Evaluation of lithium based concrete curing compound effectiveness

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Abstract

The purpose of this research was to conduct an evaluation of lithium concrete curing compounds for their relative effectiveness to decrease water evaporation and to meet other noted measures of effectiveness as well as to investigate the optimal time of application. In this regard, curing compound effectiveness was evaluated for four different curing compounds for comparative purposes as a function of differing performance indicators including moisture loss, dielectric constant, relative humidity, and compressive strength.

Based on the preliminary findings, even though the lithium curing compound seemed ineffective in limiting moisture loss from the concrete surface early in the curing cycle, the resulting compressive strengths were acceptable. A clear trend in the time of application of lithium compound places the optimal application time at 2.5 – 3 hours after casting using controlled environmental testing conditions. Differential hardening of the lithium treated concrete specimens was observed and suggests that the lithium compound should not be added to concrete prior to one hour after casting. Based on Environmental Scanning Electron Microscope (ESEM) images, the lithium compound is found to be reactive with the silicate in the mortar to form a gel-like membrane on the treated surface and later yields improved strength levels.

Introduction

Excessive early-age water evaporation from the surface of concrete pavement may induce detrimental impacts, high porosity, delamination (leading to joint spalling in concrete pavements and other similar distresses), and loss of strength on long-term performance of the pavement. Spalling involves a breakdown or dislodging of concrete segments along a crack or joint in a concrete slab within 0.6 m (2 ft) of a crack or joint, and it affects the quality of concrete pavement smoothness and riding quality.[1]

The Texas Department of Transportation (TxDOT) has recently experienced cases of spalling and delamination failures, which may be related to excessive early age evaporation worsened under the influence of certain field conditions such as high temperature, low relative humidity, high solar radiation, and high wind speed. To mitigate early-age unexpected water loss, application of curing compounds in concrete paving has been widely used to minimize evaporation. However, the TxDOT standard specifications for pavement construction (Item 360) is limited to the definition of membrane style curing compounds in terms of key characteristics such as percent solids, density, viscosity, color, and the application rate and does not specify curing performance in terms of field conditions. The current laboratory curing membrane effectiveness evaluation method ASTM C 156 has some intrinsic deficiencies: irrelevance of the test conditions to field performance, mortar's hardening effect, and questionable basis for assigning moisture loss limits. Previous research introduced a new laboratory test protocol based on changes in the surface relative humidity, moisture loss, and mortar compressive strength to evaluate the curing membrane effectiveness in controlling evaporation.[2]

The previous test protocol was used to evaluate resin and wax based curing compounds. The instrumentation (especially for surface humidly chamber) in the previous method was subsequently modified and found to be very effective in measuring curing effectiveness parameters. Lithium based compounds, which stop water loss by a different mechanism, was examined in this research by the updated test method in order to validate the applicability of the test protocol in evaluating lithium-based compounds.

Objective

The purpose of the current research is fourfold, which is listed below:

- To test lithium-based compounds using an updated test protocol in order to classify lithium compounds in terms of curing effectiveness using similar performance parameters as was previously used.
- To determine the optimal time of application of Li-compounds - to study if the time of application of lithium compound after casting play any role on changing the performance indicators.
- Use a ranking system to compare different compounds.
- To investigate the mechanism by which lithium compounds aid in the retardation of evaporation from concrete during the curing process.

Experimental Program

Materials used and test procedure employed are described below.

Materials

Four curing compounds, i.e., (i) one Lithium-based compound (LiC), (ii) one Lithium and resin based compound (LiRC), (iii) one conventional resin based compound (RC) and (iv) one high reflective resin based compound (HRRC) were used in this research. Mortar specimens of different dimensions (described next) were used for measuring relative humidly, moisture loss, dielectric constant and compressive strength. The mix design of mortar specimens used is given in Table 1. A Type I/II cement and conventional concrete sand were used.

Table 1. Mixture Propagation

Mixture	W/C	Unit Weight (lb/ft^3)		
		Water	Cement	Sand
	0.40	13.94	34.84	95.81

The sequence of mortar mixing (in accordance with ASTM C 305) is described below:

1. The total amount of water was first placed in the mixing bowl.

2. The cement is introduced and mixed at a slow speed for 30 s.

3. The required amount of aggregate is added to the mixer over a period of 30 s while the mixer continues to operate.

4. The resulting mortar is allowed to mix for an additional 30 s at a medium speed.

5. After a minute rest period, the mixing is continued for an additional minute until a homogeneous mortar with no lumps is obtained.

Test Procedure

Mortar disc specimens of 12" diameter x 4" tall are used for relative humidly, moisture loss and dielectric constant measurements whereas 2 x 2 inch mortar-cube specimens are used for compressive strength measurement.

Moisture-Loss Specimens

A square chamber assembly with two chambers was attached to the mortar disc specimen (Figure 1), using commercial silicon adhesive, to measure relative humidity (RH). The left chamber in Figure 1 represents the configuration to measure surface RH (i.e. humidity just below the curing compound) and the right chamber record RH under sealed condition. The arrangement of placing a filter paper over a perforated support in the left chamber (called a filtered chamber) creates an environment that closely mimics the situation immediately below the curing compound. Curing compound was added to the surface of the disc immediately after casting in amounts corresponding to an application rate of 180 ft^2 / gallon. The mortar discs with the humidity chamber were then placed on a scale (Figure 1) to measure moisture loss inside an environmental chamber maintained at 104±5°F, 30% relative humidity, and 10 mph wind speed.

The lithium compound (LiC – non-resin based) was applied directly to the surface of the concrete in the filtered chamber rather than on the filter since the lithium needs to be in direct contact with the concrete. Relative humidity was then recorded in the filtered chamber but the difference between it and 100% was determined to represent the relative humidity immediately below the concrete surface; a similar difference between the sealed chamber (one with no treatment) and the filtered chamber could also be considered in characterizing the effects attributable to the lithium curing compound. This method, however, returns results that may not be directly compared or consistent with the relative humidity levels of the resin-based compounds particularly in using 80% as the key indicator in compound effectiveness.

Readings were taken every 30 minutes for the first five hours, every hour for the next five hours, and then at consistent intervals up to 48 hours for the following parameters:

1. Weight reading - taken by reading digital scale
2. Filtered chamber RH and temperature - taken by inserting handheld sensor into filtered chamber assembly.
3. Sealed chamber RH and temperature - taken by inserting handheld sensor into sealed chamber assembly.
4. Ambient RH and temperature - taken by placing handheld sensor in ambient position in the environmental chamber.
5. Dielectric Constant - taken by collecting three readings with a Percometer and averaging the three readings.

Figure 1. Disc Specimen Testing Setup.

Cube Specimens for Compressive Strength

2" x 2" mortar cubes were cast by the following procedure:

Mortar was placed into each 2" x 2" opening to fill the cube mold. With a rubber tapping device the mortar was compacted 16 times, four times in four differing directions to an approximate volume equaling ½ the mold height (1"). The entire brass mold (3 – 2" x 2" cubes) was tapped approximately 10 times. Mortar was placed into each 2" x 2" mold to fill the mold to the top (2"). With a rubber tapping device the mortar was compacted 16 times, four times in four differing directions. The entire brass mold (3 – 2" x 2" cubes) was tapped approximately 10 times. The tops of the 2" x 2" molds were smoothed (finished) with a metal trowel to the top of the brass mold assembly.

Curing compounds were applied three hours after casting with the same application rate of 180 ft^2/ gallon. The cube specimens were cured under the same environmental chamber and strength data were obtained at the age of 1, 3, 7, or 28 days. Average strength of 2-3 cubes are reported as representative strength at each age.

Additional cube specimens were tested to determine the best application time for only Li compound. Lithium compound was applied until it created an uniform thick layer on cube specimen surfaces at 0, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 4.5 and 5.0 hours after casting. These cubes were cured for three days inside the same environmental conditions and compressive strength corresponding to each application time was determined. Average of three compressive strength values are reported as representative strength for each application time.

Environmental Scanning Electron Microscope (ESEM)

The appearance of Li curing compounds as well as conventional resin based compound was studied using an environmental scanning electron microscope (ESEM).

Moisture loss, RH, dielectric constant data

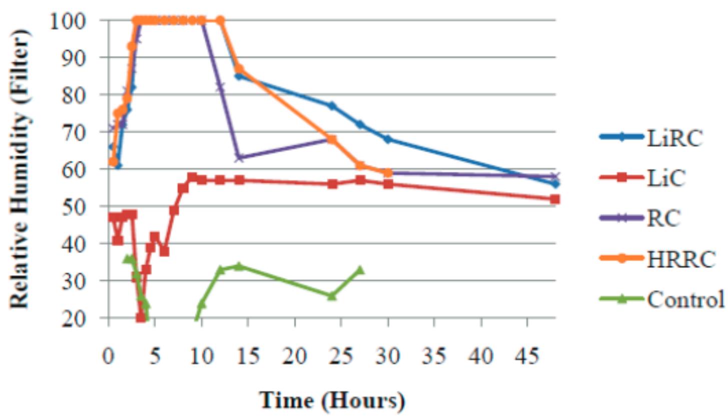

Figure 2. Relative Humidity Inside Filtered Chamber vs. Time for the Studied Curing Compounds.

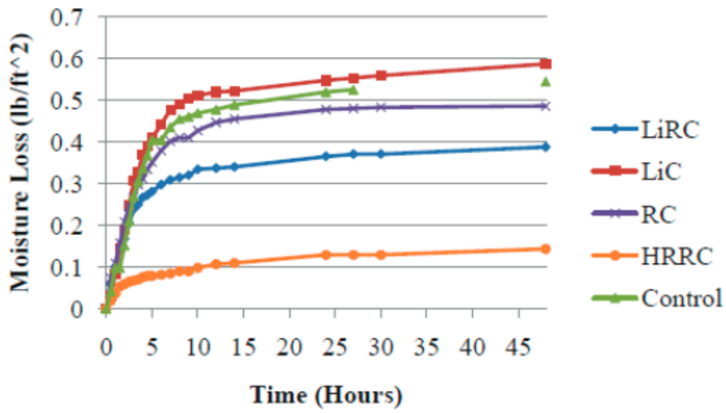

Figure 3. Moisture Loss vs. Time for the Studied Curing Compounds.

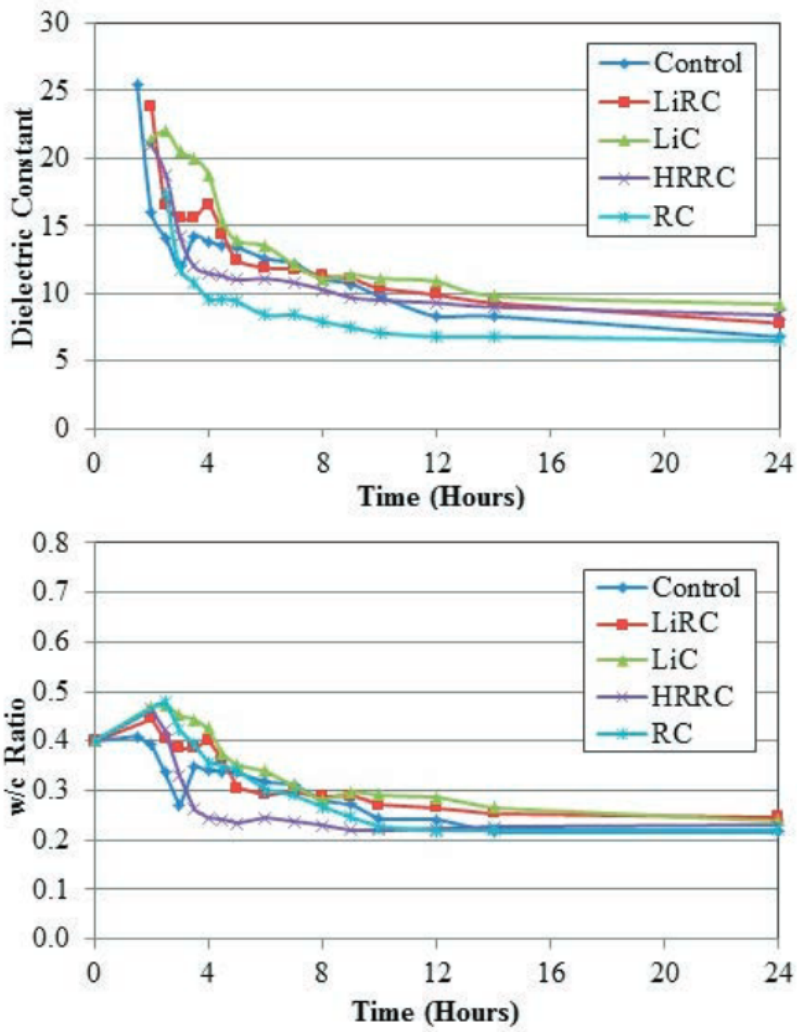

Figure 4. Dielectric Constant vs. Time for the Studied Curing Compounds.

from disc specimens, strength data using cube specimens, and selective ESEM testing were collected for each test run in Table 2. The test runs using different compounds is presented in Table 2.

Table 2. Test Combinations.

Test #	Curing Compound
1	LiRC
3	LiC
7	RC
9	HRRC

Relative humidity (i.e. cured conditions), moisture loss, and dielectric constant data was collected over a 24 hour period and summarized in Figures 2-4 for the four curing compounded listed in Table 2. In some instances, special measures were taken in order to account for the difference in how resin-based curing compounds functioned relative to non-resin-based or non surface membrane forming (i.e. reactive type) compounds. The word 'reactive' is used as a way of noting that these agents appear to react with the paste in the concrete to form a protective layer in the concrete in which to inhibit moisture loss. The reactive compounds tend to build up a protective layer gradually over time as an accelerated hydration process takes place through the depth that the curing agent penetrates the concrete surface, thus forming a hardened layer in which to inhibit the movement of moisture and facilitate strength gain in the concrete.

Relative Humidity

Relative humidity data for resin-based compounds was collected from inside a filtered chamber by placing the compound on a filtered covering which served to create a cured environment above the exposed concrete surface for sampling purposes. This test setup can be seen in Figure 1. The non-resin or reactive compounds were placed directly on the concrete surface where the difference between a RH of 100% and the measured RH immediately above the forming protective layer was observed. This observed data is presented in Figure 2. This difference is shown as a means of equivalency to the other RH data shown in the figure. The following observations were made:

- It takes around 2.0-2.5 hours for the RH inside the chamber to reach 80% for the

LiRC, RC, and HRRC compounds.

- The RH value reaches around 100% after 4-5 hours for these compounds and stays at 100% for 12-15 hours. The attainment of 100% RH is believed to be due to in part condensation inside the chamber. Nonetheless, the longer the condensation period the better the curing effectiveness. It is believed that quick decay of the condensation period is an indication of poor curing. The LiRC and HRRC both show longer duration of maintaining 100% RH followed by a slow decay till reaching 80% RH. However, RC shows a relatively shorter duration of 100% RH and a faster decay till it reaches 80% RH.
- The RH trend for the LiC compound, which is strictly a reactive type product, shows that little protection against moisture loss is not achieved until nearly 10 hours after placement. This compound, however, reaches the equilibrium RH much sooner than the resin-based or containing compounds do.

The time needed to maintain RH above 80% was found to be a useful parameter for determining curing effectiveness. The time for RC above 80% RH is around 12 hours whereas it is 18 hours for HRRC and 20 hours for LiRC. The ranking with increasing order of quality based on time above 80% RH is RC < HRRC < LiRC.

Moisture Loss

Moisture loss data was collected and is presented above in Figure 3. Moisture loss data was calculated by observing weight readings at pre-determined intervals of the disc samples to establish moisture loss curves. HRRC shows the lowest moisture loss whereas LiC shows the highest loss until 48 hours time period; the higher the moisture loss the lower the curing effectiveness. The ranking with increasing order of quality based on moisture loss is LiC < RC < LiRC < HRRC.

Dielectric Constant

Dielectric constant data of the surface concrete is presented in Figure 4. The dielectric of a material is a measure of the polarization or the electrical density that can develop within a material compared to air under an applied current. Dielectric constant readings were measured in this study using a Percometer, which produces a low frequency signal that only penetrates the concrete to a shallow depth. Consequently, the resulting dielectric reading is assumed to be applicable to only the surface concrete. Due to its sensitivity to water, high dielectric readings indicate more water is in the material than a material with a lower dielectric readings. For this reason high dielectric readings would be an indicator that water has been retained by the sample. The value indicated by the dielectric constant is considered to represent the evaporable water mainly held within the capillary pores of the concrete. The dielectric data for the different curing compounds is shown in Figure 4. As moisture is lost over time the dielectric reading diminishes as well. Based on the data shown in this figure, the dielectric reading of 15 or above corresponded to the initial water-cementitious ratio of the concrete mixture. Work by Lee showed how the surface dielectric can be used in interpret the water-cementitious ratio of the surface concrete by considering the phase of a fresh concrete in the early hardening stages.[3] The analysis for the water content was carried out on the basis that the minimum water-cementitious ratio is 0.22 but in some instances the water-cementitious ratio can exceed the initial water content during the bleeding stages.

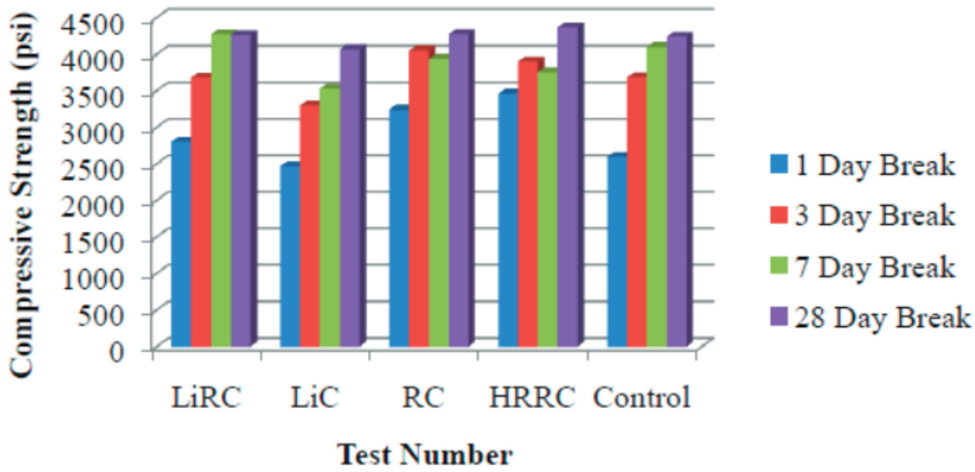

Figure 5. Strength vs. Test Number of Compressive Strength Cube Samples.

Mortar Cube Strength

Mortar cube strength data was collected on cube samples cured with the different curing compounds and presented in the bar graph above as Figure 5. The cube strength was used as a means to represent the strength of surface concrete directly exposed to the curing quality afforded by the curing compound. Based on the data shown, it is not entirely evident but the cube strengths do reflect the moisture loss and quality of curing compound to some extent.

Curing Effectiveness

Table 3 lists the performance factors considered in this study and ranks them from high to low with 1 being the best. The ranking is based on individual performance indicators for the four compounds summarized in Table 3.

This ranking shows that the HRRC curing compound showed the best results where the resin-based lithium compound showed the second best performance. Although it is not elabo-

Table 3. Ranking of Performance Indicators.

Test	Moisture Loss	Dielectric Constant (30 Hours)	Relative Humidity (Time above 80%)	Cube Strength (1 Day)
LiRC	2	2	2	3
LiC	4	3	N/A	4
RC	3	4	3	2
HRRC	1	1	1	1

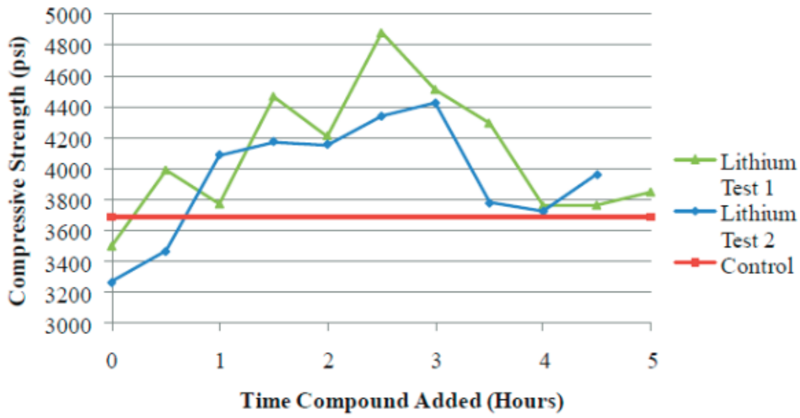

Figure 6. Strength vs. Time of Application of Lithium Compound.

rated, it is pointed out that a correlation appeared to exist between the time that the relative humidity in the filtered chamber stayed above 80% and low moisture loss. The moisture loss data showed basic trends consistent with previous curing studies carried out at the Texas Transportation Institute.[2] Samples that had less moisture loss also had longer times of relative humidity above the required 80% necessary for hydration to occur in concrete (Table 3). Additionally, samples that showed lower moisture loss also displayed signs of higher dielectric constants as expected based on results from previous work.

The primary objective of validating an approach for evaluating lithium based curing compound effectiveness characterizing the lithium compound was in this sense achieved. In regards to moisture loss, the lithium compound appears to be somewhat ineffective. The relatively high moisture loss of the lithium compound can be seen in Figure 3. These results are validated by the RC and HRRC results which are consistent with previous work.[2]

The dielectric constant readings seem to indicate that good performing compounds (low moisture loss) returned relatively high dielectric constant readings. This is consistent with previous work and further validates the data. It is noted that compounds containing lithium read higher than expected dielectric constant readings. This is believed to be due to the mechanism by which lithium interacts chemically with the concrete, subsequently elaborately.

The compressive strength of the cube samples is another indicator of performance of the curing compound. The general trend of the samples tested is consistent with the expected trends of previous work; however the magnitude of the compressive values differs by approximately a magnitude of two due to the type of sand used in the mixtures. The lithium cube strengths displayed lower cube strength characteristics in this testing series but good strength characteristics in the lithium time test studies. The general trend shows that the lithium compound (LiC) yielded the lowest strength at 1, 3, 7, and 28 days.

Characteristics of Lithium Compound Curing Mechanism

The LiRC compound tested higher than the RC product despite the fact that the LiRC product is primarily a lithium based product with a resin component; it was applied in a configuration consistent with resin based compounds. Apparently, the resin components of the LiRC held the lithium based components in place for a long enough period of time that the lithium components may have hydrated with free water in the air forming discussed gel subsequently discussed. This gel then assisted in the retardation of evaporation.

The LiC was consistently the lowest in regards to compressive strength. The LiRC displayed trends of gaining strength as curing time developed with the LiRC beginning ranked the lowest at 1 day and ending at 1 for 7 day cube strength.

Application Time for LiC

Cubes were cast and lithium compound was added at 30 minute intervals from T=0 minutes to T=300 minutes as described earlier. The results are presented in Figure 6.

The data outlined in Figure 6 shows a possible trend in the time of application of the lithium compound and the compressive strength of the concrete sample at three days. Based on the characteristics of the graph the optimal time

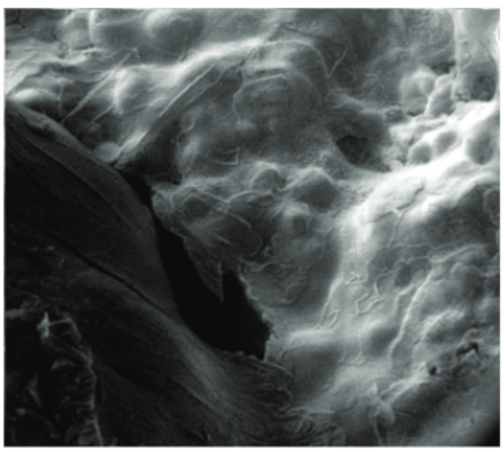

Figure 7. ESEM Image of LiC at Zero hour Application.

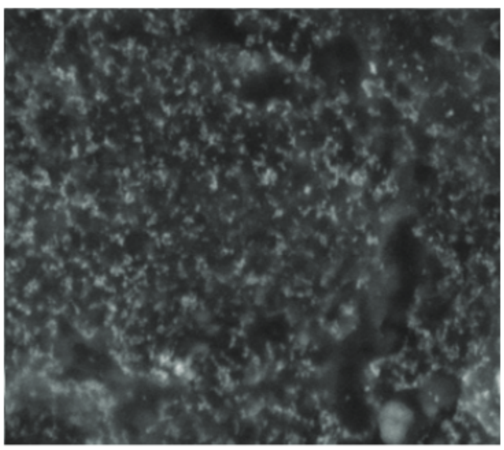

Figure 8. ESEM image of RC at Zero hour application

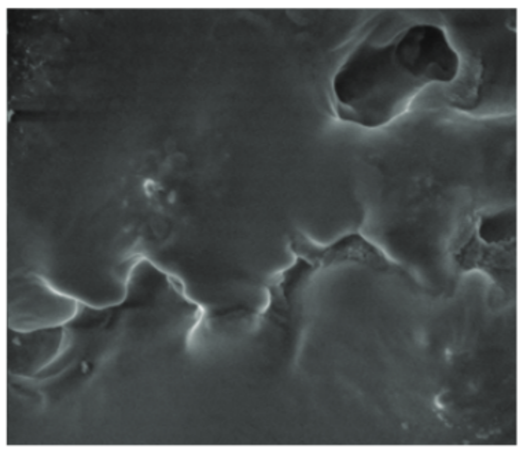

Figure 9. ESEM image of LiC at 3 Hours of application.

to apply the lithium compound is approximately 2.5-3 hours after casting based on environmental conditions.

ESEM Images of Lithium Treated Samples

The appearance of LiC at zero hours application is manifested by ESEM and presented in Figure 7. Figure 7 shows the presence of fines pores as well as cracks, which possibly facilities moisture loss. On the other hand, RC shows uniform layer without any fines pores or cracks (Figure 8).

From ESEM study, it was observed that the lithium compound formed a very tight layer without any pores or cracks (Figure 9) between application time 30 minutes and 3 hours. A compound layer of this nature has the potential to protect concrete surface from evaporation.

It was observed during testing that the lithium compounds would form a clear gel upon exposure to ambient conditions regardless of direct contact with silicates. It is believed that lithium compounds hydrate and form this gel substance with only the introduction of free water as was observed during the testing procedure. Additionally, referring to Figure 9, during investigation by scanning electron microscope the lithium was observed to have created a definite boundary layer on the treated surface of the specimen. This boundary layer offered evidence that the lithium had chemically bonded with the silicates present in the cement and that the lithium had formed a gel like substance. This gel like substance is believed to be formed when the lithium bonds with free water therefore indicating that free water was retained at the treated surface despite relatively high moisture losses. This free water held by the lithium gel would return high dielectric readings as the Percometer penetrates only the top layer of the sample.

Conclusions

Four curing compounds were evaluated for curing effectiveness based on strength, moisture loss, water retention, and relative humidity. Although drying shrinkage strain was not included in the data collection effort, the strength parameter does serve as an indicator of resistance to crack development. Nonetheless, the follow points can be made:

- Lithium curing compound (non-resin) is not effective in retarding moisture loss in the concrete curing process at an early age.
- The lithium mechanism was observed to be twofold. Evidence shows that the lithium compound does chemically combine with the silicate in the cement to form a hardened boundary layer. Evidence also shows that lithium by itself can form a hard state clear solid gel in the absence of silicate. It is believed this gel is formed from hydration with free water in the atmosphere.
- Results for RC and the HRRC were consistent with previous research.
- A trend was observed regarding a correlation between application time of lithium compound to achieve optimal compressive strength. An application time of 2.5 hours after casting was observed to return the highest compressive strength for the cube specimens.
- Lithium/resin hybrids were observed to retard moisture loss better than lithium compounds alone. This is possibly due to the resins ability to bond the lithium to the treated surface.

References

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