The Performance of Crystalline Hydrophobic in Wet Concrete Protection

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Abstract

Reflecting the need to protect concrete structures from de-icing salt and freeze-thaw loading, the study introduced in this paper springs from the uncertainty that exists in the benefit of in-situ performance Iso-butyl silane as a protection material. It is likely that environmental loading and internal moisture at the time of application are the main contributory factors for under performance. This paper deals with alternative materials, a high solids silane and an aqueous crystallization solution, operating moisture driven crystallization mechanism rather than demanding a dry application regime. The results demonstrated similar substantial reducing performance of both materials at 0-5% moisture on medium (C25:25 N/mm^2) and high strength (C40:40N/mm^2) concrete. There is greater take-up of protective materials for C25 concrete compared with C40 concrete, together with greater chloride reduction, indicating that the level of achieved dosing is a significant factor. The similarity between the absorption of water and the two protection materials relative to initial water content, points to a possible basis for predicting achievable dossing of surface applied protection materials. The crystallization material achieved greater application volume and chloride reduction than the silane material.

Key words: crystallization hydrophobic**,** impregnation, concrete protection, salt ponding, chloride content, durability, concrete bridge.

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INTRODUCTION

The UK has more than 61,000 highway and local road bridges, the majority reinforced concrete, with an annual maintenance spend of £4bn in 2012-2013, for example [DfT, 2014]. In addition, Great Britain has been recorded with the highest use of winter de-icing salts on bridges and highway structures in Europe [EC, 2002]. Whereas these are directly applied to structures, there is evidence of a secondary dossing effect at substantial elevations and distances from the dossing point [Houska, 2007]. This widens out the issue of concrete vulnerability. The average rainfall in Great Britain is also one of the highest in Europe, recorded at 1120 mm in 2014 [World Bank, 2015]. Furthermore, the correspondence between zones with prevailing wet and winter freezing conditions is strong, the West Highlands of Scotland being one example. These climatic conditions point to two important considerations in concrete protection, one obvious, the need for protection and one less well understood, the persistence of inappropriate conditions for the application of in-situ concrete impregnation treatments. Manufacturers of surface applied concrete protection products commonly stated low moisture content as a pre-requisite for application of their concrete protection products.

PROTECTIVE TREATMENTS

Reflecting the need to protect concrete structures from de-icing salt and freeze-thaw loading, the study introduced in this paper springs from the uncertainty that exists in the benefit of in-situ protection. The possibility of a wide discrepancy must be accepted between specified laboratory testing regimes for protective treatments and the onsite climatic environment that detracts from their intended performance. There is also a need to avoid the singular goal of deep penetration of protective material in concrete; lack of achievement of such being periodically reminded in published research. Reflecting climatic influences, the intention of the ongoing study is to investigate the performance of a range of material type surface applied protective treatments.

In the UK, based on observations on the reduction of half-cell potential values following applications of iso-butyl silane, this treatment material was specified for concrete repairs [Highways England, 2013]. This data was enabled from prolonged inspection of two Scottish bridges, commencing in 1986 [Brown, 1990]. It is interesting to note that based on extracted cores, site applications of silane was found to have very little surface penetration, limited to the depth of surface laitance. At the same time, water absorption was substantially reduced. Thus, it was observed, “Silane treatment was thus confirmed as a water repellent rather than a pore blocker [Brown, 1990].”

On the strength of laboratory testing for a durable (15 years) vapour permeable, water repellent surface layer, confidence grew for the application of high purity monomericalkyl (isobutyl) – trialkoxy silane, to the extent that this became specified for hydrophobic impregnation of concrete bridges. At the same time, it was recognised that other chemistries would arise and be accepted if they were compliant with a test method specified by the UK Transport Research Laboratory (TRL) [Highways England, 2013]. It is noted that the identical testing method was subsequently incorporated into the normalized European Standard covering pore-lining impregnation of concrete [BSI, 2004].

Acceptance that alternative materials should be considered, opened the gate for a range of alternatives including water based silanes, silane creams, aqueous fluoropolymer and crystallisation solutions. In the UK, their use has been granted by Highway Authorities under 'Departure from Standards', on a case by case basis. Reduced environmental impact is one of the acceptance criteria in this, which favour acceptance of the alternatives over silanes [Atkins, 2011]. Several different types of sealers have been used to protect concrete, silanes; silicates and siloxanes, epoxy, gum resins and minerals and linseed oil, stated in what is thought to be the order of decreasing effectiveness [NCHRP, 2004]. Other recent development on concrete impregnation include super hydrophobic material by incorporating waste material like waste paper sludge [Wong et al, 2015] and silica fume [Flores-Vivian et al, 2013]. Both developments highlighted limited success of improving hydrophobicity of concrete. Researchers have also reported water-resisting admixture as an integral part of concrete, but the degree of improvement in concrete protection is still unclear [Concrete Society, 2013].

Although tests have concluded that silane achieves little penetration [Wittmann et al, 2001], it has been suggested that greater than 10 mm penetration is possible using a pure high solid content silane. Such material has been tested and class II penetration achieved [Guar, 2007]. However, in the supporting testing [Syed and Donadio, 2013], it is important to note that a concrete mix water/cement ratio of 0.7 was used, which is too high to be associated with structural concrete (more typically w/c at 0.55 with a cube strength exceeding 40N/mm^2), rather pedestrian pavement construction with a 28-day cube strength in the region of 25 N/mm^2. It is reasonable to observe that such low strength, general purpose concrete is likely to favour absorption and penetration of the silane to a greater extent than achievable with structural concrete.

For the UK, a combined laboratory and treated structure assessment study was undertaken for the performance of a range of surface treatment, with the intention of facilitating the acceptance of alternative concrete impregnation materials other than monomeric alkyl (isobutyl) – trialkoxy silane as specified in the Highways Standard [Calder and McKenzie, 2008]. This would intentionally avoid the need for the previously mentioned 'Departure from Standards'. Whilst laboratory testing indicated benefits with some of the material applications, it was concluded that treated structures did not show such benefit. This influential document has since contributed to a relaxation in the requirement for hydrophobic impregnation of structures.

CONCRETE PROTECTION STUDY

Recognising the demanding climate environment for many structures in the UK and the uncertain benefit with current protective treatments, a research program is underway in the Division of Civil Engineering, Brunel University, UK to determine an effective approach to concrete protection. This takes on board the reality of site based protective material application, where moisture content levels can persist unavoidably high. Whilst laboratory based compliance testing under EN1504-2 is maintained as one performance criterion, it is irrelevant to such site application on account of excessive moisture content in the concrete. However, this test does work to safeguard the essential requirement for post-treatment vapour permeability. The need to combat chloride ion penetration and freeze-thaw damage with a durable treatment is upheld, whilst insistence on a deep penetration effect is relaxed in this work. The industry preferences for low hazard material applied as a single stage, early application is respected. To widen the potential benefit from the study, the effect of surface treatments on early thermal cracking are being investigated, respecting the concrete curing requirement. Reflecting the reality of high moisture levels in operational structural concrete, the possibility of a water reacted protective materials is under consideration, including crystallisation materials. Although there is natural preference for treatments that do not alter the visual aspects of the concrete surface, the possibility of an effective high-build protective coating is upheld in the study.

OBJECTIVES OF STUDY

The objectives of this study are:

1. To determine the effect of initial moisture content on take-up of surface applied protection material in concrete.
2. To determine the effect of initial moisture content on depth of penetration of surface applied protection materials.
3. To determine the effect of initial moisture content on chlorine ion penetration in concrete treated with surface applied protective materials.

The pre-occupation with initial moisture content in the above objectives stems from on-site experience in the application of impregnation materials to numerous concrete bridges. Testing reported in this paper supports the significance of this. Currently, whilst protection material manufacturers specify maximum water content in the concrete to be treated, there is currently no accurate site based method for confirming such. Whilst a prior 24 hour drying period may be specified, a long proceeding period of concrete saturation and prevailing high humidly at application time is possible. To help ensure worthwhile concrete protection from surface applied materials, the authors feel it is important to develop objective measurement methods, taking advantage of relevant sensing technology.

In previously reported work [Rahman et al 2013, Whiting et al 1992], pure silane (85% active content), aqueous acrylic, aqueous silane (40%) and aqueous silicate were tested, all found to reduce surface absorption rather than prevent chlorides penetration. A useful outcome during this investigation was the finding that 100 mm cubes could be used for chlorine ponding testing rather than the large test specimens previously specified for such testing [Calder and Choudhury, 1996].

The first category of alternative (to silane/siloxane) concrete protection materials under consideration are aqueous crystallisation solutions. The material patent claims that the mixture preferably works from within the concrete as well as at the surface. A water-repelling function prevents water from penetrating the concrete matrix. A hygroscopic and hydrophilic behavior of its crystallization system within a concrete matrix minimizes moisture transmission through capillaries and connected voids. As a result, the mixture promises to provide a permanent treatment for moisture related problems, such as damage caused by repeated freeze and thaw cycles and chloride ion penetration as from de-icing salts, as well as a permanent treatment for the so-called alkali-silica reactions [International Chem-Crete Corporation, 2008].

EXPERIMENTAL PROGRAM

The work reported investigates the effect of moisture content on the application of two of many available surface applied protection materials for concrete. Specifically, the study is concerned with the way that moisture content affects the achievable dossing of such materials. In the second part, the performance of these achieved dosages of protective materials, influenced by moisture content, is investigated in respect to protection against harmful chloride loading. Further publications are anticipated dealing with different types of protective materials and approaches to prediction of treatment performance with respect to achieved dosing.

Work for objectives 1 & 2: absorption issue

Materials

Whilst work is continuing with further material being tested, the first findings of the study under objectives 1 and 2 apply to two protection materials, MAT-A a high solids silane and MAT-B an aqueous crystallisation solution. Both these materials are available commercial products, both complying with the relevant European standard [Folic, 2009]. Although UK testing to standards specifies the use of C40 structural concrete (water/cement ratio 0.44), a general grade concrete C25 (water/cement ratio 0.55) has also be used to widen understanding. All concrete mixes are prepared in accordance with BS EN 1766 [BSI, 2000]. The identities of the two materials are not published because, whilst they have been commonly used for many years, the authors point to possible shortcomings with them and correspondingly wish to avoid associated legal actions by the manufacturers of them.

Each material was weighted in a high accuracy as per manufacturer recommendation and applied using a brush carefully to all surfaces of the cubes. The products were measured out in separate cups and different brushes were used to ensure that no cross contamination of the product occurred. The product was applied until the surfaces were saturated and the cubes were reweighted after application to ensure that there was approximately a 10g increase in weight. The cubes were then put aside to dry; they were propped up to allow air to circulate underneath the cube allowing the bottom surface to dry off as well. It was noted that the longer the cubes had been submerged in water the harder it was to ensure 10g of impregnate was applied to the cubes. All effort was made to make sure that all surfaces were coated equally with the impregnate.

Test Specimens

Twelve 100 mm concrete cubes were manufactured, six for each strength group, within which two where prepared at 0% moisture content, two at 2.5% and two at 5.0%. Cubes were immersed in distilled water to achieve 2.5% and 5% moisture contents. This was calculated by measuring mass before and after soaking using a high precision balance. The moisture content was determined by sorptivity test from a wetting cycles of over dried specimens. Further details of testing procedure can be found in earlier paper by the authors [Rahman et al 2014].

Figure 1 maps the number and purpose of the specimens used to investigate under objectives 1, 2 & 3. As far as possible, the protection products were applied following the respective manufacture guidelines, both at 200ml/m^2 or application to the point of refusal in the case of moisture content at the time of application.

Equipment

In the study, it is necessary to directly quantify the absorption or water and protective liquids into concrete. Whist the ISAT method has been used in previous work [Balakrishna, 2013]; the RILEM (International union of laboratories and expert in construction materials, system and structures) test [RILEM, 1980] was adopted because this was considered potentially more adaptable for on-site use.

The RILEM tube method represents a straightforward way for measuring the flow rate of liquid moving under low pressure through porous materials such as masonry, stone, and concrete [RILEM, 1980, Ibrahim et al, 1997]. The test can be either implemented in the laboratory or adapted for site to use on vertical and horizontal surfaces. After initial trials with this, it was found insensitive on account of the small contact area (5.06 cm^2) and small quantity of protective material absorbed in the cases of concrete with initial water content. To overcome this, the equipment was modified by increasing the contact area to 36 cm^2. Images of the modified RILEM test tube are shown in Figures 2 (a), (b) and (c) with a diagram of the standard device in Figure 2 (d).

The concrete face clear around the contacting rectangle is 2 cm wide, which is considered sufficient in view of the small total quantities of liquids delivered using the RILEM device.

Procedure

Test specimens were weighed immediately after 28-day curing before being placed in an oven at 105^{o}C, and then reweighed at 24 hours' intervals until they exhibited no changing weight point (dry condition). The specimens were divided into the groups previously stated and correspondingly processed in respect to the required moisture content.

The modified RILEM tube was fixed on the cube face, using a silicone sealant as shown in Figure 2c. According to the test scheme, distilled water or a protective solution was loaded to the vertical supply tube until the column reaches the "0" gradation mark. Take up of the corresponding liquid was then determined by recording its level at 5, 10, 20, 30, 40, 50 and 60 minutes after commencement. Four faces were used on each cube. The tests were applied to using four faces of each cube for the cases of 0%, 2.5% and 5% initial moisture content. The cubes were moisture conditioned between each test to avoid the effect of moisture content accummulated from apply the test to each face.

Strength effect

The influence of concrete strength on water absorption for the adopted concrete mixes is summarized in Figure 3 which shows; from left to right is each case, the results for 0%, 2.5% and 5% moisture content. The dossing rates stated in the manufacture's guidance for MAT-A and MAT-B were both exceeded for the 0% moisture content case. In all cases, the C40 concrete absorbed respectively less water or protection material than the C25 concrete. The C25 concrete absorbs an average of 35% more water than C40 concrete, 9% more MAT-A and 28% more MAT-B.

Initial moisture effect

Irrespective of any possible relationship between dosage and performance, Figure 3 shows that achievable material dossing is substantially reduced by moisture content relative to the 0% dry state. In terms of take-up volume, MAT-B out-measures MAT-A in all cases. Consistent results were observed in all specimens.

An average of 3.6 ml of water, 1.3 ml of MAT-B and 0.5 ml of MAT-A were absorbed by both concretes with 0% initial moisture content in one hour. The half saturated specimens (2.5%) absorbed an average of 0.6 ml water, 0.175 ml of MAT-B and 0.14 of MAT-A in sixty minutes, at less than 20% of the fully dry (0%) outcome. With the aid of the increased sensitivity of the modified RILEM device, the corresponding average take-ups were 0.1 ml water, 0.07 ml MAT B and 0.025 ml MAT-A. The progression of adsorption is illustrated in Figures 4 a-c with the 60-minute result shown in Figure 5.

The C45 and C25 concrete absorption of MAT-A and MAT-B declines dramatically with increased moisture content. The results for water are potentially useful because the behavior is similar but substantially more sensitive than the application response with the applied materials, in this case MAT-A and MAT-B. Figures 5 and 6 illustrate this for both concretes. This points to a potential means of predicting achievable dosages with the surface applied materials and, in turn, the possible lack of benefit that can be expected from there use on concrete with high moisture content.

Work for objectives 3: chloride penetration

Materials

The two concrete mixes and protection materials are the same as those stated in first two objectives for the absorption study.

Test specimens

Test specimens were cast to two formats, 450mm x 450mm x 50mm slabs [Calder and McKenzie, 2008, BSI, 2004] and 100mm cubes [AASHTO, 2002]. Figure 7 illustrates the large ponding slab. The use of cubes, referred to as the Unidirectional Salt Pond Test (U-SPT), gives the advantage of reducing handling weight and ease of production in large batches. A polymer sealant was used to build bunding on the cube faces. In all, six slabs and eighteen cubes were manufactured for Chloride penetration in C40 and C25 concrete. At this time, chloride penetration testing for protective materials applied under the conditions of initial water content are underway and outcomes not yet available.

Equipment

At the conclusion of salt ponding, dry drilling was used to collect dust samples. Chloride content analysis was undertaken according to BS EN 12469 (2007), using the Volhard Titration Method.

Procedure

The slabs and cubes were dried (0% moisture content) before applying the protection materials, the former divided into two groups of three, one slab of each group treated with MAT-A and MAT-B respectively. One slab of each group was held as a control specimen.

Both slabs and cube surfaces were ponded with 10% concentration sodium chloride solution for 60 days, the slabs covered by plastic sheets to reduce evaporation from the sodium chloride solution. Laboratory humidity was continuously recorded.

At 60-days, chloride content profiles were obtained by drilling the specimens in two sampling places not less than 100mm from the edge of the slabs. Dust samples were gathered between the depth intervals 0mm, 5mm, 15mm, 25mm and 40mm, using a 20 mm diameter drill bit, this diameter is equal to the maximum aggregate size. This drilling method provided a dust sample of between 1 to 5 grams, in accordance with BS EN 15629 (2007) [BSI, 2007, BSI, 2004, Highway England, 2003]. Care was exercised to avoid cross contaminate of dust samples gathered over each depth interval. Six slabs were used (untreated, MAT-A and MAT-B treated) in C25 and C40 concrete, this was to understand the influence of concrete strength on the effectiveness of the protection materials in preventing chloride penetration. Tables 1 and 2 show chloride content data for the various depth ranges for C25 and C40 respectively. Figure 8 shows this is in a graphical form.

Both MAT-A and MAT-B treatments reduce chloride ion penetration relative to the untreated cases. From Tables (1-2), it is observed that material MAT-B generally performs better than material MAT-A. For example, at 25 mm depth, MAT-B gives approximately 85% reduction in chloride ion concentration, while MAT-A gives approximately 50%.

Influence of strength

Figure 9 illustrates the effect of concrete strength on the performance of the two surface applied materials MAT-A and MAT-B. The influence of Concrete strength on Chloride content (Cc) is apparent in Figure 9 and Table 3, where untreated and treated C45 concrete is more resistance to chloride penetration than corresponding C25 cases. The increase in total chloride content with C25 compare with C45 for untreated slabs is 12.85%, 16.27% with MAT-A and 10.72% MAT-B treated concretes. Treatment with MAT-A is more influenced by concrete strength than for MAT-B.

Referring to the use of cubes for salt ponding (U-SPT ponding method) close agreement was achieved**.** The U-SPT test showed 82% agreement with the conventional salt-ponding testing method, as shown in Figure 10. This suggests that the more attractive use of cube salt ponding is a viable approach to testing. In the ongoing work, emphasis is to be given on the use of cubes, which allow large population sampling.

CONCLUSIONS

Key conclusions from the study are;

1. Increasing initial moisture content in C25 and C40 concrete has a similar substantial reducing effect on absorption of applied water and materials MAT-A and MAT-B.
2. The modified RILEM testing device developed in the reported study gives and effective means of measuring surface absorption in cases of high moisture content, where only volumes are taken up.
3. The similarity between the absorption of water and the two protection materials relive to initial water content, points to a possible basis for predicting achievable dossing of surface applied protection materials. Testing for different levels of moisture content between 0% and 5% is required to provide adequate data for such analysis.
4. The crystallization material MAT-B achieved greater application volume than the silane material MAT-A, for the three moisture content levels. However, the effect of initial moisture level on performance has yet to be concluded.
5. For the initially dry (0%) condition, both the slab and cube salt loading show that both material MAT-A and MAT-B result in reduced chlorine content in both concrete mixes. In this, MAT-B shows greater reduction than MAT-A.
6. Concrete strength influences chloride content reduction for untreated, MAT-A and MAT-B treated concrete, the C25 concrete showing greater chloride content than C40.
7. Considering the greater take-up of the protective materials for C25 concrete compared with C40 concrete, together with the greater chloride reduction achieved for the C25 concrete, indicates that the level of achieved dosing is significant. This has implications for the reduced dosing affected by initial moisture content; yet to be investigated.
8. In this testing, the crystallization material MAT-B achieves greater corresponding dosing and reduction in chloride content than the silane material MAT-A.
9. Use of 100 mm cubes for salt ponding rather than large slabs appears to be justified, giving a way to increase test specimens, which is beneficial for further testing work.

FURTHER WORK

Work is continuing towards the stated objectives, with protective materials investigated of different chemistries. Exploiting the modified RILEM test in further investigation into the influence of initial moisture content on water and protective material absorption, it is hoped that a practical, construction site method will be substantiated for predicting achievable dosing with protective materials. To support this, the effect of delivered dosing on chloride content reduction, as affected by initial moisture content, needs to be understood. The study is currently continuing in these areas.

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