

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/mm²) and high strength (C40:40N/mm²) 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 dosing 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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49 **INTRODUCTION**

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

61

62 **PROTECTIVE TREATMENTS**

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

70

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

77

78 On the strength of laboratory testing for a durable (15 years) vapour permeable, water repellent surface layer,
79 confidence grew for the application of high purity monomeric alkyl (isobutyl) – trialkoxy silane, to the extent that this
80 became specified for hydrophobic impregnation of concrete bridges. At the same time, it was recognised that other

81 chemistries would arise and be accepted if they were compliant with a test method specified by the UK Transport
82 Research Laboratory (TRL) [Highways England, 2013]. It is noted that the identical testing method was subsequently
83 incorporated into the normalized European Standard covering pore-lining impregnation of concrete [BSI, 2004].

84

85 Acceptance that alternative materials should be considered, opened the gate for a range of alternatives including water
86 based silanes, silane creams, aqueous fluoropolymer and crystallisation solutions. In the UK, their use has been granted
87 by Highway Authorities under 'Departure from Standards', on a case by case basis. Reduced environmental impact is
88 one of the acceptance criteria in this, which favour acceptance of the alternatives over silanes [Atkins, 2011]. Several
89 different types of sealers have been used to protect concrete, silanes; silicates and siloxanes, epoxy, gum resins and
90 minerals and linseed oil, stated in what is thought to be the order of decreasing effectiveness [NCHRP, 2004]. Other
91 recent development on concrete impregnation include super hydrophobic material by incorporating waste material like
92 waste paper sludge [Wong et al, 2015] and silica fume [Flores-Vivian et al, 2013]. Both developments highlighted
93 limited success of improving hydrophobicity of concrete. Researchers have also reported water-resisting admixture as
94 an integral part of concrete, but the degree of improvement in concrete protection is still unclear [Concrete Society,
95 2013].

96

97 Although tests have concluded that silane achieves little penetration [Wittmann et al, 2001], it has been suggested that
98 greater than 10 mm penetration is possible using a pure high solid content silane. Such material has been tested and
99 class II penetration achieved [Guar, 2007]. However, in the supporting testing [Syed and Donadio, 2013], it is important
100 to note that a concrete mix water/cement ratio of 0.7 was used, which is too high to be associated with structural
101 concrete (more typically w/c at 0.55 with a cube strength exceeding 40N/mm²), rather pedestrian pavement construction
102 with a 28-day cube strength in the region of 25 N/mm². It is reasonable to observe that such low strength, general
103 purpose concrete is likely to favour absorption and penetration of the silane to a greater extent than achievable with
104 structural concrete.

105

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

113

114

115 **CONCRETE PROTECTION STUDY**

116

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

131

132 **OBJECTIVES OF STUDY**

133

134 The objectives of this study are:

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

140

141 The pre-occupation with initial moisture content in the above objectives stems from on-site experience in the
142 application of impregnation materials to numerous concrete bridges. Testing reported in this paper supports the
143 significance of this. Currently, whilst protection material manufacturers specify maximum water content in the concrete

144 to be treated, there is currently no accurate site based method for confirming such. Whilst a prior 24 hour drying period
145 may be specified, a long proceeding period of concrete saturation and prevailing high humidity at application time is
146 possible. To help ensure worthwhile concrete protection from surface applied materials, the authors feel it is important
147 to develop objective measurement methods, taking advantage of relevant sensing technology.

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

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

160

161 **EXPERIMENTAL PROGRAM**

162

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

169

170 **Work for objectives 1 & 2: absorption issue**

171 **Materials**

172 Whilst work is continuing with further material being tested, the first findings of the study under objectives 1 and 2
173 apply to two protection materials, MAT-A a high solids silane and MAT-B an aqueous crystallisation solution. Both
174 these materials are available commercial products, both complying with the relevant European standard [Folic, 2009].
175 Although UK testing to standards specifies the use of C40 structural concrete (water/cement ratio 0.44), a general grade

176 concrete C25 (water/cement ratio 0.55) has also be used to widen understanding. All concrete mixes are prepared in
177 accordance with BS EN 1766 [BSI, 2000]. The identities of the two materials are not published because, whilst they
178 have been commonly used for many years, the authors point to possible shortcomings with them and correspondingly
179 wish to avoid associated legal actions by the manufacturers of them.

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

188

189 **Test Specimens**

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

195

196 Figure 1 maps the number and purpose of the specimens used to investigate under objectives 1, 2 & 3. As far as
197 possible, the protection products were applied following the respective manufacture guidelines, both at 200ml/m² or
198 application to the point of refusal in the case of moisture content at the time of application.

199

200 **Equipment**

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

205

206 The RILEM tube method represents a straightforward way for measuring the flow rate of liquid moving under low
207 pressure through porous materials such as masonry, stone, and concrete [RILEM, 1980, Ibrahim et al, 1997]. The test

208 can be either implemented in the laboratory or adapted for site to use on vertical and horizontal surfaces. After initial
209 trials with this, it was found insensitive on account of the small contact area (5.06 cm^2) and small quantity of protective
210 material absorbed in the cases of concrete with initial water content. To overcome this, the equipment was modified by
211 increasing the contact area to 36 cm^2 . Images of the modified RILEM test tube are shown in Figures 2 (a), (b) and (c)
212 with a diagram of the standard device in Figure 2 (d).

213

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

216

217 **Procedure**

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

221

222 The modified RILEM tube was fixed on the cube face, using a silicone sealant as shown in Figure 2c. According to the
223 test scheme, distilled water or a protective solution was loaded to the vertical supply tube until the column reaches the
224 "0" gradation mark. Take up of the corresponding liquid was then determined by recording its level at 5, 10, 20, 30, 40,
225 50 and 60 minutes after commencement. Four faces were used on each cube. The tests were applied to using four faces
226 of each cube for the cases of 0%, 2.5% and 5% initial moisture content. The cubes were moisture conditioned between
227 each test to avoid the effect of moisture content accumulated from apply the test to each face.

228

229 **Strength effect**

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

235

236 **Initial moisture effect**

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

240

241 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%
242 initial moisture content in one hour. The half saturated specimens (2.5%) absorbed an average of 0.6 ml water, 0.175
243 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
244 increased sensitivity of the modified RILEM device, the corresponding average take-ups were 0.1 ml water, 0.07 ml
245 MAT B and 0.025 ml MAT-A. The progression of adsorption is illustrated in Figures 4 a-c with the 60-minute result
246 shown in Figure 5.

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

252

253 **Work for objectives 3: chloride penetration**

254 **Materials**

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

257 **Test specimens**

258 Test specimens were cast to two formats, 450mm x 450mm x 50mm slabs [Calder and McKenzie, 2008, BSI, 2004] and
259 100mm cubes [AASHTO, 2002]. Figure 7 illustrates the large ponding slab. The use of cubes, referred to as the
260 Unidirectional Salt Pond Test (U-SPT), gives the advantage of reducing handling weight and ease of production in large
261 batches. A polymer sealant was used to build bunding on the cube faces. In all, six slabs and eighteen cubes were
262 manufactured for Chloride penetration in C40 and C25 concrete. At this time, chloride penetration testing for protective
263 materials applied under the conditions of initial water content are underway and outcomes not yet available.

264 **Equipment**

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

267

268 **Procedure**

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

272

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

276

277 At 60-days, chloride content profiles were obtained by drilling the specimens in two sampling places not less than
278 100mm from the edge of the slabs. Dust samples were gathered between the depth intervals 0mm, 5mm, 15mm, 25mm
279 and 40mm, using a 20 mm diameter drill bit, this diameter is equal to the maximum aggregate size. This drilling method
280 provided a dust sample of between 1 to 5 grams, in accordance with BS EN 15629 (2007) [BSI, 2007, BSI, 2004,
281 Highway England, 2003]. Care was exercised to avoid cross contaminate of dust samples gathered over each depth
282 interval. Six slabs were used (untreated, MAT-A and MAT-B treated) in C25 and C40 concrete, this was to understand
283 the influence of concrete strength on the effectiveness of the protection materials in preventing chloride penetration.
284 Tables 1 and 2 show chloride content data for the various depth ranges for C25 and C40 respectively. Figure 8 shows
285 this is in a graphical form.

286

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

290

291 **Influence of strength**

292 Figure 9 illustrates the effect of concrete strength on the performance of the two surface applied materials MAT-A and
293 MAT-B. The influence of Concrete strength on Chloride content (C_c) is apparent in Figure 9 and Table 3, where
294 untreated and treated C45 concrete is more resistance to chloride penetration than corresponding C25 cases. The
295 increase in total chloride content with C25 compare with C45 for untreated slabs is 12.85%, 16.27% with MAT-A and
296 10.72% MAT-B treated concretes. Treatment with MAT-A is more influenced by concrete strength than for MAT-B.

297

298 Referring to the use of cubes for salt ponding (U-SPT ponding method) close agreement was achieved. The U-SPT test
299 showed 82% agreement with the conventional salt-ponding testing method, as shown in Figure 10. This suggests that

300 the more attractive use of cube salt ponding is a viable approach to testing. In the ongoing work, emphasis is to be given
301 on the use of cubes, which allow large population sampling.

302 **CONCLUSIONS**

303

304 Key conclusions from the study are;

305

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

326

327 **FURTHER WORK**

328

329 Work is continuing towards the stated objectives, with protective materials investigated of different chemistries.

330 Exploiting the modified RILEM test in further investigation into the influence of initial moisture content on water and

331 protective material absorption, it is hoped that a practical, construction site method will be substantiated for predicting
332 achievable dosing with protective materials. To support this, the effect of delivered dosing on chloride content reduction,
333 as affected by initial moisture content, needs to be understood. The study is currently continuing in these areas.

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