

**Laboratory Study of
High Performance Curing
Compounds for Concrete
Pavements - Phase I**

SPR # 0092-11-05

**Ramsey Kropp
Steven M. Cramer, Ph.D., P.E.
Marc A. Anderson, Ph.D.**

**University of Wisconsin - Madison
Department of Civil and Environmental Engineering
June 2012**

WISCONSIN HIGHWAY RESEARCH PROGRAM # 0092-11-05

**Laboratory Study of High Performance Curing Compounds for
Concrete Pavement – Phase I**

FINAL REPORT

Ramsey Kropp

Steven M. Cramer, PhD, P.E.

Marc A. Anderson, PhD

University of Wisconsin-Madison

Department of Civil and Environmental Engineering

Submitted to:

THE WISCONSIN DEPARTMENT OF TRANSPORTATION

Acknowledgements

The authors gratefully acknowledge the support of the Wisconsin Highway Research Program for the financial support of this project. Accomplishment of the research was achieved through the valued contributions of a team of experts. The research team is particularly grateful for the extensive help and collaboration of the staff of the Wisconsin Department of Transportation (James Parry, P.E.), and the Wisconsin Concrete Pavement Association (Kevin W. McMullen, P.E.). The donations of materials by material suppliers and aggregate producers were greatly appreciated and without the cooperation of these companies, this research would not have been possible. Finally, the assistance of William Lang and Carole Kraak of the Structure and Materials Testing Laboratory at the University of Wisconsin-Madison, as well as the graduate and undergraduate students housed therein, is appreciated.

Disclaimer

This research was funded through the Wisconsin Highway Research Program by the Wisconsin Department of Transportation and the Federal Highway Administration under Project 0092-11-05. The contents of this report reflect the views of the authors who are responsible for the facts and accuracy of the data presented here in. The contents do not necessarily reflect the official views of the Wisconsin Department of Transportation or the Federal Highway Administration at the time of publication.

This document is disseminated under the sponsorship of the Department of Transportation in the interest of information exchange. The United States Government and the Government of the State of Wisconsin assume no liability for its contents or use thereof. This report does not constitute a standard, specification, or regulation.

The United States Government does not endorse products or manufacturers. Trade and manufacturers' names appear in this report only because they are considered essential to the object of the document.

Technical Report Documentation Page

| | | | |
|--|--|---|---|
| 1. Report No. WHRP 12-02 | 2. Government Accession No | 3. Recipient's Catalog No | |
| 4. Title and Subtitle Laboratory Study of High Performance Curing Compounds for Concrete Pavements – Phase I | | 5. Report Date June 2012 | 6. Performing Organization Code Wisconsin Highway Research Program |
| 7. Authors Ramsey Kropp Steven M. Cramer, Ph.D., P.E. Marc Anderson, Ph.D. | | 8. Performing Organization Report No. | |
| 9. Performing Organization Name and Address University of Wisconsin – Madison Department of Civil and Environmental Engineering 2205 Engineering Hall 1415 Engineering Dr. Madison, WI 53706 | | 10. Work Unit No. (TRAIS) | 11. Contract or Grant No. WisDOT SPR# 0092-11-05 |
| 12. Sponsoring Agency Name and Address Wisconsin Department of Transportation Division of Business Services Research Coordination Section 4802 Sheboygan Ave. Rm 104 Madison, WI 53707 | | 13. Type of Report and Period Covered Final Report, 10/2010 – 6/2012 | 14. Sponsoring Agency Code |
| 15. Supplementary Notes | | | |
| 16. Abstract Three emulsion-type and two sealing-type curing compounds were evaluated for their ability to impart freeze-thaw scaling resistance, and restrict evaporation, carbonation and chloride penetration to concrete specimens prepared to represent common pavement situations. Two coarse aggregate types and three cementitious material situations were the main variables in addition to the five membrane forming curing compounds (MFCC's). The final finishing and curing compound applications occurred at a fixed-time of two hours after casting. Coarse aggregate type had little or no influence on the results but the effectiveness of the MFCCs varied with the mix type. Slag cement and fly ash mixtures displayed considerably higher levels of scaling than the ordinary portland cement mix. In addition to the base durability of each type of concrete, observed differences were also likely the result of different levels of paste moisture at the surface at the time the curing compounds were applied. The moisture sealing ability of the curing compounds did not correlate with the tendency to protect from freeze-thaw scaling, suggesting that the MFCCs alter the surface layer of the concrete in multiple ways. Overall the acrylic sealing compound provided the best protection. The interaction between the MFCC and the concrete surface is complex. These results show it is not simply a question of the effectiveness of the MFCC in sealing but rather a question of the resulting microstructure at the surface of the concrete following application of the MFCC. Thus effective concrete curing guidelines should consider the mix type and the state of the concrete surface at the time of MFCC application. | | | |
| 17. Key Words Concrete, curing, curing compounds, scaling, chloride penetration, freeze/thaw durability, evaporation | | 18. Distribution Statement No restriction. This document is available to the public through the National Technical Information Service 5285 Port Royal Road Springfield VA 22161 | |
| 19. Security Classif.(of this report) Unclassified | 19. Security Classif. (of this page) Unclassified | 20. No. of Pages | 21. Price |

Executive Summary:

Curing compounds are used to ensure adequate concrete performance, having replaced for some time traditional curing methods that are not practical for paving concrete. These curing compounds are designed to prevent evaporation of free water by forming a continuous hydrophobic membrane on the concrete surface and isolating the plastic mortar from the atmosphere. Curing compounds used for pavement projects in Wisconsin require a pigment additive to increase the reflectance of the pavement surface to decrease absorbance of solar radiation. This is to slow evaporation and minimize thermal gradients as well as allow visible verification of coating quality. These materials are typically emulsions of an organic membrane forming material and pigment in water carrier. A second class of curing compounds both improves curing by membrane formation and penetrates the concrete surface to seal and possibly alter the near surface structure. These compounds do not contain reflective pigment and are not allowed for use in concrete paving even though they provide compelling performance benefits. These treatments are allowed for non-pavement concrete projects.

Several formulations of curing compounds, both pigmented and non-pigmented, were used to prepare concrete specimens for testing. Pigmented emulsions of linseed oil or poly- α -methylstyrene monomer (PAMS) were compared to a paraffin wax emulsion that was used as a control. Non-pigmented acrylic polymer or chlorinated rubber epoxy solutions in organic solvent represented the second class of compounds. Concrete specimens were prepared using cement that consisted of Ordinary Portland Cement (OPC), 30% Class C fly ash with OPC, or 30% Grade 100 blast furnace slag with OPC. For each cement type, a set of specimens was made using crushed limestone aggregate, and a second set using igneous gravel. These aggregates were chosen to represent the mineralogy of the predominant types of concrete aggregates in Wisconsin. Two hours after pouring and after finishing the specimens were treated with one of the five MFCC formulations and allowed to cure for 28 days at 25°C and 50% relative humidity. The specimens were tested for scaling resistance in the presence of deicer solution (ASTM C672), susceptibility to chloride ion penetration (AASHTO T259), susceptibility to carbonation damage (RILEM CPC-18 modified), and analyzed using scanning electron microscopy. In addition, the effectiveness of each of the 5 tested curing compounds at preventing evaporation was tested (ASTM C156).

Among the pigmented formulations PAMS treated specimens were most resistant to scaling damage. These specimens were also resistant to chloride ion penetration but were not particularly resistant to carbonation reactions. The PAMS formulation produced a membrane that was effective at preventing evaporation. Wax treated specimens were generally resistant to scaling with the exception of samples with 30% replacement of OPC by grade 100 slag. Wax treated specimens were generally poor at preventing chloride ion penetration and were not resistant to carbonation. The wax membrane was only slightly resistant to evaporation. Linseed oil based MFCC was ineffective at preventing scaling damage, although it was moderately effective at preventing chloride ion penetration. For the non-pigmented formulations, acrylic polymer was effective at preventing scaling and carbonation, and provided moderate protection from chloride ion penetration. The chlorinated rubber product was very effective at preventing scaling and carbonation, and provided moderate protection against chloride ion penetration with the caveat that it was less effective with concrete containing Class C fly ash.

A weighted grading scheme was developed to provide an aid for choosing a proper curing compound formulation. Once an MFCC type is chosen it should be applied to a well prepared concrete surface. The concrete should have completed the bleed period before trowelling and texturing to ensure performance. Determination of the end of the bleeding period is subjective and imprecise but important, and further work is recommended to reduce the subjectivity. A state standard developed from this research will result in improved performance of concrete projects.

Table of Contents

| | |
|--|-------------------------------------|
| WISCONSIN HIGHWAY RESEARCH PROGRAM # 0092-11-05 | i |
| Acknowledgements | iii |
| Disclaimer | iv |
| Technical Report Documentation Page..... | Error! Bookmark not defined. |
| Executive Summary: | v |
| Table of Contents | vii |
| 1 Problem Statement..... | 1 |
| 2 Objectives and Scope of Study | 2 |
| 3 Background..... | 2 |
| 4 Materials, Methods, and Testing | 5 |
| 4.1 Concrete Materials..... | 5 |
| 4.2 Molds | 7 |
| 4.3 Methods | 7 |
| 4.3.1 Early Age and Compressive Strength..... | 7 |
| 4.3.2 ASTM C-672: Standard Test Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals..... | 7 |
| 4.3.3 AASHTO T-259: Standard Method of Test for Resistance of Concrete to Chloride Ion Penetration 8 | |
| 4.3.4 ASTM C-156: Standard Test Method for Water Loss from a Mortar Specimen through Liquid Membrane-Forming Curing Compounds for Concrete..... | 9 |
| 4.3.5 RILEM Carbonation Depth | 9 |
| 4.3.6 SEM Imaging..... | 9 |
| 4.4 MFCC Types and Specifications:..... | 9 |
| 4.4.1 Membrane Forming Curing Compounds (MFCCs)..... | 9 |
| 4.4.2 Curing and Sealing type compounds (CSCs) | 11 |
| 5 Results | 13 |
| 5.1 Concrete properties..... | 13 |
| 5.2 ASTM C672: | 13 |
| 5.2.1 OPC concrete with crushed limestone coarse aggregate: | 14 |
| 5.2.3 30% Fly Ash concrete with crushed limestone coarse aggregate | 15 |
| 5.2.5 30% Slag concrete with crushed limestone coarse aggregate..... | 16 |
| 5.2.7 OPC Concrete with igneous gravel coarse aggregate | 17 |
| 5.2.9 30% Fly Ash Cement with igneous gravel coarse aggregate..... | 18 |
| 5.2.11 Slag Cement with igneous gravel coarse aggregate..... | 19 |
| 5.2.13 Analysis by curing compound formulation | 20 |
| 5.3 AASHTO T-259 Results..... | 22 |

| | | |
|-------|---------------------------------|----|
| 5.4 | ASTM C156 results | 23 |
| 5.5 | RILEM Carbonation depth | 23 |
| 5.6 | SEM Imaging | 24 |
| 5.6.1 | PAMS | 25 |
| 5.6.2 | Linseed..... | 25 |
| 5.6.3 | Acrylic | 25 |
| 5.6.4 | Chlorinated Rubber..... | 25 |
| 5.6.5 | Wax..... | 25 |
| 5.6.6 | Untreated - Air Dry Curing..... | 26 |
| 5.7 | SEM Images..... | 26 |
| 5.7.1 | PAMS | 26 |
| 5.7.2 | Linseed..... | 28 |
| 5.7.3 | Acrylic | 29 |
| 5.7.4 | Chlorinated Rubber..... | 30 |
| 5.7.5 | Wax..... | 32 |
| 5.7.6 | None..... | 33 |
| 6 | Conclusions | 34 |
| 7 | Recommendations: | 37 |
| 8 | Bibliography | 39 |
| 9 | Appendix | 41 |
| 9.1 | AASHTO T-259 Results..... | 41 |

1 Problem Statement

The Wisconsin Department of Transportation has observed an increased frequency of concrete scaling on urban paving projects within the first year of service. This is particularly evident in areas where chloride laden snow storage and subsequent melting occurs over the first winter season. Scaling decreases the aesthetic value of the affected surfaces of parking lanes, curbs and gutters, driveway aprons, sidewalks, and paved terraces as well as decreasing the service lifetime of concrete pavements. Symptoms of scaling include increased surface roughness, exposure of coarse aggregates, difficult snow and debris removal, and concrete scale matter accumulation. Concrete scaling is typically associated with poor curing, improper finishing, and freezing and thawing cycles. Concrete that has been properly finished and cured will be more resistant to scaling problems and provide a more durable product, which decreases the lifetime cost of the project while maintaining aesthetics. Proper mix design will ensure adequate bulk curing but evaporation can arrest hydration in a region near the surface and improper finishing can cause excessive porosity near the surface.

The exposed concrete surface is most visible to the end user and has a large effect on the perception of the quality of the project as well as pavement durability. As curing and finishing are largely determinants of near surface durability it is vital to optimize these variables. Finishing and curing practices are determined by regulation and individual contractor practices, so developing proper regulatory framework and contractor education programs is an important step to improving infrastructure quality. In paving operations curing compounds are used to control the hydraulic environment near the concrete/air interface to prevent moisture loss and decrease the likelihood of failure patterns associated with improper curing. There are a number of formulations and suppliers of curing compounds, and the performance characteristics of several formulations were compared for this study.

A number of different curing compound formulations have been experimentally compared by measuring concrete sample resistance to freeze/thaw scaling, chloride ion penetration, resistance to evaporation, and physical appearance. A total of 5 curing compound formulations were chosen, including pigmented and non-pigmented formulations. Curing compound formulation and supplier was chosen to reflect those most widely used in Wisconsin paving projects. The three pigmented curing compound types were:

- White pigmented wax based emulsion as a control,
- White pigmented boiled linseed/linseed oil emulsion, and
- White pigmented polyalphamethylstyrene monomer emulsion.

The non-pigmented formulations included:

- Acrylic monomer based product
- Chlorinated rubber epoxy product

Of these five formulations wax emulsions and linseed oil emulsions are widely used because of their low cost and historical regulatory momentum. Newer formulations (PAMS, acrylic, and chlorinated rubber) are more expensive and may not meet regulatory guidelines for use, even though their performance may exceed more traditional formulations.

2 Objectives and Scope of Study

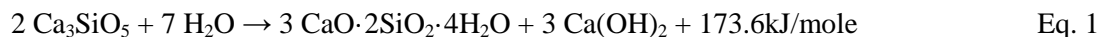
Concrete emplacement performed for WisDOT projects should provide both mechanically sound and aesthetically pleasing final products. To ensure that these goals are met WisDOT specifies performance requirements with some restrictions on material contents and sources for aggregates, cement, and other concrete-specific materials. The approved vendor list for curing compounds contains several chemistries, formulations, and suppliers, but does not specify a preferred type. Previous research has shown that there are performance differences between curing compound types and suppliers¹. This study aims to provide WisDOT with performance characteristics for several classes of curing compound with respect to cold-weather concrete performance measures. These measures include the resistance of laboratory samples to scaling, chloride ion penetration, evaporative loss of free water, and resistance to carbonation. Testing was performed on concretes prepared with Ordinary Portland Cement (OPC), 30% slag cement, and 30% fly ash cement. The two coarse aggregates, crushed limestone and igneous gravel, were chosen to represent the two primary classes of aggregates employed in Wisconsin paving operations. The specimens prepared from this matrix were treated with one of several curing compounds to provide comparison of the effectiveness of each coating formulation for each concrete type at preventing chloride related damage to exposed surfaces.

3 Background

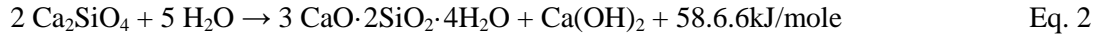
Historically, curing of the exposed surface of newly placed concrete was controlled by effective but labor intensive methods such as water ponding, covering with wet burlap, or covering with plastic sheeting. These older methods sought to improve curing by preventing the evaporation of water from exposed surfaces. The introduction of curing compounds approximately 40 years ago offered an attractive alternative to the historical curing methods. Curing compounds have developed as a class of organic membrane forming materials designed to seal the surface with a continuous hydrophobic membrane to prevent the evaporation of water in much the same way as polyethylene plastic sheeting, but provide several additional benefits.² Curing compounds require less monitoring during the initial curing period, can be applied rapidly over large areas, may contain dyes or pigments to increase aesthetic options for the designer, and form a membrane that lasts for several days to several months.

These compounds form a physical membrane that prevents the evaporation of free water in concrete. The effectiveness of these compounds depends on the integrity of the membrane, so membrane application technique is important for proper performance. If the concrete material is still producing bleed water the membrane may contain pinholes or separate from the concrete surface and fail to prevent evaporation.³ Concrete surfaces used for roadways are typically finished with an aggressive texture applied to provide adequate traction and decrease roadway noise produced by tires.⁴ This increases the surface area and accelerates the rate of evaporation. Furthermore, these textures increase the difficulty of forming a continuous membrane.^{1,5-7} The increase in surface area must be considered when applying any type of curing compound, and the sprayer should pass both parallel and perpendicular to the pavement texture to ensure that the MFCC is continuous..

The hydration reaction of cement has been studied extensively and can be summarized as shown in equations 1 and 2. Equation one is the hydration of tricalcium silicate to calcium silicate hydrate (CSH) and calcium hydroxide (CH).



Equation two is the hydration of dicalcium silicate to CSH and CH



These reactions consume large amounts of water and occur at the interface of the solid phase cement particles and liquid phase pore water. Other hydration reactions occur, but do not contribute greatly to the long-term strength of concrete.⁸ The ratio of water to cementitious material (w/cm ratio) influences both the early and long-term properties of the concrete. The stoichiometry of the hydration reactions suggests that a w/cm ratio of 0.42 provides sufficient water to completely hydrate the cement particles⁹ when considering both the chemically bound water and the gel water. In practice, other w/cm ratios are used to meet regulatory and performance targets.

The amount of liquid water present in the mortar (mixture of water, sand, and cement) dictates several early-age concrete properties. Workability describes the rheological behavior of the concrete at an early age, while the cement paste has not taken an initial set, and is principally determined by the w/cm ratio or addition of chemical admixtures. Slump cone testing is used as a field expedient method to estimate the workability of a concrete mix. Typically, easily workable concrete is easier to finish to a desired surface texture, but requires larger w/cm ratios that decrease strength and durability. Excessive workability can also hamper concrete emplacement when used with moving form machinery as in paving or curb production.

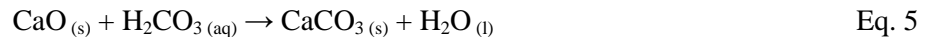
By increasing the w/cm ratio to improve workability, one can induce dramatic effects on the later properties of the concrete product. Cement mortar consists of small particles of the hydraulically active cementitious material, active fine material from the aggregates, inert fine material from the aggregate, and any inert filler, surrounded by water in the pore space. As the hydration reaction occurs and consumes water the pore space becomes a void. This void space contributes nothing to the strength of the concrete and decreases the volumetric density. The volume of the hydration products is less than that of the water initially present, so cement mortars shrink autogenously as hydration occurs, primarily at early ages before the initial formation of a gel network. This leads to an internal stress that can cause plastic cracking or increase the percolation (interconnectedness) of the final concrete pore network. Further shrinking occurs when water evaporates from gelled concrete near exposed surfaces, leading to cracking, warping, or other mechanical damage.³ Percolated voids can fill with water or de-icing solutions and will substantially decrease the concrete's resistance to freeze/thaw cycling. Water reducing admixtures (plasticizers and superplasticizers) can be added to improve the workability of the concrete slurry without increasing the w/cm ratio but complicate mix design and may affect strength and scaling resistance at high concentrations^{2,10}. Additionally, excess water not bound in pores will migrate to the concrete surfaces as "bleed water" and can delay finishing operations and increase the likelihood of forming a percolated network of surface pores⁵. The formation of bleed water is troublesome, as it delays finishing operations for concrete emplacement. The bleed water must evaporate from the surface prior to troweling to prevent an increase in the w/cm ratio near the surface and a marked loss in concrete durability.

It is not possible to completely control the w/cm ratio by simply adjusting the mass of water and cementitious materials. Other concrete components can affect the apparent w/cm ratio in the concrete pore volume. Coarse and fine aggregates physically adsorb water from the cement paste, swelling clays present in the aggregate chemically adsorb water, and pozzolanic hydration reactions can occur with the microfine fraction of the coarse or fine aggregate or with other cementitious additives. These interactions will decrease the apparent w/cm ratio and can lead to internal desiccation^{8,11}. In addition to these internal sources of water loss there is the added complication of evaporation. The hydraulic behavior of all of the concrete components and evaporation must be taken into account when considering mix design. When the concrete is in contact with a porous dry aggregate or when atmospheric water is removed from capillary space by evaporation hydration reactions are limited. Drying that occurs near an interface (between the cement paste and aggregate or at an atmospherically exposed surface) causes moisture gradients. The surface tension of water in pores causes large stress gradients in response to moisture gradients and can lead to early plastic cracking, warping, and decreased scaling resistance. This is particularly a problem in

concretes used for paving or sidewalks that have high surface area to volume ratios, and in concretes with a low w/cm ratio (0.4 or less) as would be found in continuous paving operations.¹²

Curing compounds are applied to prevent evaporation of pore water near air interfaces. These compounds consist of a hydrophobic, organic, membrane forming fraction and a carrier fraction as an emulsion or solution. After application the carrier fraction evaporates and the membrane-forming fraction is deposited onto the concrete surface. The membrane fraction may react with the atmosphere or autogenously to form chemical cross-links and increase the environmental lifetime and durability of the coating. Curing and sealing compounds (CSCs) are designed to form an evaporative barrier at the concrete surface, but also to penetrate the pore network of the concrete and further alter the near-surface pavement properties. Concretes treated with curing compounds perform differently than those cured via traditional methods, suggesting that evaporative control is not the sole method of action for concrete curing compounds¹³.

The presence of a hydrophobic membrane at the concrete surface can influence the wear, spalling, and diffusive characteristics of the pavement surface. In addition to improving the density of the near surface cement paste there is a physical barrier formed from a hydrophobic organic membrane. This membrane may act as a barrier to atmospheric carbon dioxide and limit carbonation reactions. One of the byproducts of cement hydration is solid crystals of calcium carbonate. In aqueous solutions (as found in the pore volume) this strong base will react with any acid present, such as carbonic acid formed when carbon dioxide reacts with water. In addition to the conversion of calcium hydroxide it is possible to convert calcium oxide in the CSH paste of the cured mortar (Equations 3,4 and 5).



The product of the carbonation reaction is more acidic and higher in volume than the calcium reagents. The increase in volume during this reaction creates internal tensile stress, a stress mode in which concrete is less resistant to failure. Additionally, in reinforced concrete construction the steel reinforcing bars are protected from corrosion (rusting) by a tightly coupled passive layer of iron hydroxide. The decrease in pH of the concrete system as carbonation occurs causes the conversion of iron hydroxide to weakly coupled iron oxide rust. Rust has a greater specific volume than iron or iron hydroxide, so this conversion is expansive and introduces further internal tensile stress. The combination of these stresses lead to deep spalling-type failure at locations where reinforcement is minimally covered.

The hydrophobic membrane formed by curing compounds can also serve to prevent diffusion of chloride ions into the concrete material. Chloride ions enter concrete via a number of means; bulk transport with water moving through macroscopic voids, diffusion through the pore structure, osmosis, and diffusion through the CSH paste. A consolidated and continuous membrane serves as a physical discontinuity in the system and should prevent chloride ion penetration. Chloride ions can lead to concrete damage by causing corrosion of reinforcing bars, chemical reaction with microfine fractions in the aggregates, reaction with minerals in hydrated cement paste, and reactions with bulk aggregate.

Organic materials are quickly hydrolyzed at high pH as found in concrete pore water. These reactions, coupled with accelerated weathering by sunlight and traffic, limit the environmental life span of the membrane. Sealing compounds (CSCs) that displace pore water and extend several millimeters into the surface are protected from environmental wear and will provide longer-term protection. The cumulative effects of curing compounds have substantial implication on the aesthetic and utilitarian properties of concrete. An ideal curing compound would improve all performance measures for a constructed surface

of any concrete type, but it is unlikely that any such formulation exists. Comparison of several curing compound formulations on several concrete types provides the engineer with guidelines for proper compound choice for a given project.

4 Materials, Methods, and Testing

4.1 Concrete Materials

The concrete prepared for this study consisted of a cementitious binder, a coarse aggregate fraction, and a fine aggregate fraction and conformed to WisDOT Grade A concrete for pavement. The cementitious materials were either Type (I) Ordinary Portland Cement (OPC) or a blend of OPC with 30% (mass/mass) replacement with Grade 100 ground granulated blast slag (GGBS) or Class C fly ash (FA). The total cementitious material content was 470lbs/yd³ and the coarse aggregate/fine aggregate/cementitious material ratio was 3.3:2.2:1. The coarse aggregate fraction was a WisDOT #1 stone, either crushed limestone or river run gravel. Only one source of fine aggregate was used for concrete preparation. Madison municipal water was used for all concrete mixes. A water-to-cementitious material (w/cm) ratio of 0.40 was used for OPC and GGBS concretes, but was reduced to 0.37 for FA cement blends to meet slump requirements. Low range water reducing admixture (LRWRA) was added as needed to meet the slump requirement of 3±1 in. Air entraining agent (a common synthetic polymer type) was added as needed to meet the air entrainment requirement of 6±1%.

The crushed limestone coarse aggregate obtained from southern Wisconsin was grey/green colored and had a moderately high aspect ratio of approximately 2:1 length to diameter. The material was angular and appeared soft in comparison to the gravel. The supplied aggregate met WisDOT specifications for size distribution and P200 testing as indicated in Figure 1. The igneous gravel aggregate from the Eau Claire area was multicolored and contained several mineral types including granite, basalt and a minor fraction of dolomite. The particles were nearly round with an aspect ratio of approximately 1:1 length to diameter. The material was harder and dustier than the crushed limestone from informal observation. The supplied aggregate met WisDOT specifications for size distribution and P200 content (Table 1).

Table 1: Coarse aggregate analysis

| Aggregate Type | Density | Adsorption | P200 |
|-------------------|--------------------|------------|------|
| | lb/ft ³ | % | % |
| Crushed Limestone | 164 | 1.86 | 0.8 |
| Igneous Gravel | 163 | 1.68 | 1.3 |

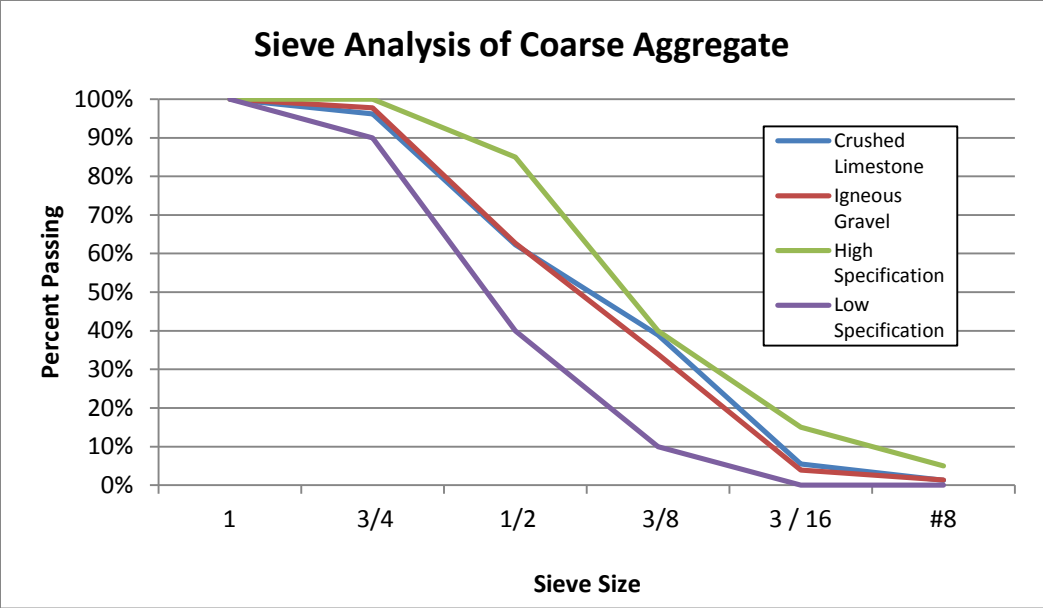


Figure 1: Sieve analysis of coarse aggregate

The fine aggregate was graded sand typical of that found in southern Wisconsin. The fine aggregate met WisDOT specifications for size analysis and P200 content (Table 2, Figure 2).

Table 2: Fine aggregate analysis

| Fine Aggregate | % Adsorption | % P200 |
|----------------|--------------|--------|
| Sand 1 | 1.21 | 3.60 |

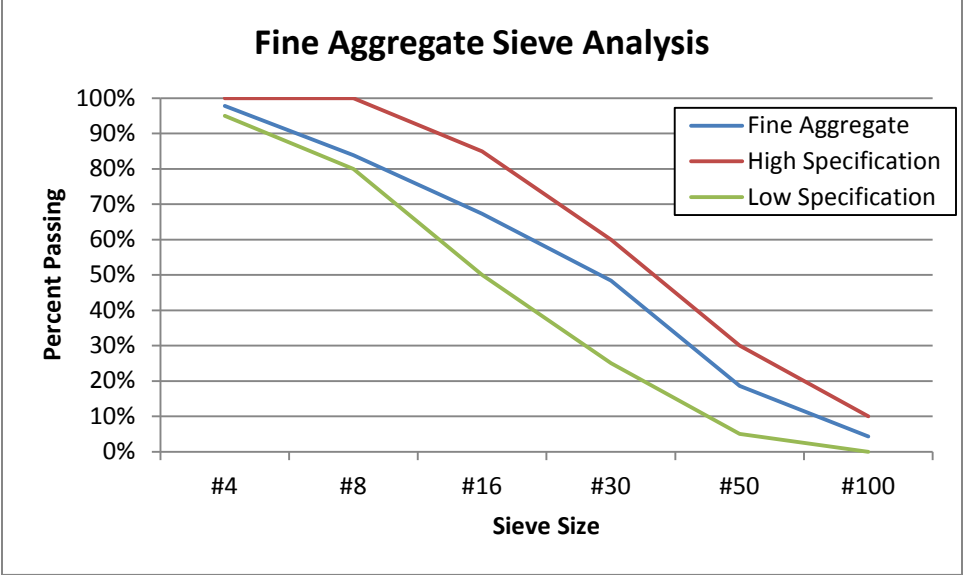


Figure 2: Fine aggregate sieve analysis

4.2 Molds

The molds used for all testing were prepared using PVC pipe of an appropriate diameter and cut to a length to provide a surface area and sample depth sufficient to meet the testing standard requirements. The PVC provided the walls of the mold and was glued to a melamine plastic coated chipboard bottom with a high strength polyurethane construction adhesive. Molds constructed in this fashion proved to be easily struck off, easily finished, non-absorbent, and highly reusable when adequate mold release compound was used.

4.3 Methods

All concrete used in this study was prepared with a rotary drum type concrete mixer in 2.5 ft³ batches as per ASTM C192. Any water reducing agent was added to approximately half of the total required batch water. The sand and coarse aggregates were mixed with the WRA/water mixture for several seconds. Cement and fly ash were then added. The air-entraining agent was combined with the remaining 1/2 of the batch water and added to the mixer. Mixing continued for two minutes after all materials were in the mix drum. The mixer was then stopped and the concrete allowed to rest for three minutes while the drum was covered to prevent evaporation. The concrete was then mixed for an additional two minutes. The concrete was poured from the mixing drum into a wheelbarrow. Any mortar sticking to the drum's interior was scraped into the concrete and hand mixed back into the concrete bulk. After hand mixing the slump and air content of the concrete were determined following test protocols ASTM C-143 and ASTM C-231. The concrete was transferred manually from the wheelbarrow to the appropriate molds using aluminum scoops. The molds were filled as per the test protocol for which they were being prepared using steel rods, rubber mallets for mold agitation, and wooden strike-off boards. Excess concrete in the molds was struck off prior to screeding. Care was taken to prevent over-working the concrete during screeding by utilizing the minimum number of passes of the striking board required for complete coverage of any coarse aggregate particles by mortar. The completed specimens were handled carefully to prevent the disturbance of any gelled network that might have formed while awaiting the end of the bleeding period.

Curing compound was applied to the surface using pressurized hand-held sprayers with nozzles chosen to provide smooth, even coverage. The specimens were placed on an electronic balance with a mask to prevent overspray from affecting the mass reading. The balance was tared and the curing compound was applied with the sprayer's nozzle perpendicular to the concrete surface, passing in alternating directions until the desired mass of curing compound was applied. The mass applied was calculated to provide a coverage rate of 200ft²/gallon for all compounds.

4.3.1 Early Age and Compressive Strength

The early age properties (slump, % entrained air, unit weight) were measured using the appropriate ASTM test and equipment prior to the preparation of any test specimens. In addition to the specimens used in the above tests a set of 4 cylinders (4 in. diameter, 8 in. height) was prepared for 28-day compressive strength testing for all mix designs. The strength specimens were demolded after 24 hours and cured in a temperature and humidity controlled space for 28 days (100% RH, 25°C). The specimens were then capped with suitable capping compound and tested to failure for compressive strength.

4.3.2 ASTM C-672: Standard Test Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals

Specimens were 12 in. diameter cylinders 3 in. deep. The specimens were leveled using a wooden strike-off board utilizing the minimum amount of finishing required to cover the large aggregate particles near the surface. The specimens were allowed to stand for a period of 2 hours prior to final finishing. Two hours was chosen as a standard time to mirror actual construction practices. High labor costs and tight

production schedules limit the time between initial emplacement of the concrete and final finish, and two hours is representative of the maximal time allowed before finishing. This period of time was insufficient to allow the complete evaporation of bleed water on the surface of some concrete blends prior to surface finishing. After the allotted time the surface was finished with three sawing motion passes of the wooden strike-off board and textured with a medium stiffness brush.

The specimens were demolded after 24 hours, labeled, and the sides and bottoms were sealed with a low-viscosity, low-modulus epoxy. Three replicates of each concrete type and treatment were prepared and allowed to cure exposed to laboratory air at $45\pm 5\%$ relative humidity and $23\pm 2^\circ\text{C}$ for 28 days. Once the specimens were prepared they were moved to a separate location for 14 days of storage at $50\pm 5\%$ RH and $23\pm 2^\circ\text{C}$ as per ASTM C672. ASTM C672 includes this second storage period to allow wet cured specimens adequate time to develop pore humidity similar to the curing compound treated specimens, as it is thought that pore humidity at the time of submersion in de-icing solution has dramatic effect on specimen performance. A 1 in. high PVC dam of 10 in. diameter was fitted to the top of the specimen to contain the deicer solution and glued in place with a polyurethane construction adhesive.

At the end of the second storage period the specimens were moved to the UW Madison Biotron facility. At this facility the specimens were subjected to a temperature cycle of 3°F for 20 hours and 93°F for four hours under a $\frac{1}{4}$ in. pond of 4% (w/w) NaCl solution. The tops of the specimen dams were loosely covered with 6 mil polyethylene film to prevent evaporation. After every fifth freeze/thaw cycle the specimens were removed from the environmental chamber and decanted over a #200 sieve. Any loose material on the specimen was rinsed off with a gentle stream of water and collected in the sieve. The scaled material was dried at 122°F in a laboratory oven and weighed with a digital balance precise to 0.01 grams. When specimens lost large fractions of surface cover it was occasionally necessary to re-glue the dam in place to prevent leaking. Care was taken to ensure that no scaled material was lost during this repair and that the dam was in the same position after repairs. The specimen was then returned to the freeze/thaw chamber. The mass of scaled material was averaged over the three specimens. No standard for an acceptable quantitative mass loss is specified for the USA, but values no greater than 500 g/m^2 are specified for Canadian roadways, and Sweden specifies a loss rate no greater than 1000 g/m^2 .¹⁴

4.3.3 AASHTO T-259: Standard Method of Test for Resistance of Concrete to Chloride Ion Penetration

AASHTO T259 specimens were 8 in. diameter cylinders 3.5 in. deep. Four specimens of each cement/MFCC type were finished and cured identically to the ASTM C672 specimens. Eight-inch diameter dams were fitted to the specimens to allow ponding under a 4% (w/w) NaCl deicer solution. After curing the specimens were moved to the UW Madison Environmental Chemistry Laboratory for long-term ponding. Every 7th day the ponding solution was removed and replaced with fresh solution. After the three-month ponding period elapsed the ponding solution was removed and the specimens were rinsed with fresh water. A rotary hammer fitted with a 0.5 in. diameter carbide-tipped masonry bit was used to pulverize the sample to a depth of 0.5 in. in 5 locations. The use of 5 drilling locations ensured that the collected material was representative of the entire sample and not unduly affected by large aggregate particles at a drill site. The pulverized material was passed through a #16 sieve, any fraction that did not pass the sieve was disposed of, and the remainder was collected for later analysis. A $\frac{3}{8}$ in. diameter bit was used to pulverize the sample in the same locations as the larger bit. The material pulverized between 0.5 in. and 1.0 in. depth was passed through a #16 sieve and collected for later analysis.

The chloride concentration of the collected material was analyzed by first digesting the cement fraction in acid and then using ion chromatographic analysis of the digesting solution to measure the acid soluble chloride concentration. Acid digestion was performed by adding 100.0 ml of 10.0% nitric acid solution, 25.0 ml of high purity water ($18.2\text{ M}\Omega/\text{cm}$ resistance), and 3.0ml of 30% hydrogen peroxide

solution to *ca.* 5 grams of pulverized sample. This mixture was then brought to a boil as quickly as possible while stirring under a watch glass. The solution was then allowed to cool to room temperature and diluted 1:10 with high purity water. This solution was analyzed for chloride ion concentration using an ion chromatography system. This system is capable of quantitative analysis at sub-ppm resolution, is automated, and high throughput in relation to electrochemical methods.¹⁵⁻¹⁸

4.3.4 ASTM C-156: Standard Test Method for Water Loss from a Mortar Specimen through Liquid Membrane-Forming Curing Compounds for Concrete

ASTM C156 mortar specimens were 6 in. diameter cylinders 1 in. in depth. These specimens were prepared from OPC and the same fine aggregate as those used for the concrete specimens. The surface was struck smooth using an oak strike off board that had been rendered non-absorbent by immersion in 200°C paraffin wax for 30 minutes. A 3mm deep groove between the specimen and the PVC mold wall was cut with a duckbill scraper and filled with paraffin wax to seal the specimen to the mold. The curing compounds were sprayed on the surface at a coating rate of 200 ft²/gallon. The specimens were weighed and inserted into an environmental chamber at 100.0±0.5°F and 32±0.5% relative humidity. After 3 days the specimens were reweighed.

4.3.5 RILEM Carbonation Depth

The mortar specimens used for ASTM C156 were placed in a 100% CO₂ atmosphere for 1 month. The specimens were then broken in half and a 1% phenolphthalein solution was sprayed on the freshly broken surface. Carbonation depth was measured as the distance from the exposed surface to the depth at which the phenolphthalein solution turned pink due to high pH. This provides a qualitative comparison of the resistance of the concrete mortar to carbonation. One drawback of this testing method is that the 100% CO₂ atmosphere was very low humidity, and humidity is known to affect the rate carbonation reactions.¹⁹

Carbonation only occurs when CO₂ is converted to carbonic acid in the presence of liquid water (Equation 3). Carbonation depth is controlled by the diffusion of CO₂ and not the rate of carbonation, while the damage due to carbonation is influenced most strongly by the rate of carbonation and the age of the concrete. Carbonation damage is therefore most likely in moist concrete, particularly if that concrete is close to a point source of CO₂. The method used to measure carbonation depth relied on a chemical indicator and not a measurement of the degree of carbonation. In well-cured concrete, particularly near the surface, the pore space is largely free of liquid water and CO₂ is free to diffuse rapidly, so the lack of humidity should not unduly affect the carbonation depth reported.

4.3.6 SEM Imaging

The specimens used for AASHTO T259 testing were sawed into 1 cm³ samples with one side as the treated surface. These specimens were exposed to deicer solution for a period of 3 months but not exposed to temperature extremes and represent mature pavement surfaces. Hydration of the specimens was halted by immersion in acetone and vacuum storage. Specimens used for imaging the interface were immersed in epoxy resin. The hardened resin and concrete was polished to expose the structure of the mortar near the interface. All samples were then sputter coated with platinum metal to increase their conductivity. SEM imaging was performed under high vacuum using a 10kV electron beam and a back-scatter electron detector and X-ray detector for elemental analysis.

4.4 MFCC Types and Specifications:

4.4.1 Membrane Forming Curing Compounds (MFCCs)

Strictly membrane forming curing compounds (MFCCs) consists of an emulsion of microscopic spheres or “bubbles” of membrane forming organic compounds and pigment suspended in water. As the water evaporates the emulsion breaks (“bubbles pop”) and deposits the organic fraction onto the surface as a continuous or semi-continuous membrane to prevent evaporation. This organic fraction can be an inert material, reactive with atmospheric oxygen, or autogenously reactive (self-polymerizing). Emulsions are prone to separation during long-term storage and should be used within the manufacturer’s specified time frame.

4.4.1.1 Wax Emulsion:

The wax based curing compound used for this study was a white-pigmented emulsion of refined petroleum wax and titanium dioxide pigment (1-5% pigment by weight) in water. It meets ASTM C-309 specifications as a Type II Class B curing compound for use on concrete slab work and flat work with a high surface area to mass ratio. The emulsion has a specific gravity between 0.97 and 1.03 g/ml, similar to the water carrier. The manufacturer specifies a 15 to 25% solid fraction. This compound has a viscosity slightly greater than that of water and little odor. As a water emulsion it emitted few VOCs and required no special handling. The manufacturer did not provide reflectance data. The product was bright white in color. This coating was easy to atomize in a sprayer. The emulsified fraction of the material had a tendency to float on the surface of bleed water at the edges of the concrete form. Additionally, on some specimens there was a network of cracks or crazing that formed in the membrane as the material dried. No trend for the formation of this crazing was found; its presence or absence appeared to be random with respect to cement or aggregate type. Crazing of this type is commonly ascribed to application of the compound to still-bleeding concrete specimens.²⁰ Crazing can also occur if the curing compound is applied late and the network of cracks from concrete shrinkage are mirrored on the curing compound surface.

4.4.1.2 Linseed Oil Emulsion:

The linseed oil based curing compound used in this study was a white-pigmented emulsion of boiled linseed oil and titanium dioxide pigment (1-5% pigment by weight) in water. It met ASTM C-309 specifications as a Type II Class B curing compound for use on concrete slab work and flat work with a high surface area to mass ratio. The emulsion had a specific gravity between 0.97 and 1.03 g/ml, similar to the water carrier. The manufacturer specified a 40 to 50% solid fraction. This compound had a viscosity markedly greater than water and emitted a mild distinctive odor. As a water emulsion it emitted few VOCs and required no special handling but has a limited shelf life. This coating provided a reflectance of 67% and had a yellowish tint. Linseed oil is a “drying oil” in which a chemical reaction occurs to consolidate the membrane on the surface. Boiling the linseed oil promotes this chemical reaction and increases the rate of chemical cross linking. This reaction is exothermic, but the small quantities involved do not affect the bulk concrete temperature. This coating was moderately difficult to atomize.

4.4.1.3 PAMS Emulsion:

The polyalphamethylstyrene (PAMS) resin based curing compound used in this study was a white-pigmented emulsion of PAMS resin and titanium dioxide pigment (1-5% pigment by weight) in water. It meets ASTM C-309 specifications as a Type II Class B curing compound for use on concrete slab work and flat work with a high surface area to mass ratio. The emulsion had a specific gravity between 0.97 and 1.03 g/ml, similar to the water carrier. The manufacturer provided a solid fraction of 52.5%. This compound has a much higher viscosity than water and has minimal odor. The manufacturer provided a reflectance value of 68%. The product had a bright white color. As a water emulsion it emits few VOCs

and requires no special handling. PAMS resin undergoes cross-linking reactions at sufficient concentration, so as the water evaporates the coating consolidates and seals the surface. This coating was the most difficult to atomize.

4.4.2 Curing and Sealing type compounds (CSCs)

The curing and sealing compounds (CSCs) used for this study were solutions of membrane forming organic compounds in mixed organic solvents. The organic solvent evaporates during application of these compounds and enters the atmosphere as VOCs. The CSCs tested met EPA standards for VOC emissions and contained no pigmentation. They are designed to both form an evaporative barrier and penetrate the porous network of the concrete to a significant degree. This seals the concrete surface and prevents the ingress of water or deicer solution. These coatings were highly hydrophilic. As they are a solution instead of an emulsion, CSCs have a longer shelf life than emulsion type MFCCs.

4.4.2.1 Chlorinated Rubber Epoxy:

The clear chlorinated rubber (CR) based curing compound used in this study is a copolymer resin of chlorinated rubber and an unspecified epoxy in an organic solvent (mixed xylenes and ethylbenzene). It meets ASTM C-309 specifications as a Type I Class A/B curing compound and also ASTM C-1315-95 Type I Class B/C sealing compound. The compound has a specific gravity of 0.97g/ml, similar to water. The manufacturer provides a solid fraction of 29%. This compound had a greater viscosity than water and a pronounced odor of organic solvents. There was no pigmentation added to the compound, but the manufacturer states that it provides a semi-gloss finish. The product was transparent with a slight yellowish tint.

The VOC emissions from this coating are very flammable and hazardous. Adequate ventilation and removal of ignition sources were necessary for safe use in the laboratory. Equipment for applying this compound should be designed to minimize ignition sources, provide provisions for remotely halting spraying and allowing access to firefighting personnel and equipment in the case of fire, and protect the operator from exposure to chemical fumes. Workers applying this compound might require personal protective equipment (PPE) if working in stagnant air or enclosed environments. The strong odor and potentially harmful nature of this compound might pose a nuisance in residential areas or near populations of sensitive individuals. ASTM specifications dictate that the surface be dry to the touch after 4 hours, so these VOC emissions occur primarily during and immediately after spraying. After one hour the VOC odor was no longer present

This compound was an organic solution of chlorinated rubber and an unspecified epoxy designed to form a membrane for evaporative control as well as penetrate the concrete surface to provide improvements to several concrete properties. As the volatile fraction evaporates the chlorinated rubber forms a membrane on the surface. It is unknown if there are cross-linking reactions in the chlorinated rubber membrane. The epoxy fraction is designed to penetrate the pore network of the concrete (up to 3/16 in. on already cured concrete as per the manufacturer) and autogenously cure. This compound was moderately difficult to atomize, and due to the lack of pigmentation it was difficult to rate the thickness or quality of the membrane formed. The low flash point, high volatility, and dense vapors made coating operations somewhat difficult, as the potential exists for vapor explosions or health effects to occur. The application rate of 200ft²/gallon was greater than the manufacturer's specification of 300 ft²/gallon.

4.4.2.2 Clear Acrylic:

The acrylic based curing compound used in this study was a clear, colorless copolymer of acrylic and methacrylate resins in an organic solvent mixture (aromatic distillates, trimethylbenzene, cumene, and mixed xylenes). It met ASTM C-309 specifications as a Type I Class A/B curing compound and also ASTM C-1315-95 Type I Class B/C sealing compound. The compound had a specific gravity of

0.91g/ml, much lower than water. The manufacturer did not provide a solid fraction, but it was calculated to be 25% based on the MSDS value for the VOC emissions and the density. The compound had a lower viscosity than water and a pronounced odor of organic solvents. There was no pigmentation added to this compound, but the manufacturer states that it provides a hard, shiny “bright” surface. No comprehensive testing of the wet and dry traction modifying behavior of surfaces with this compound were performed or known to be available, but the surface felt similar to that of the other coatings.

The VOC emissions from this coating are known to be potentially flammable and hazardous. Adequate ventilation and removal of ignition sources were necessary for safe use in the laboratory. Considerations for use of this compound include application equipment that does not introduce ignition sources, provisions for remotely halting spraying and allowing fire suppression, and protection for the operator from exposure to chemical fumes. Workers applying this compound might require personal protective equipment (PPE) particularly if working in stagnant air or enclosed environments. The strong odor and potentially harmful nature of this compound might pose a nuisance in residential areas or near populations of sensitive individuals. ASTM specifications dictate that the surface be dry to the touch after 4 hours, so these VOC emissions occur primarily during and immediately after spraying. After one hour the VOC odor was no longer present.

This compound was a solution of acrylic and methacrylate monomers in an organic solvent. It is designed to cure, seal, and harden concrete by both forming a surface membrane and penetrating the pore network of the near-surface concrete. As the organic solvent evaporates the concentration of the monomers increases until a chemical reaction occurs and consolidates the membrane. This compound was easily atomized, but the lack of pigmentation prevented rating the thickness or quality of the membrane. The low flash point, high volatility, and dense vapors made coating operations somewhat difficult, as the potential exists for vapor explosions or health effects to occur. The application rate of 200ft²/gallon was greater than the manufacturer’s specification of 300ft²/gallon.

5 Results

5.1 Concrete properties

All concrete prepared for this study exceeded 4500 PSI compressive strength at 28 days and met the required slump and air entrainment values of 3 ± 1 in. and $6\pm 1\%$ respectively. The w/c ratio was decreased for the fly ash blends to meet the slump requirements, as fly ash blends were more workable than other cement types. Air values are corrected to account for void volume in the aggregate fraction.

Table 3: Fresh concrete properties and compressive strength

| Mix | SCM Type | Coarse Aggregate Type | w/cm Ratio | Avg. Slump | % Air | Avg. Comp. Strength |
|-----|----------|-----------------------|------------|------------|-------|---------------------|
| | | | | Inches | | PSI |
| 1 | None | Limestone | 0.40 | 2.44 | 5.2% | 4590 |
| 2 | Fly Ash | Limestone | 0.37 | 2.50 | 5.1% | 5218 |
| 3 | Slag | Limestone | 0.40 | 2.19 | 5.3% | 5447 |
| 4 | None | Gravel | 0.40 | 2.15 | 5.3% | 4707 |
| 5 | Fly Ash | Gravel | 0.37 | 2.63 | 5.8% | 5097 |
| 6 | Slag | Gravel | 0.40 | 2.65 | 6.0% | 4948 |

5.2 ASTM C672:

ASTM C672 defines a test method for scaling resistance of concrete exposed to de-icing solutions. A total of 60 freeze/thaw cycles were performed on concrete specimens under a solution of 4% NaCl. The material that scaled from the surface was collected every five cycles, dried, and then weighed to provide a quantitative measure of the severity of scaling type damage. Each chart shows the cumulative mass lost and is paired with a chart of the standard deviation between the replicate samples. High standard deviation values that revert to lower values correspond to data sets in which one sample of a concrete/treatment type suffered surface failure earlier than the other replicate samples. Regions of high standard deviation that do not revert are indicative of a possible outlier for that treatment method.

5.2.1 OPC concrete with crushed limestone coarse aggregate:

OPC concrete with crushed limestone aggregate performed the best of all of the mixes. PAMS and Wax coating showed a steep initial scaling loss and continued to lose mass during the entire test. The CR and acrylic coatings performed very well, and the rate of mass loss was near zero for approximately half of the test. These specimens finished the test having only lost a small amount of material; typically thin sections of mortar over large aggregate particles near the surface. The linseed-based coating began to rapidly lose mass near the end of the testing cycle, but still finished the test with large sections of undamaged surface.

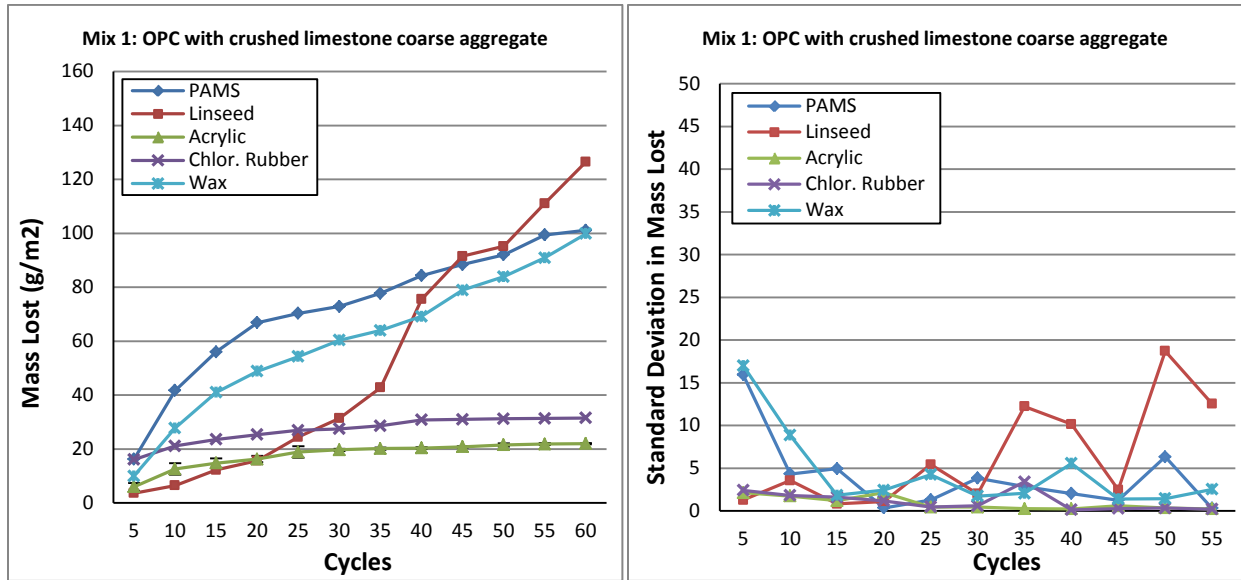


Figure 3: Scaling mass loss for OPC concrete with crushed limestone aggregate

5.2.3 30% Fly Ash concrete with crushed limestone coarse aggregate

Replacing 30% of the cementitious material with Class C Fly Ash dramatically decreased the concrete's resistance to scaling and resulted in mass losses almost an order of magnitude greater than the comparable OPC specimens. Fly ash greatly increased the slump value of the mix and necessitated the use of a lower w/cm ratio. This decreased the rate of bleeding and produced a concrete that was still very plastic after two hours. The lower w/cm ratio may have made the specimens more sensitive to surface evaporation. Concretes containing fly ash require longer curing times, as the fly ash fraction hydrates via slower pozzolanic reactions. If the OPC fraction of the cementitious material consumes the free water, the pozzolanic reactions will not occur and the paste will not develop adequate strength and have a high porosity^{21,22}.

PAMS, CR, and acrylic MFCC treated specimens lost large amounts of finished surface within the first 10 days of testing. This represented complete surface failure, in which the entire surface crumbled to a depth of several millimeters. Once the surface had failed the mass loss rate dramatically decreased. The near-surface environment, where curing was most influenced by evaporation, performed poorly in relation to adequately cured regions. This suggests that the curing compound did not adequately control the loss of water to evaporation during the curing period or altered the surface structure of the specimens. Wax specimens performed well in comparison to the other treatment types. Linseed oil treated specimens showed a similar trend to the OPC mix, in that specimens performed well for the early portion of the test but continued to lose mass during the entire testing time. Wax treated specimens performed better than the other curing compound types and had areas of undamaged surface remaining at the end of testing.

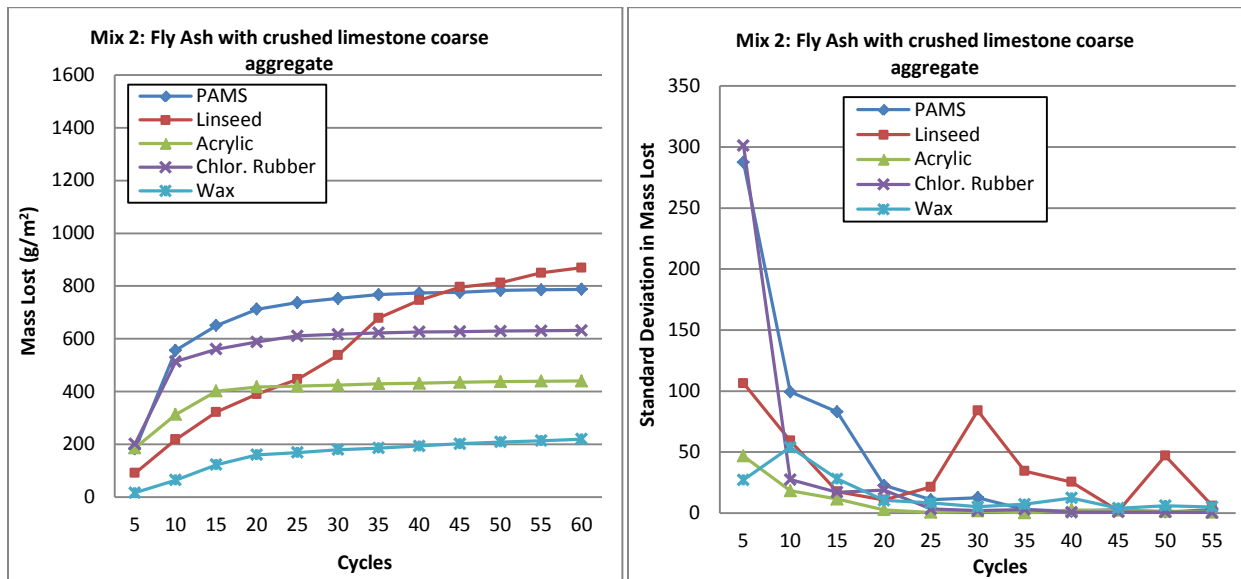


Figure 4: Scaling mass loss for 30% Fly Ash concrete with crushed limestone aggregate

5.2.5 30% Slag concrete with crushed limestone coarse aggregate

Specimens prepared with grade 100 ground-granulated blast furnace slag (slag cement) also showed a dramatic decrease in resistance to scaling but generally performed better than the fly ash specimens. A replacement rate of 30% exceeds design guidelines for concrete exposed to deicer solutions¹⁴. Slag cement is rated by grade, where the grade number is the percent ratio of the compressive strength of a mortar cube made with a 50% ground granulated blast furnace slag-cement blend to that of a mortar cube made with OPC. Slag cement is a very fine powder and produced concrete that had distinctly gelled after 2 hours. This gelling decreased the bleed rate of the concrete and the sample surface had lost the “shiny” appearance associated with bleed water at 2 hours. PAMS, CR, and acrylic MFCCs had regions of undamaged surface remaining at the end of testing, while wax and linseed treated specimens failed to a depth of several millimeters across the entire surface.

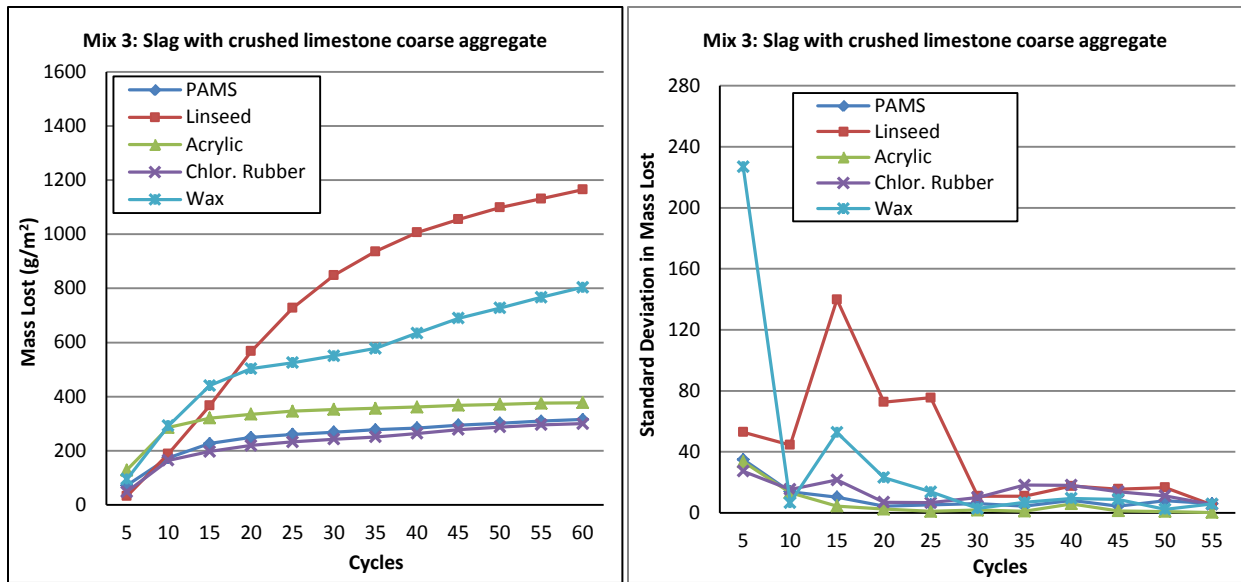


Figure 5: Scaling mass loss for 30% slag concrete with crushed limestone aggregate

5.2.7 OPC Concrete with igneous gravel coarse aggregate

With the exception of the linseed oil treated samples the OPC specimens with igneous gravel coarse aggregate behaved similarly to the specimens prepared with crushed limestone. There was some chert present in the coarse aggregate and this mineral failed under the test conditions. When aggregate particles failed near the surface it resulted in a loss of the mortar cover over the particle and eventual loss of the entire coarse aggregate piece, leaving a negative impression of the stone behind. This introduced flaws and stresses into the surface and may account for the increased mass loss rates. The wax and acrylic treated specimens finished testing with large areas of surface undamaged. The chlorinated rubber specimens lost an appreciable amount of surface early in testing but stabilized rapidly. The PAMS treated specimens performed well until beginning to lose large areas of surface cover after 50 freeze/thaw cycles. The linseed based specimens performed well until beginning to lose large areas of surface cover after 50 freeze/thaw cycles. The linseed based specimens lost cover completely to a depth of several millimeters.

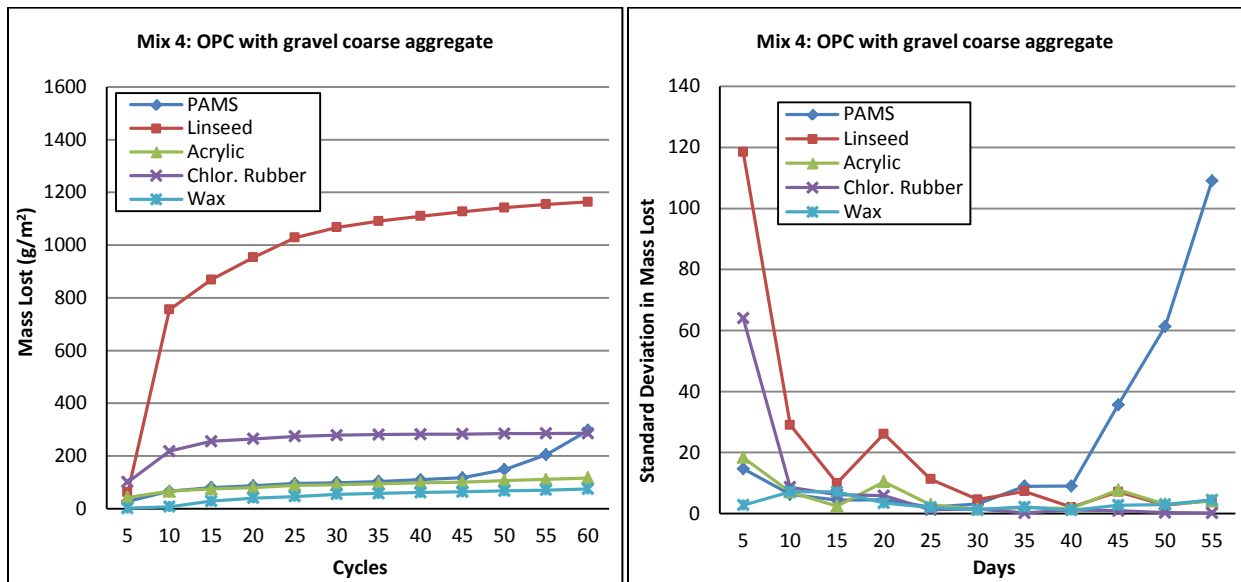


Figure 6: Scaling mass loss in OPC concrete with igneous gravel coarse aggregate

5.2.9 30% Fly Ash Cement with igneous gravel coarse aggregate

Specimens prepared from concrete with a 30% (w/w) replacement of OPC with fly ash and igneous gravel coarse aggregate performed similarly to those prepared with crushed limestone aggregate. Replacing 30% of the cementitious material with Class C Fly Ash dramatically decreased the concrete's resistance to scaling. Fly ash greatly increased the slump value of the mix and necessitated the use of a lower W/CM ratio.

Chlorinated rubber and linseed oil treated specimens lost large amounts of finished surface within the first 10 days of testing. This represented complete surface failure, in which the entire surface crumbled to a depth of several millimeters. Specimens cured with PAMS completely lost surface cover by 30 freeze/thaw cycles. Wax and acrylic cured specimens performed well in comparison to the other treatment types. The acrylic treatment appeared much more effective when used on a 30% FA concrete with gravel aggregate than on 30%FA concrete with crushed limestone aggregate.

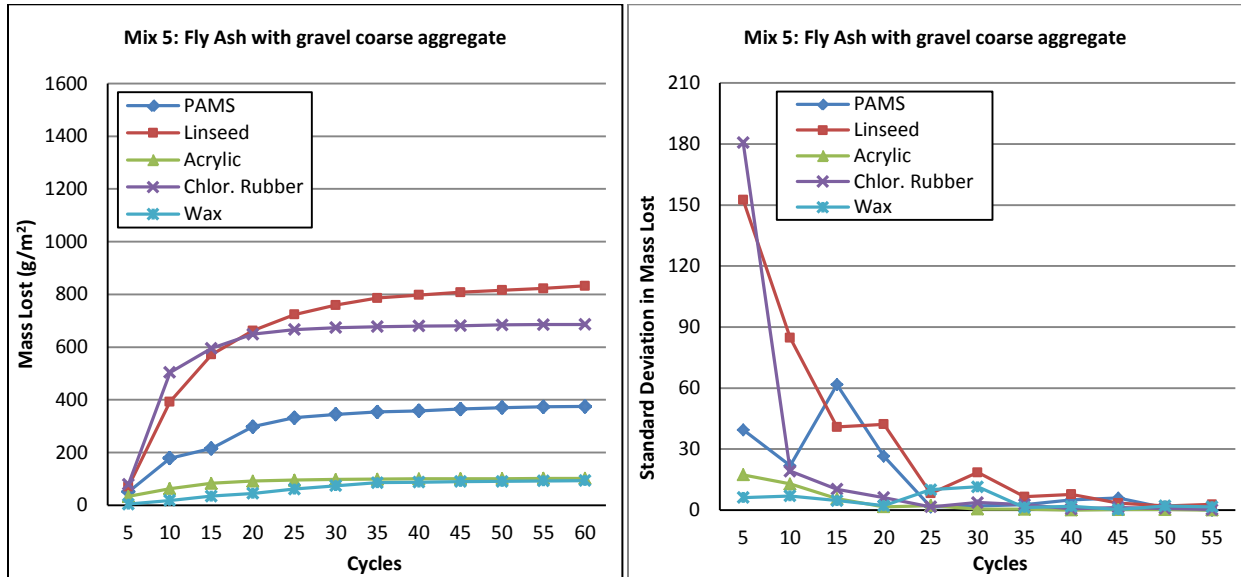


Figure 7: Scaling mass lost for 30% Fly Ash concrete with igneous gravel aggregate

5.2.11 Slag Cement with igneous gravel coarse aggregate

The performance of slag cement concretes with igneous gravel coarse aggregate was similar to that of those prepared with the crushed limestone aggregate. Specimens treated with Linseed or Wax based compounds failed completely during testing. Chlorinated rubber or Acrylic curing compounds produced concrete that was moderately resistant to scaling. The PAMS treated specimens performed very poorly in relation to the crushed limestone aggregate samples, particularly after 35 cycles. The high standard deviation in the mass lost for PAMS treated specimens shows that the failure occurred in single specimens at 25, 35, and 50 cycles, and the final specimen began to lose cover at the end of the test.

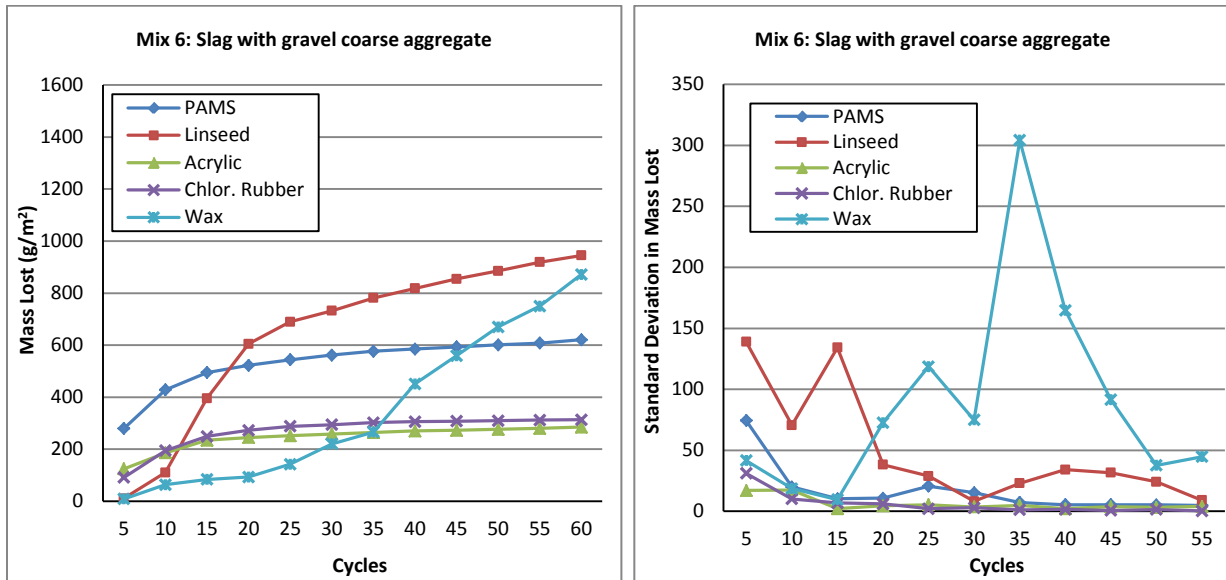


Figure 8: Scaling mass lost for 30% Slag concrete with igneous gravel aggregate

5.2.13 Analysis by curing compound formulation

Linseed oil based MFCC performed the worst of the five types tested for all concrete blends, although its performance was comparable to that of PAMS and wax based treatments for OPC concrete with limestone aggregate. Wax based MFCC was effective on fly ash concrete with crushed limestone coarse aggregate but was ineffective for preventing scaling with slag cements. PAMS MFCC was effective at moderating scaling damage on OPC concrete, but binary blend concretes were less resistant to scaling. Acrylic and chlorinated rubber epoxy based CSCs performed well in relation to the MFCCs except in the specific case of chlorinated rubber on fly ash concrete. Acrylic was the most reliable performer across the different mix types. This variability from mix to mix could reflect the differences in surface conditions at the time the curing compounds were applied.

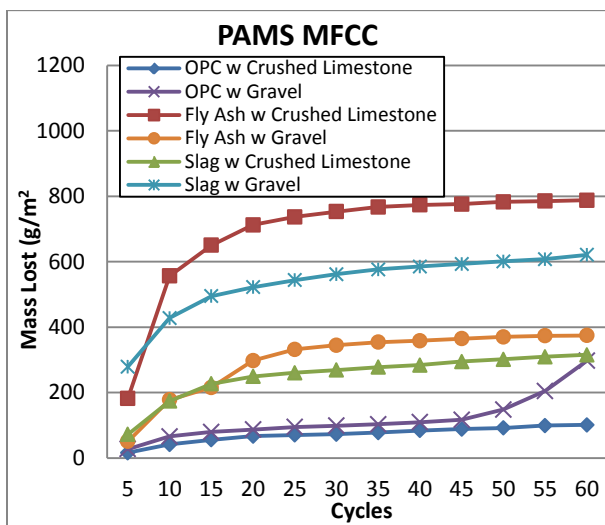


Figure 9: PAMS based MFCC performed well on concretes prepared with OPC but was less effective for binary cement mixtures

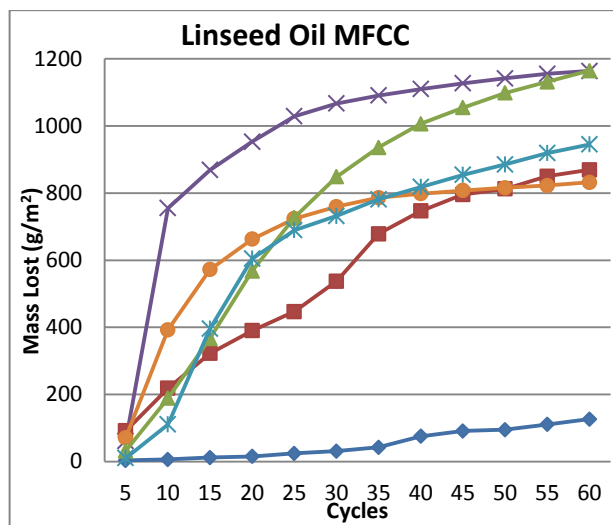


Figure 10: Linseed oil based MFCCs were extremely susceptible to scaling damage with the exception of OPC cement with limestone aggregate

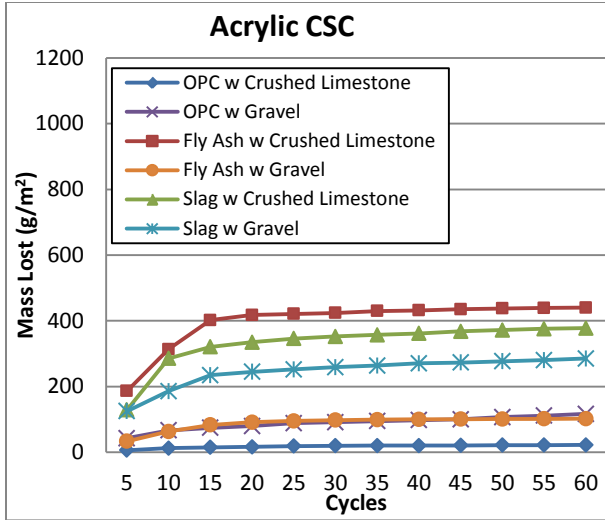


Figure 11: Acrylic based CSC produced concretes very resistant to scaling except for binary mixtures with crushed limestone

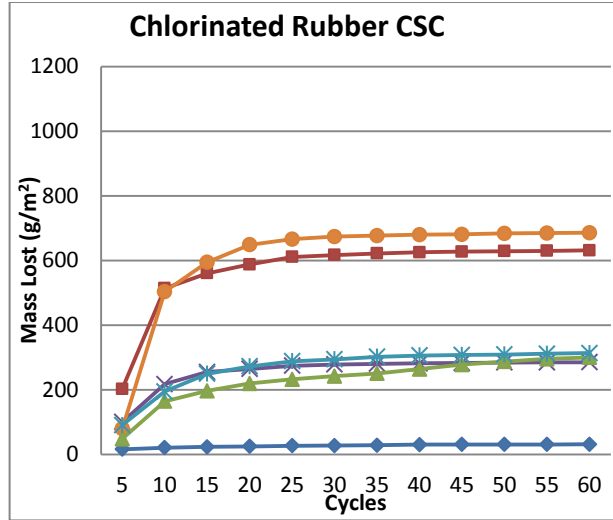


Figure 12: Concrete treated with chlorinated rubber CSC performed moderately well with the exception of fly ash cements, where performance was relatively poor

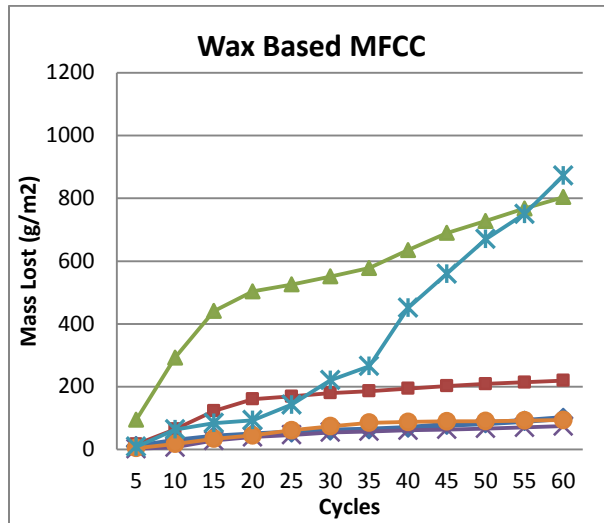


Figure 13: Concrete treated with paraffin wax based MFCC was highly resistant to scaling with the exception of binary blends with Grade 100 GGBFS

5.3 AASHTO T-259 Results

There was little variation in chloride ion penetration between cement types or curing compound applied with the exception of Wax based MFCC. Generally, there was a slight decrease in chloride ion penetration in specimens prepared with gravel coarse aggregate at both depth ranges. This may be due to the greater angularity and aspect ratio of the limestone coarse aggregate. The porous region of mortar near the mortar-aggregate interface (Interfacial Transition Zone, ITZ) may allow rapid deicing solution transport along aggregate surfaces and specimens with a large aspect ratio, as these aggregates pack less densely, extend further into the bulk concrete, and provide a highly porous pathway deep into the concrete specimen. Another possible explanation for the increased chloride ion penetration is the presence of reactive microfine material decreasing the strength of the aggregate-mortar bond and allowing bulk transport along decoupled interfaces.

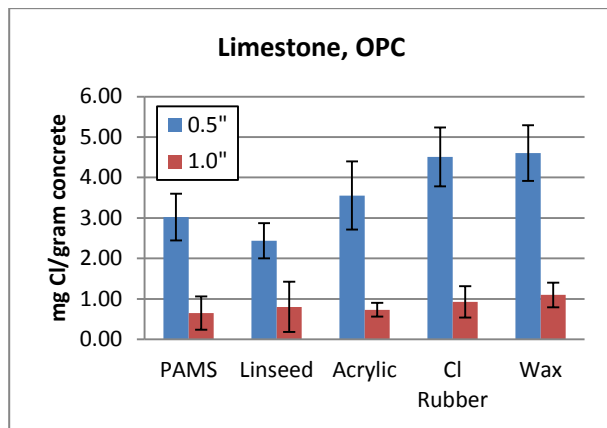


Figure 14: There is little variation between the curing compounds for OPC/Limestone specimens. Chlorinated rubber and wax emulsion appeared slightly more susceptible to chloride ion penetration

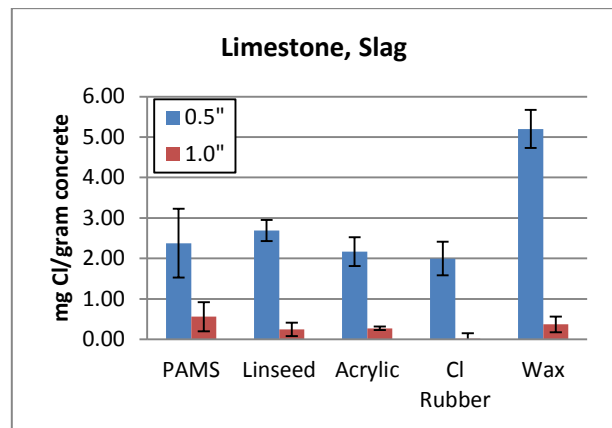


Figure 16: Wax emulsion MFCC was appreciably more susceptible to chloride ion penetration

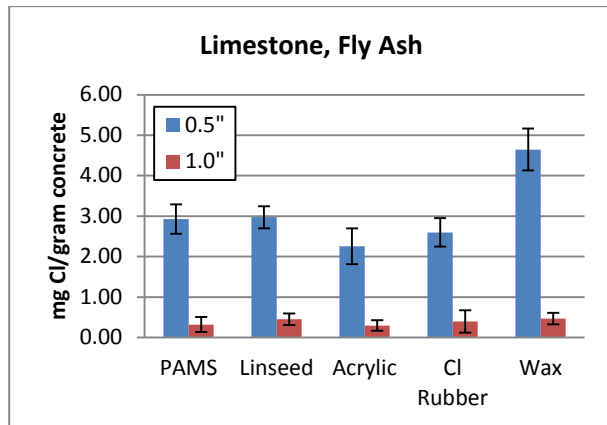


Figure 15: Wax emulsion MFCC was appreciably more susceptible to chloride ion penetration for OPC/Limestone concrete specimens

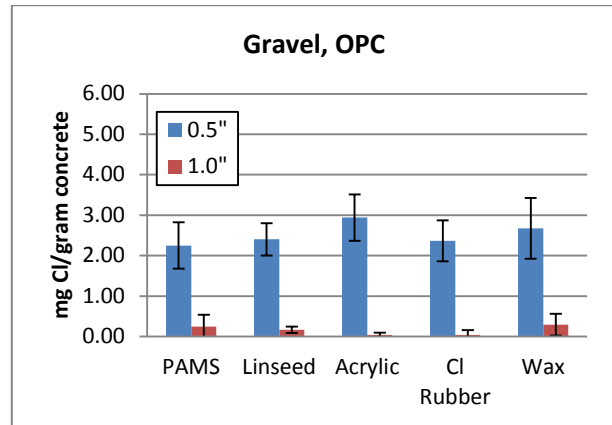


Figure 17: OPC/gravel concrete was slightly less susceptible to chloride ion penetration, including specimens treated with wax emulsion. The lower 1 in. depth concentration suggests improved resistance in the first 0.5 in. of mortar

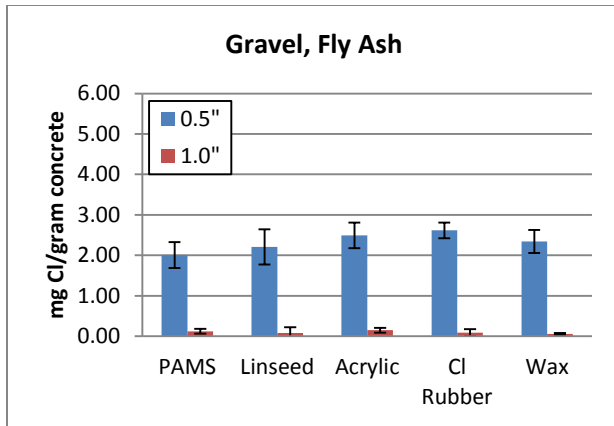


Figure 18: Fly Ash/gravel concrete was slightly less susceptible to chloride ion penetration, especially when treated with wax MFCC

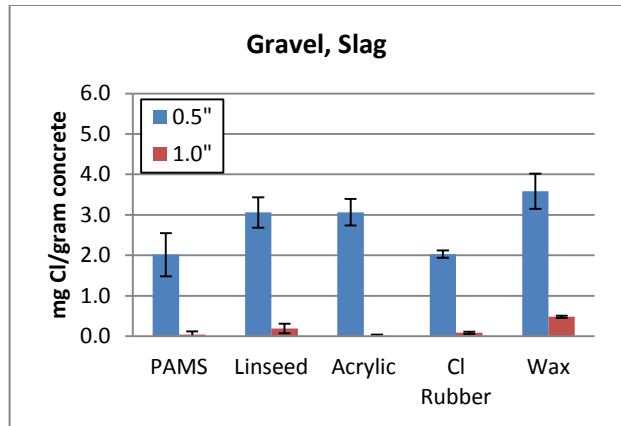


Figure 19: Wax MFCC treated specimens were slightly less susceptible in gravel coarse aggregate specimens

5.4 ASTM C156 results

OPC mortar specimens were prepared with each curing compound type and the evaporative control performance for each formulation was measured. Samples that were untreated were included as an additional reference point. Wax and Linseed oil based MFCCs were very slightly effective at preventing moisture loss. PAMS MFCC was the most effective of the strictly curing compound formulations, decreasing evaporation by 28%. The CSCs produced specimens much more resistant to evaporative loss.

Table 4: Evaporative mass lost during 3 day curing period in 37.8°C and 32% RH air

| Coating Type | Coating Rate | Water Loss Rate | S.D. | % Reduction in water loss rate |
|--------------------|----------------------|-------------------|-------------------|--------------------------------|
| | ft ² /gal | kg/m ² | kg/m ² | |
| None | NA | 3.69 | 0.16 | Reference |
| PAMS | 200 | 2.65 | 0.14 | 28% |
| Wax | 196 | 3.39 | 0.22 | 8% |
| Linseed | 201 | 3.11 | 0.11 | 16% |
| Chlorinated Rubber | 199 | 0.81 | 0.03 | 78% |
| Acrylic | 197 | 1.16 | 0.04 | 69% |

5.5 RILEM Carbonation depth

The specimens prepared for ASTM C156 testing were left in the mold after three days exposure to 37.8°C and 32% RH air. They were placed in a sealed polyethylene bag and the atmosphere was swapped several times with CO₂. Every 3 days, for a period of one month, the atmosphere of the bag was exchanged with CO₂. After 28 days exposure to 100% CO₂ the specimens were removed from the sealed mold, broken in half, and sprayed with a 1% phenolphthalein solution. This solution is pink in basic media and changes to colorless in carbonated concrete. The depth of carbonation is measured from the surface to the pink/clear

transition point. This accelerated protocol provided a rapid, quantitative comparison of the carbonation damage between the specimens used for this test, but should not be compared with other research groups without correction for atmospheric composition. A potential drawback of this accelerated testing protocol is that the CO₂ atmosphere did not contain any humidity, which is known to affect carbonation reaction rates.¹⁹

The MFCCs actually decreased the specimen's resistance to carbonation in comparison to an untreated specimen, in particular the linseed treatment. CSCs did provide protection against carbonation reactions, decreasing the carbonation depth by 50%. This is likely due to the treatment penetrating the mortar pore structure near the surface, sealing it, and protecting the concrete from the atmosphere.

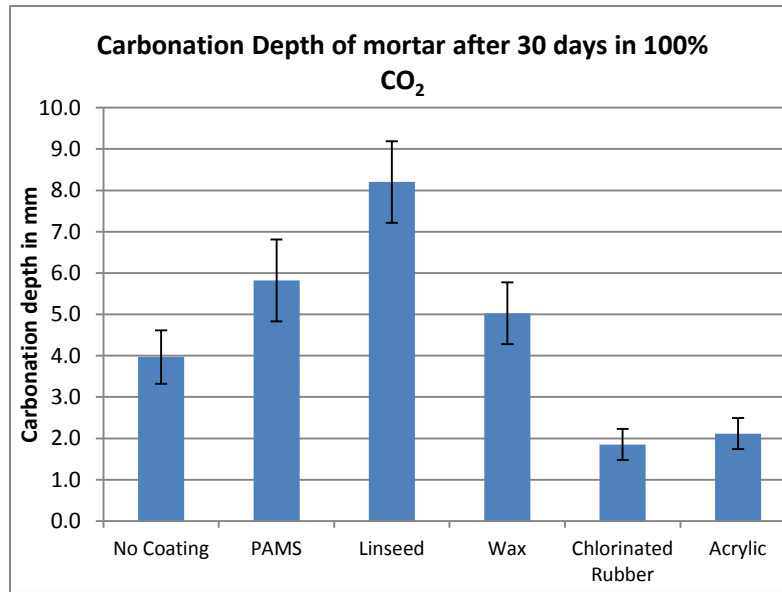


Figure 20: Carbonation depth of mortar specimens prepared with OPC and treated with each curing compound type

5.6 SEM Imaging

SEM analysis was performed for OPC specimens with crushed limestone aggregate to detect microstructural changes in the concrete near the air interface due to curing with the differing curing compound types. The differences in performance in the ASTM C672 scaling testing for the different curing types was not well correlated with performance in ASTM C156 moisture loss resistance testing, implying that evaporative control is not the sole mechanism by which curing compounds influence concrete performance. Images were taken near the air interface, as well as at a depth of 0.5in to compare the near surface effects for each curing compound as well as any potential bulk concrete effects.

The specimens used for imaging were obtained from the mix 1(OPC and crushed limestone coarse aggregate) specimens after AASHTO T259 was complete. These specimens were stored for three months under deicer solution at room temperature, and as such they represent mature specimens that had not been stressed by thermal cycling. Large specimens were sectioned on a diamond concrete saw to produce 0.5×0.5×0.5in. cubes with the air interface as one surface. These small specimens were submerged in acetone and vacuum dried to halt hydration. They

were then embedded in a low viscosity epoxy resin, exposed to medium vacuum ($\sim 10^{-4}$ Torr) for several minutes to remove air from the pore space, then re-exposed to atmospheric pressure to force a large fraction of the epoxy into the evacuated pores. The specimens were sliced on a precision sectioning saw to expose a concrete plane that ran perpendicular to the air interface. The coarse sawed surface was polished to a high degree of smoothness using a combination of wet sanding, diamond lapping, and a final polish of $0.3\mu\text{m}$. The smooth surface was sputter coated with platinum metal to increase the conductivity of the surface and aid in imaging.

5.6.1 PAMS

Specimens that had been cured with PAMS MFCC exhibited fair resistance to scaling. Figure 21 is a low magnification image of the near surface concrete showing voids from the entrained air. There is some porosity near the surface that suggests inadequate moisture retention leading to poor curing (Figure 22). The mortar paste was a generally well-consolidated structure of 20nm crystals (Figure 23).

5.6.2 Linseed

Specimens that had been cured with Linseed oil based MFCC performed very poorly in scaling resistance testing in relation to the other compounds. The cement paste surface was very porous, even in relation to the dry cured specimen (Figure 24). The microstructure consisted of poorly consolidated particles between 20 and 200nm with a high porosity (Figure 25). The extent of the porosity suggests that the pore network is percolated and will readily allow deicer to enter the pore space.

5.6.3 Acrylic

The specimens treated with acrylic CSC performed very well in ASTM C672 scaling resistance testing. There is some evidence that the compound penetrated the top $10\mu\text{m}$ of the surface, filling the voids and protecting the bulk (Figure 26). There were some voids that showed evidence of inadequate curing and partial hydration (Figure 27), but the microstructure of the paste was well consolidated and moderately porous (Figure 27 and Figure 28). The porous network was not well connected. The lack of percolation and the protective layer of the compound in the near surface prevented water migration and therefore improved the scaling performance.

5.6.4 Chlorinated Rubber

The specimens treated with chlorinated rubber curing and sealing compound performed very well in ASTM C672 scaling resistance testing as well. There was no evidence that the curing and sealing compound penetrated to any appreciable depth (Figure 29). There was a good deal of micron scale porosity, but the pores were evenly distributed and widely separated (Figure 30), which will prevent water migration. The paste was well consolidated and non-porous (Figure 31).

5.6.5 Wax

The Wax MFCC treated specimen was well consolidated and had few pores in the micron scale (Figure 32). The microstructure of the near surface paste was well consolidated and had low porosity (Figure 33). Note that the fracturing in Figure 33 was due to electron beam induced stress due to poor sample conductivity.

5.6.6 Untreated - Air Dry Curing

Specimens cured in dry air had a surprising lack of porosity normally associated with improper curing due to lack of water (Figure 34). This is likely due to continuing hydration of the specimen while in deicer solution, as there was no membrane to prevent the ingress of moisture. The microstructure consisted of densely packed and well-consolidated crystals from 30 to 100nm in diameter (Figure 35).

5.7 SEM Images

5.7.1 PAMS

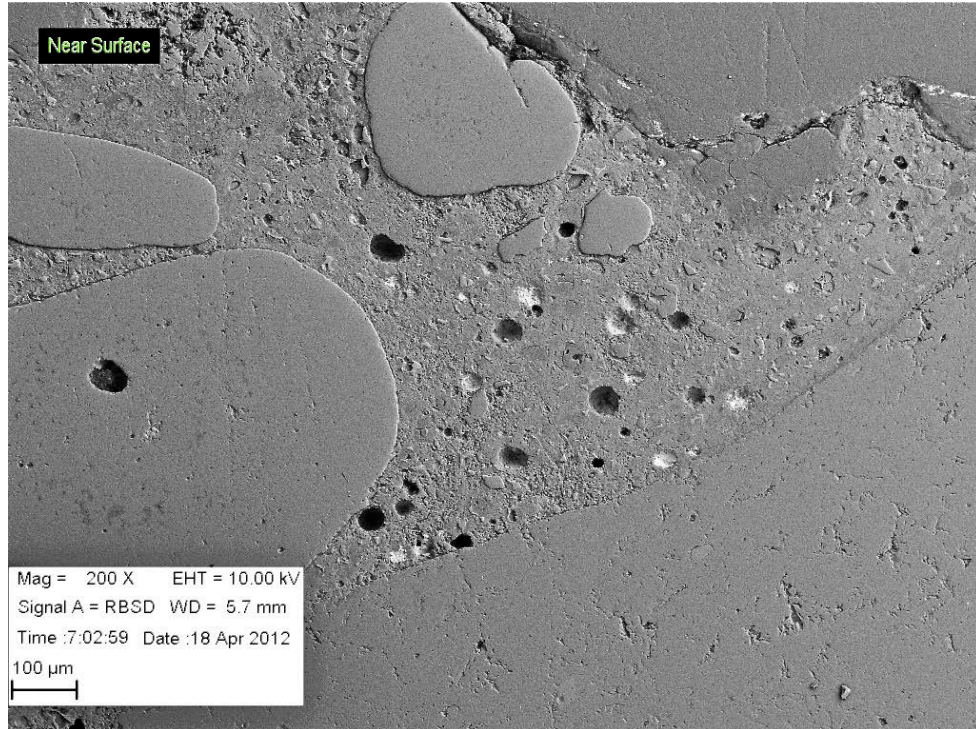


Figure 21: Wide view of the near-surface concrete of PAMS treated specimen.

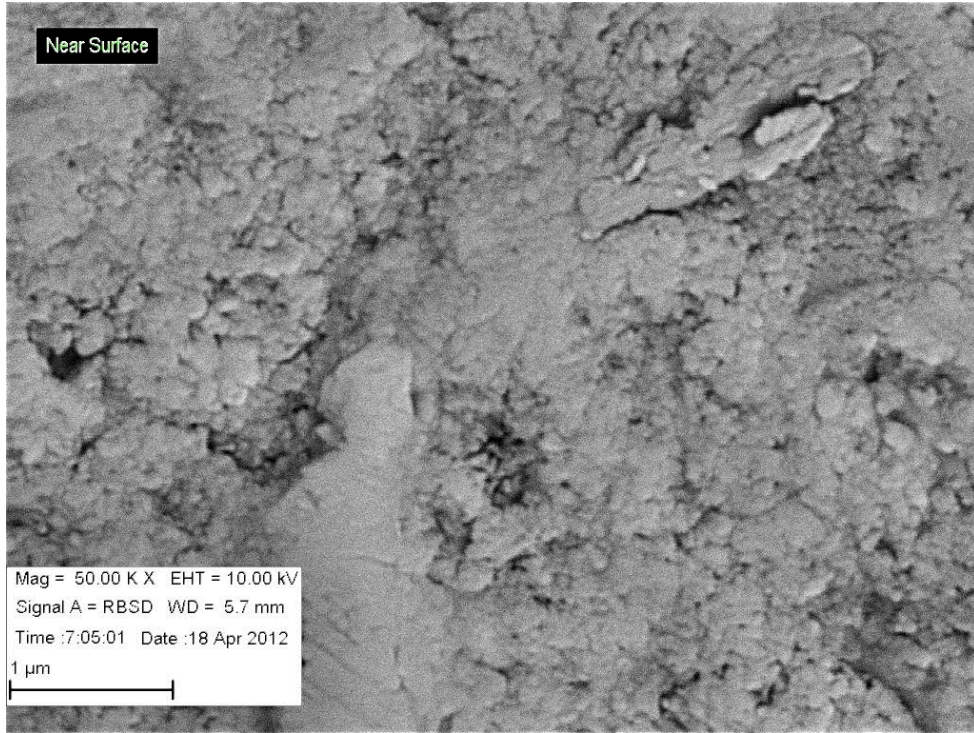


Figure 22: Micron scale structures in the near interface region of the PAMS specimen

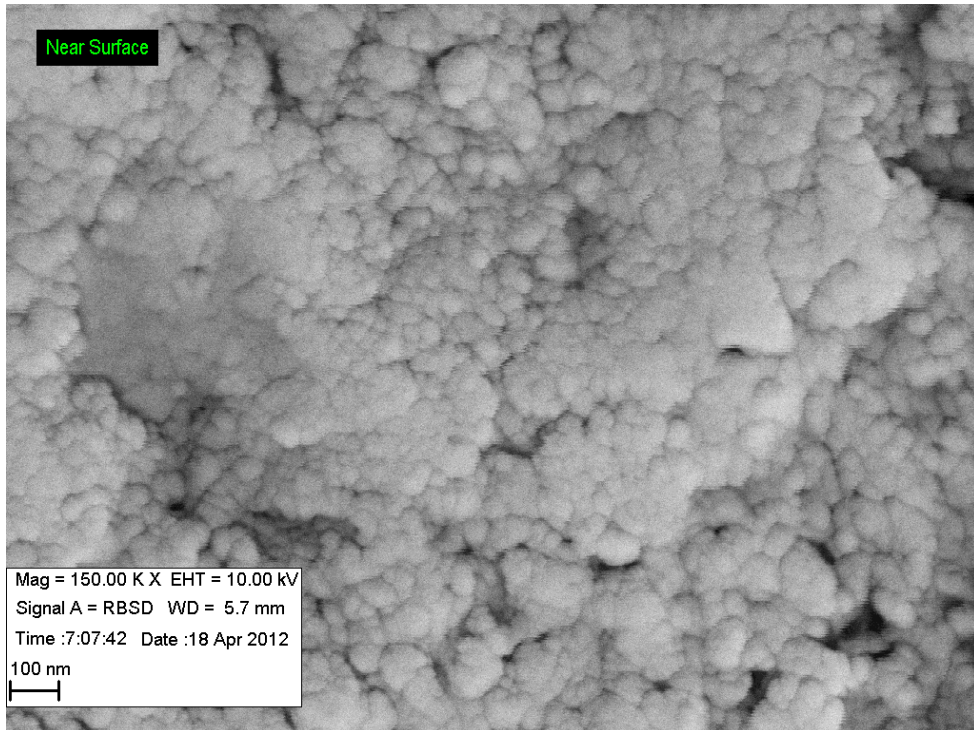


Figure 23: High magnification view of the microstructure of the mortar paste.

5.7.2 Linseed

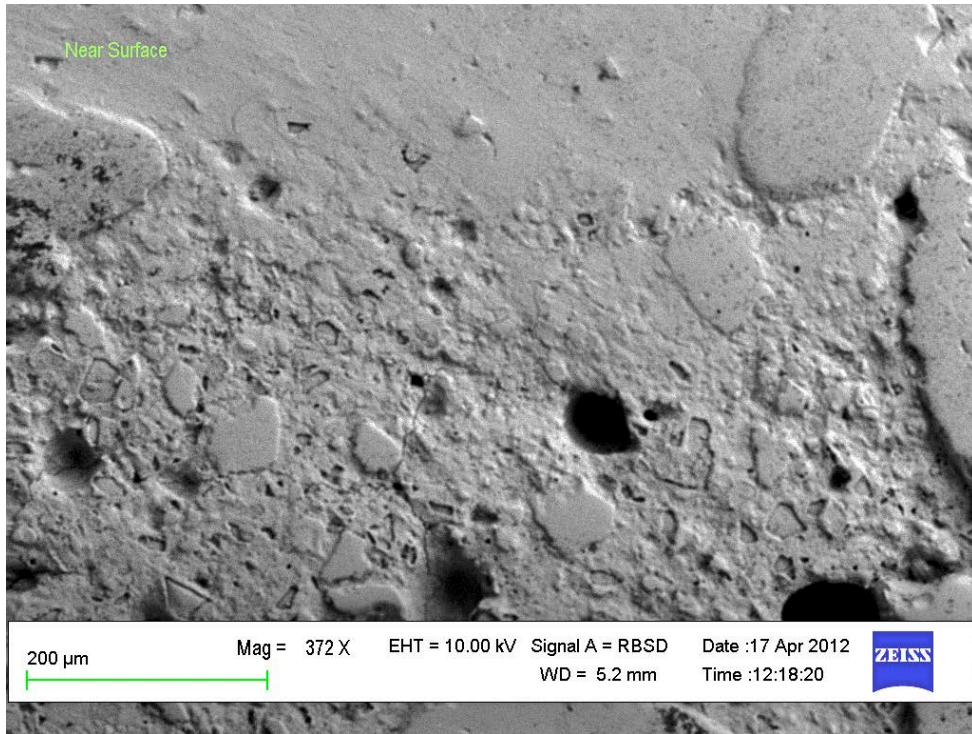


Figure 24: Low magnification view of the concrete near the interface of the Linseed oil MFCC treated concrete

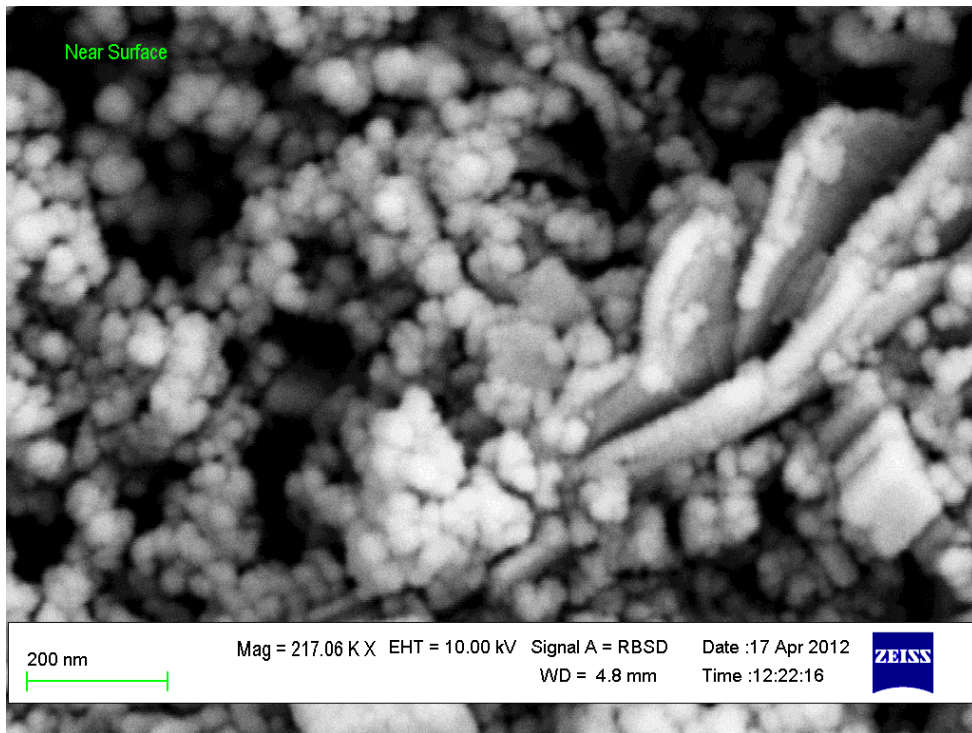


Figure 25: Microstructure of the cement mortar near the interface of the Linseed MFCC treated specimen. Note the high porosity and poor consolidation

5.7.3 Acrylic

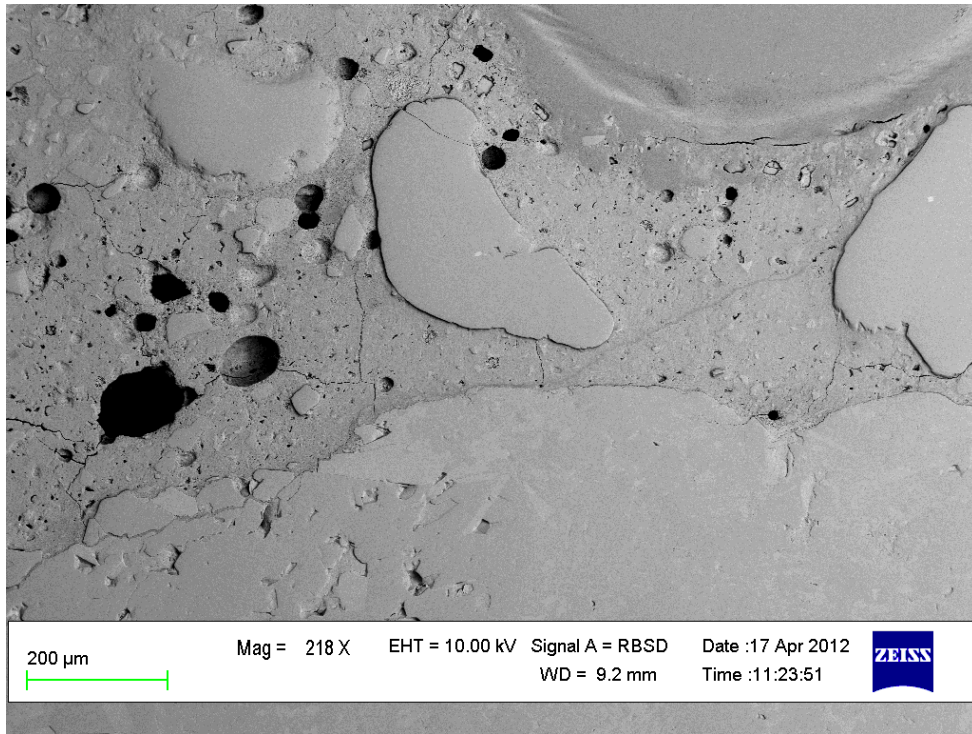


Figure 26: Low magnification view of the interface of the Acrylic treated specimen

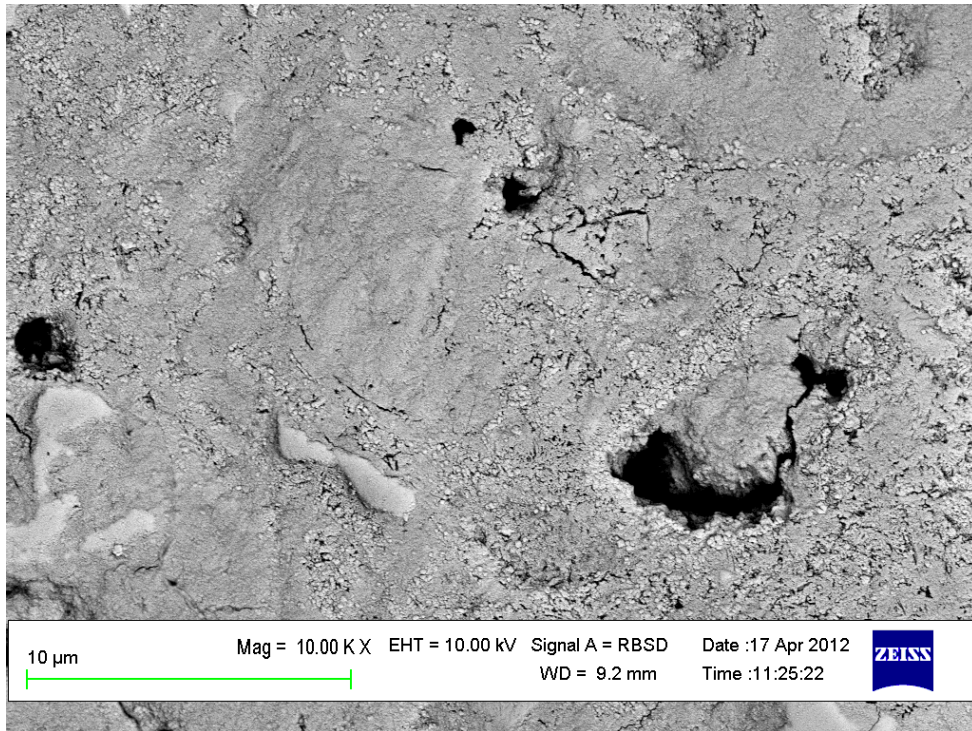


Figure 27: Micron scale structures in the cement mortar of the Acrylic MFCC treated specimen

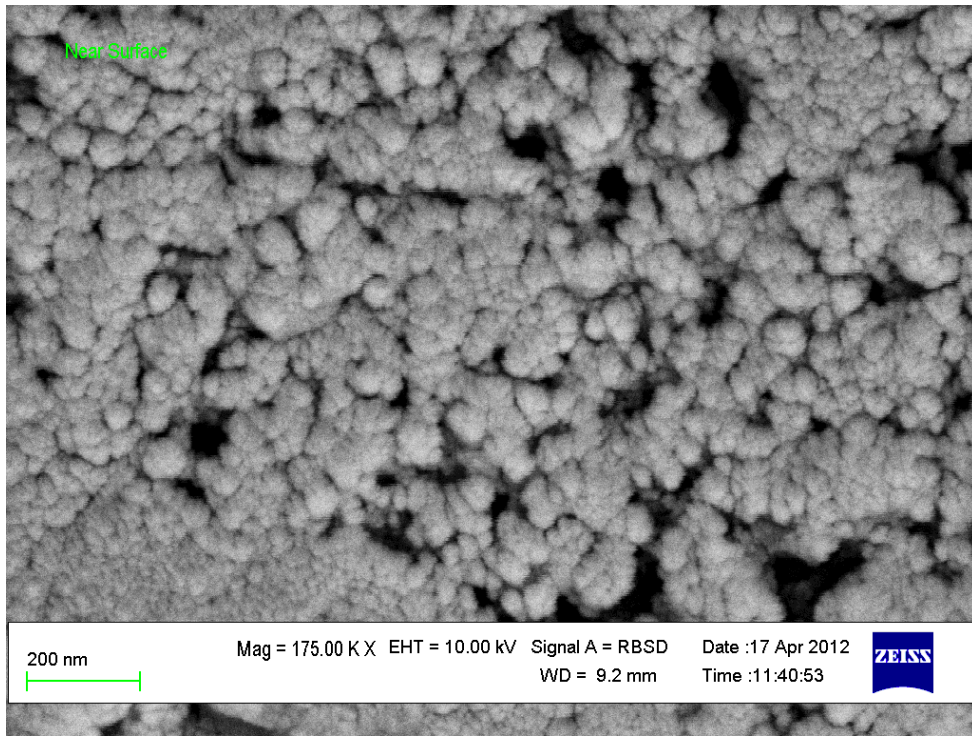


Figure 28: Microstructure of the cement mortar near the interface

5.7.4 Chlorinated Rubber

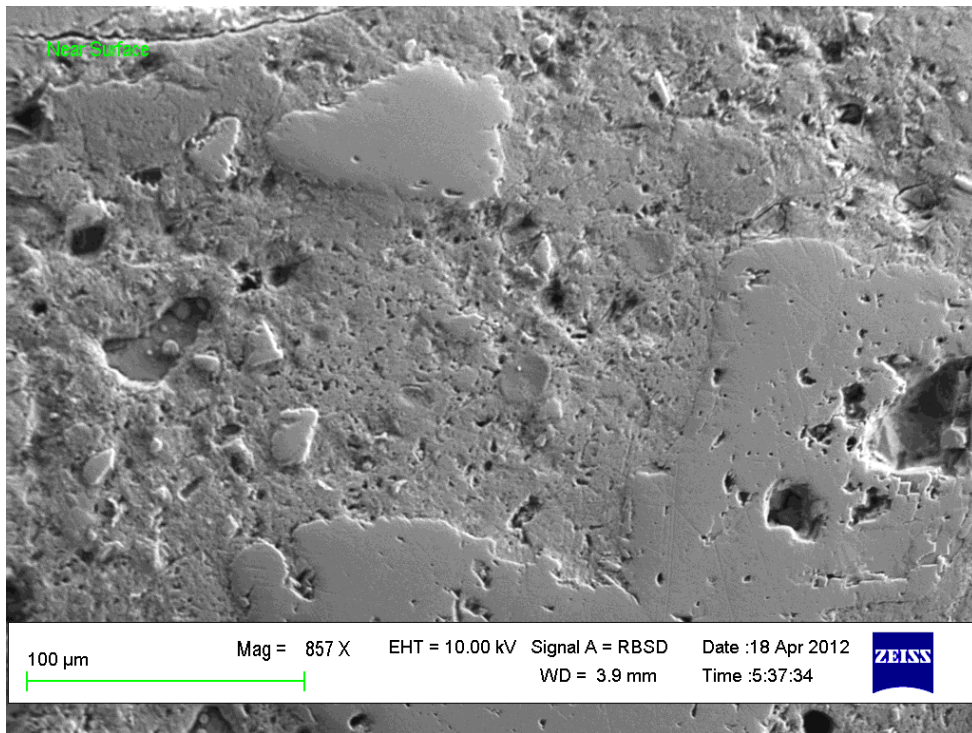


Figure 29 : Low magnification image of the Chlorinated Rubber MFCC treated concrete near the air interface



Figure 30: Micron scale structure of the mortar paste near the surface of Chlorinated Rubber MFCC treated concrete

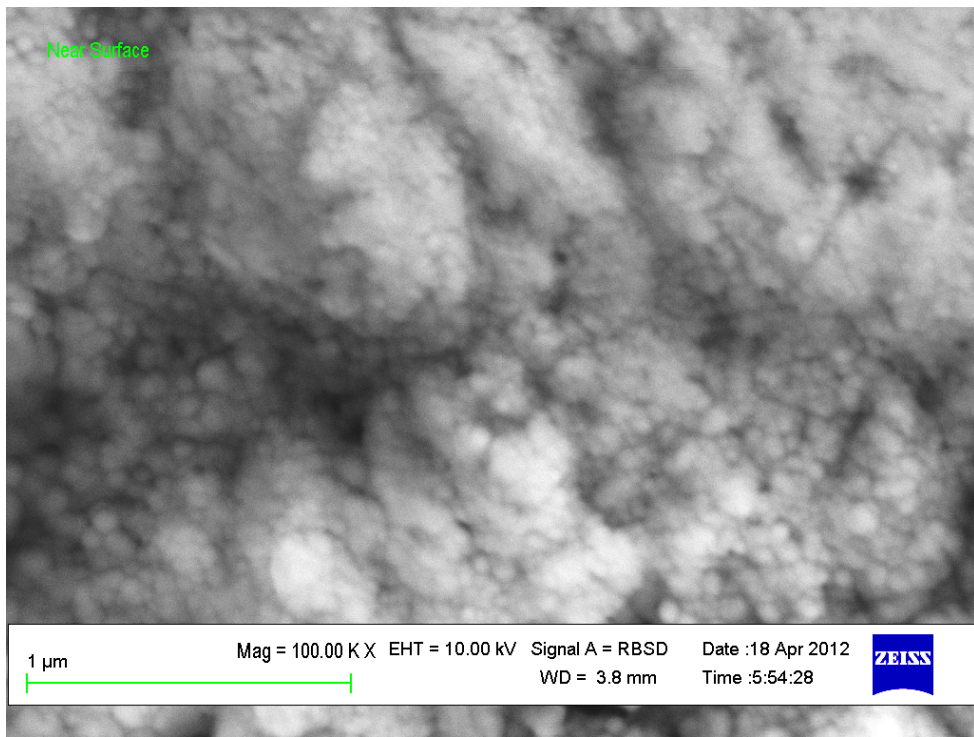


Figure 31: Microstructure of the cement mortar near the interface of the Chlorinated Rubber MFCC treated specimen

5.7.5 Wax

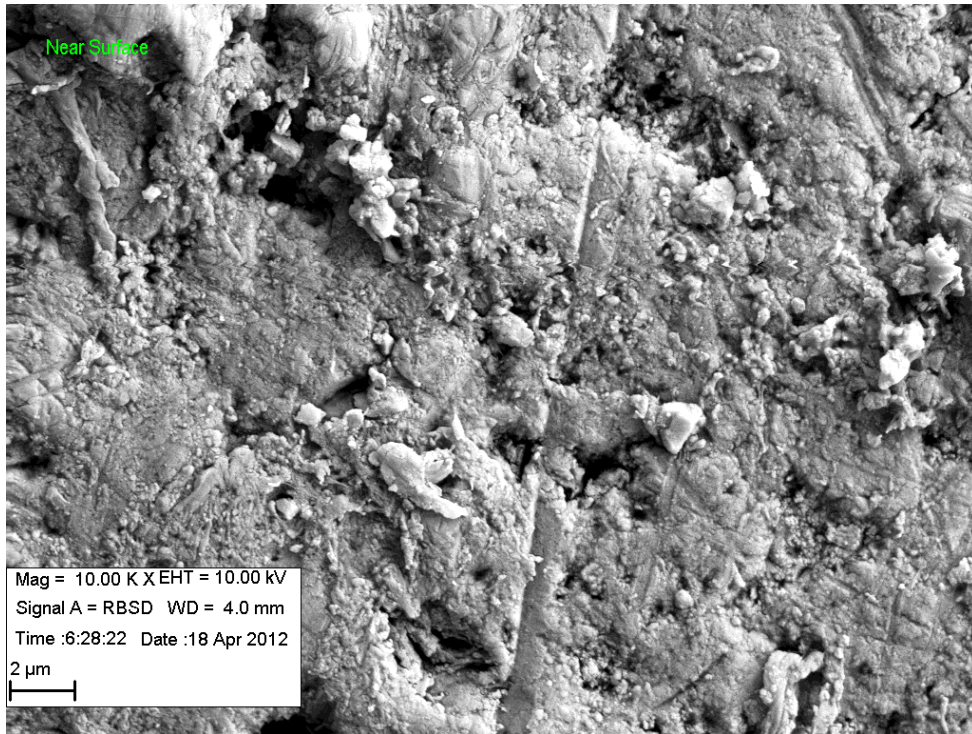


Figure 32: Micron scale structure of the cement paste near the interface of the Wax MFCC treated specimen

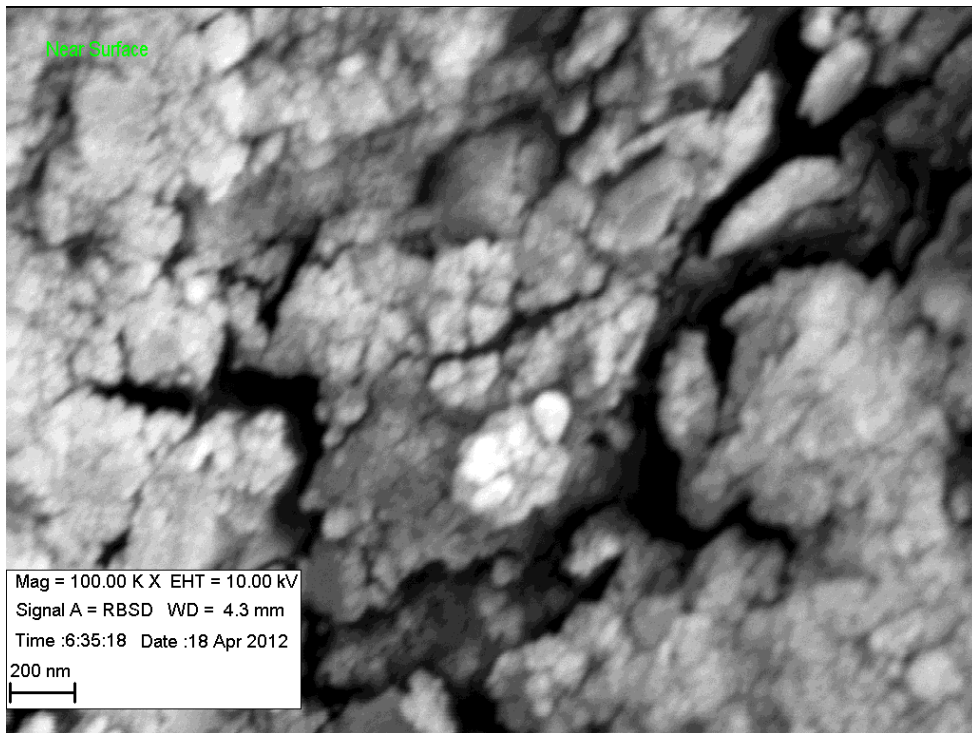


Figure 33: Microstructure of the cement paste near the interface of the Wax treated specimen. This image over represents the porosity of the structure because of electron beam induced fracturing

5.7.6 None

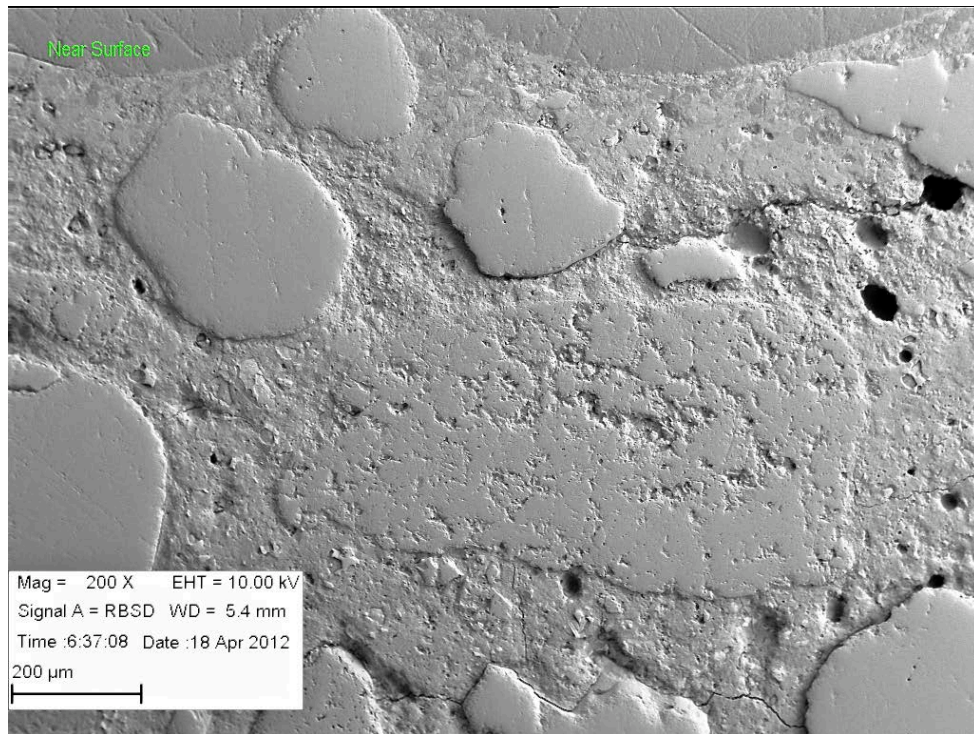


Figure 34: Near interface low magnification image of the untreated (dry room) cured concrete

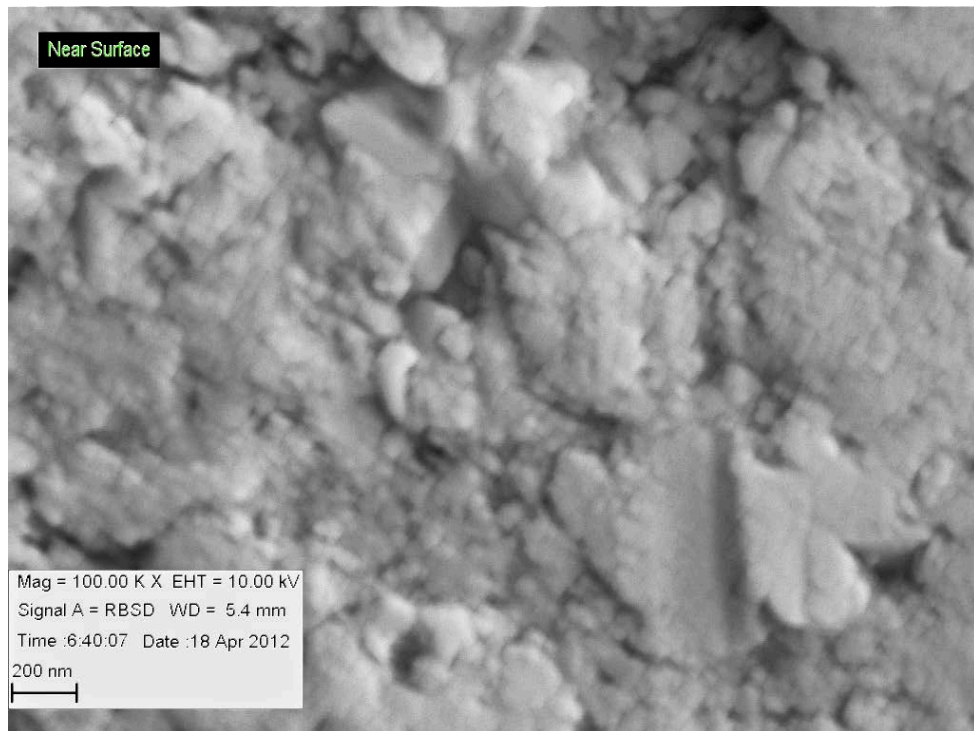


Figure 35: Microstructure of the near-surface mortar of the untreated specimen

6 Conclusions

Concrete projects represent significant capital expenditure and should maintain their aesthetic and practical utility during their design lifetime or beyond. Surface scaling and damage from chloride ion penetration decrease the utility of the emplacement and impact public perception of the project. Concrete carbonation is a concern mostly on projects with very long projected lifespans, but can impact internal stresses and reinforcement corrosion that leads to failure^{19,23}. Of these effects, deicer mediated surface scaling is the most visible to the end-user and can happen over a single winter in middle latitudes^{24,25}. This decreases public confidence in the quality of emplacements performed by the state, can destroy the surface texture applied to a pavement for traction and noise modification, and increases the difficulty of removing snow and debris.

Proper concrete curing can increase the resulting concrete's resistance to scaling by allowing full hydration and minimizing porosity of the near-surface region of the slab^{7,20,21,26-28}. Curing is typically viewed as being a result of maintaining adequate pore space humidity to support complete hydration of the cement particles in the paste. The hydration reaction increases the interconnectedness of the cement particles and decreases the degree of percolation in the pore space.⁸ Curing compounds are believed to support hydration reactions by preventing the loss of moisture to evaporation, but it is clear from our results that evaporative control is not the only mechanism by which curing compounds protect concrete from scaling. Wax MFCC was less effective at preventing moisture loss from mortar specimens than linseed oil based formulations when applied identically but produced concrete specimens that were more resistant freeze-thaw scaling damage.

Concrete is a complex material composed of simple ingredients. Replacing some fraction of Portland cement with reclaimed cementitious material like slag or fly ash can decrease the energy and monetary cost of construction while increasing the later age durability and strength but introduces a great deal of complexity and uncertainty to the concrete design process. This already challenging situation is further muddied by the use of chemical admixtures to control rheology, hydration kinetics, and air entrainment. Optimizing the mixture for certain characteristics may introduce or exacerbate design shortcomings in other performance metrics. It is therefore useful to present the conclusions of this study in the form of a chart in which the design element (curing compound) is rated against the design parameters to allow the end user to easily choose the treatment with the most favorable balance of outcomes. By choosing numeric values for the performance of each coating in each performance metric of interest, deciding on a weight of each metric, and producing a weighted average it is possible to create a decision matrix to choose a "best general fit" curing compound (Tables 5 and 6). An example is provided below, where the weight of scaling damage is twice that of chloride penetration resistance.

This decision table could be expanded to include carbonation resistance, cost, and aesthetic concerns as long as the additional performance metrics are given an appropriate numerical ranking and criterion weight. Large numeric rankings for unacceptable performance could also be replaced with go/no go rankings to exclude coatings if failure in that criterion outweighs good performance in the others. As an example, scaling losses greater than 1000g/m² could exclude a coating even if it were effective at preventing carbonation and chloride penetration with low cost.

Table 5: General guidance for concrete engineers. Low magnitude represents higher resistance. Note: the highlighted section represents the scaling resistance of the Limestone/OPC system, which performed dramatically better than the other 5 systems, and would rate as excellent in comparison.

| Aggregate Type | Cement Type | Property | Coating Type | | | | | |
|----------------|------------------|---------------|--------------|------------|------------|------------|------------|------------|
| | | | PAMS | Linseed | Acrylic | Cl. Ru. | Wax | |
| Limestone | OPC | Scaling | 3 | 3 | 1 | 1 | 3 | |
| | | Chloride Res. | 2 | 2 | 2 | 3 | 3 | |
| | 30% Fly Ash | Scaling | 4 | 5 | 3 | 5 | 2 | |
| | | Chloride Res. | 2 | 2 | 1 | 2 | 3 | |
| | 30% Slag | Scaling | 2 | 10 | 2 | 2 | 5 | |
| | | Chloride Res. | 1 | 2 | 1 | 1 | 3 | |
| Gravel | OPC | Scaling | 2 | 10 | 1 | 2 | 1 | |
| | | Chloride Res. | 1 | 1 | 2 | 1 | 2 | |
| | 30% Fly Ash | Scaling | 2 | 5 | 1 | 4 | 1 | |
| | | Chloride Res. | 1 | 1 | 1 | 1 | 1 | |
| | 30% Slag | Scaling | 4 | 5 | 2 | 2 | 5 | |
| | | Chloride Res. | 1 | 2 | 2 | 1 | 2 | |
| | Total | | | 14 | 29 | 10 | 14 | 16 |
| | Weighted Average | | | 2.1 | 4.0 | 1.6 | 2.1 | 2.6 |

Table 6: Criteria for ratings

| Scaling | | | Chloride ion penetration | | |
|--------------|------------------|----------------|--------------------------|--------------------|----------------|
| Rating | g/m ² | Numeric Rating | Rating | mg Cl / g concrete | Numeric Rating |
| Unacceptable | >1000 | 10 | Poor | >4 | 3 |
| Poor | 800<x<1000 | 5 | Good | 2<x<4 | 2 |
| Fair | 600<x<800 | 4 | Excellent | <2 | 1 |
| Good | 400<x<600 | 3 | | | |
| Very Good | 200<x<400 | 2 | | | |
| Excellent | <200 | 1 | | | |

In addition to the guidance provided by the decision matrix several trends were observed in the performance characteristics of the curing compounds:

1. MFCCs containing pigment are required for use in WisDOT roadway emplacement but are less effective than CSCs at maintaining pore humidity and preventing scaling.
 - a. Linseed oil based formulations produced concrete with poor scaling resistance.
 - b. PAMS resin MFCC produced concretes that were largely resistant to scaling damage.
 - c. Wax based curing compound was ineffective at preventing scaling in concretes containing 30% slag cement but performed well on all other mix designs.
2. The lack of pigment, high VOC load, potentially hazardous application operations, lack of regulatory framework, and high cost currently prevents the use of CSCs on large scale projects like roadways, even though they offer significant performance benefits like increased resistance to evaporation, resistance to scaling, and resistance to carbonation.
 - a. CSCs generally perform better than purely membrane forming compounds across several performance metrics.
 - b. Concretes containing OPC or 30% slag were highly resistant to scaling and carbonation when cured using chlorinated rubber epoxy CSCs
 - c. There appears to be some chemical incompatibility between the chlorinated rubber CSC and fly ash concrete that results in concrete with low scaling resistance. This combination should be avoided.
 - d. Concrete specimens treated with acrylic monomer curing and sealing compounds were not the most scaling resistant of those tested, but their strong performance across all performance metrics for all mix designs suggests that they are the best general use treatment.

Curing compound formulation is not the sole determinant of concrete quality; poor finishing practices can produce poor concrete surfaces even with suitable curing agent choice. Concrete finishing (troweling and texturing) should occur after concrete bleed water is no longer being produced to prevent an increase in the apparent water/cementitious material ratio of the near surface cement paste. This will prevent concrete failure modes associated with excessive porosity, as working the surface after bleeding is finished will destroy the pores left by bleed water migration. Monitoring the concrete bleed rate is a better practice than allowing a set time to pass before finishing or simply observing the surface for moisture, as evaporation can dry the concrete surface faster than bleed water replenishes it. An apparently dry surface (from rapid evaporation) might prompt finishing work to commence prior to the actual end of concrete bleeding and result in poor surface performance. Bleeding occurs until formation of an ettringite network causes initial set, after which the concrete is no longer plastic and pore water is trapped in the paste by the gel network. Formation of this network occurs anywhere from within several minutes for concretes prepared with accelerators, silica fume, or at high temperature to tens of hours for concrete with significant fractions of supplemental cementitious materials with slow hydration kinetics. This high variability in time elapsed before finishing increases the uncertainty of the contractor in scheduling and budgeting for labor and provides an incentive for the contractor to take shortcuts like beginning finishing too early. The variability in performance of curing compounds across mix types could well be associated with the variability in bleeding rates rather than a direct consequence of differences in mix constituents.

7 Recommendations:

The use of a suitable design matrix will allow the project designer to specify an effective and appropriate curing compound to be chosen. This will improve surface performance and decrease lifetime cost even if the curing compound chosen is more expensive than the alternative. White pigmented curing compounds are required for concrete pavements in the state of Wisconsin, even though they may perform poorly in relation to non-pigmented formulations (including CSCs). White pigment decreases the absorbance of solar radiation, which will slow evaporation, assist in limiting high surface temperature during curing and prevent the formation of thermal gradients at exposed surfaces. The exclusion of curing and sealing compounds from the list of approved curing compounds might exclude highly effective formulations.

This study utilized specimens prepared and coated in a laboratory environment in which solar thermal forcing of the surface was not present. A study comparing the marginal benefit of thermal control of the concrete surface for pigmented compounds against the improved performance of curing and sealing compounds should be performed. Surface temperature of specimens in direct sunlight should be measured for each curing compound type, as there are significant color and albedo differences between them (i.e. linseed is yellowish while PAMS is stark white). Embedded thermocouple arrays could be used to determine the severity and magnitude of thermal gradients in curing concrete. The scaling resistance of specimens prepared and cured in bright, direct sunlight should be compared with the results of laboratory studies to determine the relative performance of pigmentation.

Curing compound choice is but one aspect of ensuring adequate performance, as the MFCC must be applied to a concrete surface. Therefore, surface preparation is an important concern in pavement or concrete design. Freshly placed concrete bleeds water to the surface. As the concrete settles and gelling begins, pore water is excluded from the pore space and migrates to concrete/form and concrete/air interfaces. This bleeding process can induce additional porosity because the migrating water must clear channels through which to flow. The bleed water can form standing water on the surface if the evaporation rate is slower than bleeding, resulting in a shiny, wet looking surface. If the evaporation rate is approximately equal to or greater than the rate of bleeding the concrete may present an entirely dry surface, even though significant water migration is still occurring. Application of materials designed to prevent evaporation to a bleeding surface can result in poorly adhering membrane coatings, or pinholes and crazing that decrease the effectiveness of the curing compound.

1. Each cement blend produced concretes with different gel times, times to initial set, and bleeding periods.
2. In highly evaporative conditions (strong direct sunlight, wind, low humidity) the concrete can still be producing significant bleed water but present a dry surface for inspection.
3. Applying curing compounds will prevent evaporation and a layer of bleed water can separate the curing compound from the surface, cause pinhole formation, or cause crazing of the membrane (in the case of wax MFCC). This will limit the effectiveness of the curing compound for improving later age concrete performance.
4. Scientifically justifiable guidelines and procedures for monitoring concrete bleeding time are feasible to develop. Such guidelines could provide contractors with justification for delaying finishing practices until the optimal time even if waiting incurs greater labor costs, decreasing the incentives for improper early finishing.

5. Delayed finishing may increase the cost of production marginally but should provide significant improvements in project durability. Further study of the bleeding problem is required to quantify the magnitude of this effect.
6. An effort to develop pigmented CSCs that meet state requirements for reflectance but provide the scaling resistance, carbonation protection, and decreased chloride diffusion characteristics of acrylic or chlorinated rubber formulations would enhance the options available to the state to improve concrete pavement durability.

Earlier work has focused on the effect of curing compound application, and the best practice principles identified by these studies should be used to develop WisDOT regulations.¹ Application of the curing compound by sprayer can result in poor coverage in high winds. Coating quality is also compromised if the spray equipment is poorly designed or spraying occurs in a single direction. This is exacerbated when aggressive surface texture, such as randomly space parallel tining, is applied for traction or road noise prevention.²⁹⁻³¹ Spraying equipment should have a nozzle pattern with sufficient overlap to prevent streaking and incomplete coverage but not cause pooling or drip formation. The coating rate should be adjusted to reflect the actual surface area of the concrete after texture is applied rather than as the simple geometric road area to prevent areas of thin coating that would be less effective at preventing evaporation. The best curing compound formulations can be ineffective if poorly applied.

Curing compounds are used to improve concrete curing and project durability, primarily through evaporative control, but formulations that are equally effective at preventing evaporation (in ASTM C156 testing) produce concrete with different performance properties. Curing compound formulation must be chosen with an eye on cost, compatibility with the cement used, and performance for the most important design criteria. A weighted decision matrix provides a tool for the project designer to use when specifying curing compound type. Once a curing compound type is chosen it must be applied to a suitable surface. Surface finishing operations should only occur after bleeding has halted, which may not be readily apparent even to trained workers. After finishing, the curing compound must be applied in a thin, even layer at the manufacturers specified rate after taking account of the increased surface area of textured concrete surfaces. Concrete prepared in this method stands the largest chance of providing the long term durability and environmental stability needed to ensure the project meets design life targets.

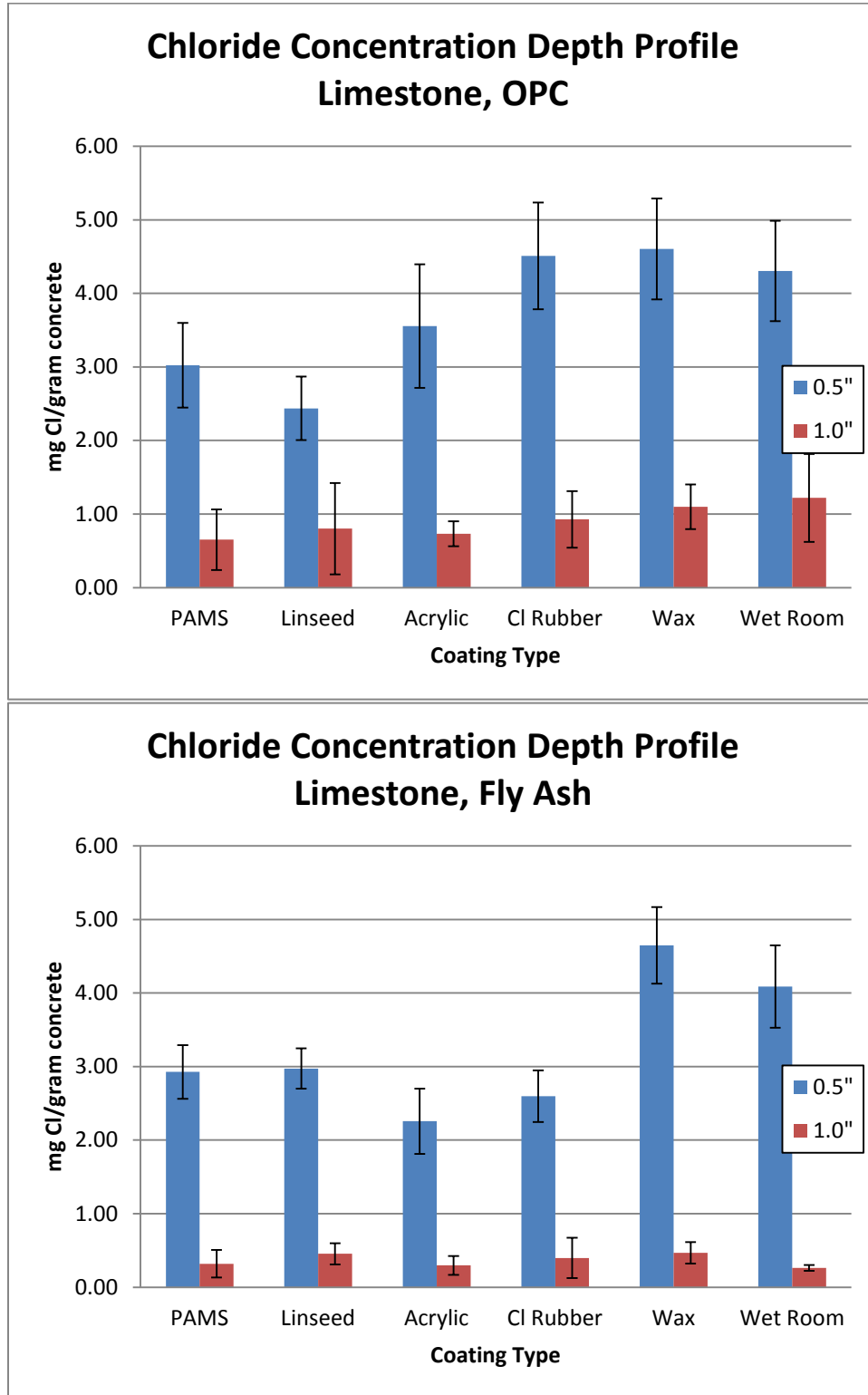
8 Bibliography

1. Vandenbossche, J. A Review of the Curing Compounds and Application Techniques Used by the Minnesota Department of Transportation for Concrete Pavements. 38 (Minnesota Dept. of Transportation: Office of Research Services, 1999).
2. Mindess, S., Young, J. F. & Darwin, D. Concrete. (Prentice Hall: 2002).
3. Nassif, H., Suksawang, N. & Mohammed, M. Effect of curing methods on early-age and drying shrinkage of high-performance concrete. Transportation Research Record: Journal of the Transportation Research Board 1834, 48–58 (2003).
4. Snyder, M. B. Pavement surface characteristics: a synthesis and guide. (2006).
5. Kovler, K. & Roussel, N. Properties of fresh and hardened concrete. Cement and Concrete Research 41, 775–792 (2011).
6. Bentz and Stutzman - 2006 - Curing, hydration, and microstructure of cement pa.pdf.
7. Whiting, N. M. & Snyder, M. B. Effectiveness of Portland Cement Concrete Curing Compounds. Transportation Research Record: Journal of the Transportation Research Board 1834, 59–68 (2003).
8. Bentz, D. P. & Stutzman, P. E. Curing, hydration, and microstructure of cement paste. ACI materials journal 103, 348–356 (2006).
9. Jensen, O. M. & Hansen, P. F. Water-entrained cement-based materials: I. Principles and theoretical background. Cement and Concrete Research 31, 647–654 (2001).
10. Neuwald, A., Krishnan, A., Weiss, J., Olek, J. & Nantung, T. E. Concrete curing and its relationship to measured scaling in concrete containing fly ash. Transportation Research Board. Available at: http://cobweb.ecn.purdue.edu/*concrete/weiss/publications/r_conference/RC-019.pdf. Cited 5, (2007).
11. Bentur, A., Igarashi, S. & Kovler, K. Prevention of autogenous shrinkage in high-strength concrete by internal curing using wet lightweight aggregates. Cement and Concrete Research 31, 1587–1591 (2001).
12. MacGregor, J. G., Wight, J. K., Teng, S. & Irawan, P. Reinforced concrete: mechanics and design. (1997).
13. Zhang, M. H., Bilodeau, A., Shen, G. & Malhotra, V. H. De-Icing Salt Scaling of Concrete Incorporating Different Types and Percentages of Fly Ashes. 1, (1998).
14. LaBarca, I., Foley, R. & Cramer, S. Effects of Ground Granulated Blast Furnace Slag in Portland Cement Concrete – Expanded Study. 88 (Department of Civil and Environmental Engineering: University of Wisconsin, Madison, 2007).
15. Dhir, R. K., Jones, M. R. & Ahmed, H. E. H. Determination of total and soluble chlorides in concrete. Cement and Concrete Research 20, 579–590 (1990).
16. He, X. & Shi, X. Chloride Permeability and Microstructure of Portland Cement Mortars Incorporating Nanomaterials. Transportation Research Record: Journal of the Transportation Research Board 2070, 13–21 (2008).
17. Mangat, P. S., Khatib, J. M. & Molloy, B. T. Microstructure, chloride diffusion and reinforcement corrosion in blended cement paste and concrete. Cement and Concrete Composites 16, 73–81 (1994).
18. Stanish, K. D., Hooton, R. D. & Thomas, M. D. A. Testing the chloride penetration resistance of concrete: a literature review. Dep. Of Civil Eng., University of Toronto (1997).
19. Chi, J. M., Huang, R. & Yang, C. C. Effects of carbonation on mechanical properties and durability of concrete using accelerated testing method. Journal of Marine Science and Technology 10, 14–20 (2002).
20. Almusallam, A. A., Khan, F. M., Dulaijan, S. U. & Al-Amoudi, O. S. B. Effectiveness of surface coatings in improving concrete durability. Cement and Concrete Composites 25, 473–481
21. Abdul Razak, H., Chai, H. K. & Wong, H. S. Near surface characteristics of concrete containing supplementary cementing materials. Cement and Concrete Composites 26, 883–889 (2004).

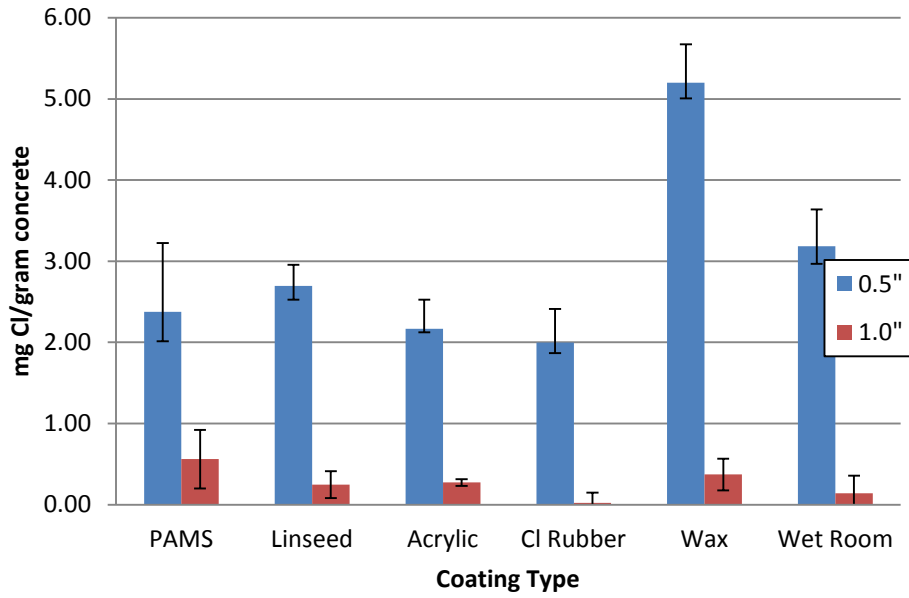
22. Gebler, S. H. & Klieger, P. Effect of Fly Ash on Some of the Physical Properties of Concrete. (1986).
23. Hansson, C. M., Poursee, A. & Jaffer, S. J. Corrosion of Reinforcing Bars in Concrete. PCA R&D 3013, (2007).
24. Lamond, J. F. & Pielert, J. H. Significance of tests and properties of concrete and concrete-making materials. (ASTM International: 2006).
25. Sutter, L., Dakota, S. & Institute, M. T. T. The deleterious chemical effects of concentrated deicing solutions on portland cement concrete. (South Dakota Dept. of Transportation: 2008).
26. Radlinski, M., Olek, J. & Nantung, T. Effect of Mixture Composition and Initial Curing Conditions on Scaling Resistance of Ternary (OPC/FA/SF) Concrete. *Journal of Materials in Civil Engineering* 20, 668 (2008).
27. Stark, J. & Ludwig, H. M. Freeze-thaw and freeze-deicing salt resistance of concretes containing cement rich in granulated blast furnace slag. *ACI Materials Journal* 94, (1997).
28. Pigeon, M., Talbot, C., Marchand, J. & Hornain, H. Surface microstructure and scaling resistance of concrete. *Cement and Concrete Research* 26, 1555–1566 (1996).
29. Chandler, J. W. E., Phillips, S. M., Roe, P. G. & Viner, H. E. Quieter concrete roads: construction, texture, skid resistance and noise. TRL REPORT TRL 576 (2003).
30. Caestecker, C. Test Sections of Noiseless Cement Concrete Pavements. *Routes/Roads* 299, 53–65 (1998).
31. Cackler, E. T., Harrington, D. S. & Ferragut, T. Evaluation of US and European Concrete Pavement Noise Reduction Methods. (2006).

9 Appendix

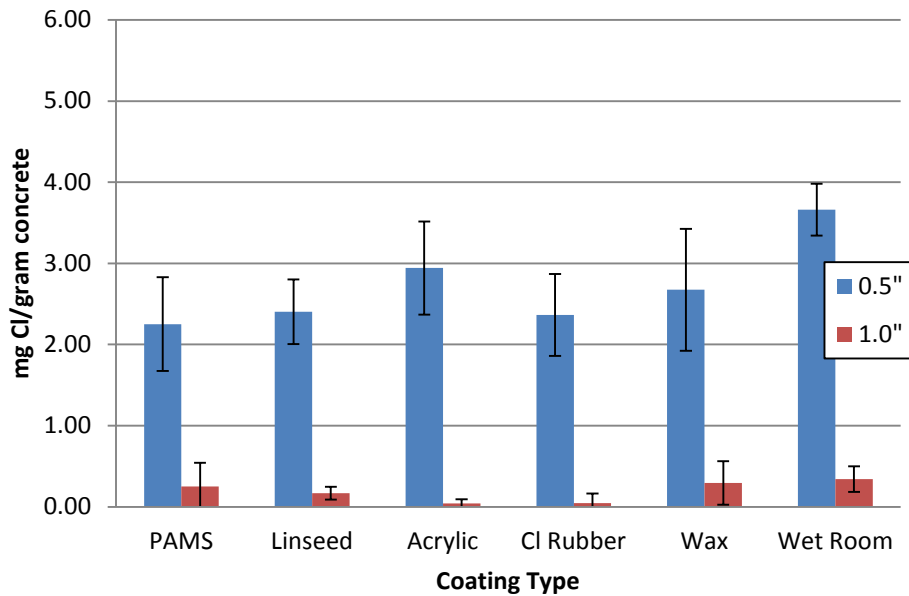
9.1 AASHTO T-259 Results



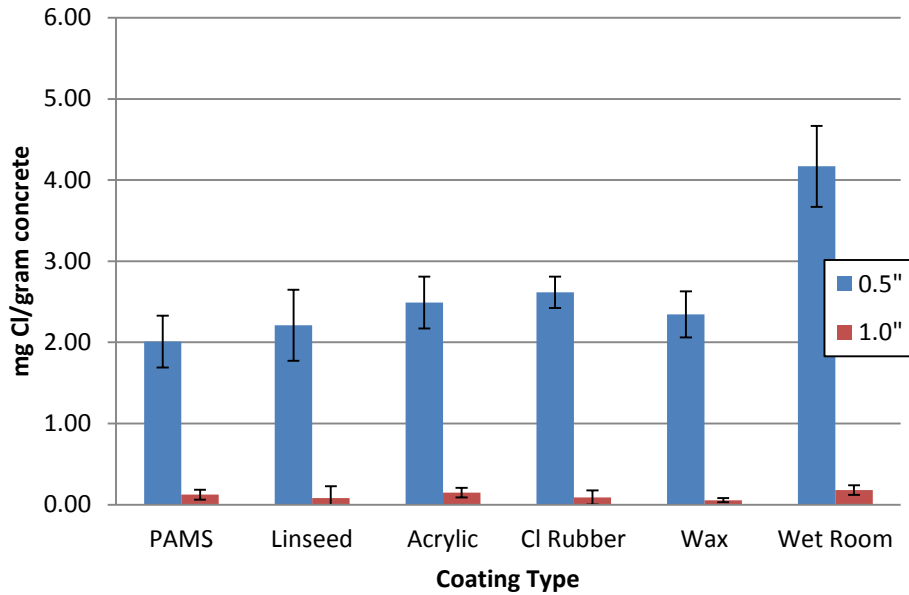
Chloride Concentration Depth Profile Limestone, Slag



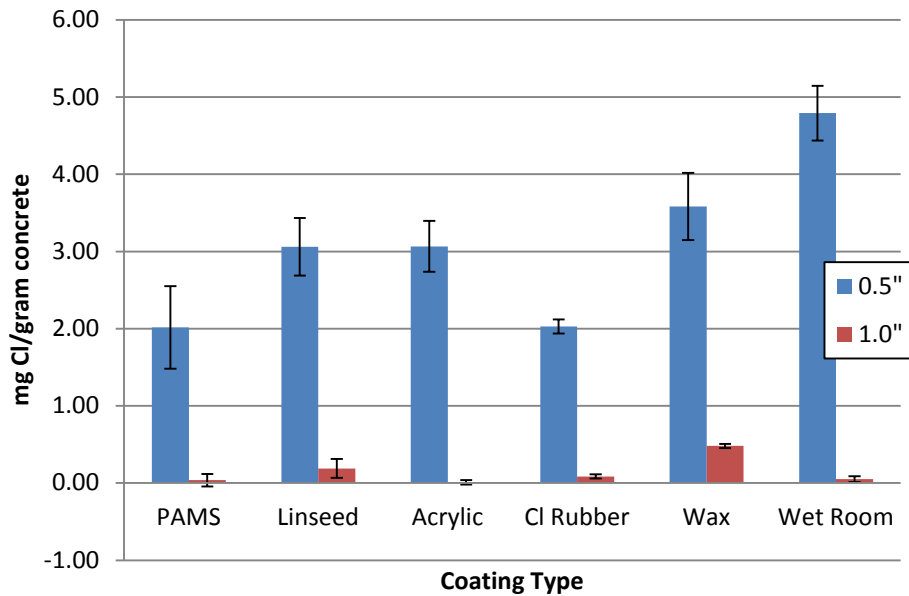
Chloride Concentration Depth Profile Gravel, OPC



Chloride Concentration Depth Profile Gravel, Class C Fly Ash



Chloride Concentration Depth Profile Gravel, Grade 100 Slag



Wisconsin Highway Research Program
University of Wisconsin-Madison
1415 Engineering Drive
Madison, WI 53706
608/262-3835
www.whrp.org