

Silane Performance: Testing Procedures and Effect of Concrete Mix Design

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The performance of a silane-penetrating water-repellent material applied to three concrete mix types was evaluated using alternative test procedures. One of the mix types was similar to standard reference concrete often used in laboratory testing. The other two mixes represented structural deck concrete and high-density overlay concrete, with correspondingly lower water-cement ratios and varying mix designs. Performance was evaluated with respect to depth of penetration, absorption, water vapor permeability, and chloride ingress. Test procedures and mix type were both found to affect the parameters used to evaluate the performance of the treated concretes. Results indicate the need to represent field conditions (such as mix design) to the extent possible in order to predict better field performance with laboratory tests.

The problems that arise from water and chloride-laden water intrusion into concrete bridge decks are often costly to repair. These problems range from scaling due to freeze-thaw cycles to corrosion of reinforcement and subsequent spalling of the concrete cover (1). The problems typically occur where the concrete bridge deck is exposed to freeze-thaw cycles and intermittent wetting, especially where deicing chemicals are used (2).

Over the past decade the use of silane to protect concrete bridge decks has grown in popularity because of its desirable performance characteristics, namely, reduction of chloride and water ingress, penetration into the concrete (which is useful on wearing surfaces and offers protection from the deteriorating effects of ultraviolet radiation), and unaffected skid resistance (3). However, the implementation of silane technology has been hindered by the lack of consensus concerning standardized testing procedures. This situation is costly to the various departments of transportation, in many cases because of uncertainties and concerns related to the effects of various commonly encountered field conditions often not considered in the testing procedures. Many current testing procedures use a standard laboratory reference concrete with a water-cement ratio (w/c) of approximately 0.50. However, structural concrete used for bridge decks usually has a somewhat lower w/c ratio, often about 0.45. Other mixes, such as high-density overlay concrete, may have even lower w/c 's. Variations in mix design for concrete in the field may be significant, and the projected performance based on laboratory testing of a reference concrete mix type may vary greatly from the field where a different concrete type is used.

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This study examines the effect of mix design on the performance of a silane water-repellent treatment material, with alternative testing procedures used to evaluate performance. Physical parameters studied in the tests include water absorption, chloride ingress, depth of penetration, and moisture vapor permeability (MVP). The effect of concrete moisture content at the time of application was also examined since the tests used different treatment conditions. Results of the first phase of a research project sponsored by the Oklahoma Department of Transportation (ODOT) are presented in this paper. It is anticipated that ODOT will use results to improve existing screening methods and to develop criteria to promote better field performance of penetrating water-repellent treatment materials.

SILANE WATER-REPELLENT MATERIAL

When silane is applied to the surface of concrete, the molecules are absorbed to the depth of penetration because of capillary suction forces (4). In the presence of moisture the hydrolysis process starts and produces silanol molecules, which are unstable and condense to form a Si-O bond with the silicate molecules in the concrete (5). The hydrocarbon group is in turn bonded to the silane molecules with a Si-C chemical bond. The hydrocarbon group is responsible for water and chloride repellency and lines the micropores of the concrete matrix, producing a hydrophobic layer by reducing the concrete surface tension (3).

The silane water-repellent treatment material used in the study was produced by the Chemistry Department of the University of Oklahoma to ensure the material's quality and reproducibility. The solution contained 40.3 percent (by weight) isobutyltrimethoxysilane in an isopropyl alcohol carrier, with a recommended application rate of 3.07 m²/L (125 ft²/gal). This rate was used for the research program.

EXPERIMENTAL PROGRAM

Concrete Mix Types

Three mix types were investigated and designated Types A, AA, and HD (high-density overlay). Specifications and properties for the mixes are shown in Table 1.

Test Procedures

Two basic test series were performed for each mix type: ODOT series tests and tests based on *NCHRP Report 244 Series II*

TABLE 1 Specifications and Measured Properties of Mixes

Mix Type	A	AA	HD
W/C Ratio	0.49	0.44	0.33
Cement Factor (kg/m ³)	335	390	490
Specified Slump (mm)	25-75	25-75	13-25
Specified Air Content (%)	5-7	5-7	5.5-7.5
Measured Slump* (mm)	50, 50	38, 25	13, 19
Measured Air Content* (%)	6.5, 5.2	4.9, 4.5	5.2, 6.0
Maximum Size Aggregate [†] (mm)	13	13	13
Coarse Aggregate [‡] (kg/m ³)	950	950	964
Fine Aggregate [‡] (kg/m ³)	869	802	640
28 Day Compressive Strength (MPa)	39.3	46.2	56.5

(1 kg/m³ = 1.68 lb/cyd, 1 mm = 0.039 in, 1 MPa = 0.145 ksi)

* Values for each of two batches

[†] Maximum aggregate size used in laboratory concrete specimens

[‡] Batch weights at saturated surface dry condition

(6). Brief descriptions of the tests performed for each series follow.

ODOT Series Tests

The ODOT series tests consisted of procedures to evaluate depth of penetration, MVP, absorption, and chloride ion intrusion. For all tests except chloride ion intrusion, test specimens were broom-finished blocks 200 × 200 × 50 mm (8 × 8 × 2 in.). The chloride ion intrusion test (salt ponding) used blocks 300 × 300 × 75 mm (12 × 12 × 3 in.).

- Depth of penetration—OHD-L34 (7): oven-dried specimens were treated and broken, and the depth of the hydrophobic layer was measured at multiple locations.

- MVP—OHD-L35 (8): specimens were treated after 48 hr of immersion in deionized water. The weight of water lost during oven drying was measured to provide an indication of the vapor transmissibility through the silane treatment.

- Absorption—ASTM C642-81: absorption through the top (unwaxed) specimen surface after 48-hr and 50-day immersion in water was measured for treated and untreated blocks.

- Chloride ion intrusion—AASHTO T259-80/T260-84: chloride contents were determined after 90-day ponding with a 3 percent NaCl solution. Powder samples were taken at depths of 1.6 to 13 mm (1/16 to 1/2 in.) and 13 to 25 mm (1/2 to 1 in.) using a rotary hammer. Chemical analysis was performed according to AASHTO T260, and results were reported as total chlorides absorbed (total chlorides minus total base chlorides in control specimens).

NCHRP Report 244 Series II Tests

The basic test procedure described in *NCHRP Report 244 (6)*, with slight modifications, was used for the major part of the

study. Two variations of the procedure were also examined.

Treated and untreated cubes [100 mm (4 in.)] of each mix type were immersed in a 15 percent NaCl solution for 21 days and then air-dried for 21 days. Specimen preparation specifics included light sandblasting to remove surface laitance and 21 days of self-curing in plastic bags with a 5-day air-drying period before treatment. Weight recordings were performed every 3 days during the immersion and air-drying periods. Triads of cubes were used for each test group instead of pairs of cubes, as were used in the original NCHRP study. Chloride samples taken at the conclusion of the test procedure were obtained by rotary hammer drilling into three faces of the cube at two depth intervals, similar to the AASHTO T259/T260 procedures. This differs from the original NCHRP study, in which samples were obtained by crushing half of a cube to a fineness sufficient for chloride analysis.

Two variations of the procedure were carried out in parallel with the basic test procedure. In one variation, the cubes were not sandblasted before treatment. The second variation differed from the basic procedure in that the cubes were oven-dried, rather than air-dried, at the conclusion of the 21-day immersion period.

Other Depth-of-Penetration Tests

The moisture content of the concrete, among other factors, influences the depth of penetration of silane water-repellent treatment materials. The various tests performed used a wide range of moisture contents at treatment (from oven dry to saturated surface dry), so depth of penetration was measured for selected specimens (in addition to the primary purpose of the original test). Measurements were taken using absorption specimens, cubes, and MVP specimens. A pilot study was conducted to examine the effect of retreatment on specimens that initially exhibited negligible penetration. Previously untreated specimens from earlier tests were treated at random

moisture contents to examine the effect on penetration, and a controlled study was performed using Mix Type AA.

RESULTS AND DISCUSSION OF RESULTS

Test results are presented to give insight into the behavioral parameters of depth of penetration, water absorption, chloride ingress, and MVP. All results presented are averages of individual test specimens for each mix type.

Depth of Penetration

Primary depth of penetration results were obtained from the ODOT test, which is designed solely for that purpose. Additional results were obtained as described earlier. The depth of penetration and absorption tests specified treatment after oven drying (zero moisture content), whereas the MVP test used treatment at a nearly saturated surface dry state. The high moisture content of the MVP specimens resulted in negligible penetration depth for all mix types. To examine the feasibility of retreatment, the MVP specimens were oven-dried, and silane reapplied at full [(3.07 m²/L (125 ft²/gal)] and half [6.14 m²/L (250 ft²/gal)] coverage rates.

Results for all mix types are shown in Table 2. The depth of penetration was consistently higher for Mix Type HD, despite the low w/c of the mix. This could be due to the mix configuration and pore size. Mix Type A (w/c = 0.49) yielded higher depth-of-penetration values than Type AA (w/c = 0.44). Thus, results of these tests suggest that the w/c alone does not provide a relative indication of performance. Retreatment of specimens that originally had no measurable penetration led to significant improvement. Retreatment at full coverage rate resulted in penetration comparable to the other dry-treated specimens. It must be recognized that the retreated specimens were oven-dried before silane was reapplied and that the scope of this portion of the study was very limited.

Results of the preliminary study using surplus and originally untreated specimens treated at random moisture contents are shown in Figure 1. Because of various factors, the moisture contents could in some cases be determined only approximately; however, the results shown in Figure 1 indicate that concrete moisture content at the time of treatment can have a dramatic impact on the depth of penetration. Results of a controlled study for Mix Type AA are shown in Figure 2. Significant reductions in penetration occurred for moisture contents higher than about 1 percent.

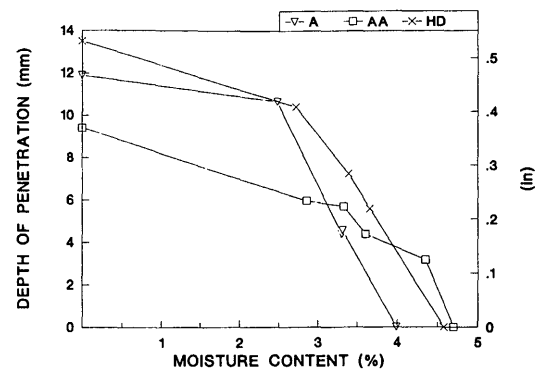


FIGURE 1 Influence of moisture content on penetration: preliminary study.

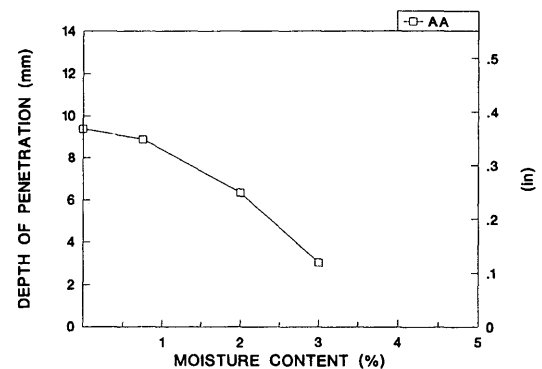


FIGURE 2 Influence of moisture content on penetration: controlled study.

Water Absorption

ODOT Series Test

Absorption results, obtained from tests following ASTM C642, are shown in Table 3. Mix Type A absorbed more water than Type AA, even though Type A had a higher depth of penetration. In general, the Type HD mix absorbed the least water. The percentage reduction in absorption due to treatment was higher for Type AA than the other two mixes. The percentage improvement due to treatment dropped after 50 days, reflecting the difference in absorption rates between untreated and treated specimens. For the untreated specimens, approxi-

TABLE 2 Depth of Penetration Results For Dry Application (mm)

Mix Type	Depth of Penetration Test	Absorption Specimens	MVP Specimens, 1/2 Rate Reapplied	MVP Specimens, Full Rate Reapplied
A	11.9	11.7	9.1	11.9
AA	9.4	8.6	8.4	11.9
HD	13.5	15.5	10.2	14.5

(1 mm = 0.039 in)

TABLE 3 Results of ODOT Series Absorption Tests

Mix Type	48 hr Absorption (%)			50 day Absorption (%)		
	Untreated	Treated	Improvement (%)	Untreated	Treated	Improvement (%)
A	4.28	0.55	87	5.25	2.20	58
AA	4.12	0.17	96	5.11	0.79	85
HD	3.98	0.36	91	4.68	0.77	84

mately 80 percent of the 50-day moisture gain was absorbed during the first 48 hr of soaking, whereas the treated specimens absorbed only 20 to 45 percent of the 50-day moisture gain during the first 48 hr.

NCHRP Report 244 Series II Test

Weight gained or lost was normalized with respect to the cube weight immediately before immersion, because dry weight of the untreated cube is not obtained from the test procedure. Approximate moisture contents at time of treatment, obtained from oven-dried cubes, were 3.3, 4.0, and 3.6 percent for Mix Types A, AA, and HD, respectively. Average weight gained or lost for each mix type is presented for untreated cubes in Figure 3 and for treated cubes in Figure 4. A statistical comparison (*t*-statistic) between means of the weight-gained curves (at selected times after immersion) confirmed that differences in behavior of the mixes were significant at the 5 percent level.

Mix Type AA gained more moisture than the other two mix types for treated and untreated specimens, although the difference was not great for the treated specimens. The rate of weight gain was also higher for the Type AA mix. Compared with the other two mix types, the treated and untreated Type HD specimens absorbed the least water on immersion.

The percentage improvements in absorption due to treatment (at 21-day immersion) do not compare directly to the values obtained from the ODOT series absorption tests, because the durations of immersion and moisture contents at the time of treatment are different for the two tests. And the cubes were immersed in a salt solution instead of water. The values, given in the following table, indicate that in general,

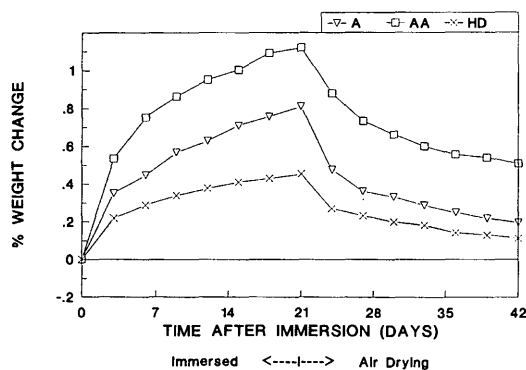


FIGURE 3 Weight gain or loss of untreated cubes (air-dried).

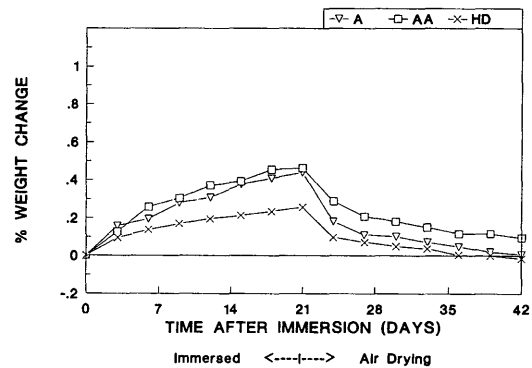


FIGURE 4 Weight gain or loss of treated cubes (air-dried).

the improvement in moisture repellency was greater when silane was applied to oven-dried specimens, as with the ODOT absorption tests (Table 3).

Mix Type	Improvement (%)
A	59.0
AA	47.5
HD	44.4

Cubes that were not sandblasted before immersion exhibited absorption characteristics similar to those of sandblasted cubes.

Moisture Vapor Permeability

For the ODOT series test, the depth of penetration was negligible because of the high specimen moisture content at the time of application. Moisture loss due to evaporation before the completion of treatment resulted in a large variation of results; this part of the testing program was deemed inconclusive. MVP behavior of the treated NCHRP 244 cubes can be seen in Figure 4 (air-dried cubes) and Figure 5 (oven-dried cubes). The absence of sandblasting had little effect on the vapor transmission characteristics of the specimens; those results are not presented here.

Treated air-dried cubes (Figure 4) lost much, if not all, of the weight absorbed during immersion by the end of the 21-day drying period. Mix Types A and HD lost 93 and 108 percent, respectively, of the water absorbed during immersion; Mix Type AA lost 75 percent of its absorbed water. The better vapor permeability characteristics of treated A and HD cubes were partially inherent in the mixes themselves. Untreated cubes reflected the same trend as treated cubes (Figure 3). The percentages of absorbed moisture lost on air drying were higher for the treated cubes than for the untreated cubes,

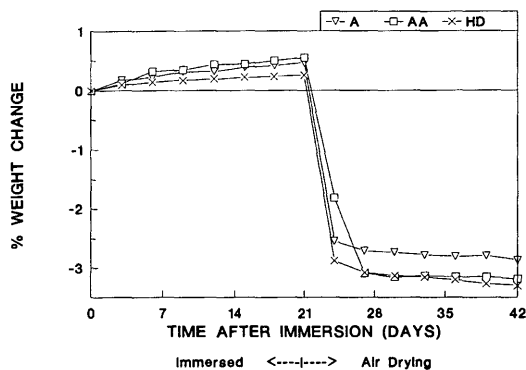


FIGURE 5 Weight gain or loss of treated cubes (oven-dried).

indicating that the vapor permeability characteristics of the mixes were largely unaffected by the silane. It must also be recognized that the specimens were immersed in salt water. It would be expected that not all of the weight gained during immersion would be lost upon drying, because some salt could remain in the concrete. The amount of actual moisture lost upon drying may be slightly higher than predicted by the test.

Curves for the oven-dried cubes are shown in Figure 5. As expected, the immersion branches of the curves are essentially identical to those of the air-dried cubes (Figure 4). The results reflect the different moisture contents of the mixes at the time of immersion. For example, Mix Types AA and HD exhibited the largest negative weight change upon oven drying, largely because of their higher moisture contents at time of immersion.

Absorbed Chlorides

Results for AASHTO salt ponding specimens and NCHRP cube specimens are shown in Figures 6 and 7, respectively. These figures contain means for the individual data. Because of the fairly large variability observed in the data, statistical comparisons were performed. Differences in means were tested using the *t*-statistic at the 5 percent level.

Considering differences between treated and untreated mixes for the AASHTO salt ponding tests (Figure 6), at the first

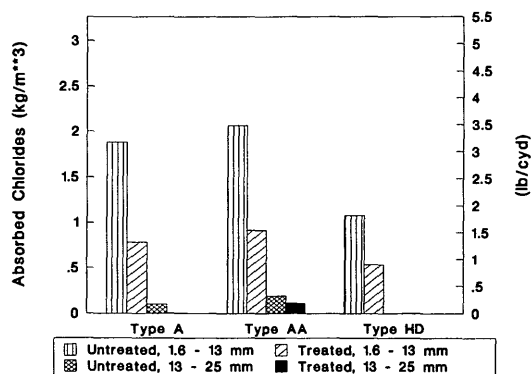


FIGURE 6 Absorbed chlorides, ODOT series test (AASHTO T259/T260).

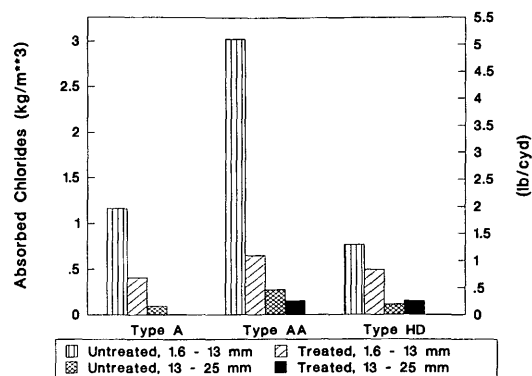


FIGURE 7 Absorbed chlorides, NCHRP 244 series test.

depth the Type HD mix absorbed the least chlorides, and Types A and AA appear similar. These observations were verified statistically, except no difference was observed between Types HD and A. An unexplained larger variation in the data occurred for Type A, particularly for the untreated specimens, which contributed to this statistical observation. No statistical difference was observed between mixes at the second depth, for untreated or treated concrete.

Comparison between treated and untreated slabs (for a given mix) indicated that the difference for Types AA and HD was significant at the first depth. The absence of difference due to treatment for Mix Type A is again explained by the large variability in the data. No statistical difference was observed between treated and untreated concrete, regardless of mix, at the second depth.

Examining results from the NCHRP cubes (Figure 7), the untreated Mix Type AA absorbed considerably more chlorides at the first depth than did Types A and HD, which were statistically similar. No statistical difference was found between treated mixes at the first depth or between treated or untreated mixes at the second depth. Considering treated versus untreated cubes for a given mix, the only statistical difference observed was for Mix Type AA at the first depth.

Comparing the results from the AASHTO slabs to those of the NCHRP cubes yielded no statistically significant differences in absorbed chlorides for samples of the same mix, depth, and treatment. However, for both test procedures, the difference between chlorides measured at the first and second depths was significant for all mixes for a given treatment condition. The small measured chlorides at the second depth, regardless of treatment or mix, suggest that chloride absorption in the upper 13 mm (1/2 in.) may be a more useful indicator of performance using these tests. The observed variability in the chloride data also suggests that the cost of testing and importance of the results should be closely examined in selecting the number of specimens to be sampled.

Although variability in the data limited statistical conclusions, in all but one case (NCHRP test, HD mix, second depth), mean chlorides absorbed by treated mixes were lower than for corresponding untreated mixes. This trend was consistent and should not be ignored. The fact that treatment lowered chloride absorption is clear, despite statistical conclusions, although the degree of improvement is subject to debate. This dilemma results from the fact that observations

concerning macrolevel behavior are being based on microlevel sampling. Large variability is commonly encountered in such cases. Similarly, the lack of statistical difference between treated and untreated mixes at the second depth should not be interpreted to mean that silanes do not reduce chlorides at depths below 13 mm (½ in.). Instead, it may indicate that the tests performed do not produce sufficient chloride levels at this depth to assess adequately the improvement due to treatment.

SUMMARY AND CONCLUSIONS

The performance of a silane-penetrating water-repellent material applied to three concrete mix types was evaluated using tests based on ODOT procedures and *NCHRP Report 244* Series II procedures. The three mixes were representative of standard reference concrete (Type A), structural concrete (Type AA), and high-density overlay concrete (Type HD).

1. Depth of penetration was good for all mixes when treatment was applied to dry (0 percent moisture) concrete. Significant reduction in penetration was observed with increasing moisture content at treatment.

2. From the results of statistical analyses, absorbed chlorides in the first 13 mm (½ in.) depth were significantly reduced by silane treatment for Mix Types AA and HD according to the AASHTO salt ponding test. However, the NCHRP cube tests indicated that only Mix Type AA was improved by treatment at this depth. Small amounts of absorbed chlorides were measured at the second depth for all mixes, regardless of treatment. Mean absorbed chlorides were consistently reduced by treatment for all mixes at both depths (with one minor exception). The variability in chloride data should be closely considered in selecting numbers of test samples, developing screening limits, and interpreting results.

3. The treated HD mix generally outperformed treated specimens of the other two mixes, exhibiting greater depth of penetration and lower water and chloride absorption. Improvement in performance resulting from silane treatment was usually not as great as for the other mixes; tests on untreated specimens indicated inherently better performance of the HD mix.

4. Performance of treated Mix Types A and AA was fairly similar, with some relative behavior affected by the test procedure used to obtain the physical parameter of interest. For treated NCHRP 244 cubes, Type A slightly outperformed Type AA with respect to absorption and vapor permeability, but they had similar absorbed chlorides. In the ODOT test series, Type A exhibited greater depth of penetration and similar chloride absorption but higher water absorption—despite its greater depth of penetration and contrary to the trend observed in the NCHRP 244 cube tests.

5. Performance of untreated Mix Types A and AA was similar for the ODOT test series. The NCHRP series suggested superior performance of untreated Type A over Type AA in terms of moisture gain and chloride absorption.

6. Results of the tests indicate that the interrelationship between performance characteristics of treated concretes is not straightforward. Use of a more “dense” mix (lower w/c and higher cement factor) did not necessarily reduce penetration or absorption, and smaller depth of penetration did

not necessarily lead to higher absorption. There is a need for a more thorough understanding of the mechanisms responsible for performance.

7. Of the test procedures examined, the *NCHRP Report 244* Series II cube tests more closely represented field conditions, at least in terms of specimen moisture content at the time of silane application. Moisture content was shown to have a dramatic impact on depth of penetration; it is not known to what degree other performance characteristics are influenced by this variable. However, variability in test conditions exists since moisture content at the time of silane application is not controlled.

8. Results of this study point to the need to duplicate field conditions in the laboratory, to the extent possible, in order to better predict field performance of concrete treated with silane. Use of standard reference concrete for product screening purposes may be advantageous in comparing data from various testing agencies. However, silane performance of treated standard reference concrete may not predict field performance, because of the myriad variables introduced (of which mix type is but one).

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