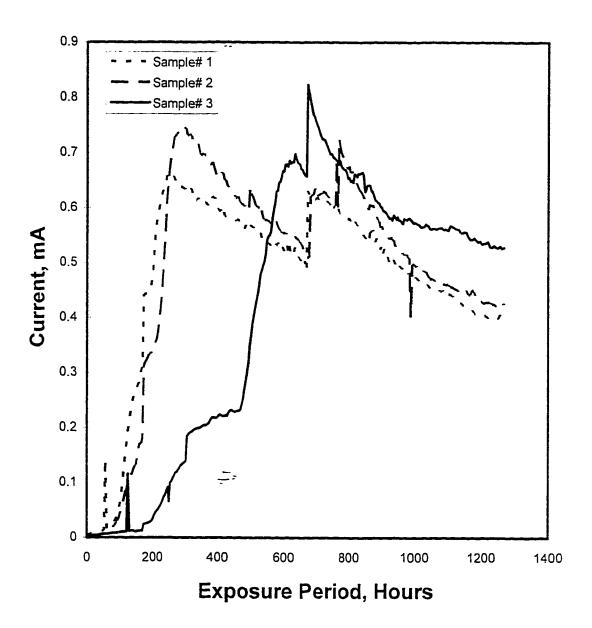


Figure 4.4.2 : Variation of Current in the Concrete Specimens Coated with Silicone Resin Solution

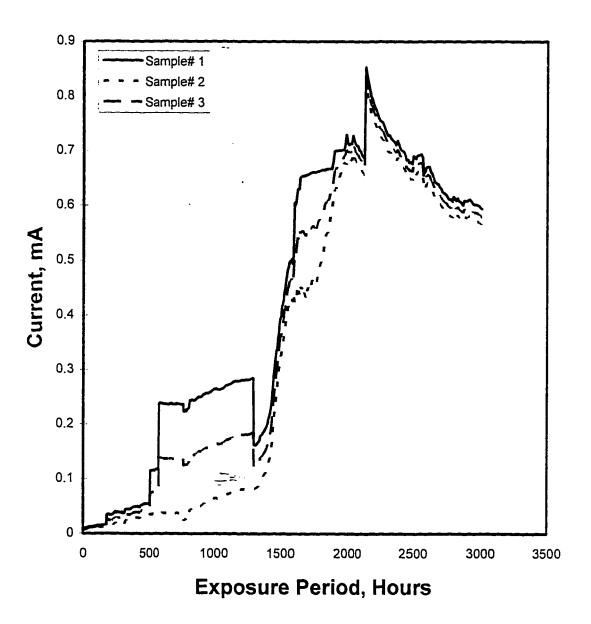


Figure 4.4.3 : Variation of Current in the Concrete Specimens Coated with Silane/Siloxane

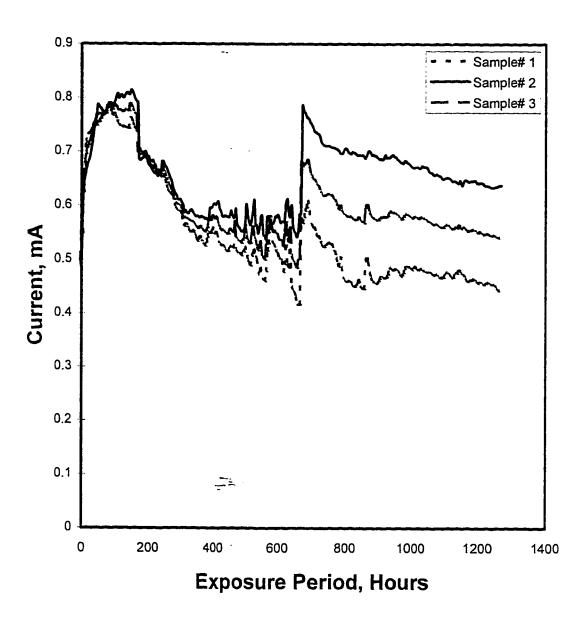


Figure 4.4.4 : Variation of Current in the Uncoated Concrete Specimens

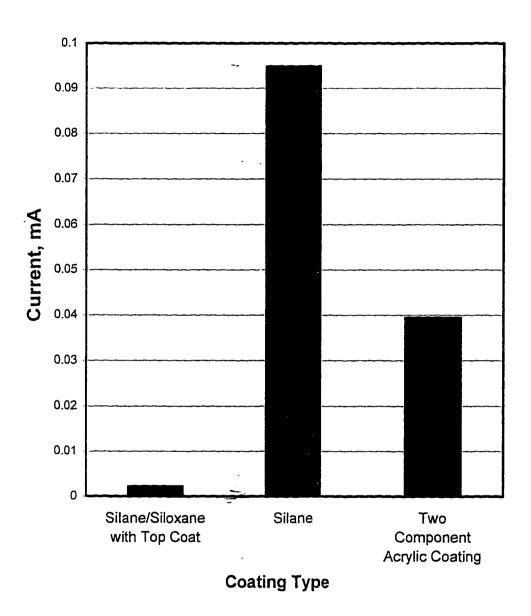


Figure 4.4.5 : Current Required to Maintain a Potential of +2 V in the Uncracked Coated Concrete Specimens

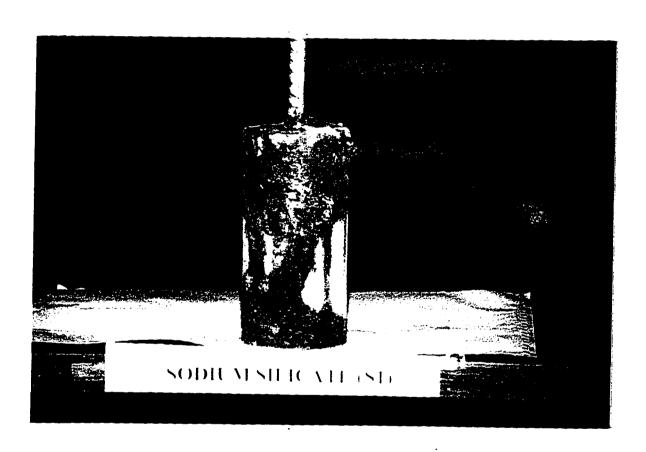


Plate 4.4.6: Concrete Specimen Coated with Sodium Silicate Subjected to an Impressed Anodic Potential of 2 Volts

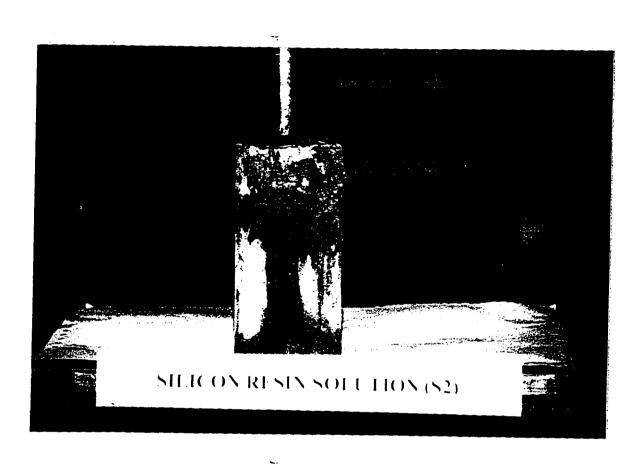


Plate 4.4.7: Concrete Specimen Coated with Silicone Resin Solution Subjected to an Impressed Anodic Potential of 2 Volts

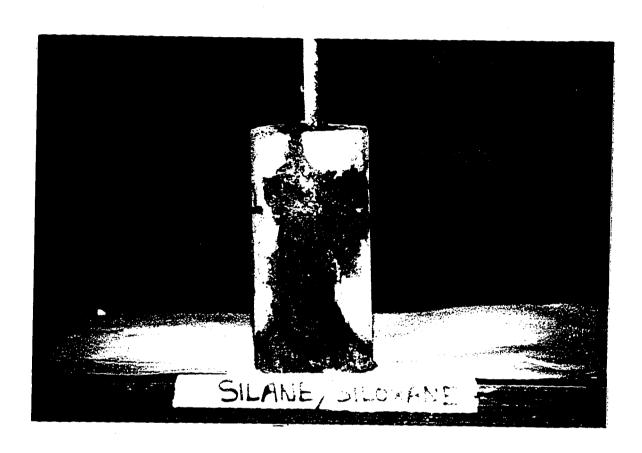


Plate 4.4.8: Concrete Specimen Coated with Silane/Siloxane Subjected to an Impressed Anodic Potential of 2 Volts

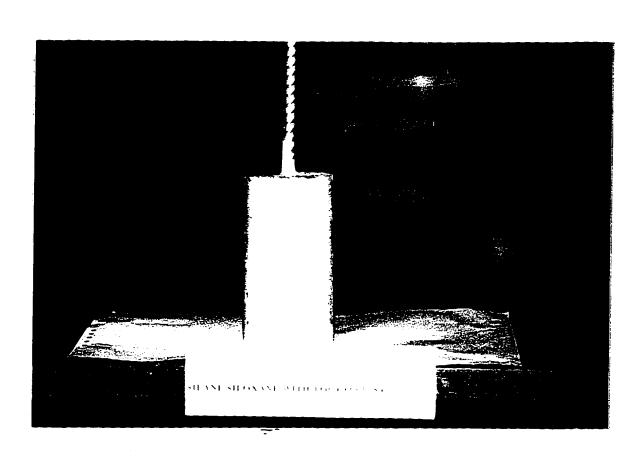


Plate 4.4.9: Concrete Specimen Coated with Silane/Siloxane with top coat Subjected to an Impressed Anodic Potential of 2 Volts

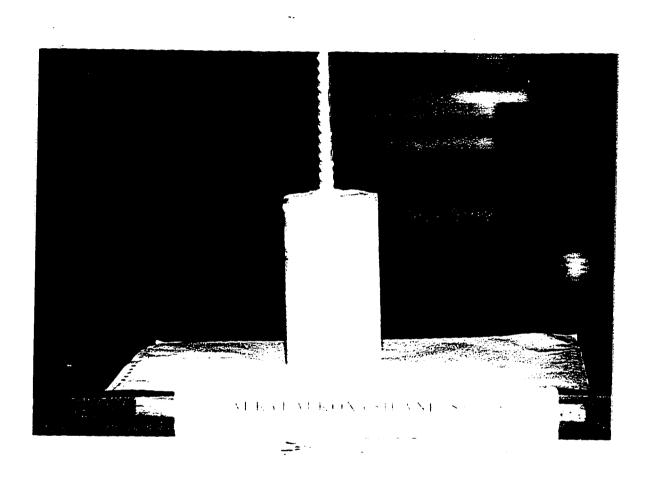


Plate 4.4.10 : Concrete Specimen Coated with Silane Subjected to an Impressed Anodic Potential of 2 Volts

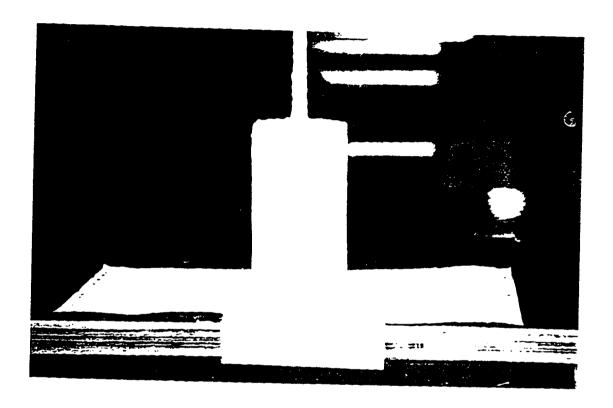


Plate 4.4.11: Concrete Specimen Coated with Acrylic Coating Subjected to an Impressed Anodic Potential of 2 Volts

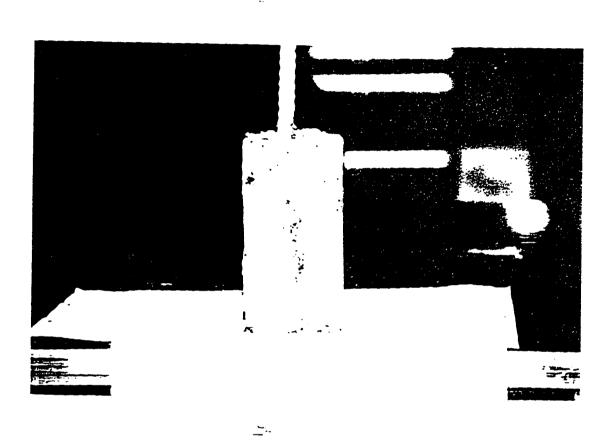


Plate 4.4.12: Uncoated Concrete Specimen Subjected to an Impressed Anodic Potential of 2 Volts

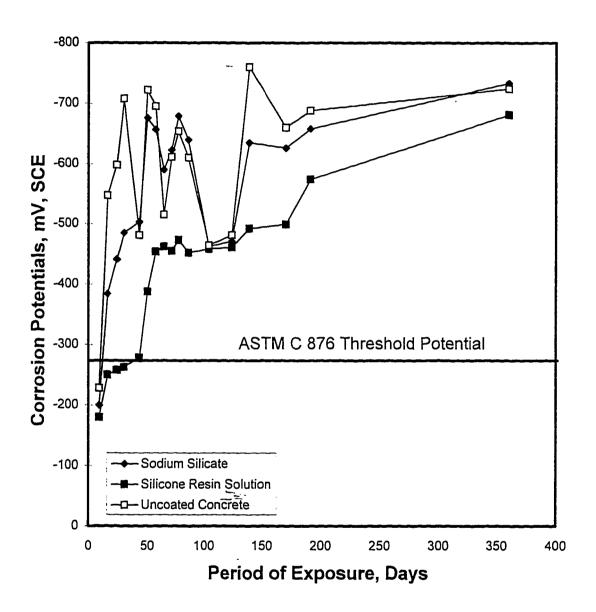


Figure 4.5.1 : Variation of Corrosion Potentials on steel in the Coated and Uncoated Concrete Specimens

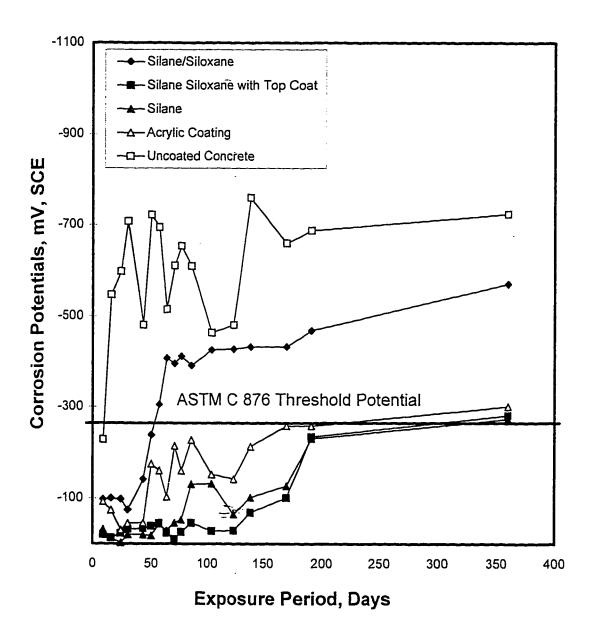


Figure 4.5.2 : Variation of Corrosion Potentials on steel in the Coated and Uncoated Concrete Specimens

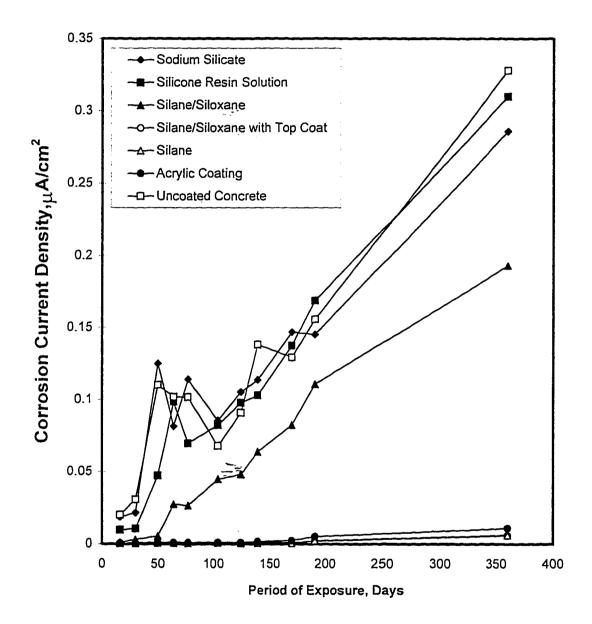


Figure 4.5.3 : Variation of Corrosion Current Density on Steel in the Coatedd and Uncoated Concrete Specimens

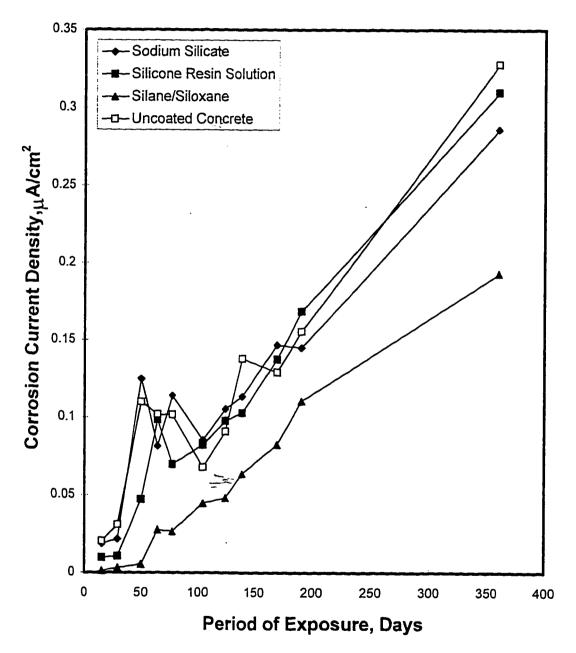


Figure 4.5.4: Variation of Corrosion Current Density on Steel in the Coated and Uncoated Concrete Specimens

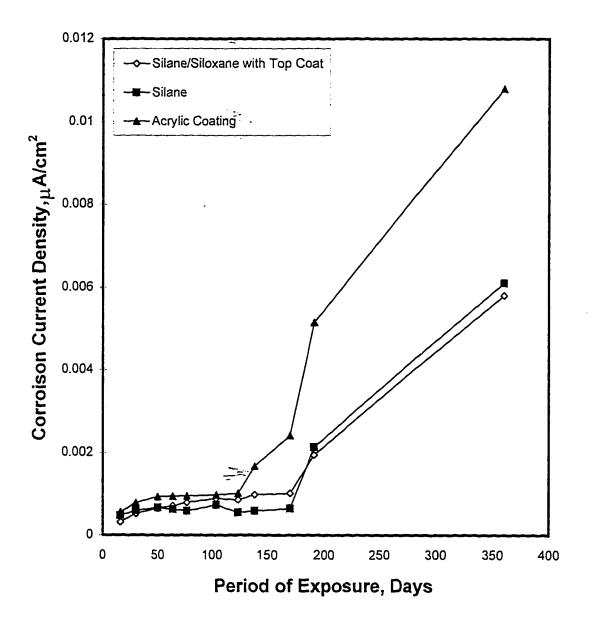


Figure 4.5.5 : Variation of Corrosion Current Density on Steel in the Coated Concrete Specimens

Table 4.5.1.1: Time to initiation of reinforcement corrosion, based on ASTM C 876

Sealer/ Coating	Time to initiation of reinforcement corrosion,
	days
Uncoated Concrete	10
Sodium Silicate	12
Silicone Resin	40
Silane/Siloxane	55
Silane	340
Acrylic Coating	225
Silane/Siloxane with top coat	320

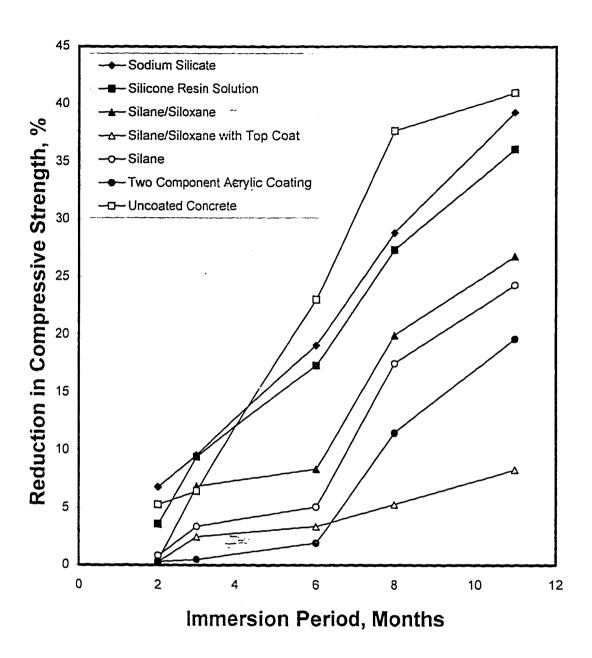


Figure 4.6.1: Reduction in Compressive Strength in the Coated and Uncoated Concrete Specimens Exposed to Sulfate Solution

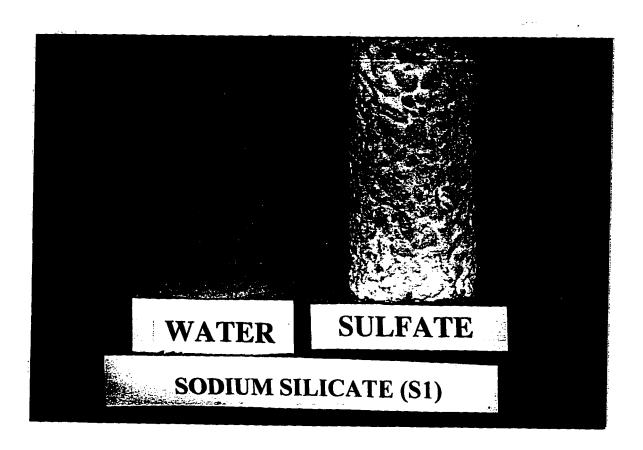


Plate 4.6.2: Concrete Specimens Coated with Sodium Silicate Immersed in the Sulfate Solution and Water for 330 days

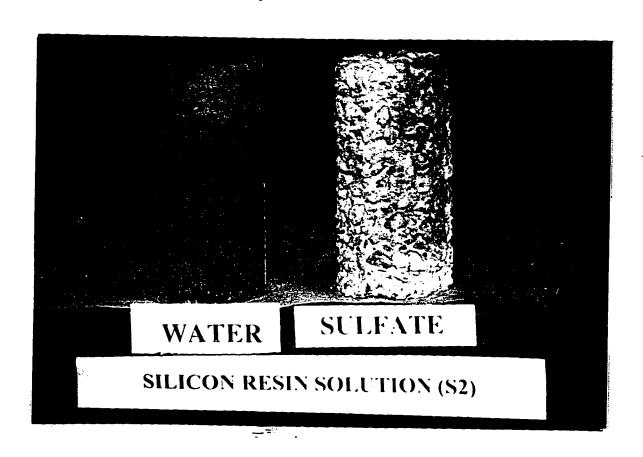


Plate 4.6.3: Concrete Specimens Coated with Silicone Resin Solution Immersed in the Sulfate Solution and Water for 330 days

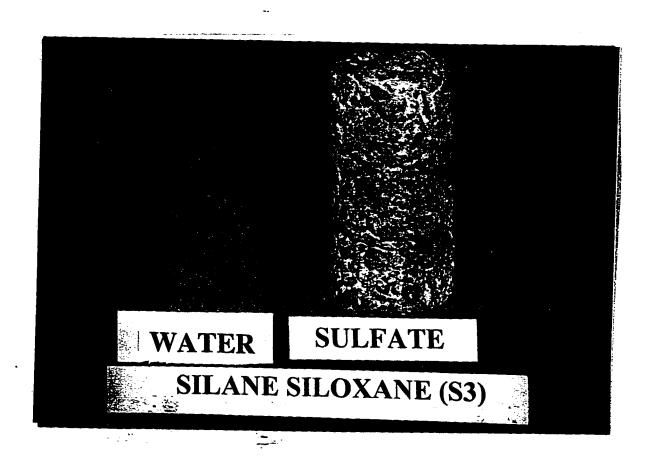


Plate 4.6.4: Concrete Specimens Coated with Silane/Siloxane Immersed in the Sulfate Solution and Water for 330 days

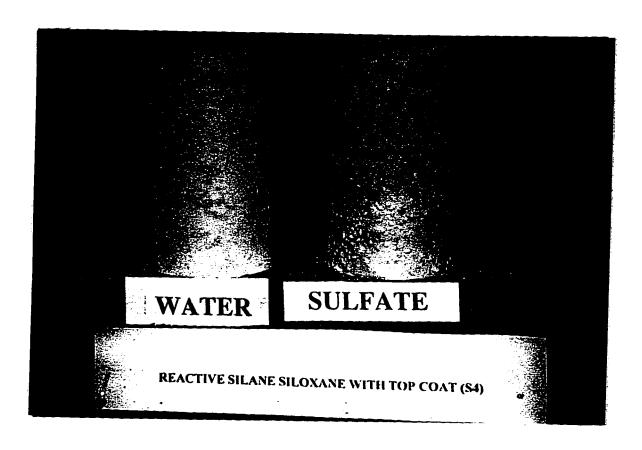


Plate 4.6.5: Concrete Specimens Coated with Silane/Siloxane with top Coat Immersed in the Sulfate Solution and Water for 330 days

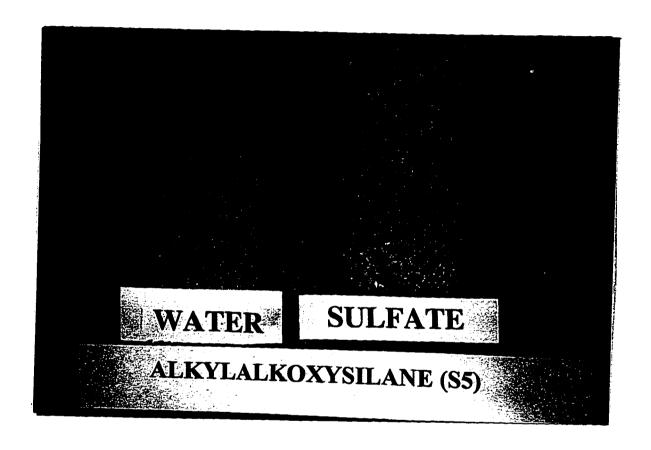


Plate 4.6.6: Concrete Specimens Coated with Silane Immersed in the Sulfate Solution and Water for 330 days



Plate 4.6.7: Concrete Specimens Coated with Acrylic Coating Immersed in the Sulfate Solution Water for 330 Days

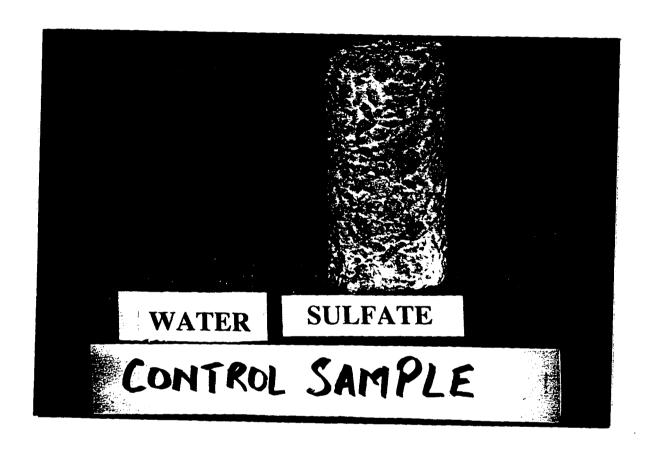


Plate 4.6.8: Uncoated Concrete Specimens Immersed in the Sulfate Solution and Water for 330 days

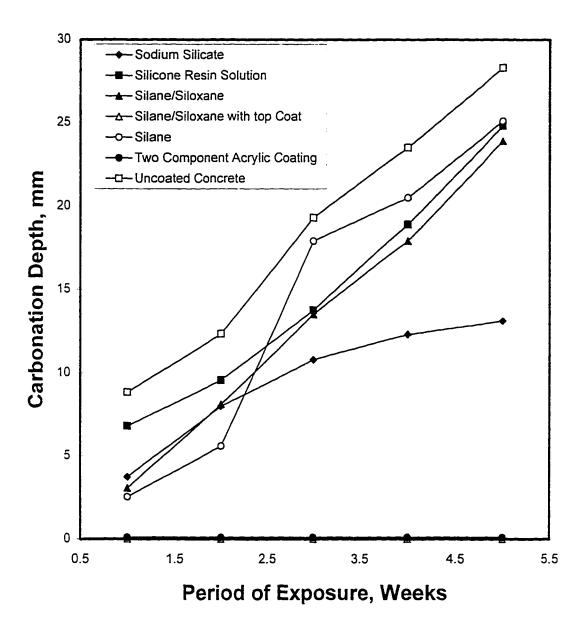


Figure 4.7.1 : Carbonation Depth in the Uncoated and Coated Concrete Specimens

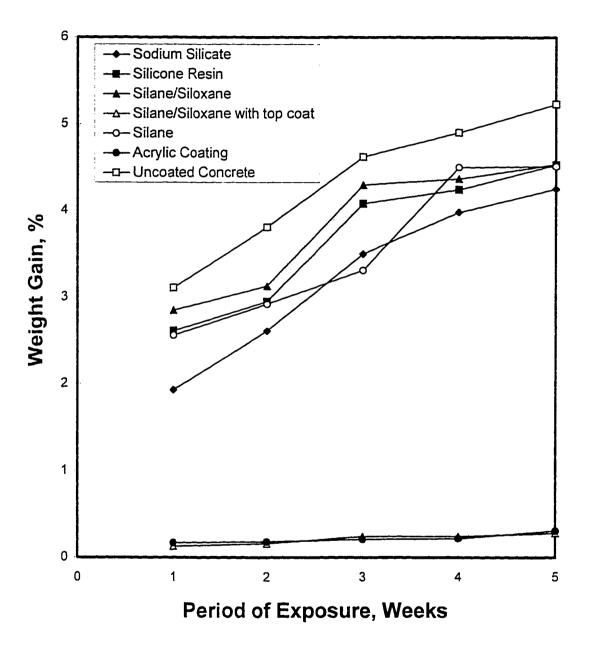


Figure 4.7.2 : Weight gain in the Coated and Uncoated Concrete Specimens



Plate 4.7.3: Carbonation in the Concrete Specimens Coated with Silane/Siloxane with Top Coat

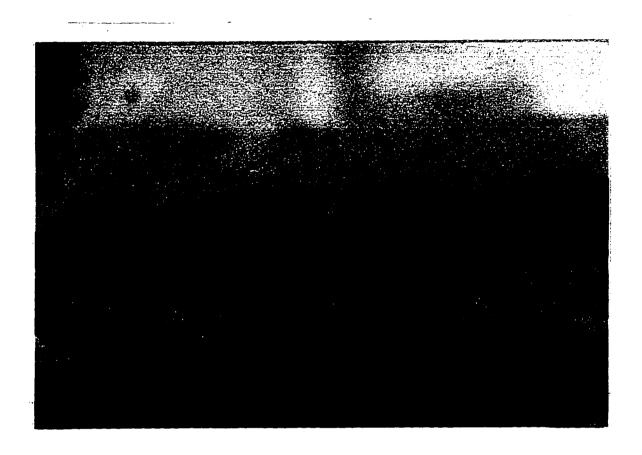


Plate 4.7.4: Carbonation in the Concrete Specimens Coated with Two Component Acrylic Coating

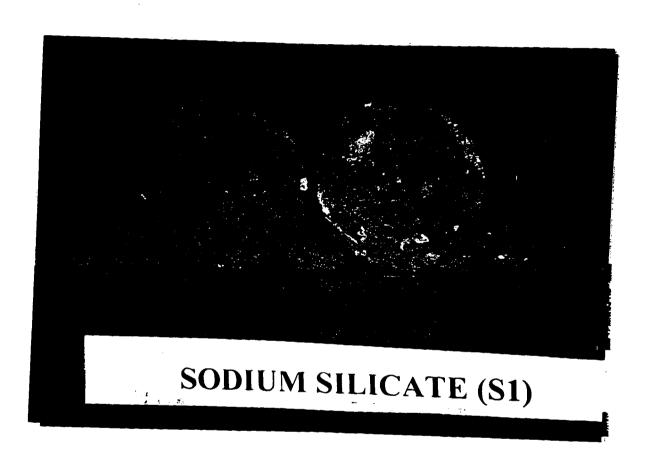
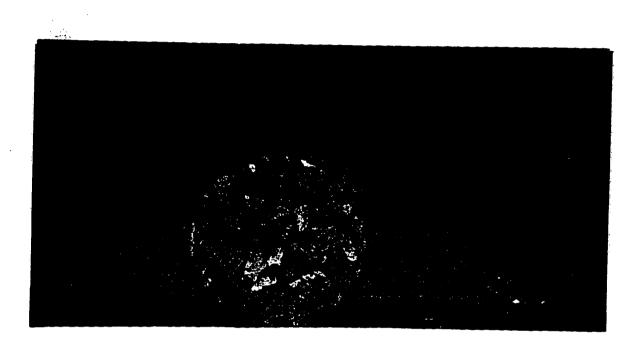
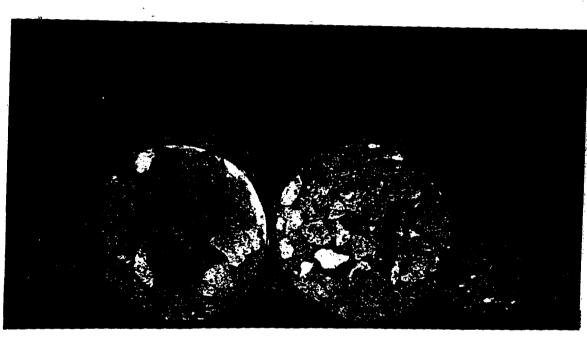


Plate 4.7.5: Carbonation in the concrete Specimens Coated with Sodium Silicate



SILICON RESIN SOLUTION (S2)

Plate 4.7.6: Carbonation in the Concrete Specimens Coated with Silicone Resin Solution



SILANE SILOXANE (S3)

Plate 4.7.7: Carbonation in the Concrete Specimens Coated with Silane/Siloxane

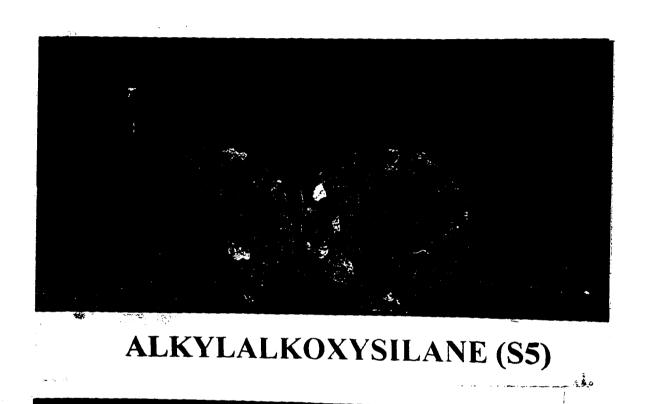


Plate 4.7.8: Carbonation in the Concrete Specimens Coated with Silane



CONTROL SAMPLE

Plate 4.7.9: Carbonation in the Uncoated Concrete Specimens

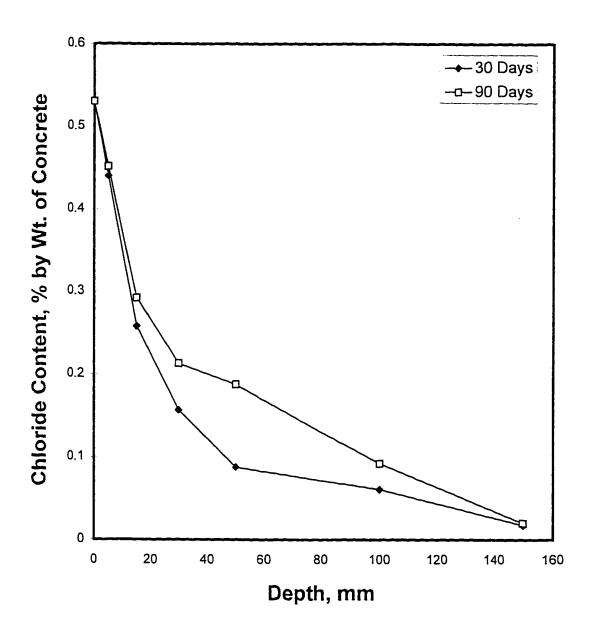


Figure 4.8.1 : Chloride Profile in the Concrete Specimens Coated with Sodium Silicate

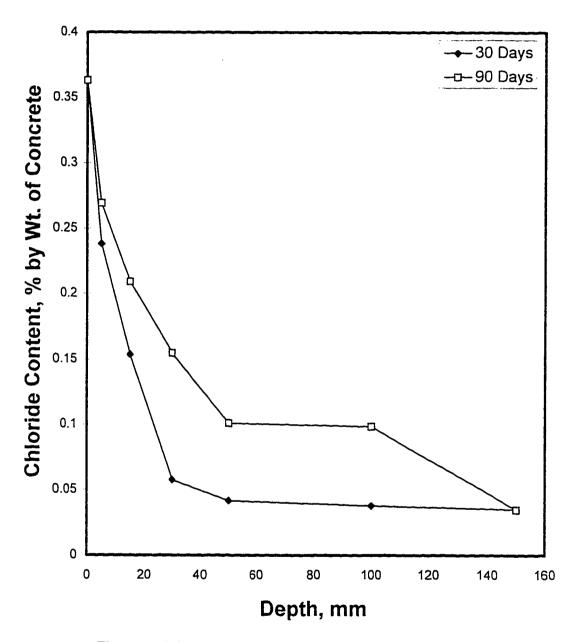


Figure 4.8.2 : Chloride Profile in the Concrete Specimens Coated with Silicone Resin Solution

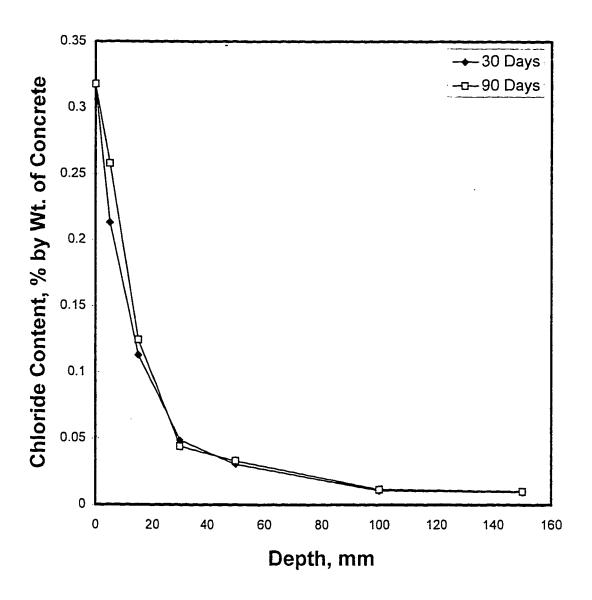


Figure 4.8.3 : Chloride Profile in the Concrete Specimens Coated with Silane/Siloxane

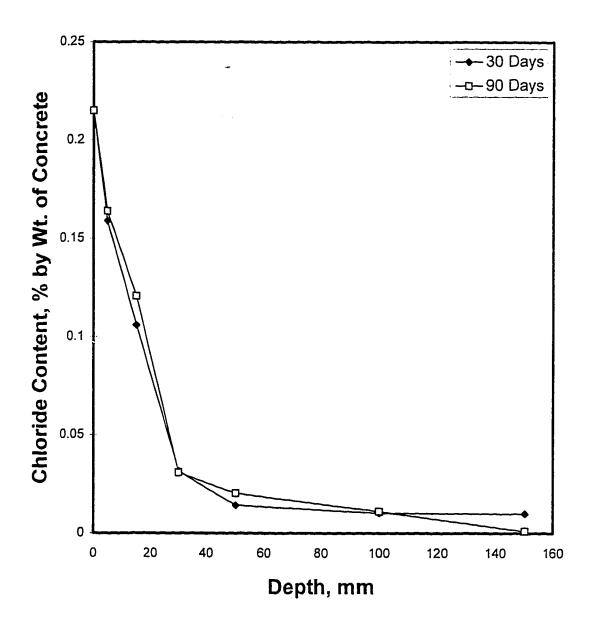


Figure 4.8.4 : Chloride Profile in the Concrete Specimens Coated with Silane/Siloxane with top coat

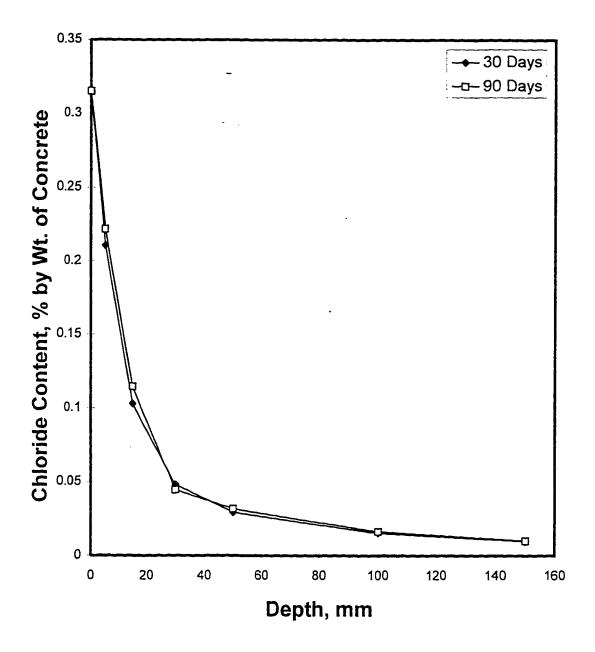


Figure 4.8.5 : Chloride Profile in the Concrete Specimens
Coated with Silane

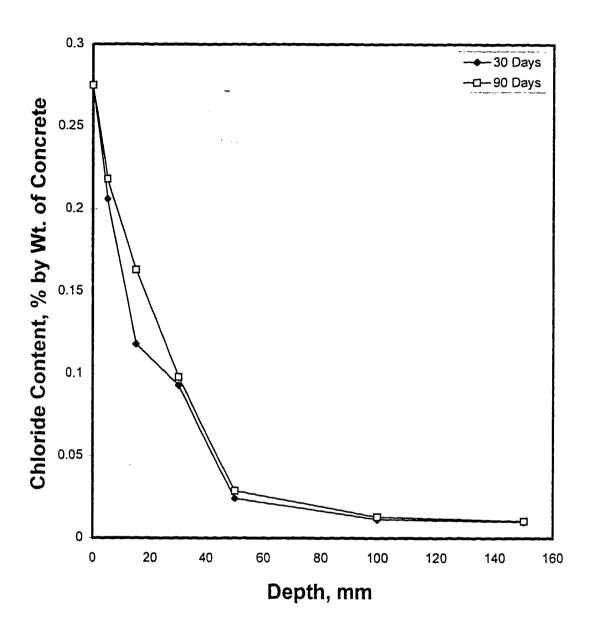


Figure 4.8.6 : Chloride Profile in the Concrete Specimens
Coated with Acrylic Coating

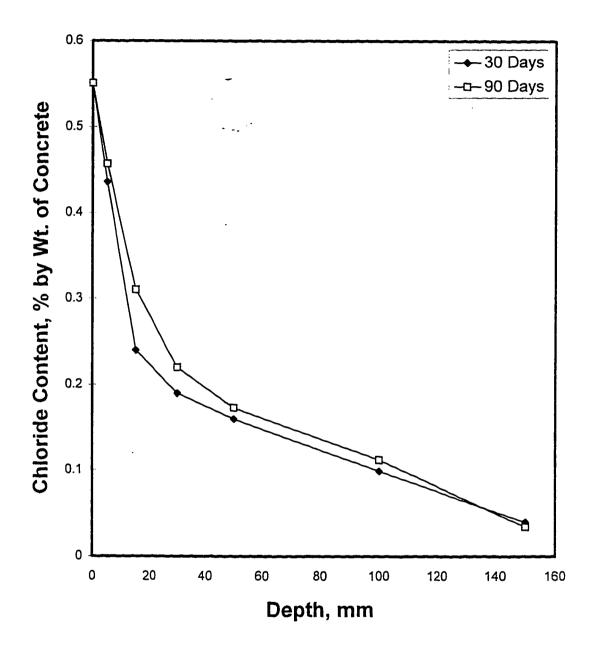


Figure 4.8.7 : Chloride Profile in the Uncoated Concrete Specimens

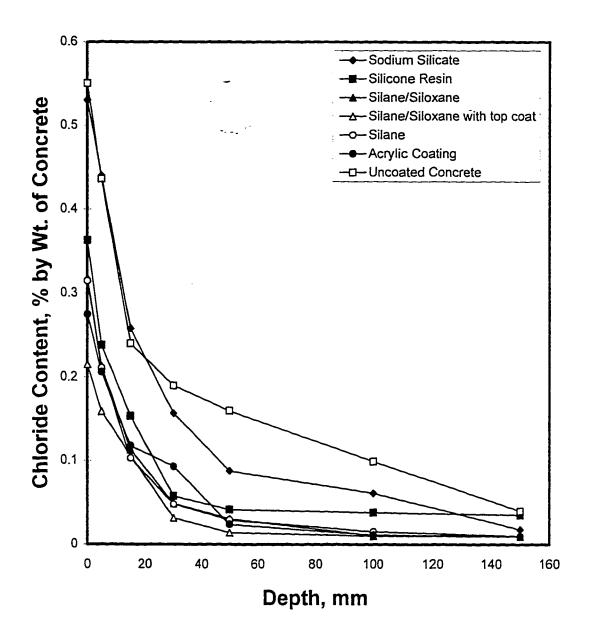


Figure 4.8.8 : Chloride Profiles of the Uncoated and Coated Concrete Specimens Exposed for 30 days

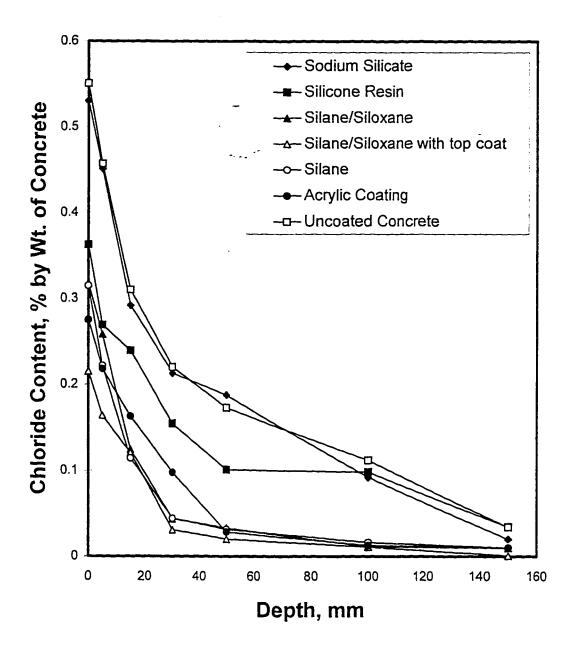


Figure 4.8.9 : Chloride Profile of Coated and Uncoated Concrete Specimens Exposed for 90 days

Table 4.8.1: Chloride Diffusion Coefficients in the Coated and Uncoated Concrete Specimens

Sealer/Coating	Diffusion Coefficient, cm²/sec
Sodium Silicate	20.06 x 10 ⁻⁸
Silicone Resin Solution	16.86 x 10 ⁻⁸
Silane/Siloxane	14.83 x 10 ⁻⁸
Silane/Siloxane with Topcoat	7.83 x 10 ⁻⁸
Silane	9.86 x 10 ⁻⁸
Acrylic Coating	8.18 x 10 ⁻⁸
Uncoated Concrete	21.83 x 10 ⁻⁸

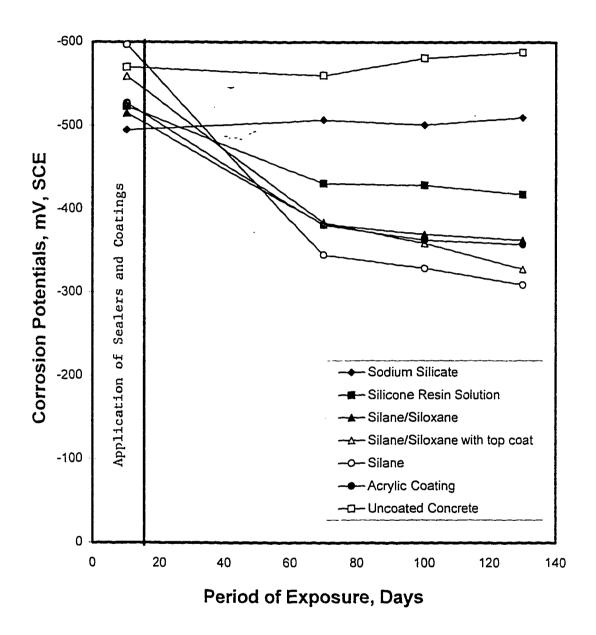


Figure 4.9.1: Variation of Corrosion Potentials in the Concrete Specimens before and after the Application of Sealers and Coatings

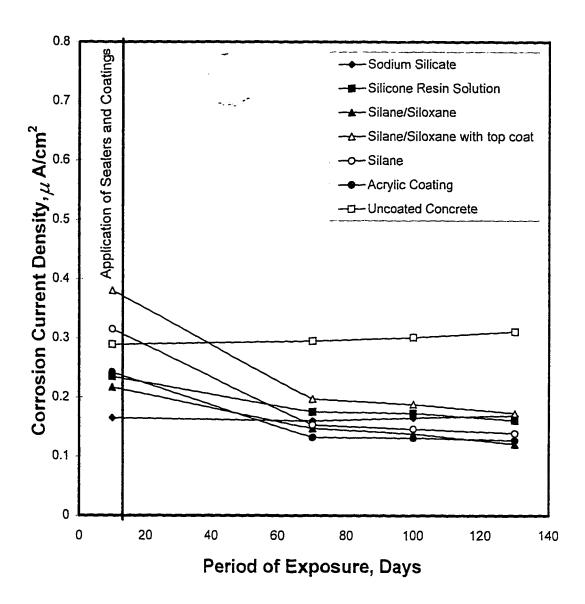


Figure 4.9.2: Variation of Corrosion Current Density on Steel in the Concrete Specimens before and after the Application of Sealers and Coatings

CHAPTER 5

DISCUSSION

5.1 EFFECT OF HEAT-COOL AND WET-DRY CYCLING ON THE WATER ABSORPTION AND COMPRESSIVE STRENGTH

The effect of heat-cool and wet-dry cycling on the water absorption and compressive strength of the uncoated and coated concrete specimens was evaluated over a period of 4 months. Tables 5.1.1 and 5.1.2 summarize the water absorption and reduction in compressive strength of specimens subjected to wet-dry and heat-cool treatment after 4 months. The increase in the water absorption with the increasing heat-cool and wet-dry cycles may be attributed to the formation of micro cracks within the concrete due to thermal and moisture variations.

Among all the surface treatment systems, silane, silane/siloxane with top coat and two component acrylic coating were the most effective in improving the properties of hardened concrete. The improved performance of these three surface treatment systems may be attributed to an effective barrier against the moisture

ingress into the concrete matrix. Another important conclusion from these results is that the effect of heat-cool and wet-dry cycling is minimal in the specimens coated with these specimens. The performance of concrete specimens coated with sodium silicate was not better than the uncoated concrete specimens. The probable explanation for the poor performance of concrete specimens coated with sodium silicate is that, it can react with calcium hydroxide present in the concrete to form secondary calcium silicate hydrate. This calcium silicate hydrate is not stable and leaches out of concrete in wet environments, there by increasing the permeability of the concrete. Among all the sealers and coatings investigated in this study, silane, silane/siloxane with top coat and acrylic coating were effective in minimizing the damage due to thermal and moisture variations.

5.2 REINFORCEMENT CORROSION

The effectiveness of surface treatment systems in reducing reinforcement corrosion was evaluated by measuring the corrosion potentials and corrosion current density on steel in the concrete specimens exposed to 5% NaCl solution. The time to cracking of concrete under an impressed potential of +2 V was evaluated. Table 5.2.1 summarizes the time to cracking of uncoated concrete specimens and those coated with sodium silicate, silicone resin solution, silane/siloxane, silane/siloxane with top coat and acrylic coating. Concrete

specimens coated with silane, silane/siloxane with top coat and two component acrylic coating did not crack at all, while the time to cracking in the specimens coated with other coatings was more than that in the uncoated concrete specimens. Table 5.2.2 shows the summary of corrosion current density on steel in the uncoated and coated concrete specimens, after about 360 days of exposure, to 5% NaCl solution. The data in Tables 5.2.1 and 5.2.2 indicate that, sodium silicate, silicon resin solution and silane/siloxane may prolong the time to initiation of reinforcement corrosion, but its rate may not be much different from the uncoated concrete specimens. The low corrosion activity in the concrete specimens coated with silane/siloxane with top coat, silane and acrylic coating is mainly because of reduced supply of oxygen and moisture content to concrete.

The results obtained from the accelerated reinforcement corrosion by impressing an anodic potential of 2 Volts and corrosion current density on steel in the reinforced concrete specimens exposed to 5% NaCl solution are consistent. This indicates that the impressed potential system can be used to quickly evaluate the performance of any material in reducing reinforcement corrosion.

Table 5.2.3 shows the average corrosion current in the uncracked concrete specimens after 4350 hours (180 days) of impressed potential. These specimens

were coated with silane, silane/siloxane with top coat and acrylic coating. The average current was well below the cracking current, which was around 0.7 mA. Low current requirement in the concrete specimens coated with silane/siloxane with top coat, silane and acrylic coating indicates that the cathodic reaction are not supported due to lack of oxygen. Among the penetrating sealers, silane was the most effective in reducing reinforcement corrosion. The probable explanation for this may be attributed to its enhanced penetration into the concrete matrix, thus inhibiting the ingress of oxygen and moisture.

5.3 EFFECTIVENESS OF SEALERS AND COATINGS IN REDUCING SULFATE ATTACK

Table 5.3.1 summarizes the reduction in compressive strength, in the coated and uncoated concrete specimens immersed in the sulfate solution for 330 days. The strength reduction in the specimens coated with silane, silane/siloxane, acrylic coating and silane/siloxane with top coat was less than the uncoated concrete specimens and those coated with sodium silicate and silicone resin solution. Minimum strength reduction was noted in the concrete specimens coated with silane/siloxane with top coat. The effective performance of silane/siloxane with top coat could be attributed to the fact that, top coat forms a layer over the concrete

and provide a barrier against the diffusion of sulfate ions. Further, silane/siloxane provide additional protection.

5.4 EFFECTIVENESS OF SURFACE TREATMENT SYSTEMS IN PREVENTING INGRESS OF CARBONDIOXIDE

Table 5.4.1 summerizes the depth of carbonation in the coated and uncoated concrete specimens, after 5 weeks of exposure to accelerated carbondioxide environment. No carbonation was detected in the specimens coated with acrylic coating and silane/siloxane with top coat even after about 210 days of exposure. All the penetrating sealers were ineffective in inhibiting the diffusion of carbondioxide. In all the specimens coated with the penetrating sealers, the carbonation depth was higher than the usual cover provided over the reinforcing steel, particularly in the slabs. This may reduce the pH of the pore solution and make the protective oxide layer unstable. However, the coatings investigated in this study, namely silane/siloxane with top coat and acrylic coating were effective in reducing the ingress of CO₂.

5.5 EFFECTIVENESS OF SURFACE COATINGS IN REDUCING DIFFUSION OF CHLORIDE IONS

Table 5.5.1 shows the chloride concentrations, in the coated and uncoated concrete specimens at 5 mm depth, exposed to a period of 90 days. The reduction in the chloride concentration in the specimens coated with silane, silane/siloxane with top coat and acrylic coating, as compared to the uncoted concrete specimens. may be attributed to reduced permeability of concrete. Furthermore, the chloride diffusion coefficient in the uncoated concrete specimens, made with a cement content of 350 kg/m³ and a w/c ratio of 0.45 w/c ratio is in agreement with the data reported previously [51].

5.6 EFFECTIVENESS OF SEALERS AND COATINGS IN REDUCING REINFORCEMENT CORROSION IN IN-SERVICE STRUCTURES

Table 5.6.1 shows the change in corrosion current density on steel in the coated and uncoated concrete specimens after 4 months of the application of surface treatment materials. The reduction in corrosion current density on steel in the concrete specimens coated with silane/siloxane, silane/siloxane with top coat.

silane and acrylic coating was 44, 54, 55 and 47%, respectively. The corrosion current density on steel in the concrete specimens coated with the silicone resin solution reduced to a lesser degree, while an insignificant change was observed in the uncoated specimens and those coated with sodium silicate.

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The reduction in the corrosion current density on steel, in most of the coated concrete specimens, may be due to reduction in the oxygen content at steel-concrete interface. Therefore, a reduction in corrosion current density on steel in the concrete specimens coated with silane, silane/siloxane with top coat and acrylic coating indicates that, these surface treatment materials can be used to rehabilitate corroding structures.

5.7 COMPARISON AND EVALUATION OF DIFFERENT SURFACE TREATMENT SYSTEMS

The basic purpose of using surface treatment materials was to prevent rapid deterioration of concrete structures by retarding penetration of water, chloride ions. sulfate ions, oxygen and carbondioxide. The experimental program was designed to extensively test these surface treatment materials in all aspects of durability of concrete. It has been established that there is no ideal sealer which can completely protect the concrete from deterioration. However, some existing sealers can protect concrete from deterioration exposed to a particular environment. The following discussion focuses on the merits and disadvantages of the sealers tested in this study.

5.7.1 Sodium Silicate

Among all the surface treatment systems used in this study, the performance of sodium silicate treated concrete was significantly inferior. According to all the experimental results, except carbonation depth, the performance of sodium silicate was even worse than the uncoated samples. It was partly effective in reducing carbonation of concrete. The poor performance of sodium silicate may be

attributed to the fact that it is readily soluble in water, thereby its leaching in wet environment is possible.

5.7.2 Silicon Resin Solution

The performance of silicon resin solution was also not good and it failed to reduce the deterioration of concrete. However, it performed slightly better than sodium silicate. The penetration depth of silicon resin solution was very less because of the fact that these materials are much higher in molecular weight than silanes and siloxanes. Normally, these materials are not reactive and dry by solvent evaporation to leave a surface film of resin. Therefore, it is not advisable to use silicon resin solution to protect concrete from deterioration for long period of time.

5.7.3 Silane/Siloxane

Siloxanes are more correctly described as oligomeric alkyl alkoxy siloxanes. The performance of silane/siloxane treated concrete in this study was moderate. According to all the experimental results, silane/siloxanes were effective in reducing the deterioration of concrete only for a short period of time. The

siloxanes have all the advantages of silane with respect to reactivity and water repellency, but their large size makes them less effective in penetrating concrete.

This may be the factor which affected the performance of silane/siloxane treated concrete.

5.7.4 Silane

Silane is more commonly known as alkyl alkoxy silane. Among all the penetrating sealers used in this study, the performance of silane treated concrete was significantly superior. However, it did not performed satisfactorily in the sulfate and Co₂ environment. Silane contains relatively smaller size molecules dissolved in a volatile solvent. When applied to concrete, silane penetrates and obtains its water repellent characteristics from two stage chemical reaction. Most of the sealers available in the market protect the concrete by filling the capillary pores thereby decreasing the permeability of concrete. Ludwig and Sideris [54] have determined the reaction of a particular alkyl alkoxy silane as follows:

Hydrolysis in an acid or alkaline medium: (1)

$$OC_2H_5$$
 OH OC_2H_5 + $3H_2O$ CH₃ - SI OH + $3C_2H_5OH$ OH OC_2H_5 OH (methyl-triethoxy silane) (methyl-silanol) (ethanol)

Reaction with Substrate Surface: (2)

(substrate surface) (bonding of silanol group onto substrate surface)

As seen in the above equations, the reaction products form a water repellent surface. Consequently, the absorptivity of concrete treated with silane is markedly reduced.

5.7.5 Silane/Siloxane with acrylic top coat

Among the two coating materials used in this study, the performance of silane/siloxane with top coat was better than two component acrylic coating. The specimens coated with silane/siloxane with top coat performed well in the impressed current testing, the current being 0.002 mA. It is clearly evident from

the experimental data that, in all aspects of durability of concrete silane/siloxane. with top coat performed well and it was effective in reducing deterioration of concrete for a considerable span of time. But one of the disadvantages was that, at high temperatures air bubbles appeared within the top coat and it increased the water absorption. Silane/siloxane with acrylic top coat, is a combination system. formulated to provide maximum protection possible to concrete structures. According to the results obtained in this study, this system can provide excellent chloride-resistance and inhibit diffusion of CO₂ gas. The specimens coated with this combination system, completely prevented the carbonation process and the reduction in the compressive strength, was the lowest compared to other coated specimens when exposed to sulfate-bearing environment. The better performance of silane/siloxane with top coat may be attributed to its low viscosity and volatility due to which it penetrates the substrate and gives the system its resistance to aggressive species, such as water and water soluble salts. The acrylic top coat forms a protective layer over the surface of concrete and gives the system its resistance to aggressive species including moisture, oxygen and carbondioxide.

5.7.6 Two Component Acrylic Coating

The effectiveness of two component acrylic coating in preventing deterioration of concrete was acceptable, but not as good as silane/siloxane with top coat. Unlike silane/siloxane with top coat, the two component acrylic coating did not perform well in the sulfate-bearing environment. In other aspects of durability of concrete, such as reinforcement corrosion, water absorption, resistance to thermal and moisture variations, carbonation and chloride diffusion, its performance was satisfactory. The two component acrylic coating retards the deterioration of concrete by blocking the pores and forming a thick layer of coating on the surface of concrete.

5.8 ECONOMICS

The durability performance of concrete can be improved by the following approaches:

- 1) Using High Quality Concrete,
- 2) Applying Protective Concrete Surface Coatings,
- 3) Using Fusion Bonded Epoxy Coated Reinforcement,
- 4) Applying Cathodic Protection and
- 5) Using Special Concrete Admixtures.

Although, every method has its own advantages and disadvantages, the use of protective concrete surface coatings can be applied to old and new concrete. The selection of a particular method or combination mostly depends on several factors including cost ease of application, effectiveness in retarding deterioration of concrete and maintenance.

One of the main difficulties with the concrete surface coatings is that, with the wide range of such coatings available in the market, it becomes extremely difficult to choose the right type of material for a particular exposure condition. The choice and the selection of the surface treatment system is most important and depends on the specific requirements of the structure. There are several factors to be considered before selecting a particular surface treatment material.

They are:

- a) Cost,
- b) Carbondioxide diffusion resistance,
- c) Chloride ion ingress,
- d) Water vapor diffusion resistance,
- e) Ultra violet light resistance,
- f) Crack bridging ability,

- g) Chemical resistance,
- h) Abrasion resistance,
- I) Ease of application and
- j) Aesthetic appearance.

The total cost for using these materials generally includes, material, cost of the surface preparation, and application. Table 5.6.1, shows the cost of the materials used in this study. The cost of surface preparation and application are not included.

Although, most of the materials used in this study, were not effective in all aspects of durability of concrete, some of them can be specified to suite a particular situation. Among all the penetrating sealers, silane proved to be the most effective and can be used in many cases, except in situations where the environment is saturated with sulfate-salts and carbondioxide. Silane/siloxane with top coat, was also effective and can be used virtually in all the situations. The cost of silane/siloxane with top coat, is slightly above the cost of other materials. However, the advantages gained may be many.

Table 5.1.1: Summary of Water Absorption in the Uncoated and Coated Concrete Specimens Subjected to Wet-Dry and Heat-Cool Treatment after 4 months of Exposure

	WATER ABSORPTION, %	
SEALER/COATING	WET-DRY	HEAT-COOL
Sodium Silicate	5.133	5.891
Silicone Resin Solution	2.68	2.506
Silane/Siloxane	2.723	1.781
Silane/Siloxane with Topcoat	0.663	1.68
Silane	1.12	1.75
Acrylic Coating	0.338	1.239
Uncoated Concrete	5.04	5.51

Table 5.1.2: Reduction in Compressive Strength in the Uncoated and Coated Concrete Specimens Subjected to Wet-Dry and Heat-Cool Treatment after 4 months of Exposure

	REDUCTION IN COMPRESSIVE STRENGTH, %	
SEALER/COATING	WET-DRY	HEAT-COOL
Sodium Silicate	12.3	16.36
Silicone Resin Solution	10.13	13.76
Silane/Siloxane	8.23	8.73
Silane/Siloxane with Topcoat	6.46	8.49
Silane	6.86	6.81
Acrylic Coating	5.78	7.81
Uncoated Concrete	12.78	17.63

Table 5.2.1: Summary of Time to Cracking of Concrete Specimens Subjected to a Impressed Anodic Potential of 2 Volts.

Sealer/Coating	Time to Cracking, Hours
Control Specimen	144
Sodium Silicate	176
Silicone Resin Solution	672
Silane/Siloxane	2125
Silane/Siloxane with Topcoat	No Cracks
Silane	No Cracks
Acrylic Coating	No Cracks

Table 5.2.2: Summary of Corrosion Current Density on steel in the Coated and Uncoated Concrete Specimens after 360 days of exposure to 5% NaCl Solution for 360 days.

Sealer/Coating	CORROSION CURRENT DENSITY,		
-	μ A/cm²		
Uncoated Concrete	0.328		
Sodium Silicate	0.286		
Silicone Resin Solution	0.31		
Silane/Siloxane	0.193		
Silane/Siloxane with Topcoat	0.0058		
Silane	0.0061		
Acrylic Coating	0.0108		

Table 5.2.3 Average Current in the Uncracked Coated Concrete Specimens

Sealer/Coating	CORROSION CURRENT, mA
Silane .	0.095
Silane/Siloxane with Topcoat	0.0023
Acrylic Coating	0.0396

Table 5.3.1: Summary of Reduction in Compressive Strength in the Uncoated and Coated Concrete Specimens Immersed in the Sulfate Solution for 330 days.

Sealer/Coating	REDUCTION IN COMPRESSIVE STRENGTH, %
Uncoated Concrete	41
Sodium Silicate	39.3
Silicone Resin Solution	36.1
Silane/Siloxane	26.8
Silane/Siloxane with Topcoat	8.3
Silane	24.3
Acrylic Coating	19.63

Table 5.4.1: Summary of Carbonation Depth in the Coated and Uncoated Concrete Specimens after 5 Weeks of Exposure

Sealer/Coating	CARBONATION DEPTH, mm
Uncoated Concrete	28.3
Sodium Silicate	13.1
Silicone Resin Solution	24.8
Silane/Siloxane	23.9
Silane/Siloxane with Topcoat	0
Silane	25.1
Acrylic Coating	0

Table 5.5.1: Chloride Concentration in the Coated and Uncoated Concrete Specimens, at 5 mm Depth, Exposed to a Period of 90 days

Sealer/Coating	CHLORIDE CONCENTRATION, %
Uncoated Concrete	0.457
Sodium Silicate	0.451
Silicone Resin Solution	0.269
Silane/Siloxane	0.258
Silane/Siloxane with Topcoat	0.164
Silane	0.2216
Acrylic Coating	0.218

Table 5.6.1: Change in corrosion current density after 4 months of application of sealers and coatings

			
	Corrosion Current Density, µA/cm²		
TYPE OF COATING	Before Application	After 4 Months of Application	Change in Corrosion Rate*, %
Uncoated Concrete	0.289	0.311	+7
Sodium Silicate	0.165	0.169	+2.4
Silicone Resin Solution	0.235	0.161	-31
Silane/Siloxane	0.217	0.121	-44
Silane/Siloxane with Topcoat	0.38	0.173	-54
Silane	0.315	0.139	-55
Acrylic Coating	0.242	0.127	-47

⁺ Indicates Increase in Corrosion Current Density
- Indicates Reduction in Corrosion Current Density

Table 5.7.1: Material Costs of Sealers and Coatings Used in this Study

Sealer/Coating	MATERIAL COST, SR/m ²
Sodium Silicate	7
Silicone Resin Solution	6
Silane/Siloxane	8
Silane/Siloxane with Topcoat	14
Silane	9
Acrylic Coating	18

CHAPTER 6

CONCLUSIONS & RECOMMENDATIONS

The effectiveness of surface treatment materials in reducing deterioration of concrete and corrosion of embedded reinforcement was evaluated. The effect of concrete surface treatment systems on the compressive strength, water absorption. initiation of reinforcement corrosion, cracking due to corrosion and depth of carbonation were investigated. The performance of these surface treatment materials were evaluated under heat-cool, wet-dry, high chloride, high sulfate and carbonation environments. The effectiveness of these materials in reducing reinforcement corrosion was evaluated by impressing an anodic potential of 2V and measuring the time to cracking of concrete specimens. The performance of these materials, in reducing corrosion activity, in chloride-environment was evaluated by immersing them in 5% NaCl solution, and monitoring the corrosion potentials and corrosion current density at periodic intervals. Also, the chloride diffusion characteristics of concrete specimens coated with these surface treatment materials was evaluated.

From the data developed in this study, the following conclusions can be drawn:

- 1. The water absorption in the concrete specimens coated with sodium silicate was 10% more than that in the uncoated concrete specimens. There was 42 and 51% reduction in water absorption was observed in the concrete specimens coated with silicone resin solution and silane/siloxane, as compared to the uncoated concrete specimens. Among all the penetrating sealers, silane was the most effective in reducing water absorption in concrete and there was 90% reduction in water absorption was indicated in these specimens, as compared to the uncoated concrete specimens. The water absorption in the concrete specimens coated with silane/siloxane with top coat and two component acrylic coating was only 0.67 and 0.29%, respectively, which indicates 83 and 93% reduction, as compared to the uncoated concrete specimens.
- 2. The water absorption in the uncoated and coated concrete specimens continuously increased from 10 to 40 wet/dry cycles. The performance of uncoated concrete specimens and those coated with sodium silicate was similar. After 40 wet/dry cycles, the water absorption in the concrete specimens coated with sodium silicate was the highest among all the other treated specimens. The best performance was indicated by silane, silane/siloxane with

top coat and two component acrylic coating. The performance of concrete specimens coated with silicone resin and silane/siloxane was average.

- 3. A continuous increase in the water absorption and decrease in the compressive strength was noted in the coated and uncoated concrete specimens subjected to heat-cool cycling. The water absorption in the uncoated and coated concrete specimens subjected to heat-cool cycling was higher than that in the specimens subjected to wet-dry cycling. In this test also, the performance of uncoated concrete specimens and those coated with sodium silicate was similar. Sodium silicate and silicone resin solution were not effective in minimizing damage due to continuous heating and cooling. Among the coating materials, acrylic coating performed better. Silane and silane/siloxane, proved to be more effective, among all the penetrating sealers used in this study. Silane/siloxane with top coat, performed well and was also effective in minimizing the damage caused by heat-cool cycling.
- 4. The uncoated concrete specimens cracked within 144 hours of testing when an impressed potential of +2V was applied. The performance of sodium silicate was not satisfactory and the concrete specimens coated with this material cracked after about 172 hours of exposure. The performance of concrete

specimens coated with silicone resin solution was average. Silane/siloxane, a penetrating sealer was effective in reducing reinforcement corrosion for long period of time and the concrete specimens coated with this coating cracked after 2125 hours. In all the uncoated and coated concrete specimens, cracking occurred only when the current exceeded 0.7 mA. Among all the penetrating sealers, the performance of silane was the best and the concrete specimens coated with this material did not crack after 4350 hours of exposure. The average current in the specimens coated with silane was around 0.095 mA after about 4350 hours of testing. Silane/siloxane with top coat and two component acrylic coatings were the most effective in preventing reinforcement corrosion and the average current was around 0.004 and 0.04 mA, respectively.

5. The corrosion potentials on steels in the concrete specimens coated with silane/siloxane with top coat, silane and two component acrylic coating were less as compared to the threshold potential, even after 360 days of exposure. Sodium silicate, silicone resin solution and silane/siloxane were not effective in delaying the time to initiation of reinforcement corrosion. The behavior of uncoated concrete specimens and those coated with sodium silicate was similar.

- 6. The penetrating sealers investigated in this study were not effective in reducing the deterioration of concrete due to sulfate attack. However, silane/siloxane and silane were partly effective in resisting sulfate attack. Among these two coatings, silane/siloxane with top coat was the most effective in reducing the sulfate attack. The reduction in the compressive strength, due to sulfate attack. for the concrete specimens coated with silane/siloxane with top coat was 8.3%, whereas it was 41% in the uncoated concrete specimens. Two component acrylic coating also performed satisfactorily upto 8 months of exposure.
- 7. None of the penetrating sealers studied were totally effective in preventing carbonation of concrete. However, silane/siloxane with top coat and acrylic coating performed better than other sealers and coatings. The depth of carbonation in these concrete specimens was very minimal even after 6 months of continuous storage in the carbonation chamber. The depth of carbonation in the concrete specimens coated with sodium silicate was 13.1 mm as against 28.3 mm measured in the uncoated concrete specimens. After 5 weeks of exposure, the carbonation depth in the concrete specimens coated with silicone resin solution, silane/siloxane and silane was 24.8, 23.9 and 25.1 mm. respectively.

- 8. The chloride concentration and diffusion coefficients were found to be strongly influenced by the type of coating. The performance of concrete specimens coated with silane, silane/siloxane with top coat and acrylic coating was improved with reduction in water permeability. The rest of the coating materials were not effective in retarding the diffusion of chloride ions.
- 9. Silane, silane/siloxane with top coat and acrylic coating were most effective in reducing reinforcement corrosion in in-service structures. The highest reduction in corrosion current density on steel, was observed in the concrete specimens coated with silane.

In general, the performance of sealers and coatings investigated in this study is of the following order:

Silane/Siloxane with top coat > Acrylic coating > Silane > Silane/Siloxane > Silicone Resin solution > Sodium Silicate

The preferred sealers/coatings for various environments are summerized below:

Table 6.1: Preferred sealers and coatings to suite environmental conditions

Exposure Conditions	Preferred Sealers/Coatings
Wet-Dry	Silane/Siloxane with top coat, Acrylic Coating, Silane
Heat-Cool	Silane/Siloxane with top coat, Acrylic Coating, Silane
CO ₂	Silane/Siloxane with top coat, Acrylic Coating, Sodium Silicate
Sulfate	Silane/Siloxane with top coat
Chloride	Silane/Siloxane with top coat, Silane, Acrylic coating

Based on this study and previous conclusions, the following recommendations, pertaining to the future study, can be made:

- 1. The performance of sealers and coatings in actual service environment has to be studied.
- 2. The effectiveness of sealers and coatings in combination with the blended cements can be investigated.
- 3. Also, the effectiveness of sealers and coatings exposed to UV radiation should be elucidated.

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