Evaluation of Curing Compound Application Time on the Surface Durability of Concrete

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EVALUATION OF CURING COMPOUND

APPLICATION TIME ON THE SURFACE

DURABILITY OF CONCRETE

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16. Abstract

The effect of curing compound application time after concrete finishing was examined in this study. Times of 30 minutes, 2 hours and 4 hours were considered and repeatability was evaluated with comparisons to a Phase I portion of the study. Scaling resistance varied with mix type and curing compound type. None of the curing compounds provided scaling resistance that approached that of wet room curing. The effectiveness of most curing compounds appeared tied to the surface condition of the concrete. The presence of bleed water varied with mix type and if bleed water was present, the effectiveness of the curing compounds were typically compromised. Polyalphamethylstyrene (PAMS) resin based curing compound appeared to be equally effective regardless of application time but this was not true for wax-based, linseed oil and acrylic curing compounds. Generally for these compounds 2 to 4 hours was needed for best results depending on cementitious additives.

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Executive Summary

Premature scaling of concrete pavements has sporadically and repeatedly occurred in Wisconsin over the past decade. This premature distress can threaten public infrastructure investments long before replacement or repair strategies should typically be contemplated. With some certainty that these issues are not associated with concrete mix design and admixtures, the logical focus has been to examine pavement finishing and curing, as these practices are known to impact surface layer freeze-thaw durability of concrete. Membrane forming curing compounds (MFCCs) are used primarily to minimize concrete water evaporation so that hydration occurs to the fullest degree possible and to minimize costs associated with curing the large surfaces associated with pavements. Ideally the curing compounds lead to conditions that mimic wet curing whereby curing commences after initial set such that the water is neither drawn out nor added to the concrete surface hydration. Timing is critical as application too soon may either be ineffective or disrupt the hydration in the concrete surface layer. Application too late can mean that significant drying has occurred at the concrete surface inhibiting the full hydration of the cement paste. Either situation can lead to a surface that has reduced freeze-thaw resistance and is susceptible to scaling.

The primary goal of this research was to evaluate the influence of MFCC application time on the freeze-thaw scaling damage resistance of concrete made with materials common to Wisconsin paving. A partial factorial experiment was designed to probe the effect of MFCC application time on scaling damage as measured using ASTM C672. Three emulsion-based curing compounds, Linseed Oil, Wax, and Poly-alpha-methyl-styrene (PAMS) were evaluated at three application times (30 Minutes, 2-hour, and 4-hour after finishing) on concrete specimens prepared with one of two sources of coarse aggregate and one of three cementitious materials. An Acrylic solvent-based sealing compound was evaluated with respect to concrete scaling resistance

at two application times. Untreated specimens from each mix type were cured in a wet room and tested as controls.

A secondary goal of this project was to evaluate a new method for determining the presence of bleed water on a concrete surface by designing and constructing a device to detect changes in the relative humidity of the air above the concrete surface over time to more reliably determine the cessation of the bleeding than current methods. To achieve this goal, a device was built and its operational capability was tested during the study.

Concrete mixes were designed and specimens were prepared according to Wisconsin Department of Transportation (WisDOT) procedures. Freeze-thaw testing was performed and data was recorded. Properties such as fresh concrete slump, air content, 28-day compressive strength, and curing compound application rates were also measured.

Results from this study indicate that the influence of MFCC application time on the scaling resistance of concrete is dependent upon the selected compound and the concrete mix. The Linseed Oil and Acrylic formulations displayed improved scaling resistance with an increase in application time. The PAMS formulation was generally equally effective regardless of application time. Increase in the time before application with the Wax based compound did not consistently yield improved scaling resistance. Results from the untreated wet room cured specimens indicate that curing compounds do not replicate scaling resistance levels that are comparable to wet room curing. Scaling resistance was also influenced by the composition of the concrete, especially with respect to the coarse aggregate and cementitious material choice. Elevated levels of ambient relative humidity at the time of specimen manufacture appeared to decrease the scaling resistance, regardless of application time. The first generation humidity detection device was found to be unreliable for monitoring the presence of bleed water. Analysis

of the data collected by the device showed that future modifications could be made to improve its operational capability.

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Chapter 1 **Problem Statement**

Roadways that are both durable and aesthetically pleasing are primary goals of Wisconsin Department of Transportation (WISDOT) paving projects. Recently, Portland Cement Concrete (PCC) pavement projects constructed by WISDOT have experienced increased amounts of scaling within the first year or two of service life. Scaling is a form of surface damage that can lead to decreases in the aesthetic appeal, functionality, and service life of a PCC pavement. Pavements that have scaled typically demonstrate symptoms including surface roughness, exposed aggregates, collection of loose scaled material on the surface, and discontinuities in both the outward appearance and level of the top layer of the pavement.

Membrane forming curing compounds (MFCCs) are used in paving applications to properly ensure that the mix water within the mortar that comprises the top ¼ to ½ inch of the concrete pavement surface does not prematurely evaporate and inhibit hydration. When sprayed onto the surface of the pavement, the compounds form a hydrophobic membrane that retains and protects the mix water from evaporative factors such as direct solar radiation and air currents at the exposed surface. However, pavement projects using any one of several different WISDOT approved curing compounds still have experienced scaling within a few years of construction.

A WISDOT study, through the Wisconsin Highway Research Program (WHRP) recently completed the evaluation of a variety of curing compounds (Kropp et al. 2012). This study will be referred throughout this document as 'Phase I.' A total of four curing compounds applied to six different concrete mix types were evaluated for scaling resistance. The results from this study's analysis showed significant variability in scaling resistance not between specimens of the same curing condition, but between the uses of the same curing compound on different mix types and between uses of different curing compounds on the same mix type.

In order to fully form a protective membrane, both curing compound manufacturers and the American Concrete Institute instruct operators to ensure the concrete surface is free of bleed water prior to curing compound application. If the surface still has bleed water present, or is hydraulically active, there is the potential that the curing compounds, which are typically less dense than the bleed water, may segregate and allow for weaknesses within the curing surface to develop. Within the Phase I study, all compounds were applied two hours after finishing operations were completed. However, it was unclear as to whether bleed water was present or the bleeding process had ceased at this two hour application time for all the mix types involved. Therefore, it is theorized that those specimens that saw increased amounts of scaling had imperfections within the curing compound membrane due to the presence of bleed water during curing compound application.

Chapter 2 **Objectives and Scope of Study**

The primary objective of this study (Phase II) was to determine the impact of the application time of curing compounds on the scaling resistance of concrete. The application time was measured as the time between the final finishing of the fresh concrete surface and the curing compound application. It is hypothesized that the presence of concrete bleed water at the time of curing compound application negatively impacts the durability of the concrete surface by preventing the curing compound from forming a complete water-retaining membrane necessary to ensure adequate curing. The length of the curing compound application time was chosen as the primary variable to reflect the time dependency of fresh concrete bleeding.

A secondary objective of this study was to determine the repeatability of curing compound performance with comparisons to the results from Phase I.

The tertiary and most difficult objective of this research was the determination of a process that quickly and reliably identifies the optimal time for curing compound application based upon the extent of the bleeding process. Using a thermohygrometer apparatus, an investigation into the feasibility of developing a robust relationship between the presence of bleed water and the relative humidity of the air immediately above the concrete surface was performed.

All materials used in the manufacturing of the concrete specimens studied were representative of materials used in Portland cement concrete (PCC) pavement projects within Wisconsin. All mix designs used in the manufacturing of the concrete specimens studied within this project were in accordance with the Wisconsin Department of Transportation Grade A, Grade A-S, and Grade A-F mix designs listed in the WISDOT Standard Specifications (WISDOT 2013). Within the specimen manufacture mix matrix, the primary factor separating the mix types was the coarse aggregate; followed by the secondary factor of cementitious material used (Table A-1). Two coarse aggregate types, representative of geology found in the Northern and Southern

regions of Wisconsin were chosen. In addition, one fine aggregate source was used. Finally, three cementitious materials conditions were used: ordinary Portland cement (OPC), OPC and Ground Granulated Blast Furnace Slag (referred to as slag cement or slag), and OPC with Class C Fly Ash. This resulted in a total of six concrete mix types. All mix types included one brand of air entrainment agent and one brand of water reducer. Chapter 4 contains more detail on the aggregates used and the mix design.

To evaluate the effect of MFCC application time on scaling resistance, five curing conditions were studied: three curing compounds, one sealing compound, and one untreated, wet room cured condition. Despite fundamental differences between sealing and curing compounds, they are all referred to as curing compounds for consistency within this document. Two application times were used for all concrete specimens within this study, 30 Minutes and 4 Hours. A third application time of 2 Hours for three of the compounds only on mixes containing OPC or slag cement was included to provide a link back to the Phase I study which only used 2 Hours. These three times corresponded to anticipated stages of the bleeding process: 30 Minutes to represent a time when bleeding was active, 4 Hours to represent where the bleeding process had ended and bleed water was no longer present, and 2 Hours to represent where the bleeding process might be either complete or incomplete. In total, 66 total mix type-curing compound-application time treatments were evaluated according to ASTM C672 for scaling resistance to assess the hypothesis.

A thermohygrometer apparatus was designed, built, and an operational procedure was developed to assess the feasibility of the secondary goal of this project. The design and operation are presented in further detail in Section 4.6.

Chapter 3 **Background**

3.1 **Membrane Forming Curing Compounds**

The curing environment of concrete has been known to influence surface hydration of concrete flatwork and thus the development of strength, durability, freeze-thaw scaling damage resistance, permeability, aesthetic appearance, and other important properties. For high surface area-to-volume structures such as roadway slabs, the fresh concrete comprising these structures is often poured when solar radiation, wind, high temperature, and low relative humidity are unavoidable. Ideal curing environments for exposed fresh concrete surfaces historically involved labor and time intensive practices such as ponding or covering with damp burlap (Vandenbossche, 1999). This was necessary to ensure the concrete mix water required for hydration of the cement binder was not removed by highly evaporative environmental conditions.

Membrane forming curing compounds (MFCCs) were developed to create a similarly effective evaporative barrier that could be sprayed quickly over large swaths of the exposed surface, reducing the labor and time required of previous methods. These compounds typically are solutions containing a solid fraction suspended in a water carrier or an organic solvent. Upon placement, the water or solvent fraction evaporates, depositing the solid fraction on the concrete surface that forms the membrane (Vandenbossche, 1999). If applied properly, curing compounds provide an even, uninterrupted physical membrane that prevents the egress of mix water during curing and the ingress of external chemicals like deicers. Many states require pigmentation of the MFCCs used on roadway projects, as they provide the additional benefits of increasing solar radiation reflectance and boosting the aesthetic quality of the surface (Vandenbossche, 1999).

The effectiveness of a curing compound is governed by the integrity of the membrane it forms. Several factors can impact the integrity of the membrane: the quality of the curing compound material (Choi et al., 2012), the amount applied, the application time, the curing compound selected (Kropp et al., 2012), the surface texture (Vandenbossche, 1999), concrete bleeding (Jana, 2007), and environmental conditions (Ye, Shon, Mukhopadhyay, & Zollinger, 2010). Many of these factors can be introduced when poor application procedures are used. American Concrete Institute recommendations stipulate that the optimum time to apply MFCCs is after final finishing, when the concrete surface is free of bleed water (ACI Comittee 308, 2001). This guidance is complicated by evaporative conditions that when too high can prematurely signal the end of bleeding; or when too low extend the period of free water on the surface. This condition has been particularly troublesome, as multiple studies have noted that pinholes and cracks can form in the membrane as bleed water segregates the freshly placed curing compound (Valenza II & Scherer, 2007b; Vandenbossche, 1999). Current application procedures outlined in WISDOT Standard Specification do not provide guidance on how to accurately assess compliance with minimum spray rates (WISDOT 2013). As a result, application has been controlled by subjective visual inspection. If spray nozzle patterns are adequately positioned (Vandenbossche, 1999) and flow rates accurately measured, application rates can be easily obtained as a function of the application cart speed, removing the subjectivity of the operator (Choi et al., 2012). The membrane integrity can also be improved if the application carts have adequate wind shielding to prevent loss of material during application (Vandenbossche 1999; Choi et al. 2012). Moisture retention is an important function of an MFCC, as adequate moisture is required for proper strength and durability development in the concrete surface layer. However, studies have cast doubt on the effectiveness of the current ASTM C 156 procedure for evaluating the moisture retention of MFCCs and its accuracy for use on actual concrete surfaces (Vandenbossche, 1999; Ye et al., 2010).

3.2　Concrete Surface Formation and Microstructure

. While many hydration reactions occur due to the complexity of Portland cement (H. F. W. Taylor, 1997), the two that are most attributed towards the creation and hardening of the paste that binds the constitutive parts of concrete are the hydration of tricalcium silicate ($3CaO \cdot SiO_2$, or C_3S) and dicalcium silicate ($2CaO \cdot SiO_2$, or C_2S) with water (H_2O) (Mamlouk, 2006); shown as equations (1) and (2) respectively.

$$2C_3S + 6H_2O \rightarrow 3C\text{-}S\text{-}H + 3CH \tag{1}$$

$$2C_2S + 4H_2O \rightarrow 3C\text{-}S\text{-}H + CH \tag{2}$$

The products of these hydration reactions are calcium silicate hydrate ($CaO \cdot SiO_2 \cdot 3H_2O$, or CSH) and calcium hydroxide ($Ca(OH)_2$, or CH). CSH gel formation is very important not only for its strength and binding properties, but the physical structure of the paste itself can greatly impact the chemical resistance and durability of the concrete surface. To develop an adequate CSH gel network, both of these reactions need water as shown in the hydration equations. This should be provided by proper mix design and adequate curing.

A fully mature hardened concrete surface requires time for the hydration of the CSH gel layer to form in the presence of enough hydraulically active water. The microstructural development of this layer determines the strength and durability properties of the exposed concrete surface (Snyder & Bentz, 2004). Immediately after placement, the concrete paste can be thought of as a suspension of rigid particles in water (Bentz, 2008). Solid particles such as coarse cement particles, fine and coarse aggregates will migrate towards the bottom of the concrete due to gravity, while mix water will rise, referred to as bleeding once it reaches the surface (Radocea, 1992). Gradients develop within depth of

the concrete, with the top layer characterized by a lower density, higher (w/c), and a coarser capillary pore network that is initially filled with water (Bentz, 2008). Hydration is intended to connect the original cement particles into as strong a network of hardened CSH as possible, while disconnecting these original water-filled capillary pores (Bentz and Stutzman 2006). Autogenous shrinking of the paste occurs as the hydration products occupy less volume than the water consumed during the reactions. While this shrinkage can reduce the size, coarseness, and interconnectivity of the capillary pore network, it can also lead to tensile stresses that eventually result in cracking that reconnect the capillary pore network. If pore space where water was pulled from empties, the pore becomes a void; which in tandem with resulting nearby unhydrated cement particles, reduces the strength, durability, and density of the surface layer by a process called self-desiccation (Bentz, 2008). The porosity of the hardened paste ultimately determines the permeability of the surface. Decreasing the (w/c) ratio results in poor pore network connectivity, which greatly reduces the permeability and increases the durability of the paste (Bentz and Stutzman 2006). The introduction of aggregates, both coarse and fine, can reduce strength by increasing the porosity through two means: increasing the diameters of the capillary pores (Winslow & Liu, 1990), and the entrapment of voids around the aggregates by 'honeycombing' (Jana, 2007). All sources of porosity can increase the level of permeability, which is a determinant of how resistant the concrete is to ingress of aggressive agents. This ultimately influences the durability of the concrete surface (Song & Kwon, 2007).

3.3 Concrete Bleeding

Bleeding of concrete has effects on the microstructural development of the surface layer. Research has determined that the primary properties of concrete bleeding are the rate and the total amount of accumulated bleed water. There are many factors that influence the process and the resulting properties: water content of the mix, fineness of the cementing materials (Radocea, 1992),

slab thickness, retarding admixtures (Jana, 2007), aggregate absorption, aggregate fines (Topçu & Elgün, 2004), air entrainment, cement composition, and the use of supplementary cementitious materials (SCMs) such as ground granulated blast furnace slag cement or fly ash (Valenza II & Scherer, 2007b). Increased bleeding can reduce surface strength, increase permeability, and delay the onset of finishing operations; while decreased bleeding can increase plastic shrinkage and poor surface hydration (Topçu & Elgün, 2004).

Bleeding is a time dependent process, with the highest rates of bleeding in OPC concrete typically occurring early within the first 30 minutes after placement, followed by a steady decline in the rate over the following hour as the concrete begins to harden (Josserand 2004; Josserand et al., 2006). The use of slag and fly ash can result in an increase in the total amount of bleed water by bleeding for a longer period than typical OPC concrete (Afrani & Rogers, 1994; Wainwright & Rey, 2000). It is hypothesized that this is due to the SCMs requiring longer hydration times that extend the time to initial set leading to more bleeding in SCM concretes (Valenza II & Scherer, 2007b). Entrained air bubbles used for freeze/thaw resistance reduce bleeding in the paste by adhering to cement particles, inhibiting downward settlement through increased buoyancy. The cement particles hydrate and form a hardened network that blocks bleeding channels, reducing the amount of bleeding (Valenza II & Scherer, 2007b).

Construction practices are impacted by a concrete's bleeding behavior. This manifests itself in the ACI guidance for finishing and MFCC use as stated previously. The evaporative environment can mask when the true cessation of bleeding occurs. A reduction in the relative humidity from 80% to 50% can lead to an increase in evaporation of 100%; while increasing the wind speed can increase the evaporation nearly 200% (Topçu & Elgün, 2004). Finishing prior to the cessation of bleeding increases the amount of water in the surface of the paste, which reduces the strength and durability by

changing the increasing the (w/c) ratio and microstructure of the gel and air void network as it

matures (P. C. Taylor et al. 2004; Bouzoubaâ et al. 2008). This increase in (w/c) ratio of the paste is

also thought to occur when curing compounds are applied early, despite the disassociation of the

membrane discussed previously (Ye et al., 2010). Because both ACI and WISDOT offer guidance on

the construction practices in response to bleeding, determining when bleeding is complete along with

the absence of bleed water on the surface of the concrete is important. ASTM C232 offers a test for

evaluating the amount of mix water that will bleed. However, studies have shown that results from

this procedure correlate poorly with the actual bleeding behavior of concrete (Choi et al., 2012) and

that it does not account for environmental evaporative conditions even when the ACI-issued

nomograph for evaporation conditions is used (Uno, 1998). While several proposed methods show

promising results at offering a replacement, practical problems exists within their procedures for

implementation on construction sites (Radocea 1992; Josserand et al. 2006; Ye et al. 2010; Choi et al.

2012).

3.4   Concrete Scaling Damage

Concrete Scaling is defined as 'local flaking or peeling away of the near-surface portion of

hardened concrete or mortar' (ACI Comittee 308, 2001). Severity of scaling is often identified by

visual inspection of the removal of mortar that exposes the near-surface aggregates; or by measuring

the depth and mass of the lost material.

In order for concrete freeze-thaw scaling to occur, several conditions must be present. First,

water with deicing chemicals must pool on the concrete surface (Valenza II & Scherer, 2007b).

Second, scaling can occur if the minimum temperature is less than -10°C (14°F) (Valenza II &

Scherer, 2007a). Most importantly, scaling damage only occurs when the concentration of the deicing

chemical within the pooled water is approximately 3% by mass of the total solution; an amount called

the pessimum concentration (Verbeck & Klieger, 1957). The Glue Spall method was developed by Valenza and Scherer to explain both the mechanism of scaling and the purpose of the requirement of the pessimum concentration (2006). As pooled water with deicer freezes on a concrete surface, a mechanical bond forms between the layer of ice and the concrete. As the temperature drops further, the thermally expanding ice will crack where there are pockets of deicing brine that weaken the ice. Because the bond between the ice and the roughened concrete surface is stronger than the concrete itself, the crack in the ice will penetrate into the concrete layer (Çopuroğlu & Schlangen, 2008). In successive cycles, the brine that occupies the crack in the concrete will pull out pore water, diluting the solution and allow for further cracking to develop as ice forms. Once the crack reaches the top layer of coarse aggregate, the crack will propagate approximately parallel to the surface of the concrete due to the poor bond between the mortar layer and coarse aggregate at the interfacial transition zone (ITZ) (Ollivier et al., 1995). Similarly-propagating cracks will eventually weaken ITZ bond due to thermal stresses, resulting in the layer removal referred to as scaling (Valenza & Scherer, 2006). The Glue Spall theory uses the strength of the ice to explain the existence of the pessimum concentration, regardless of what deicer is used. Pooled solutions of pure water or less than 3% deicer do not cause scaling because the ice layer is too strong for cracks to form with the ice. For solutions with concentrations higher than 5%, the ice is typically too soft to bond with the concrete and develop fracturing stresses during thermal expansion (Sun & Scherer, 2010; Valenza & Scherer, 2006; Wu, Shi, Gao, Wang, & Cao, 2014). If the depth of the ice layer is large, the tensile cracking forces transferred into the concrete surface are larger, resulting in higher cracking stresses within the concrete surface layer (Çopuroğlu & Schlangen, 2008).

3.5   Scaling Damage Resistance of Concrete

Concrete resistant to scaling requires a surface microstructure that is strong, air entrained, and of low permeability. As the temperature drops, pore water migrates towards entrained air voids where ice nucleation can occur. This has the dual effect of preventing frost action damage due to pore water expansion in the pore network, while also contracting the bulk mortar layer; counteracting the expansive stresses from the bonded ice layer that would result in Glue Spall cracking (Sun & Scherer, 2010; Valenza II & Scherer, 2007b). Air voids should typically be less than 300 μm in diameter, and spaced properly for scaling resistance. A critical spacing factor for air voids in the mortar layer has been identified to correspond well with scaling resistance. Scaling mass loss is roughly proportional to the amount the spacing factor between air voids exceeds the critical amount (Valenza II & Scherer, 2007b). While it has been determined that the compressive and tensile strength of the bulk concrete has little impact on the scaling resistance, (Afrani & Rogers, 1994) it is recommended that the concrete be matured to at least 4000 PSI prior to first exposure of deicing salts (Jana, 2007). A lower (w/c) ratio corresponds with decreased porosity and increased strength, resulting in a more durable surface. At a (w/c) ratio of 0.3, it has been found that no air entrainment in the mortar layer is necessary to prevent scaling due to the density of the microstructure (Valenza II & Scherer, 2007a, 2007b).

Aggregate and SCM use can impact the scaling resistance of concrete. The use of slag or fly ash can result in poor microstructural development by hindering the concrete's ability to entrain air and retain adequate spacing factors between air voids; resulting in increased permeability and scaling susceptibility (Giergiczny, Glinicki, Sokołowski, & Zielinski, 2009; Valenza II & Scherer, 2007b). Carbonation of hydration products in concretes containing slag can destabilize the fresh CSH, ultimately reducing the scaling resistance (Battaglia et al.2010). Aggregates that have high porosity,

high absorption, and low moduli of elasticity are particularly susceptible to aggregate popout scaling, especially when critically saturated as a result of high permeability of the mortar paste. Aggregate popout is a form of scaling where the aggregate underlying the paste fractures due to thermal expansion when saturated with water during freezing. This results in upwards crack propagation, towards the concrete surface, resulting in localized scaling immediately above the fractured aggregate (Jana, 2007).

There is limited research on the impact that curing compound use has on the ultimate scaling resistance of concrete. Previous research showed that curing compound use results in more scaling compared to wet burlap or other moist curing methods (Kropp et al. 2012). In addition, curing compounds delayed the onset of scaling damage only slightly, with significant mass loss occurring after only 5 freeze/thaw cycles in ASTM C672 testing.

ASTM C672 is a standard testing procedure for measuring concrete scaling. The procedure involves pooling a ¼-inch layer of 4% by weight sodium chloride (NaCl) solution on a concrete surface, subjecting it to freezing and thawing cycles, and measuring the cumulative mass lost by sieve collection and subjective visual inspection. There is concern that this testing procedure does not produce results that are the same as that from in-situ slabs. As with many accelerated concrete tests, the ASTM C672 procedure produces more severe damage than a typical natural freeze-thaw situation would induce (Boyd & Hooton, 2007; Valenza II & Scherer, 2007b). In addition, the visual rating system outlined in ASTM C 672 introduces inter-operator variability due to its subjective nature (Taylor et al. 2004; Valenza II and Scherer 2007a; Bouzoubaâ et al. 2008).

Chapter 4 Materials, Methods and Testing Procedures

4.1 Concrete Materials

The concrete tested within this study was prepared following the guidelines of the WISDOT Concrete Grades A, A-FA and A-S for concrete pavements containing cementitious materials consisting of either Ordinary Portland Cement (OPC, and a blend of either OPC and Class C Fly Ash (FA) or Grade 100 Ground Granulated Blast Furnace Slag (GGBFS), respectively. For each mix, the total cementitious material content was 565 lbs/yd^3. In addition to the cementitious material, coarse aggregate and fine aggregate fractions were used with Madison Municipal tap water for all mixes. A water to cementitious materials ratio (w/c) of 0.40 was used for all mix types, except for three batches of Mix Type 4 where a (w/c) of 0.41 was used to improve workability. The mix designs are provided in Appendix D. Two chemical admixtures were added to each mix: a low range water reducing agent (WRA) was used to meet the slump requirement of 3±1 inch and an air entrainment agent (AEA) to meet the entrained air requirement of 6±1% (WISDOT 2013).

Two types of coarse aggregate were used to reflect the predominant aggregates used in concrete pavements throughout Wisconsin. The first aggregate was a crushed limestone from Waukesha County; while the second was a glacial gravel from Eau Claire County. The crushed limestone was angular, homogenous in particle composition, and had an approximate 2:1 to 2.5:1 length to width ratio. The glacial gravel was smoother and rounder, with more particles having a 1:1 length to width ratio. The gravel contained several mineral types and was dustier than the limestone based upon visual observation. Despite this, both aggregates met the WISDOT Standard Specifications for gradation sizes (see Figs. 4-1 and 4-2), P200 content, and absorption (WISDOT 2013) as shown in Tables 4-1 and 4-2.

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Table 4-1: Coarse Aggregate Properties

Coarse Aggregate Type	Specific Gravity	Absorption	P200
Glacial Gravel	2.658	0.94%	0.50%
Limestone	2.712	1.51%	0.20%
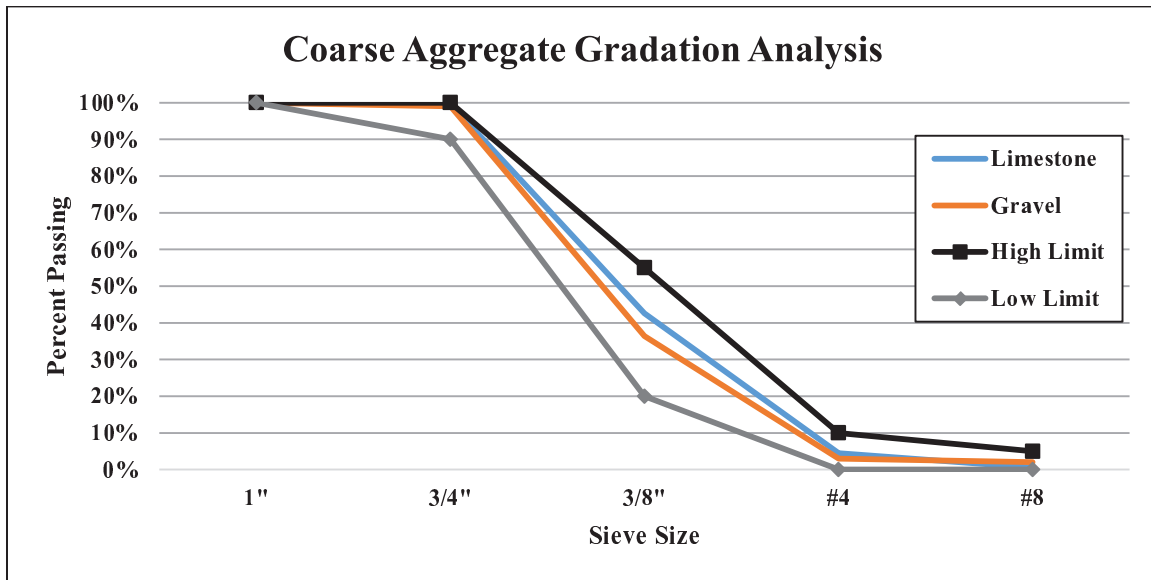
Figure 4-1: Coarse Aggregate Sieve Analysis

A single fine aggregate source from Southern Wisconsin was used for all mixes. It met the WISDOT specifications for gradation, P200 content, and absorption.

Table 4-2: Fine Aggregate Properties

Fine Aggregate Type	Absorption	P200
Sand 1	0.19%	1.17%

Fine Aggregate Gradation Analysis

A chart titled "Fine Aggregate Gradation Analysis" with Y-axis "Percent Passing" (0% to 100%) and X-axis "Sieve Size" (3/8", #4, #16, #50, #100). Three series shown: Fine Aggregate, Low Limit, High Limit.
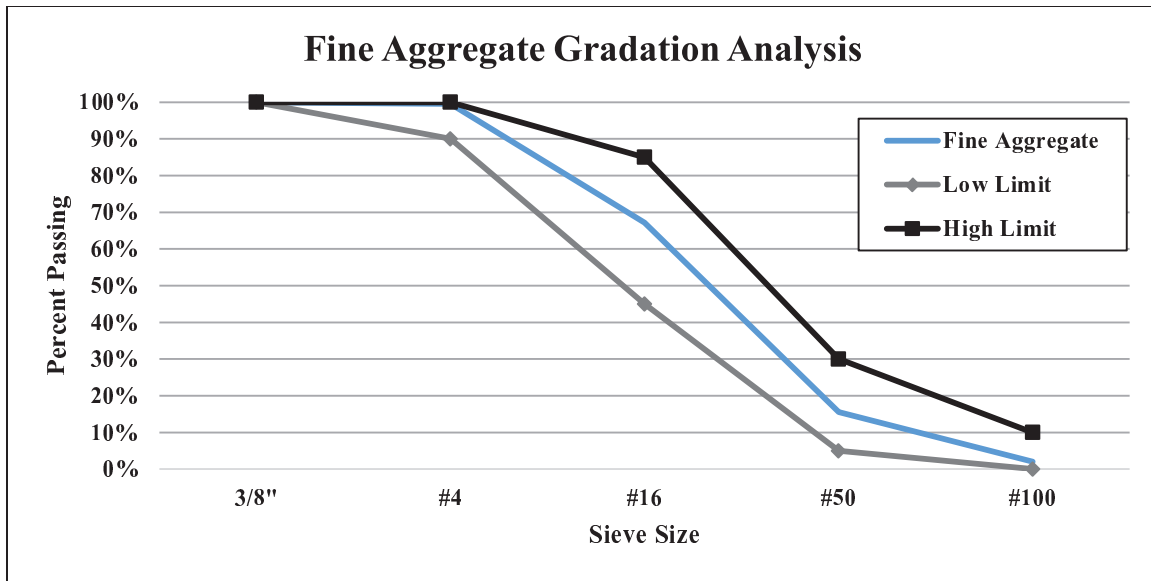
Figure 4-2: Fine Aggregate Sieve Analysis

4.2   Specimen Molds

ASTM C672 requires specimen dimensions of at least 72 in^2 of exposed concrete surface, with a depth of at least 3 inches. The molds used to fabricate these specimens were created using 12 inch inner diameter Schedule 40 PVC pipe sections, cut into three inch segments. These segments were adhered to a melamine coated particle board with polyurethane construction adhesive. These proved to be durable, reusable, non-absorbent, they allowed for a smooth strike off surface, produced uniformly-sized specimens, and were easy to remove with air pressure applied to the non-testing side. Prior to mixing, each mold was coated lightly with a mold release agent to aid in the demolding process.

4.3   Methods

4.3.1   Mixing Procedure and Fresh Concrete Tests

A rotary drum mixer located within a temperature controlled, humidity monitored laboratory environment was used to mix all concrete for this project following the protocol set in ASTM C192. Prior to batches that were used to pour specimens, a butter batch of the same mix components but totaling a concrete volume of 0.66 ft^3 was used to coat the inside of the mixture to prevent concrete loss and to test the slump and air content following the ASTM test protocols C143 and C231 respectively. Calibration of the air content pressure meter was performed with each coarse and fine aggregate mixture to calculate the Air Correction Factor following the procedure in Item 6 of ASTM C143. These are listed in Table 4-3.

Table 4-3: Air Content Correction Factor for Mixes

	Gravel and Sand	Limestone and Sand
Air Content Correction Factor	1.2%	1.1%

Each concrete batch were either 2.7 ft^3 or 2.0 ft^3 in volume to allow for the slump, air content test, three 4-in by 8-in compressive strength cylinders, one 3-in x4-inx16-in prism for the ARCHERS test, and nine or six ASTM C672 specimens, respectively. These batches were large so as to ensure that the slump and air content test concrete was not used in specimen manufacture; and that there was additional concrete remaining.

The coarse and fine aggregates were dried in an oven on the day prior to mixing, and portioned out by weight after enough time had passed to allow them to cool to room temperature while covered. Recordings of the laboratory temperature and relative humidity were taken before

proceeding with the mix. The batch was weighed out and split into two buckets, so that the WRA and AEA could be dispersed among the batch water independent of one another. The coarse and fine aggregates were added to the rotary mixer; then the water bucket containing the WRA was added. The mixer was turned on, then immediately the cementitious materials were added followed by the remaining water. The concrete mixed for three minutes, then the mixer was stopped for three minutes and covered to prevent evaporation. The mixer was turned on for an additional two minutes, then poured in to a wheelbarrow. Any excess concrete remaining in the mixer was scraped out into the wheelbarrow, where it was mixed for 30 seconds with a hand scoop. The slump and the air content of the fresh concrete were then determined by ASTM C-143 and C-231 respectively. After evaluating that both of these fresh concrete tests fell within their acceptable limits as indicated in the work plan; specimen manufacture began.

The specimen molds were filled by scoop in one layer, and were rodded once per ever two square inches of specimen surface area on the exposed side, per ASTM C-672. The sides of the mold were then tapped with a rubber mallet fifteen times to ensure adequate consolidation; followed by a troweling of the surrounding mold walls to prevent adherence to the wall surface during demolding. Finally, a wood strike off board was used to screed the surface of each mold, using between 12-15 passes in order to ensure a surface level with the height of the mold walls. Once each ASTM C672 specimen was made, three 4-in by 8-in compression cylinders were made using the remaining concrete in accordance with ASTM C192.

4.3.2   Curing Compound Application

Curing compounds were applied for each mix type at three separate times after the screeding effort was complete; unless otherwise noted: 30 minutes, two hours, and four hours. Three replicate specimens for each curing compound application time for each mix type, for a total of nine specimens

per batch were made. The curing compounds were sprayed on the specimens using handheld pressurized nozzle sprayers with the ability to provide even coverage over the specimen. The specimens were placed on an electronic digital scale capable of 0.1 g accuracy. Alternating perpendicular passes over the specimens were used to ensure an even coating of the entire specimen surface. A wooden mask was used to prevent overspray of the mold wall and base. Each specimen was coated with an amount of curing compound as close to the manufacturers recommended spray rate, as is noted in the WISDOT Standard Specifications. Care was taken to ensure that each specimen's surface condition was not disturbed when moving to and from the curing location within the laboratory and the digital scale.

4.3.3　Specimen Preparation

Each specimen was cured for 24 hours in laboratory air, then demolded and labeled. After demolding, each specimen had a 10" inner diameter PVC dam of one inch in height affixed to the sprayed surface with polyurethane construction adhesive. Following the curing time for the construction adhesive, a two part low modulus/low-viscosity sealing epoxy was applied to the non-testing surfaces of the specimens. Once the epoxy had hardened, the edges between the PVC dam and the outside rim of the specimen were sealed with a silicone based caulk to prevent leakage of the deicing solution during testing. After curing for the required amount of time, the specimens were transported to the UW-Madison Biotron building where the freeze-thaw testing commenced.

4.3.4　Compressive Strength

In accordance with ASTM C192, three 28-day compressive strength cylinders were made in each mix where ASTM C672 specimens were made. These cylinders were demolded after 24 hours, placed in a humidity controlled wet room with 100% relative humidity for 28 days. After this curing

time, the cylinders were capped with a sulfur-based capping compound; and tested to failure for compressive strength following the procedure in ASTM C39.

4.4   ASTM C672: Standard Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals

After each sample was taken to the Biotron, a picture of the surface was taken to indicate the visual condition of the surface prior to the test. With the dams adhered to the surface of each specimen, a ¼-in deep (approximately 330 mL) of 4% by weight sodium chloride (NaCl) solution was added to the surface of each specimen. The solution was carefully agitated without harming the surface to prevent settling of undissolved NaCl crystals. The top of the dams of each specimen were covered with a non-restrictive polyethylene sheet to prevent evaporation, and then placed within the Biotron freeze-thaw chamber. Within this chamber, the temperature cycled between $32^{o}C$ $(90^{o}F)$ for four hours and $-16^{o}C$ $(3^{o}F)$ for twenty hours. Every fifth cycle during the thaw phase, each specimen was taken out of the environmental chamber and the solution was decanted over a #200 sieve. A gentle stream of water was used to rinse off the surface of each specimen. Once the scaled mass for each specimen was collected, the solution was replaced, and the specimen was returned to the freeze-thaw chamber. Any specimens that had noticeable leaking were resealed with three-hour caulk, and returned the same day. Each sample was subjected to 60 cycles. Pictures of each specimen were taken after 30 cycles and 60 cycles, to show visual scaling damage progression at the halfway point and full testing period, respectively.

Once the scaled material had been collected, it was transferred into heat resistant test tubes, then dried out at $80^{o}C$ $(176^{o}F)$ in a laboratory oven for at least 24 hours. Each sample of scaled

material was then weighed out on a digital balance with a precision of 0.1 grams. The scaled material for each mix type-curing compound-application time set was averaged over three specimen duplicates, and analyzed.

ASTM C672 includes a procedure for assessing the surface damage progression by assigning a number rating based upon visual inspection. The researchers felt that the mass loss evaluation procedure was more meaningful for the purposes of determining scaling resistance, and that visual inspection by the operator was inherently too subjective to have scientific merit.

4.5   MFCC Types and Specifications

Each curing compound treatment types used on this project either classified as emulsion-based or curing/sealing type. The spray rates for each treatment type were generated based upon the manufacture's recommendation of spray rate, converted from ft^2/gallon to grams/area of specimen based upon the estimated density of the curing compound provided by the manufacturer.

4.5.1   Emulsion MFCCs

The emulsion type MFCCs consist 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 autogenically reactive (polymerizing).

4.5.1.1   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. The manufacturer specified a 40 to 50% solid fraction. This compound had a viscosity greater than water and emitted a mild distinctive odor. As a water emulsion it emitted few volatile organic compounds (VOCs) and requires no special handling. This coating provides a reflectance of 67% and upon application provided a yellowish tint. The spray rate was 14.9 grams/specimen.

4.5.1.2   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 met ASTM C-309 specifications as a Type II Class A 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. The manufacturer specified a 15 to 25% solid fraction. This compound had a viscosity slightly greater than that of water and emitted little odor. As a water emulsion it emitted few VOCs and requires no special handling. The manufacturer did not provide reflectance data. The product was bright white in color. The spray rate was 14.9 grams/specimen.

4.5.1.3   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 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 has a specific gravity

between 0.97 and 1.03 g/ml. 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 yellowish-white appearance. As a water emulsion it emitted few VOCs and requires no special handling. PAMS resin undergoes cross-linking reactions at sufficient concentration, so as the water carrier evaporates the coating consolidates and seals the surface. The spray rate was 14.9 grams/specimen.

4.5.2   Curing/Sealing type MFCC's

The curing/sealing MFCC used in this study was a solution of membrane forming organic compounds in mixed organic solvents. The organic solvent evaporated during application of this compound and was known to emit VOCs. The curing/sealing compound tested met EPA standards for VOC emissions and contained no pigmentation. It was designed to form an evaporative barrier and penetrate the porous network of the concrete surface. This is intended to seal the concrete surface and prevent the ingress of water or deicer solution.

4.5.2.1   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. 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 has a lower viscosity than water and a pronounced odor of organic solvents. There was no pigmentation added to this compound, it provided a surface with a minor shine. The VOC emissions from this coating are very flammable and hazardous, so coating operations generally require adequate

ventilation and removal of ignition sources. From the manufacturer's description, as the organic solvent evaporated the concentration of the monomers increased until a chemical reaction occurred with the pore water and consolidated the membrane. The spray rate was 9.1 grams/specimen.

4.6 Air Reconditioning Concrete Humidity Evaporation Research System (ARCHERS)

An objective of this study was the investigation of a practical method for determining the termination of the bleeding process of concrete. Currently, ASTM C232 exists as a method for determining the portion of mix water that will bleed by extracting bleed water with a pipette mechanism. This procedure is labor intensive, time consuming, and impractical for most job sites. Other methods have been proposed, but are equally labor and time intensive.

The Air Reconditioning Concrete Humidity Evaporation Research System, or ARCHERS, was created as part of this project to indicate the presence of bleed water on a concrete surface over time. The primary operational assumption behind the ARCHERS is that there is detectable gaseous water vapor in the air directly above a liquid due to evaporation. It was hypothesized that the evaporation of liquid bleed water could be evaluated as a change in the relative humidity immediately above the concrete surface. By monitoring the changes in the relative humidity of the air above the concrete over time, it was theorized that the cessation of bleeding could be identified by the gradual reduction in the relative humidity following the final evaporation and dispersion of liquid bleed water.

An enclosed box made of a lightweight, non-absorbent acrylic, containing a hygrometer (humidity probe) and thermocouple were used measure the relative humidity and temperature directly above the concrete. This is referred to as the contactor in Figure 4-3. The contactor minimized measurement interference from atmospheric conditions such as temperature, solar radiation, wind, or

humidity changes. Despite reducing ambient interference, a contactor constructed without an outlet would restrain the evaporated bleed water within, resulting in a constant near-maximum reading of relative humidity. To avoid this problem, a source controllable air flow, treated to operator-set constant temperature would have to replace the air immediately above the concrete surface to obtain a better relative humidity profile above the surface over time.

The ARCHERS layout containing the acrylic contactor is shown in Figure 4-3. The ARCHERS included a data acquisition instrument (DAQ) used to monitor and record the operational process variable of relative humidity, to adjust the primary output: the Fan Duty Cycle (FDC) of the inlet fan in Figure 4-3. This is done through the use of a Proportional-Integral-Derivative (PID) feedback loop within the DAQ software interface on a computer terminal. The DAQ measures a difference in relative humidity before and after a portion of air passes over the concrete surface ΔH, and compares that to a relative humidity difference set point, ΔH_{set}. The error between ΔH and ΔH_{set} is evaluated through the PID logic, changing the output of the FDC in the following time step. The value of the FDC versus time is continually plotted over time, visible to the operator on a graph on the computer terminal. This plot is used by the operator to determine the stage of the bleeding process by evaluating the FDC over time.
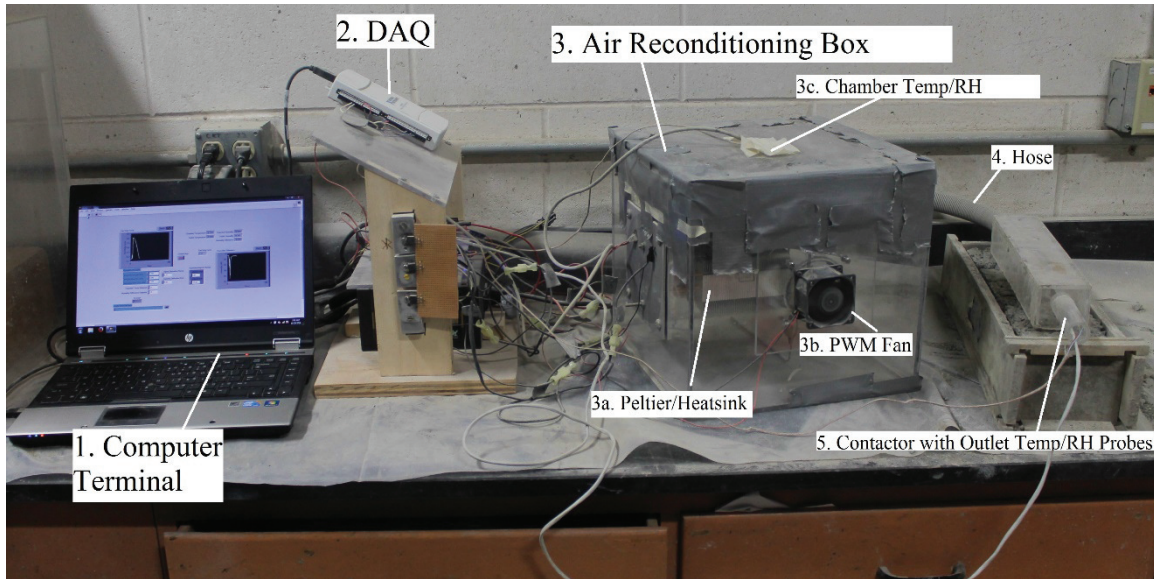
Figure 4-3: ARCHERS Layout

The operational procedure of the ARCHERS contains the following actions:

A. The computer running the DAQ software (1), DAQ (2), and Air Reconditioning Box (ARB) (3) are turned on. This includes power to the temperature and humidity probes.

B. Within the LABVIEW program, a ΔH_{set}, a temperature of the reconditioned air T_{set}, PID parameters, FDC limits and a time step for averaging readings is set by the operator.

C. Once the concrete is poured and the contactor (5) lightly positioned upon the concrete surface, the data logging procedure is turned on. This turns on the Peltier Thermoelectric Chip which is attached to a heat sink outfitted with a fan to disperse heat (3a) and inlet fan attached to the ARB (3b). This fan pushes the treated air to through a hose (4) to the contactor past the outlet temperature and humidity probes (5).

D. Temperature, Relative Humidity, and the FDC readings are collected within the DAQ and recorded to the computer. An output of the FDC versus time is plotted on the Computer.

E. The difference in humidity between the outlet humidity reading and the humidity within the ARB, ΔH (H_{Out}-H_{In}) is compared to the ΔH_{set}. PID logic within the LABVIEW control system evaluates the different between ΔH and ΔH_{set}:

    I. If $\Delta H > \Delta H_{set,}$ the FDC is increased by the PID gains, unless the FDC is already at its maximum limit

    II. If $\Delta H < \Delta H_{set}$, the FDC is decreased by the PID gains, unless the FDC is already at its minimum limit

    III. If $\Delta H = \Delta H_{set}$, the FDC remains constant

F. The system will continue to run for the duration of the curing period. As the amount of bleed water decreases, the FDC will eventually drop to the minimum limit, and is expected to stay there. After the FDC has remained at the minimum limit for an appropriate amount of time, the operator shuts off the program, concluding the test. A spreadsheet of the recorded data is then saved.

While the theory and construction of the ARCHERS remained the same during the research effort, parameters within the logic in the DAQ software were continually adjusted to mitigate operational challenges found in both the tuning phases and during actual bleed water testing on concrete mixes. Figure 4-4 shows a screenshot of the operational interface of the ARCHERS, followed by explanation of the operator inputs.
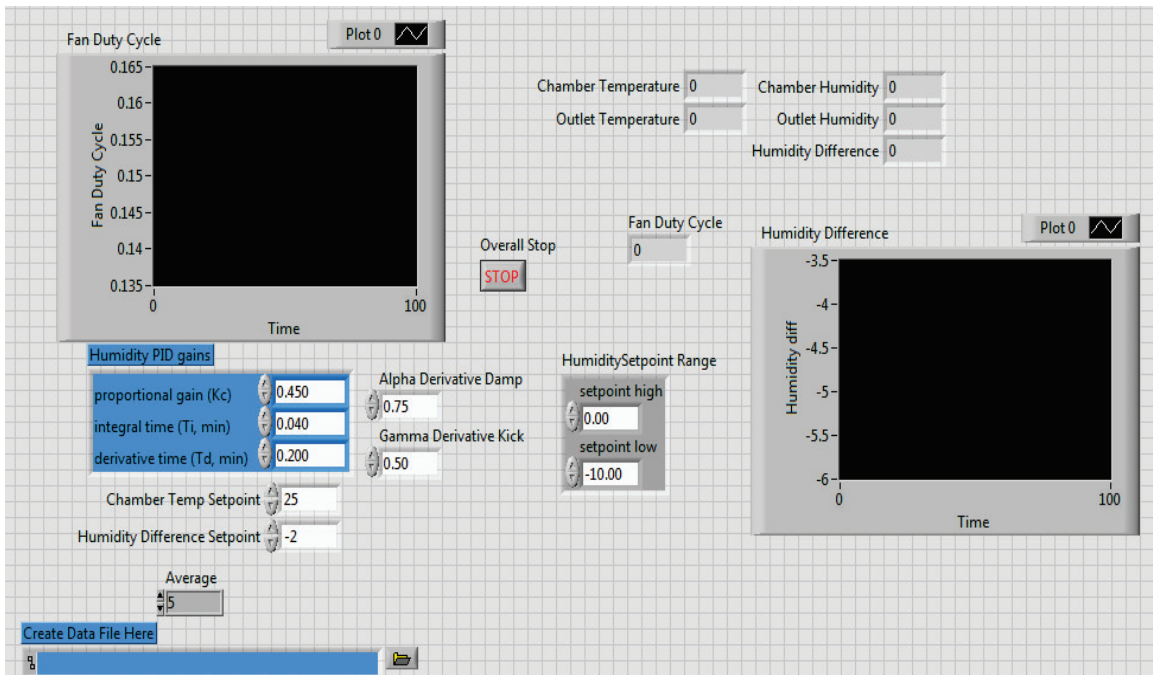
Figure 4-4: ARCHERS Computer Interface

- Humidity Difference Set Point: The process variable that drove the PID to change the FDC. This could be adjusted to react to environmental conditions and technical issues.

- Humidity PID Gains: These are values that govern FDC response to a difference in the humidity difference and the set point. These can be tuned manually or by established tuning methods to idealize the response of the system.

- Derivative Damp/Kick: Limits the instantaneous impact of the derivative term on PID output. It is helpful for reducing the response that individual spikes in the process variable may have on the FDC

- Temperature Set point: This controlled the Peltier heating chip to heat or cool off the air in the ARB based upon thermocouple readings

- Humidity Set Point Range: With the one humidity set point value, the response of the system would frequently 'ring' or oscillate around this set point. This range works in

tandem with the set point value by providing an upper limit slightly more than the set point. For example: if the set point humidity was 8% and the limits were from 0 to 10%, the FDC would not increase until the humidity was larger than 10%. This greatly eliminated perturbations in the data that would cause ringing.

- Average: Samples were acquired from the DAQ once every second. This parameter allows the operator to choose the period of averaging the data before it is plotted. Five second running averages were used for this project.

- Create Data File: Once the test had concluded, the operator could name a file and save it for further analysis.

Alterations to the various program parameters could be made with relative ease to tune the operational capacity of the ARCHERS to achieve better performance, or to notify the operator of potential problems with the device. Results and recommendations for the improvement of system performance are discussed in Section 5.6.3.

Chapter 5 **Results**

5.1   **Fresh Concrete Properties**

Five concrete batches per mix type were created corresponding to the five curing conditions examined in this study. Table 5-1 displays the fresh concrete properties averaged over the five batches per mix type. Complete fresh concrete properties are given in Appendix B. Each batch was designed to have a (w/c) ratio of 0.4; with the exception of Mix Type 4. This particular mix type created difficulties meeting the low end of the 3±1 inch slump requirement, even with the inclusion of large amounts of water reducer admixture. As a result, the (w/c) ratio was increased to 0.41. Average air content of the fresh concrete did fall within the 6±1% amount as mandated for WISDOT pavement projects. Mixes 4-6 were poured at times where the relative humidity within the laboratory was heightened, despite near constant temperature. Finally, all mixes prepared for this project exceeded 5000 PSI average 28-days compressive strength.

Specimens were labeled with a four digit identification number, with the first two being most important for the purpose of this data analysis. The first digit (1-6) indicated both the coarse aggregate and supplementary cementitious material (SCM) used, the second digit (A-E) identified the curing compound used. These are listed in Table 5-1 and Table 5-2, respectively. The third digit and fourth digits indicated the application time and replicate number, respectively.

Table 5-1: Fresh Concrete Mix Properties

Mix Type Number	Coarse Aggregate	Cementitious Material (by weight)	w/cm Ratio	Average Slump (inches)	Average Air Content (%)	Ambient Relative Humidity (%)	Compressive Strength (PSI)
1	Crushed Limestone	OPC	0.4	1.25	6.5%	24%	6200
2	Crushed Limestone	30% Slag	0.4	1.50	6.4%	27%	6110
3	Crushed Limestone	30% Fly Ash	0.4	2.50	6.2%	31%	5813
4	Glacial Gravel	OPC	0.41	1.50	6.4%	38%	5348
5	Glacial Gravel	30% Slag	0.4	1.25	5.8%	53%	6129
6	Glacial Gravel	30% Fly Ash	0.4	2.75	5.9%	52%	5817

Table 5-2: Curing Compound Designations

Wet Room	Emulsion			Curing/Sealing
None	Linseed	Wax	PAMS	Acrylic
A	B	C	D	E

5.2 ASTM C672 Results by Individual Mix Type and Curing Compound

The amount of scaling damage was measured by collecting and drying the mass of the scaled material that was collected on a #200 sieve every five freeze-thaw cycles, per ASTM C672. The scaled material was then divided by the exposed area of the specimen and converted to a grams-per-square-meter value. For each mix type-curing compound-application time, the scaled material per square meter value was generated by averaging three replicate specimens. Scaling damage accumulation curves were compiled by summing these average damage values over the 60 cycles of the ASTM C672 test.

Complete scaling damage data is located within Appendix C. Tables are included alongside scaling accumulation charts in this section that include the relative humidity at the time of specimen manufacture, the total amount scaled at 30 and 60 cycles, the percent of damage occurring within the first 15 cycles, and the percent change in scaling damage between the 30 Minute specimens and the 2 and 4 Hour specimens.

5.2.1 Effect of Application Time on Mix Type 1: Crushed Limestone and Ordinary Portland Cement

Mix Type 1 batches were made with crushed limestone aggregate and Ordinary Portland Cement. For all charts in this section, a scale up to 700 g/m^2 was used for consistency.

5.2.1.1 Wet Room Cured

Table 5-3 shows that the wet room cured specimens for Mix Type 1 suffered an average of 17.1 g/m^2 in scaling damage. In Figure 5-1, no significant scaling accumulation was detected from 15 cycles to 45 cycles. This set was predicted to have the least amount of scaling of all specimens sets from Mix Type 1 by virtue of being cured in an ideal 100% relative humidity environment prior to freeze-thaw exposure.

Table 5-3: Mix 1-A Scaling Data from ASTM C672 Tests

Mix 1-A Wet Room Cured			
Relative Humidity	Scaled Mass at 30 Cycles (g/m^2)	Scaled Mass at 60 Cycles (g/m2)	Damage Occuring in First 15 Cycles (%)
27%	2.0	17.1	12%
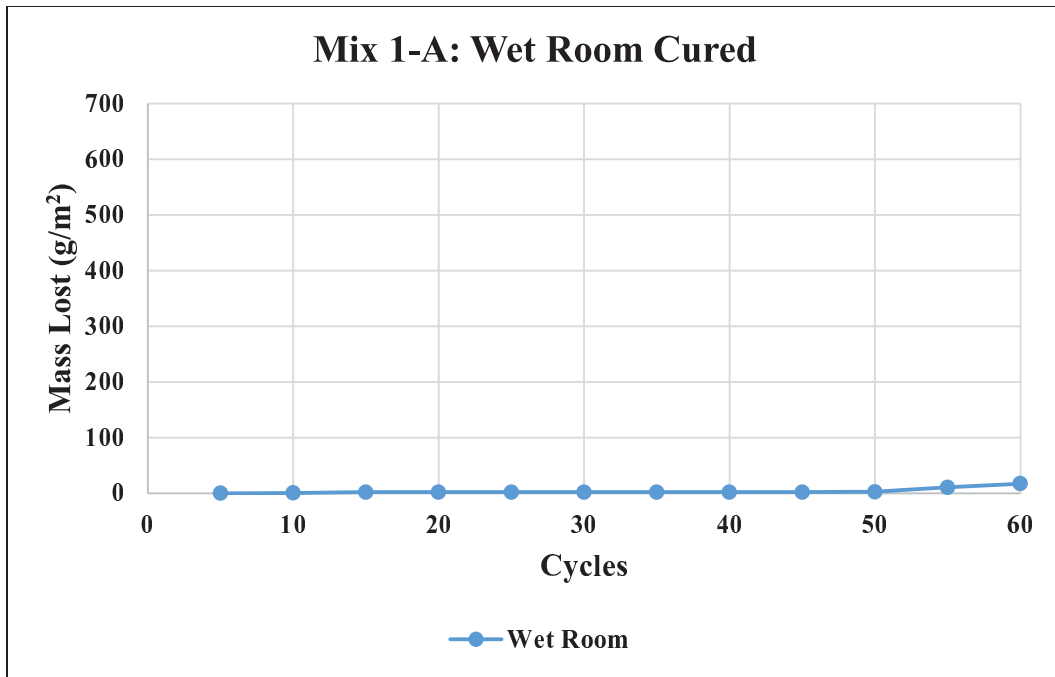
Figure 5-1: Mix 1-A Cumulative Scaling from ASTM C672 Tests

5.2.1.2   Linseed Oil

The scaling data for Mix 1-B specimens is located in Table 5-4 and the damage accumulation curves are shown in Figure 5-2. For the 30 Minute and 2 Hour specimens, the mass loss rate was very low initially, then accelerated during the last 40 cycles. The 4 Hour application time also possessed low initial scaling, but delayed the increase in mass loss rate until after 30 cycles. Lower amounts of total scaling were exhibited in specimens with longer application times.

Table 5-4: Mix 1-B Scaling Data from ASTM C672 Tests

Mix 1-B Linseed Oil						
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m^2)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
21%	30 Minutes	149.3	-----	479.5	-----	4%
	2 Hours	135.5	-9%	446.0	-7%	7%
	4 Hours	30.3	-80%	344.0	-28%	4%
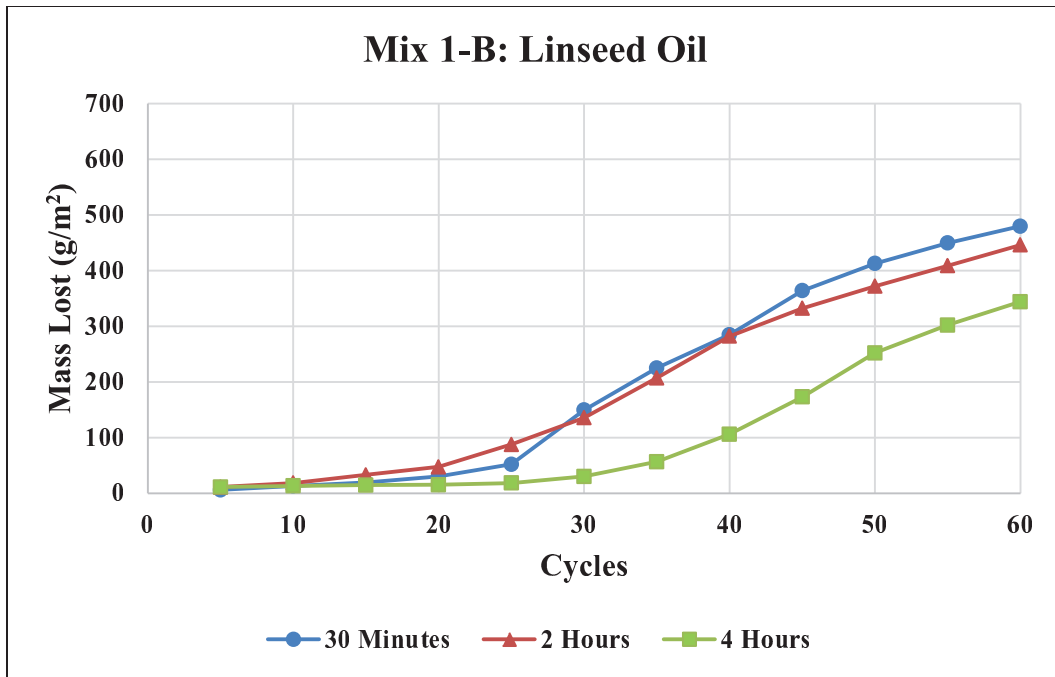
Figure 5-2: Mix 1-B Cumulative Scaling from ASTM C672 Tests

5.2.1.3   Wax

The damage accumulation curves for Mix 1-C in Figure 5-3 indicate that specimens at all three application times scaled very slowly for the first 45 cycles, then rapidly lost mass during the final 15 cycles. In Table 5-5, the 4 Hour specimens exhibited marginal reductions in scaling damage as compared to the 30 Minutes specimens. However, the 2 Hour specimens suffered over twice the amount of total scaling damage after 60 cycles as the 30 Minutes specimens. Possible reasons why the 2 Hour specimens possessed greater scaling damage than the 30 Minute and 4 Hour specimens are discussed in 5.4.2.

Table 5-5: Mix 1-C Scaling Data from ASTM C672 Tests

Mix 1-C Wax						
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m^2)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
	30 Minutes	53.9	-----	301.9	-----	15%
25%	2 Hours	105.2	95%	621.6	106%	9%
	4 Hours	56.6	5%	259.8	-14%	13%
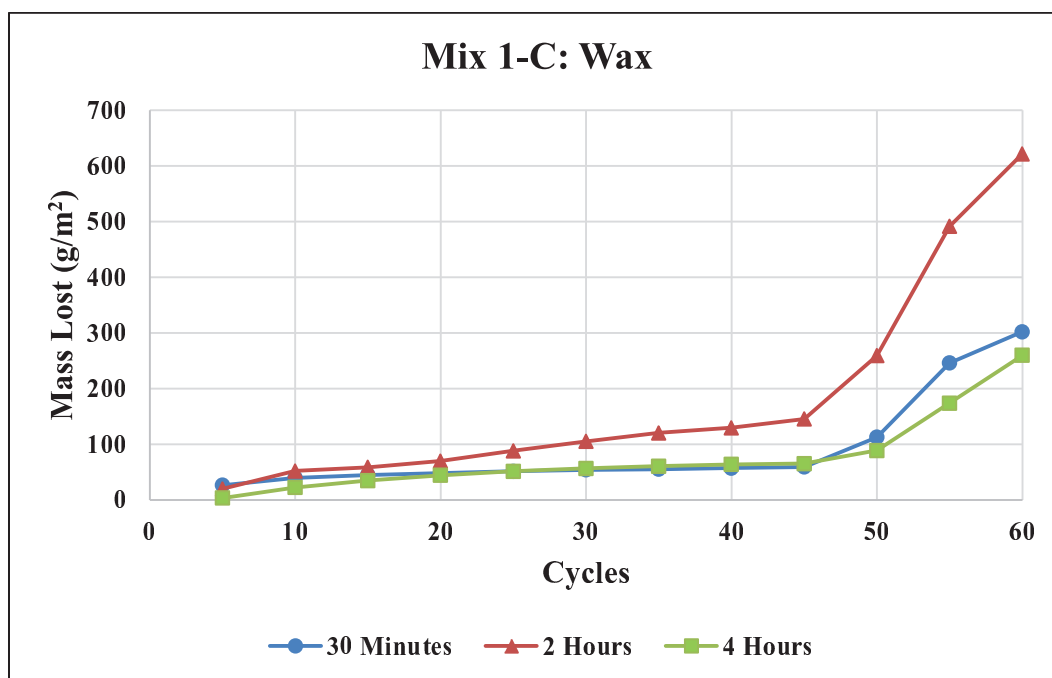
Figure 5-3: Mix 1-C Cumulative Scaling from ASTM C672 Tests

5.2.1.4 PAMS

The scaling accumulation curves in Figure 5-4 indicated that an increase in the application time led to an increase in scaling damage for Mix 1-D specimens. Table 5-6 shows that all three specimen sets exhibited the majority of the damage they would sustain during the first 15 cycles, with damage rates slowing down for the remainder of the test to near flat line levels. Possible reasons for why the application time scaling patterns were reversed from the expected trend are discussed in 5.4.4.

Table 5-6: Mix 1-D Scaling Data from ASTM C672 Tests

			Mix 1-D PAMS			
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m^2)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
21%	30 Minutes	46.0	-----	53.9	-----	68%
	2 Hours	83.5	81%	87.5	62%	85%
	4 Hours	121.7	164%	133.5	148%	80%
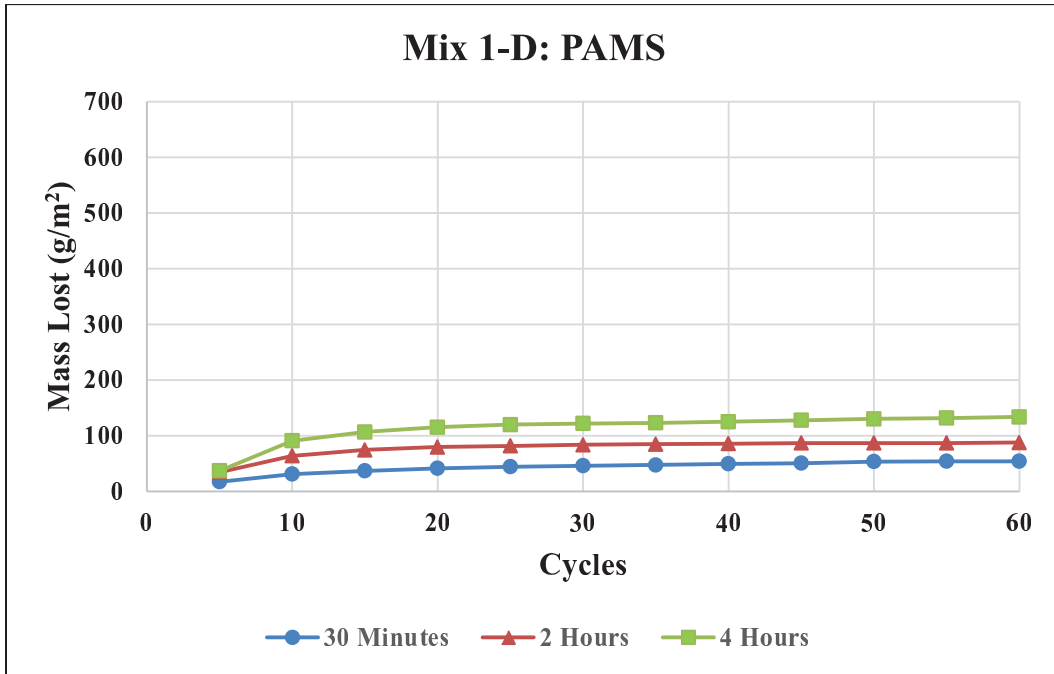
Figure 5-4: Mix 1-D Cumulative Scaling from ASTM C672 Tests

5.2.1.5   *Acrylic*

In Figure 5-5, both the 30 Minute and 4 Hours specimen sets of Mix 1-E scaled at approximately the same amount per every five cycles; the main difference was that the 30 Minutes specimens had a higher amount of initial scaling. Results in Table 5-7 indicate the 4 Hour specimens accumulated approximately half the scaling damage of the 30 Minute specimens. The total amount of scaling for the 30 Minute specimens at the end of 60 Cycles did not exceed 150 g/m^2.

Table 5-7: Mix 1-E Scaling Data from ASTM C672 Tests

Mix 1-E Acrylic						
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m^2)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
27%	30 Minutes	100.6	-----	142.1	-----	56%
	4 Hours	48.0	-52%	77.0	-46%	45%
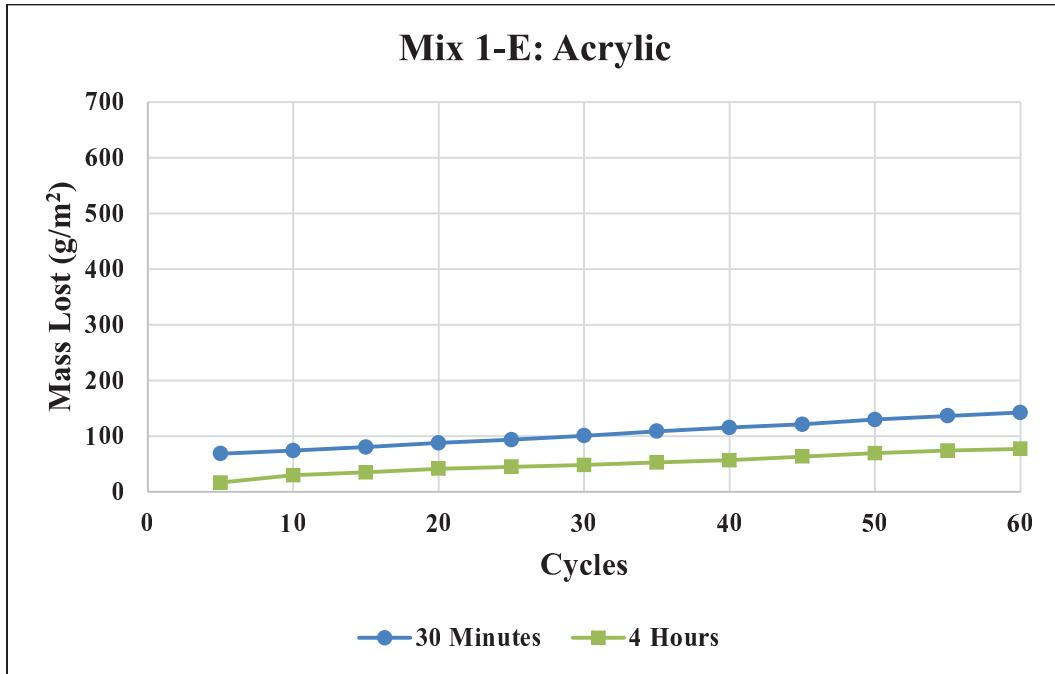
Figure 5-5: Mix 1-E Cumulative Scaling from ASTM C672 Tests

5.2.2 Effect of Application Time on Mix Type 2: Crushed Limestone and 30% Replacement Slag

Mix Type 2 contained a crushed limestone coarse aggregate and a 30% replacement by weight of Portland Cement with Grade 100 Ground Granulated Blast Furnace Slag. All charts in this section used a scale up to 1400 g/m^2.

5.2.2.1   Wet Room Cured

The scaling data in Table 5-8 indicates that the Wet Room specimens for Mix Type 2 exhibited nearly 180 g/m^2 of average scaling damage after 60 cycles, with nearly half the damage occurring within the first 15 cycles. The damage accumulation pattern in Figure 5-6 displays a progressive increase in the scaling damage over time for Mix 2-A after the first 15 cycles.

Table 5-8: Mix 2-A Scaling Data from ASTM C672 Tests

Mix 2-A Wet Room Cured			
Relative Humidity	Scaled Mass at 30 Cycles (g/m^2)	Scaled Mass at 60 Cycles (g/m2)	Damage Occuring in First 15 Cycles (%)
24%	121.0	180.9	49%
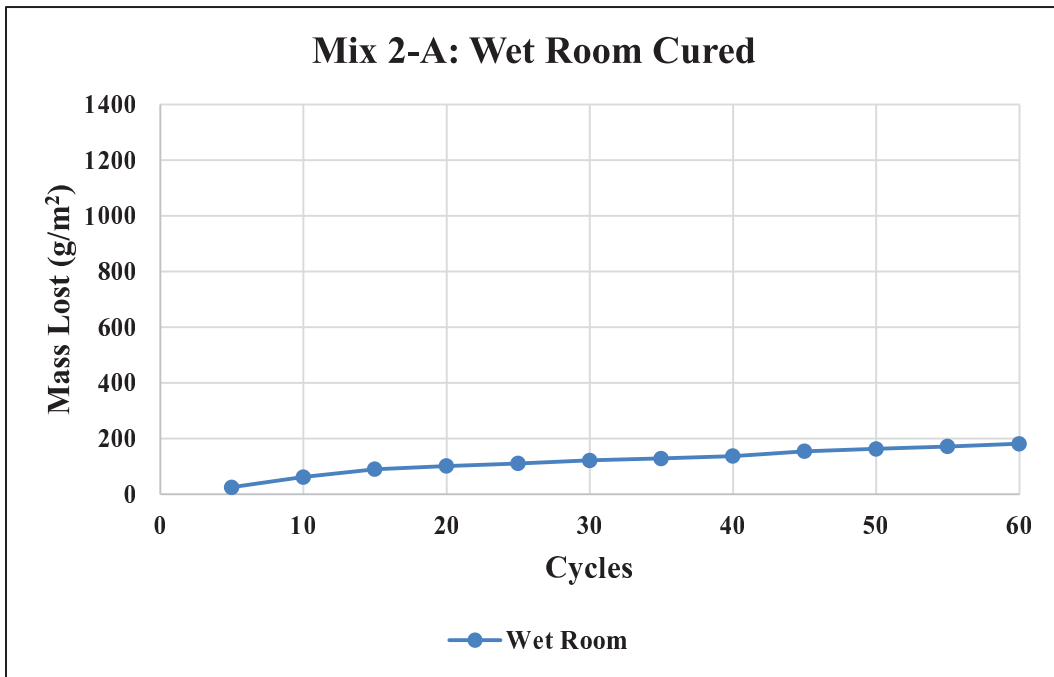
Figure 5-6: Mix 2-A Cumulative Scaling from ASTM C672 Tests

5.2.2.2 Linseed Oil

In Table 5-9, the total amount of scaling damage exhibited by Mix 2-B's 30 Minute specimens was over 1000 g/m^2; an amount that was significantly reduced in the 2 and 4 Hour specimens. The damage accumulation curves for the 2 and 4 Hour specimens in Figure 5-7 show nearly identical amounts of scaling accumulation over 60 cycles. All three application times resisted early scaling damage, with accelerated accumulation after 15 cycles.

Table 5-9: Mix 2-B Scaling Data from ASTM C672 Tests

Mix 2-B Linseed Oil						
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m^2)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
23%	30 Minutes	440.7	-----	1032.1	-----	12%
	2 Hours	128.9	-71%	587.4	-43%	4%
	4 Hours	104.6	-76%	586.8	-43%	4%
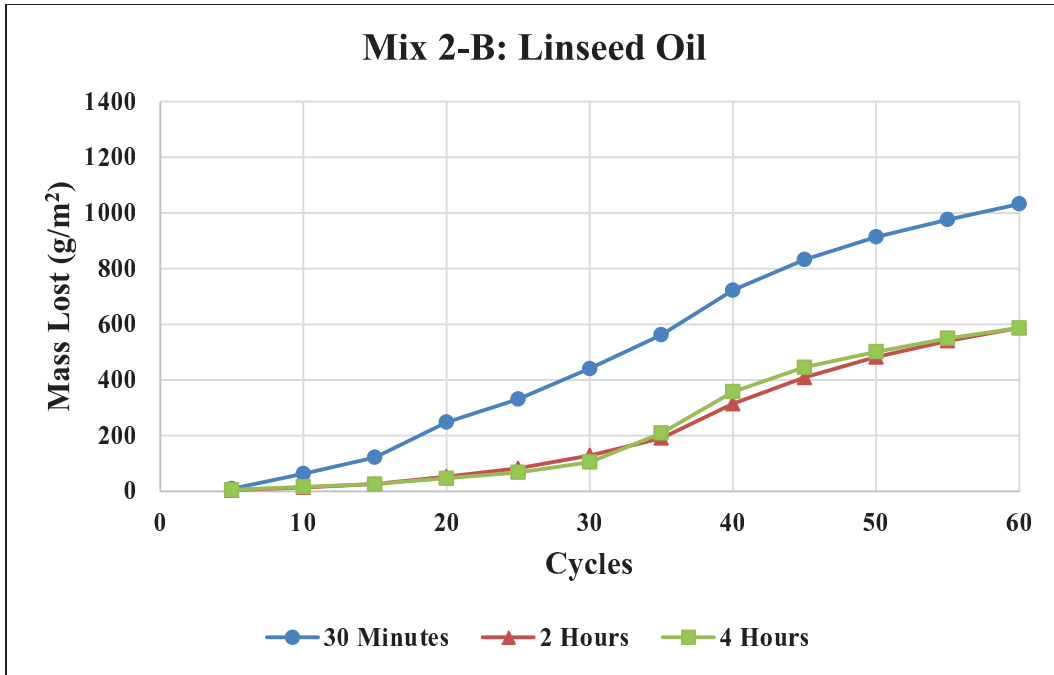
Figure 5-7: Mix 2-B Cumulative Scaling from ASTM C672 Tests

5.2.2.3 Wax

The scaling accumulation curves for Mix 2-C specimens in Figure 5-8 show nearly identical amounts of scaling damage throughout the first 20 cycles for the 30 Minute and 2 Hour specimens, followed by accelerated damage accumulation in the 2 Hour specimens for the final 40 cycles. The 4 Hour specimens exhibited much less initial scaling than the 30 Minute and 2 Hour specimens. However, both the 30 Minute and 4 Hour specimens appeared to have the same damage accumulation rates throughout the 60 cycles, offset by the initial scaling amounts. In Table 5-10, the 2 Hour specimens accumulated 50% more total scaling than the 30 Minute specimens, while the 4 Hour specimens suffered slightly more than half the amount of scaling of the 30 Minute specimens. Possible explanations for the higher scaling amounts in the 2 Hour specimens are discussed Sections 5.4.1 and 5.4.2.

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Table 5-10: Mix 2-C Scaling Data from ASTM C672 Tests

Mix 2-C Wax						
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m²)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
31%	30 Minutes	363.1	-----	546.0	-----	62%
	2 Hours	416.4	15%	818.3	50%	45%
	4 Hours	178.3	-51%	305.2	-44%	52%
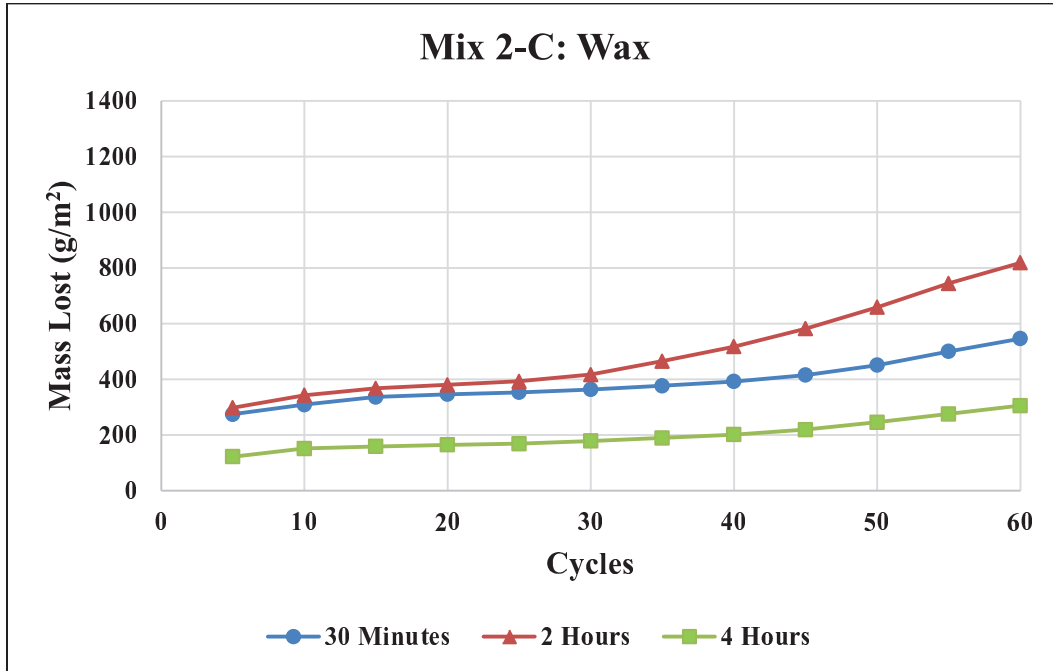
Figure 5-8: Mix 2-C Cumulative Scaling from ASTM C672 Tests

5.2.2.4   PAMS

As noted in Table 5-11, all three specimen sets for Mix 2-D suffered nearly 80% of their total scaling damage within the first 15 cycles. Each set scaled more than 850 g/m², with the 2 Hour specimens scaling over 1200 g/m², a 43% increase in damage over the 30 Minute specimens. The damage patterns in Figure 5-9 show that the 30 Minute and 4 Hour specimens scale at nearly the same rate and amounts for the entire test. Possible explanations for the scaling behavior of this slag-containing mix type are discussed Section 5.4.1.

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Table 5-11: Mix 2-D Scaling Data from ASTM C672 Tests

		Mix 2-D PAMS				
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m²)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
31%	30 Minutes	740.0	-----	867.0	-----	79%
	2 Hours	1063.0	44%	1238.0	43%	82%
	4 Hours	790.0	7%	851.2	-2%	88%
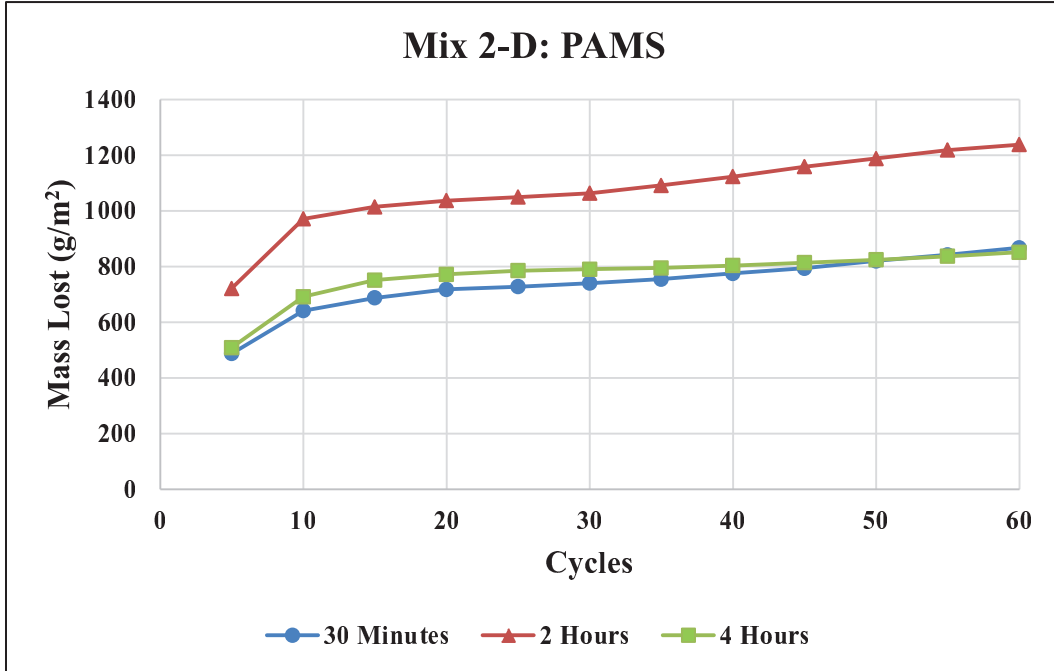
Figure 5-9: Mix 2-D Cumulative Scaling from ASTM C672 Tests

5.2.2.5 *Acrylic*

The Mix 2-E scaling data in Table 5-12 shows a nearly 26% decrease in scaling damage for the 4 Hour specimens versus the 30 Minute specimens. Both sets of specimens suffered most of their total damage within the first 15 cycles. Figure 5-10 shows that the 30 Minute and 4 Hour specimens scaled at approximately the same rates after the first 15 cycles.

Table 5-12: Mix 2-E Scaling Data from ASTM C672 Tests

Mix 2-E Acrylic						
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m^2)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
24%	30 Minutes	528.9	-----	617.0	-----	73%
	4 Hours	352.6	-33%	454.5	-26%	63%
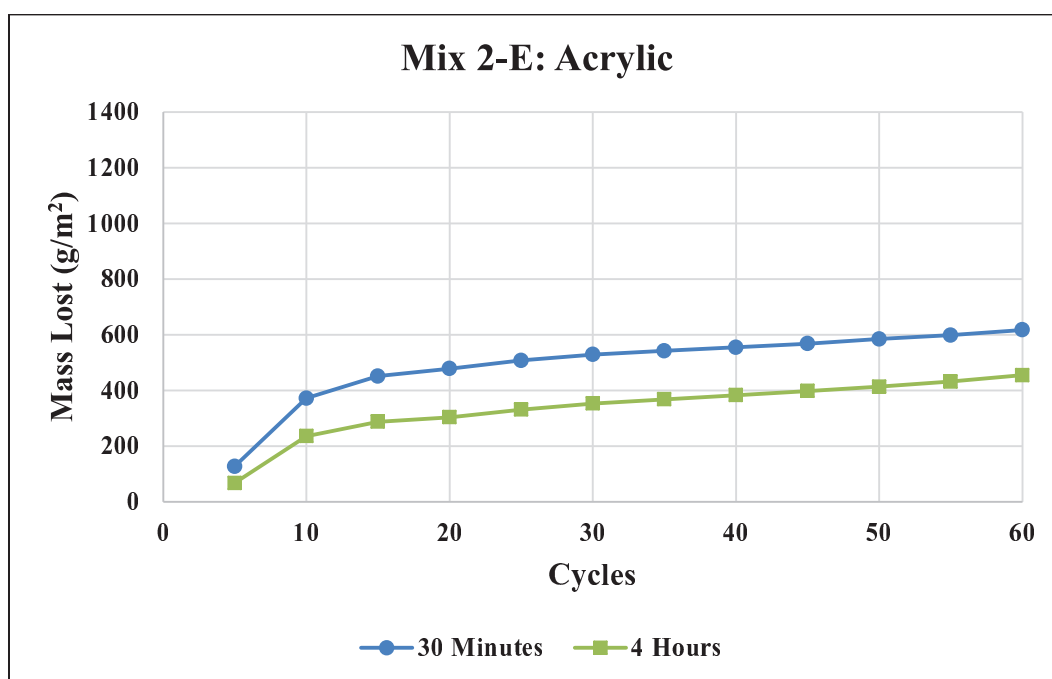
Figure 5-10: Mix 2-E Cumulative Scaling from ASTM C672 Tests

5.2.3   Effect of Application Time on Mix Type 3: Crushed Limestone and 30% Replacement Fly Ash

Mix Type 3 contained the crushed limestone and a 30% replacement by weight of Portland Cement with Class C Fly Ash. Only two application times were evaluated for this set: 30 Minutes and 4 Hours. As shown in Table 5-1, Mix Type 3 batches possessed slumps higher than the crushed limestone batches that contained just OPC and Slag Cement. All charts in this section used a constant mass loss scale up to 1600 g/m^2.

5.2.3.1 *Wet Room Cured*

Table 5-13 shows that the Wet Room specimens for Mix Type 3 most of their total scaling damage occurring within the first 15 cycles. This is also shown in Figure 5-11, where very little scaling accumulation occurred after the initial 15 cycles.

Table 5-13: Mix 3-A Scaling Data from ASTM C672 Tests

Mix 3-A Wet Room Cured			
Relative Humidity	Scaled Mass at 30 Cycles (g/m^2)	Scaled Mass at 60 Cycles (g/m2)	Damage Occuring in First 15 Cycles (%)
32%	81.6	94.7	69%
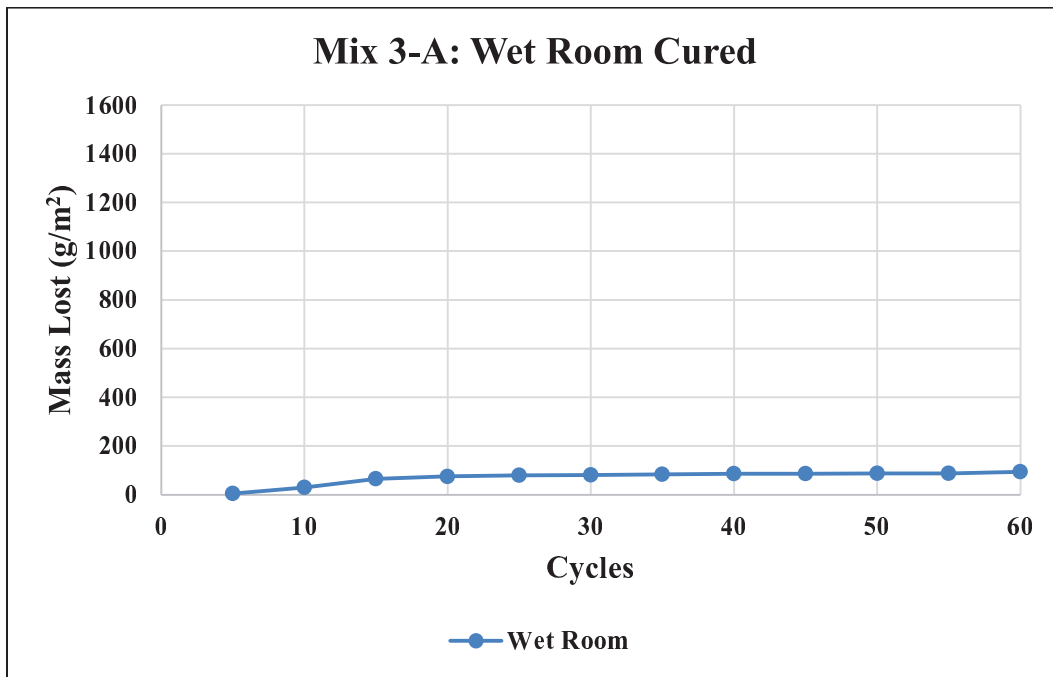
Figure 5-11: Mix 3-A Cumulative Scaling from ASTM C672 Tests

5.2.3.2   Linseed Oil

In Figure 5-12 for Mix 3-B, a large amount of scaling in the 30 Minute specimens within the first 15 cycles contrasted with the small amount of scaling accumulation in the 4 Hour specimens during that same time. Both sets of specimens scaled at approximately the same rates during the following 45 cycles. The total scaling damage in the 30 Minute specimens was reduced by over half by extending the application time to 4 Hours, as shown in Table 5-14.

Table 5-14: Mix 3-B Scaling Data from ASTM C672 Tests

Mix 3-B Linseed Oil						
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m^2)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
29%	30 Minutes	1151.2	-----	1479.4	-----	68%
	4 Hours	268.4	-77%	642.7	-57%	7%
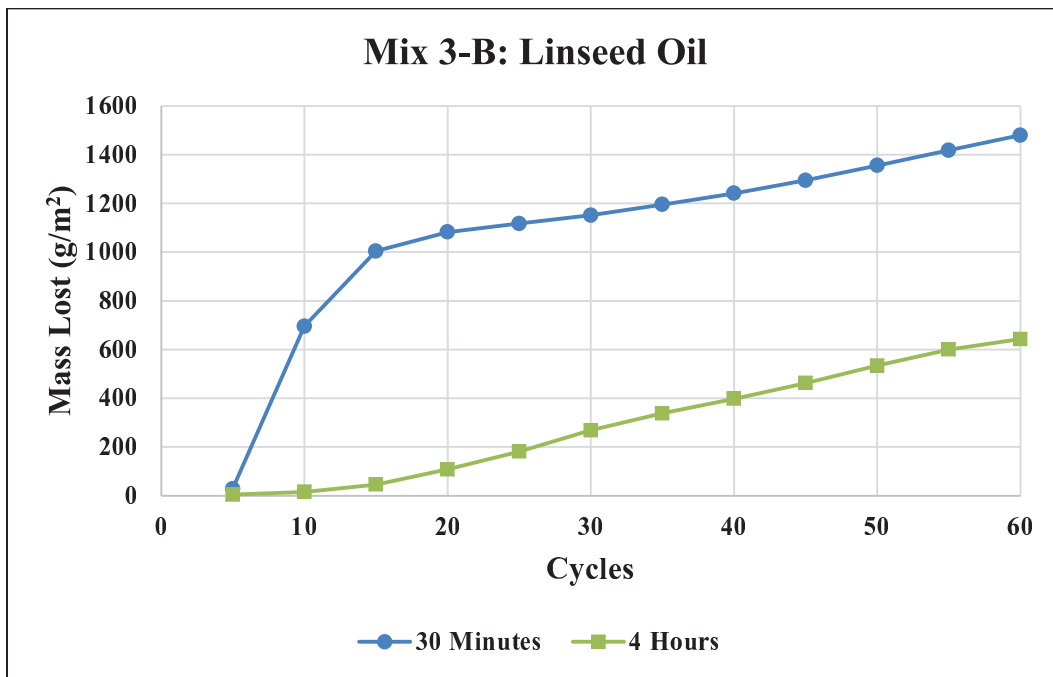
Figure 5-12: Mix 3-B Cumulative Scaling from ASTM C672 Tests

5.2.3.3   *Wax*

Mix 3-C scaling data in Table 5-15 shows that the total amount of scaling the 30 Minute specimens was reduced by over half by increasing the application time to 4 Hours. The scaling accumulation curves for both sets of specimens in Figure 5-13 were nearly identical in magnitude for the first 20 cycles. While both set of specimens exhibited increased rates of scaling damage occur during the final 40 cycles, the 30 Minute specimens suffered higher rates of damage accumulation than the 4 Hour specimens. Extending the application time to 4 hours limited this acceleration of scaling accumulation in the Mix Type 3 Wax coated specimens.

Table 5-15: Mix 3-C Scaling Data from ASTM C672 Tests

Mix 3-C Wax						
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m^2)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
32%	30 Minutes	225.6	-----	847.9	-----	16%
	4 Hours	157.9	-30%	394.0	-54%	33%
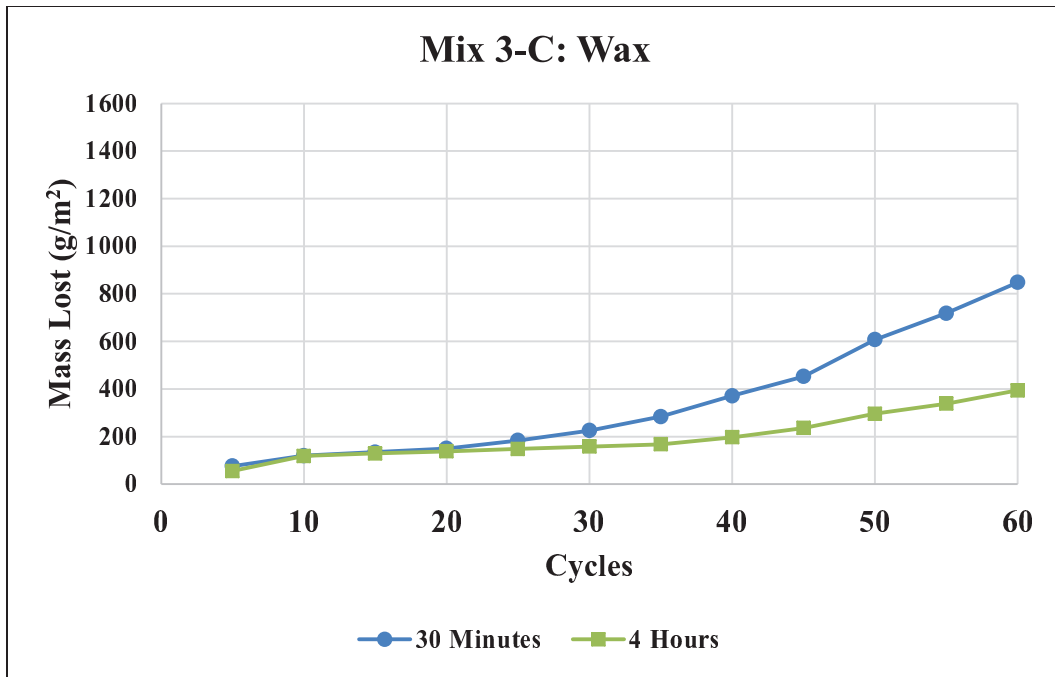
Figure 5-13: Mix 3-C Cumulative Scaling from ASTM C672 Tests

5.2.3.4 PAMS

Scaling data for Mix 3-D in Table 5-16 shows that the PAMS-coated specimens exhibited levels of scaling resistance within both 30 Minutes and 4 Hours specimen sets averaging total amounts of damage less than 310 g/m^2. The scaling accumulation curves for both application time specimen sets in Figure 5-14 were roughly identical in magnitude for the first 40 cycles, with the 30 Minute specimens suffering slightly higher rates of scaling than the 4 Hour specimens during the final 20 cycles.

Table 5-16: Mix 3-D Scaling Data from ASTM C672 Tests

Mix 3-D PAMS						
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m^2)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
27%	30 Minutes	120.4	-----	309.2	-----	26%
	4 Hours	113.8	-5%	211.2	-32%	45%
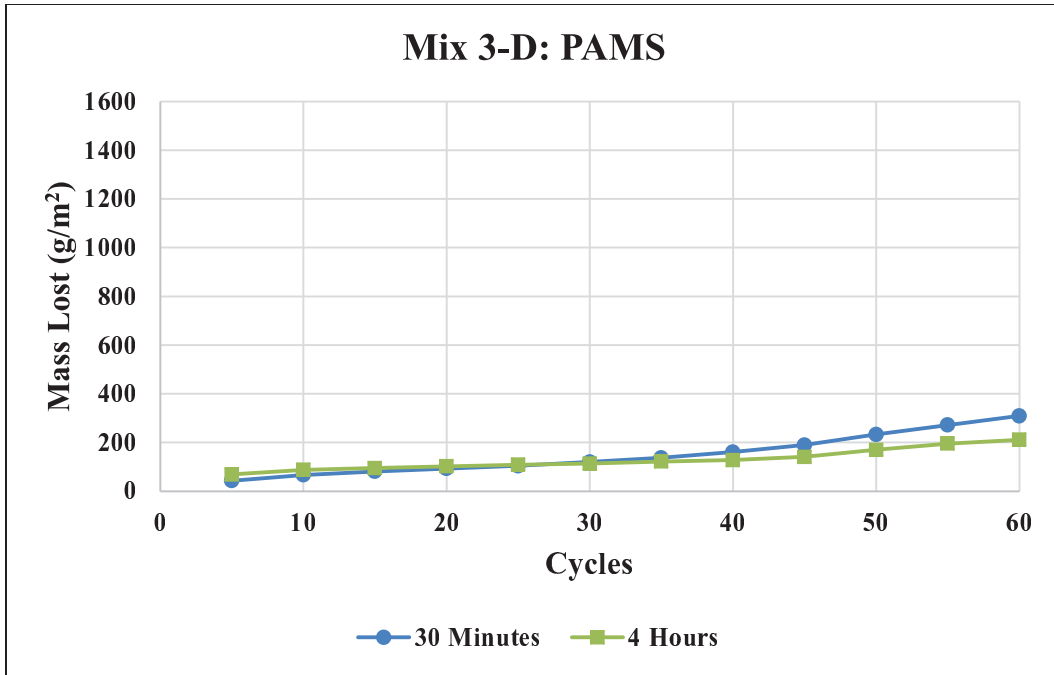
Figure 5-14: Mix 3-D Cumulative Scaling from ASTM C672 Tests

5.2.3.5 *Acrylic*

The scaling damage accumulation curves for Mix 3-E acrylic coated specimens in Figure 5-15 indicate that the 30 Minute specimens exhibited significant surface loss between 5 and 10 cycles, while the 4 Hour specimens did not. This is important, as during the final 50 cycles the damage accumulation rates for both the 30 Minute and 4 Hours specimens were both very low. As shown in Table 5-17, extending the application time to 4 Hours resulted in a decrease in total scaling damage of over 80%, or approximately 600 g/m^2.

Table 5-17: Mix 3-E Scaling Data from ASTM C672 Tests

Mix 3-E Acrylic						
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m^2)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
36%	30 Minutes	671.0	-----	734.1	-----	83%
	4 Hours	109.9	-84%	130.9	-82%	71%
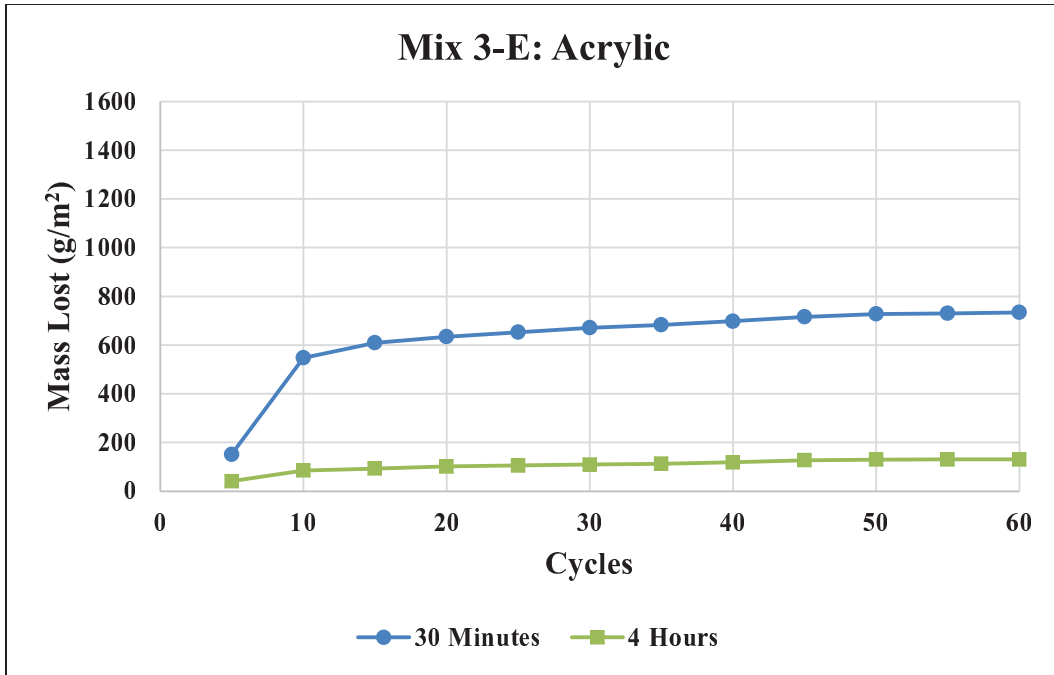
Figure 5-15: Mix 3-E Cumulative Scaling from ASTM C672 Tests

5.2.4 Effect of Application Time on Mix Type 4: Glacial Gravel and Ordinary Portland Cement

Mix Type 4 consisted of the Glacial Gravel coarse aggregate in OPC. Due to workability concerns, batches 4-B, 4-C, and 4-D required an increase in the water/cement ratio to 0.41 from 0.40 to ensure workability requirements were met. In addition to a higher (w/c) ratio, most of these mixes were mixed in environments with lower relative humidity, possessed slightly higher air contents, and lower compressive strength than the other gravel-containing mixes, Mix Types 5 and 6 as summarized in Table 5-1. All charts in this section used a mass loss scale up to 700 g/m^2, the same as for the limestone OPC specimen results.

5.2.4.1   Wet Room Cured

Table 5-18 shows that the Wet Room specimens for Mix Type 4 exhibited less than 60 g/m^2 of average total scaling, with only 26% of this damage occurring in the first 15 cycles. This is also reflected in Figure 5-16, where the accumulation of scaled material was relatively constant throughout the duration of the exposure to deicing chemicals.

Table 5-18: Mix 4-A Scaling Data from ASTM C672 Tests

Mix 4-A Wet Room Cured			
Relative Humidity	Scaled Mass at 30 Cycles (g/m^2)	Scaled Mass at 60 Cycles (g/m2)	Damage Occuring in First 15 Cycles (%)
35%	37.5	57.2	26%
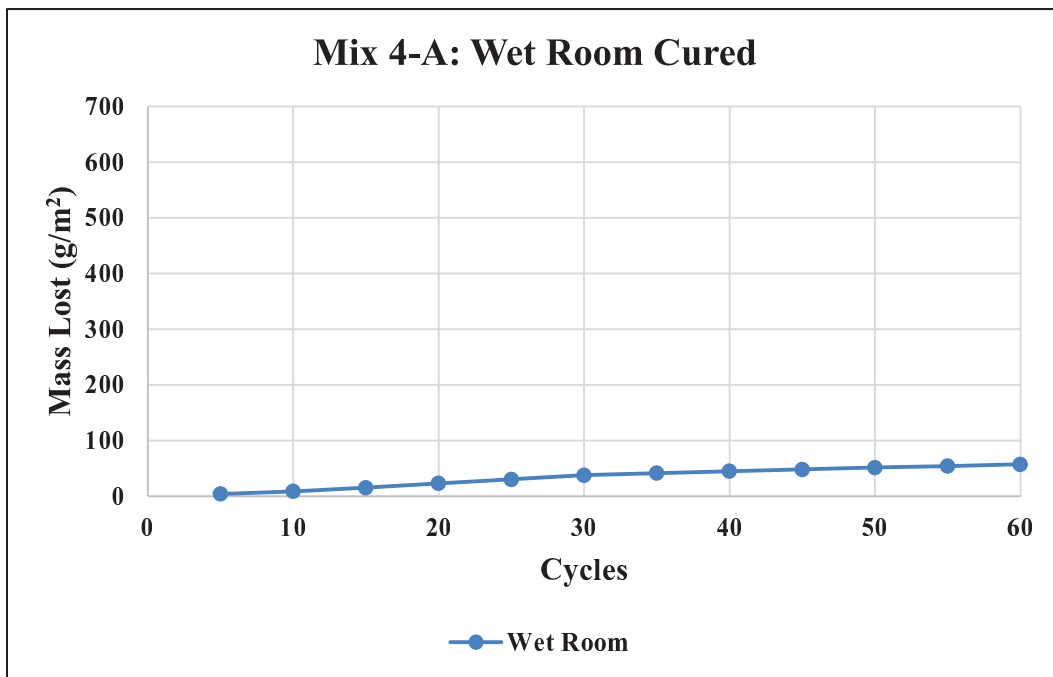
Figure 5-16: Mix 4-A Cumulative Scaling from ASTM C672 Tests

5.2.4.2   Linseed Oil

Figure 5-17 shows that the Mix 4-B 30 Minute specimens lost over half their total damage within the first 30 cycles, while the 2 Hour and 4 Hour specimens exhibited very little scaling damage during that time. After 30 cycles, all three specimen sets appeared to scale at similar rates. Table 5-18 shows that increasing the application time to 2 Hours reduced the scaling damage in half as compared to the 30 Minute specimens; while an application time of 4 Hours decreased the scaling damage by nearly two thirds.

Table 5-19: Mix 4-B Scaling Data from ASTM C672 Tests

		Mix 4-B Linseed Oil				
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m^2)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
	30 Minutes	381.5	-----	609.1	-----	13%
27%	2 Hours	69.7	-82%	300.6	-51%	4%
	4 Hours	23.7	-94%	217.1	-64%	2%
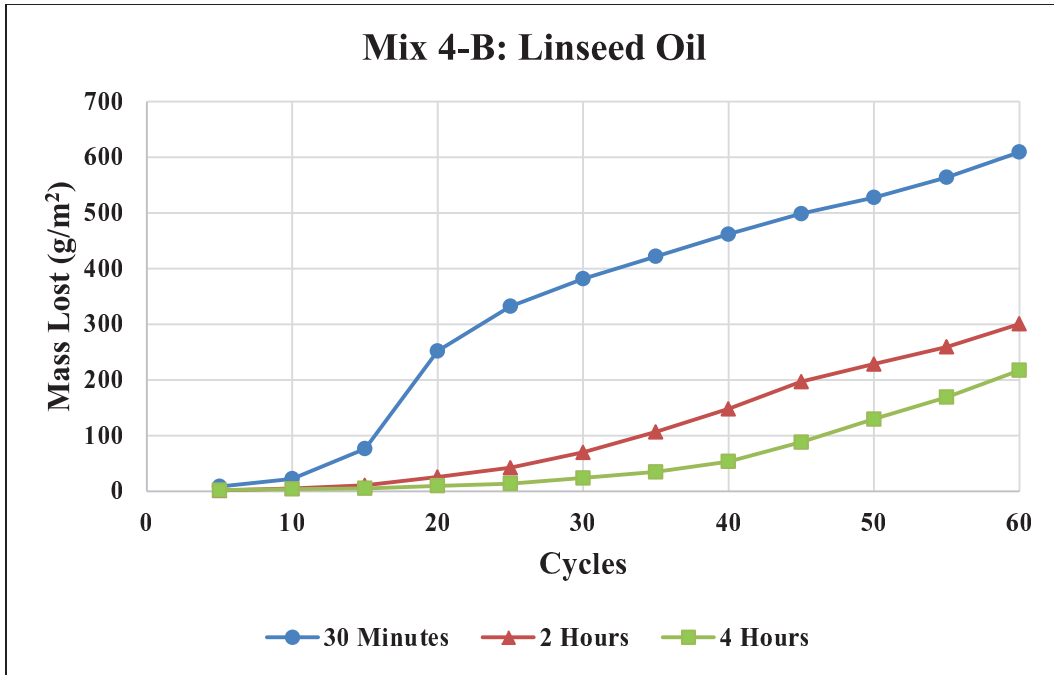
Figure 5-17: Mix 4-B Cumulative Scaling from ASTM C672 Tests

5.2.4.3   Wax

Scaling data for Mix 4-C in Table 5-20 shows that the while the 4 Hour specimens scaled less than the 30 Minute specimens, the 2 Hour specimens exhibited an increase in scaling damage of 78% over the 30 Minute specimens. The damage accumulation curves in Figure 5-18 show that while all specimens from all three application times scaled at roughly the same rate from 10-60 cycles, the 2 Hour specimens exhibited higher amounts of scaling within the first 10 cycles than the specimens from the other two application times. Possible reasons as to why the 2 Hour specimens lost more mass than the other application time specimens are discussed in Section 5.4.2.

Table 5-20: Mix 4-C Scaling Data from ASTM C672 Tests

		Mix 4-C Wax				
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m^2)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
	30 Minutes	139.5	-----	178.9	-----	66%
25%	2 Hours	260.5	87%	317.7	78%	73%
	4 Hours	91.4	-34%	147.3	-18%	38%
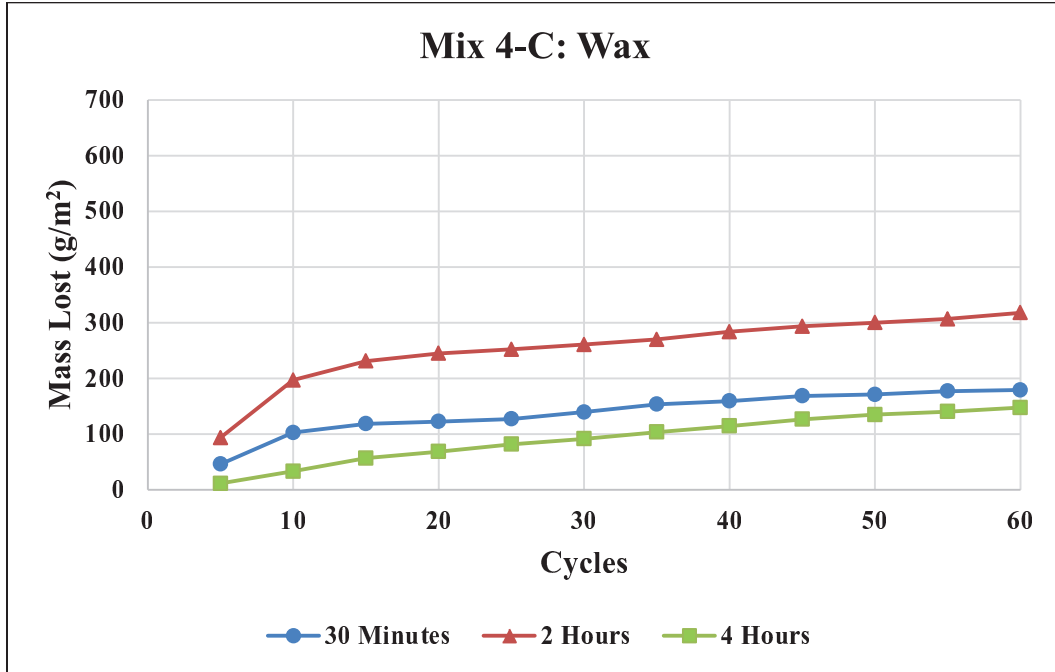
Figure 5-18: Mix 4-C Cumulative Scaling from ASTM C672 Tests

5.2.4.4 PAMS

In the scaling data for Mix 4-D in Table 5-21, the percentage of total damage occurring within the first 15 cycles decreased with an increase in the application time. This is also shown in Figure 5-19, where the scaling damage accumulation curves show that the total amount of scaling damage was heavily dependent on the amount of scaling that occurred within the first 10 cycles. It should be noted that while the 4 Hour specimens exhibited nearly 29% less scaling damage than the

30 Minute specimens after 60 cycles, the 2 Hour specimens only exhibited a reduction in scaling of about 1%.

Table 5-21: Mix 4-D Scaling Data from ASTM C672 Tests

		Mix 4-D PAMS				
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m^2)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
28%	30 Minutes	531.5	-----	599.9	-----	77%
	2 Hours	444.0	-16%	591.4	-1%	59%
	4 Hours	246.7	-54%	426.9	-29%	36%
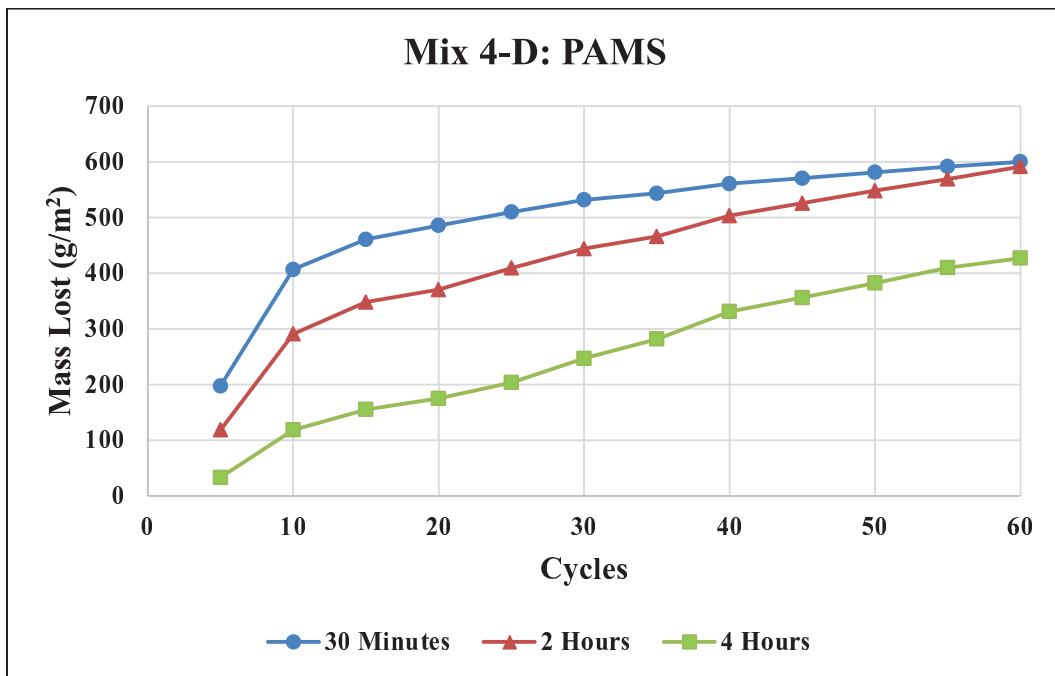
Figure 5-19: Mix 4-D Cumulative Scaling from ASTM C672 Tests

5.2.4.5   *Acrylic*

For the Acrylic-coated Mix 4-E specimens within Table 5-22, applying the acrylic at 4 Hours reduced the amount of scaling by over 300 g/m^2 compared to the 30 Minute specimens. In Figure 5-20, the 30 Minute specimens both lost more surface mass initially, and had higher rates of scaling

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throughout the remainder of the test than the 4 Hour specimens. Both specimen sets suffered nearly half their total damage within the first 15 cycles.

Table 5-22: Mix 4-E Scaling Data from ASTM C672 Tests

Mix 4-E Acrylic						
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m^2)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
76%	30 Minutes	277.6	-----	386.1	-----	58%
	4 Hours	35.5	-87%	48.0	-88%	48%
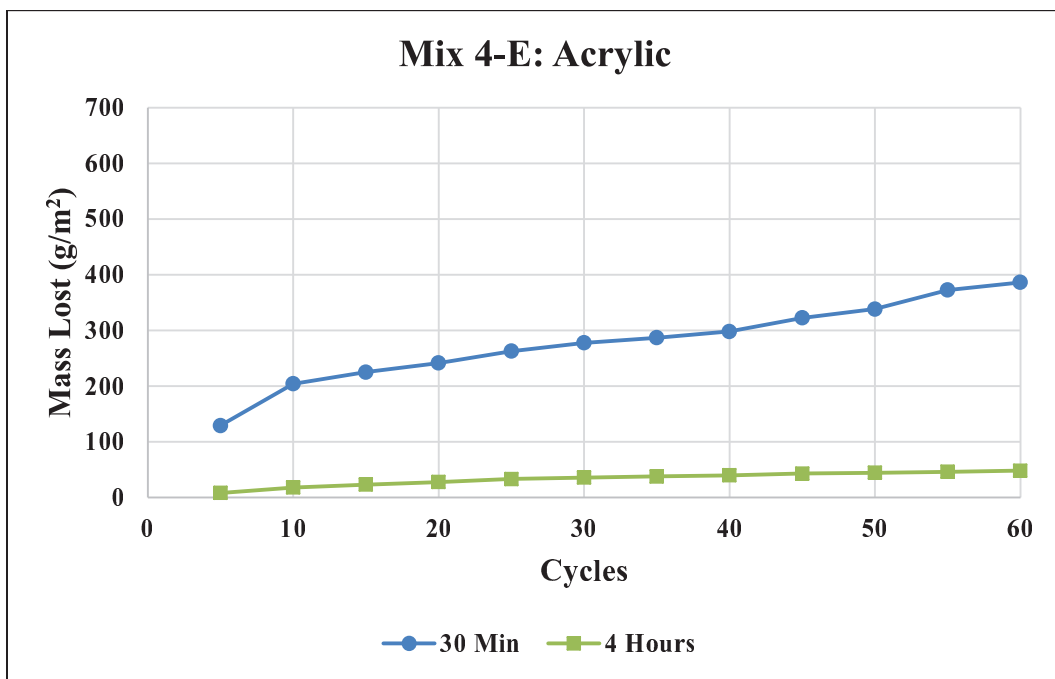
Figure 5-20: Mix 4-E Cumulative Scaling

5.2.5 Effect of Application Time on Mix Type 5: Glacial Gravel and 30% Replacement Slag

Mix Type 5 contained the glacial gravel in addition to a 30% replacement of OPC with Grade 100 Slag Cement. These mixes typically coincided with lower air content values than Mixes 1-4, and

the Wet Room, Wax, and Acrylic specimens were poured in environments where the relative humidity exceeded 60%, as noted in Appendix B. All charts in this section use a mass loss scale of up to 1600 g/m^2.

5.2.5.1 *Wet Room Cured*

The scaling damage data in Table 5-23 indicates the average total mass loss amount for Mix 5-A Wet Room specimens was over 200 g/m^2, with nearly a third of the damage accumulating in the first 15 cycles. As shown in Figure 5-21, the progressive scaling accumulation was relatively constant over the 60 cycles.

Table 5-23: Mix 5-A Scaling Data from ASTM C672 Tests

Mix 5-A Wet Room Cured			
Relative Humidity	Scaled Mass at 30 Cycles (g/m^2)	Scaled Mass at 60 Cycles (g/m2)	Damage Occuring in First 15 Cycles (%)
64%	90.1	202.6	29%
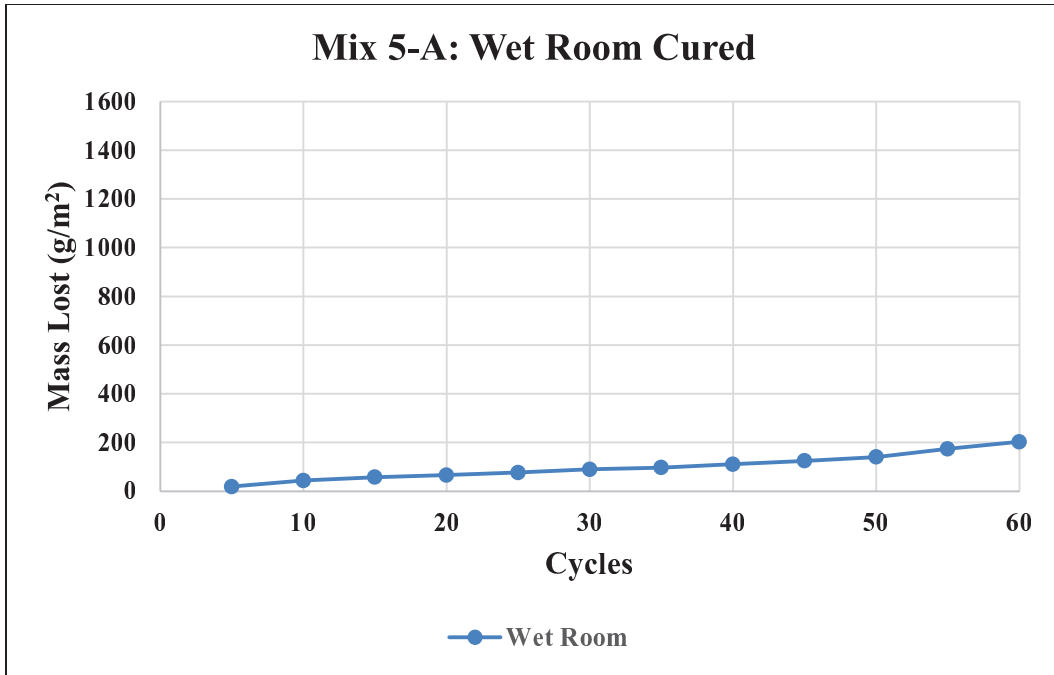
Figure 5-21: Mix 5-A Cumulative Scaling from ASTM C672 Tests

5.2.5.2 *Linseed Oil*

In the scaling data for Mix 5-B in Table 5-24, the 30 Minute specimens exhibited an average of 1295 g/m^2 of total damage. By applying the Linseed Oil either at 2 or 4 Hours, total damage was reduced by nearly two-thirds to nearly 480 g/m^2. However, no additional significant scaling resistance was exhibited by increasing the application time from 2 Hours to 4 Hours. The scaling damage accumulation curves in Figure 5-22 show that the 30 Minute specimens lost surface mass much faster than the other two specimen sets, losing over 1000 g/m^2 after 30 cycles.

Table 5-24: Mix 5-B Scaling Data from ASTM C672 Tests

Mix 5-B Linseed Oil						
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m^2)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
33%	30 Minutes	1001.8	-----	1295.2	-----	38%
	2 Hours	276.9	-72%	480.2	-63%	33%
	4 Hours	168.4	-83%	478.9	-63%	5%

Figure 5-22: Mix 5-B Cumulative Scaling from ASTM C672 Tests

5.2.5.3 Wax

The scaling results from Mix 5-C's specimens in both Table 5-25 and Figure 5-23 show that all specimen sets from Mix 5-C suffered nearly 70% of their total damage within the first 15 cycles. Additionally, all three specimen sets exhibited scaling damage on average in excess of 1000 g/m^2. The 30 Minute specimens scaled the least throughout the test, while the 2 Hour specimens scaled the most of the three sets. It is important to note that these specimens have three potential explanations

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for the magnitude of scaling suffered and the perceived independence of scaling resistance from application time: the inclusion of slag, the use of Wax curing compound and a high relative humidity at time of specimen manufacture. A discussion on the potential effects of these three sources on the poor scaling performance are discussed in Sections 5.4.1, 5.4.2, and 5.4.3, respectively.

Table 5-25: Mix 5-C Scaling Data from ASTM C672 Tests

		Mix 5-C Wax				
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m²)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
68%	30 Minutes	881.5	-----	1082.1	-----	75%
	2 Hours	1078.8	22%	1427.4	32%	69%
	4 Hours	1003.8	14%	1331.4	23%	69%
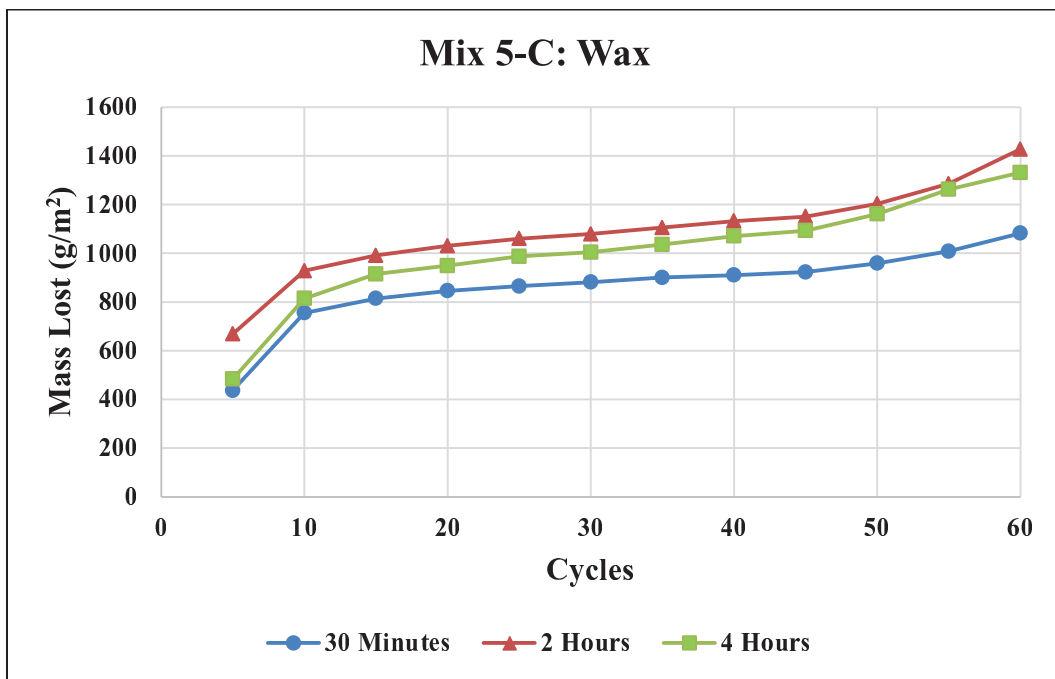
Figure 5-23: Mix 5-C Cumulative Scaling from ASTM C672 Tests

5.2.5.4   PAMS

In Figure 5-24, all three application time scaling accumulation curves for Mix 5-D scaled nearly identically after 5 cycles and possessed approximately the same rates of mass loss accumulation after 15 cycles. However, Table 5-26 illustrates that the 2 and 4 Hour specimen sets exhibited 64% and 21% more total scaling damage than the 30 Minute specimens, respectively. This is primarily due to the rapid degradation of the concrete surface of both the 2 and 4 Hour specimen sets between 5 and 10 cycles. Possible explanations for why these scaling patterns occurred are discussed Section 5.4.1.

Table 5-26: Mix 5-D Scaling Data from ASTM C672 Tests

Mix 5-D PAMS						
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m^2)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
26%	30 Minutes	578.9	-----	655.8	-----	77%
	2 Hours	959.1	66%	1074.8	64%	82%
	4 Hours	691.3	19%	791.3	21%	82%
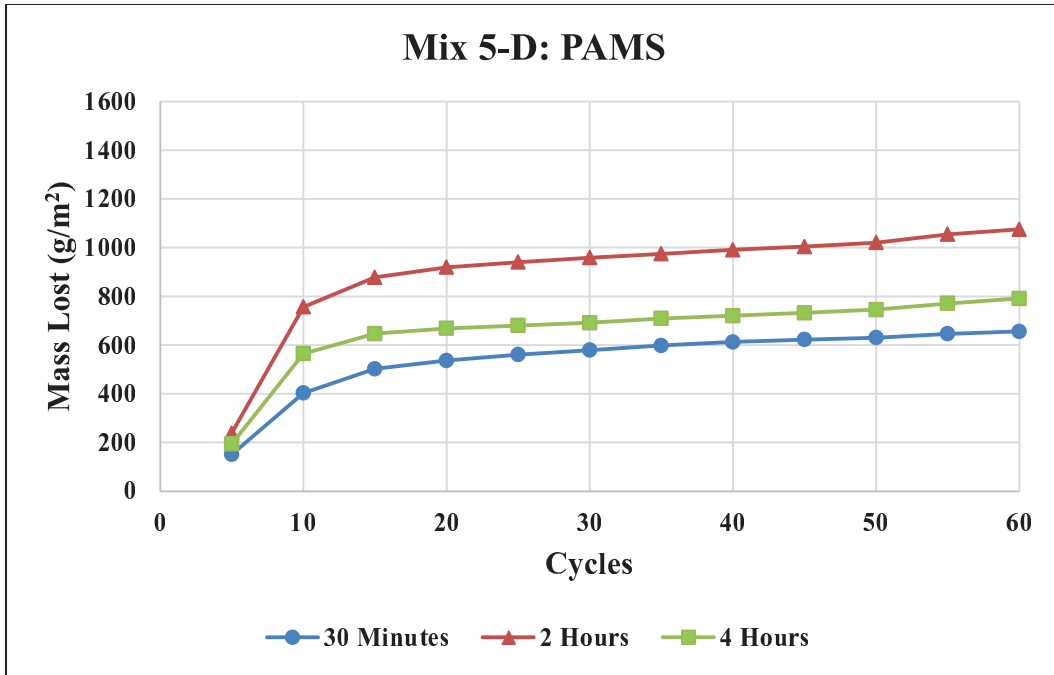
Figure 5-24: Mix 5-D Cumulative Scaling

5.2.5.5 *Acrylic*

In the scaling data for Mix 5-E listed in Table 5-27, the total mass loss from the 30 Minute application time was reduced by 45% or nearly 360 g/m^2 when the application time was extended to 4 Hours. While both sets of specimens suffered the majority of their total damage early within the first 15 cycles, Figure 5-25 shows that the 30 Minute damage accumulation curve was slightly more severe over the entire 60 cycles than the 4 Hour specimens.

Table 5-27: Mix 5-E Scaling Data from ASTM C672 Tests

Mix 5-E Acrylic						
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m^2)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
75%	30 Minutes	536.1	-----	808.4	-----	58%
	4 Hours	380.9	-29%	444.0	-45%	73%
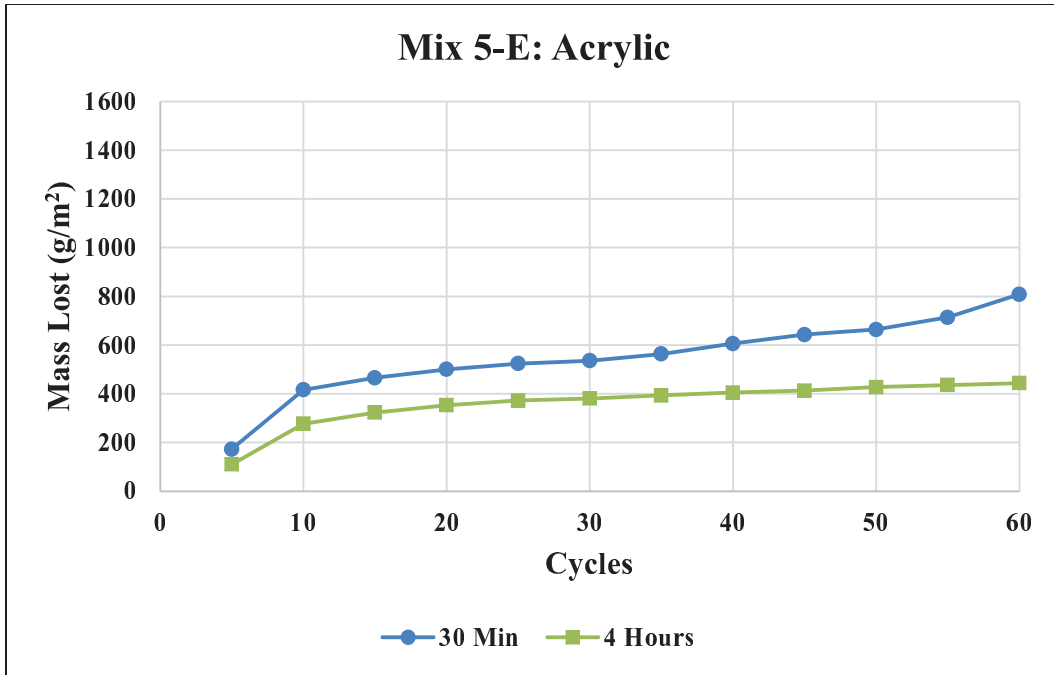
Figure 5-25: Mix 5-E Cumulative Scaling from ASTM C672 Tests

5.2.6 Effect of Application Time on Mix Type 6: Glacial Gravel and 30% Replacement Fly Ash

Mix Type 6 contained Glacial Gravel with a 30% by weight replacement of cement with Class C Fly Ash. The scaling performance for two application times was evaluated for this mix type: 30 Minutes and 4 Hours. As noted in Table 5-1, these concrete batches were characterized by high slumps and the Wet Room, Wax, and Acrylic batches were manufactured during periods of higher laboratory relative humidity. All charts in this section have a mass loss scale up to 2000 g/m^2.

5.2.6.1   *Wet Room*

As noted in Table 5-28, the Wet Room cured specimens for Mix Type 6 scaled an average amount of 312.5 g/m². It should be noted that the relative humidity at the time of manufacture was high at 83%. The scaling accumulation trend in Figure 5-26 exhibited approximately stable rates of progressive damage accrual.

Table 5-28: Mix 6-A Scaling Data from ASTM C672 Tests

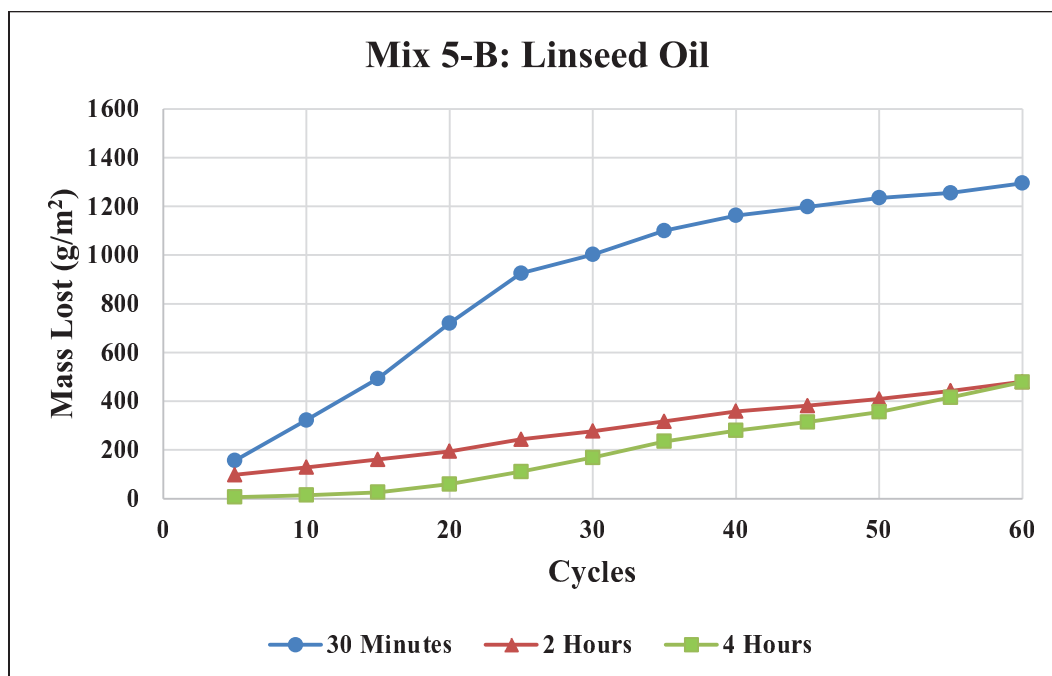| Mix 6-A Wet Room Cured			
Relative Humidity	Scaled Mass at 30 Cycles (g/m²)	Scaled Mass at 60 Cycles (g/m2)	Damage Occuring in First 15 Cycles (%)
83%	125.6	312.5	21%
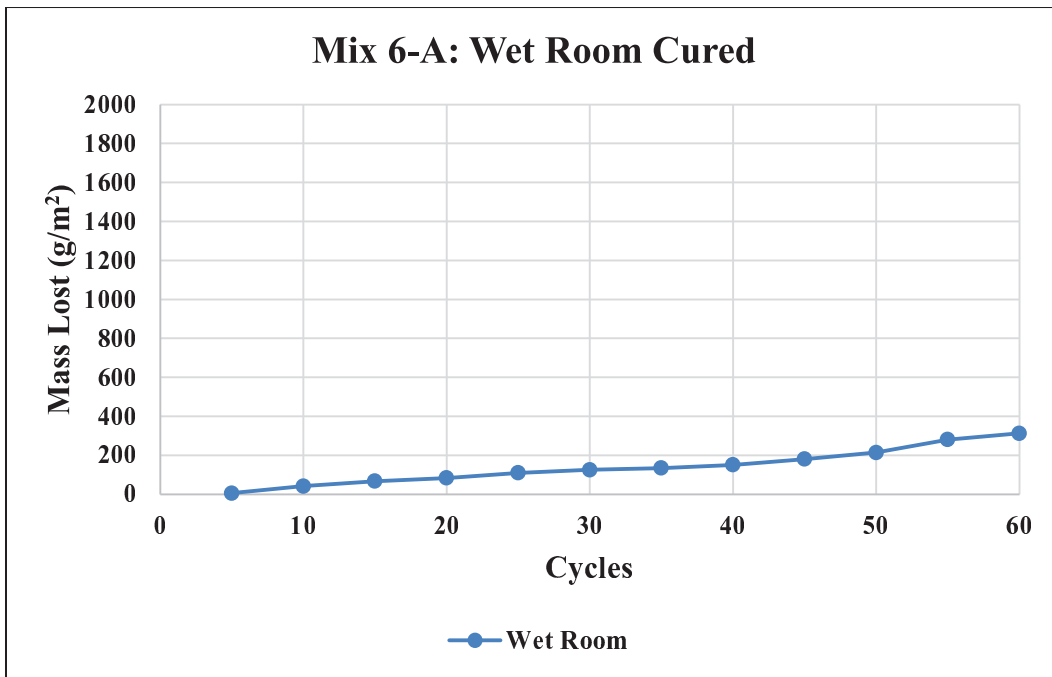
Figure 5-26: Mix 6-A Cumulative Scaling from ASTM C672 Tests

5.2.6.2 *Linseed Oil*

In Table 5-29, the Mix 6-B 30 Minute specimens on average exhibited scaling in excess of 1400 g/m^2, while extending the application time to 4 Hours reduced the scaling damage by approximately 50% to an average of approximately 726 g/m^2. Figure 5-27 shows that the 30 Minute specimens suffered significant mass loss immediately, with very high rates of loss throughout the test. In contrast, the 4 Hour specimens exhibited low scaling damage during the first 20 cycles, followed by progressive mass loss at rates nearly equal to the 30 Minute specimens during the final 40 cycles.

Table 5-29: Mix 6-B Scaling Data from ASTM C672 Tests

		Mix 6-B Linseed Oil				
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m^2)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
31%	30 Minutes	854.5	-----	1461.6	-----	36%
	4 Hours	176.3	-79%	726.2	-50%	3%
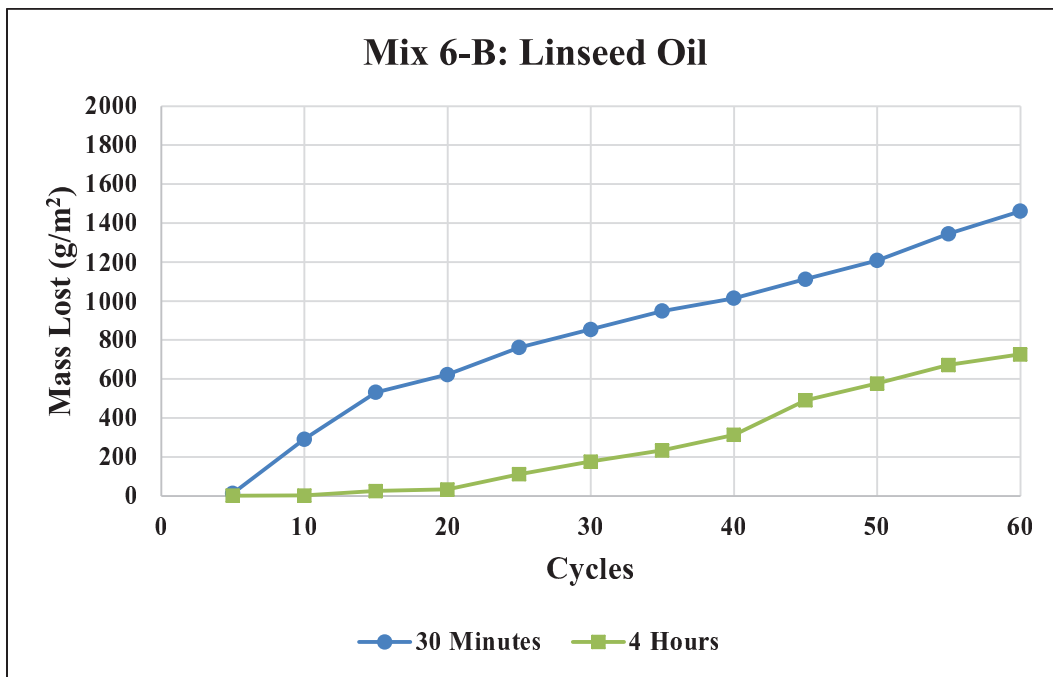
Figure 5-27: Mix 6-B Cumulative Scaling from ASTM C672 Tests

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5.2.6.3 *Wax*

As shown in Table 5-30, the total scaling amounts of both the 30 Minute and 4 Hour Mix 6-C specimens were nearly identical, with the 4 Hour specimens suffering slightly more damage than the 30 Minute specimens. Throughout the test, the 4 Hour specimens scaled more than the 30 Minute specimens, as shown by Figure 5-28. Both sets of specimens exhibited average total scaling damage levels exceeding 780 g/m^2. The specimens from this batch of concrete were manufactured and coated when the laboratory air had an elevated relative humidity level of 65%. The potential effects of the Wax curing compound and the elevated laboratory relative humidity on the scaling patterns are discussed in Sections 5.4.2 and 5.4.3, respectively.

Table 5-30: Mix 6-C Scaling Data from ASTM C672 Tests

Mix 6-C Wax						
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m²)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
65%	30 Minutes	380.2	-----	783.4	-----	37%
	4 Hours	506.5	33%	801.2	2%	44%
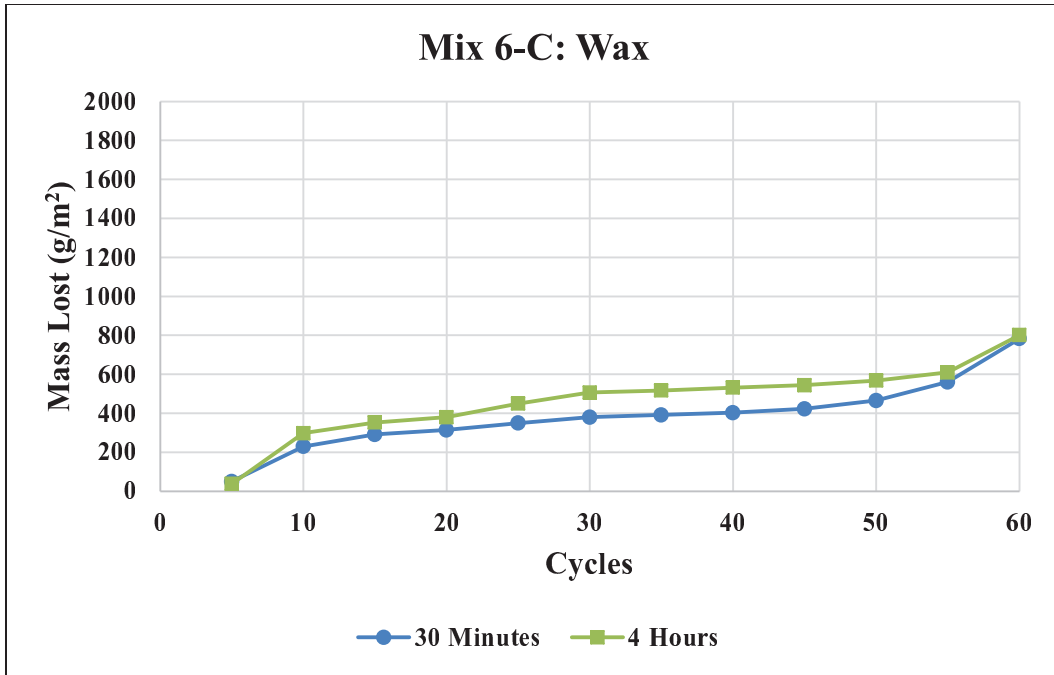
Figure 5-28: Mix 6-C Cumulative Scaling from ASTM C672 Tests

5.2.6.4 PAMS

Both the 30 Minute and 4 Hour specimens for Mix 6-D exhibited total scaling damage levels under 260 g/m^2, as shown in Table 5-31. The 4 Hour specimen set suffered slightly less total damage than the 30 Minute specimens. The scaling damage accumulation curves in Figure 5-29 show very little difference in scaling damage between the two specimen sets over the 60 cycles. Possible explanations for why the scaling amounts for the two application times were so similar are discussed in Section 5.4.4.

Table 5-31: Mix 6-D Scaling Data from ASTM C672 Tests

Mix 6-D PAMS						
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m^2)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
30%	30 Minutes	164.5	-----	257.2	-----	37%
	4 Hours	229.6	40%	251.3	-2%	64%
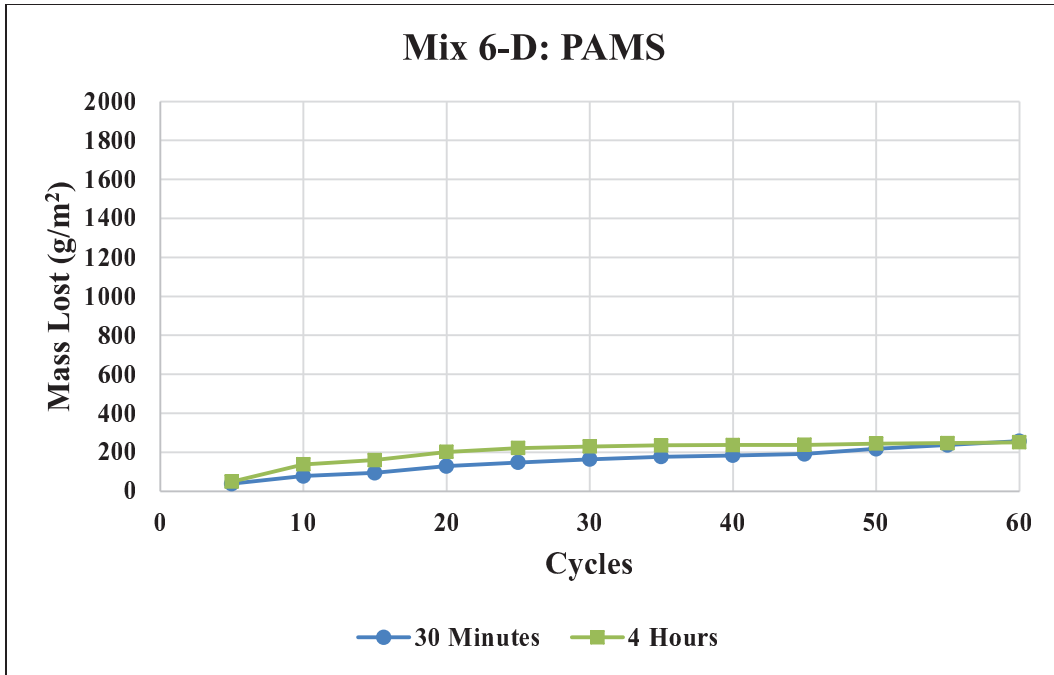
Figure 5-29: Mix 6-D Cumulative Scaling from ASTM C672 Tests

5.2.6.5 *Acrylic*

As shown in Table 5-32, the Mix 6-E 30 Minute specimens exhibited amounts of scaling damage at an average in excess of 1900 g/m^2, while the 4 Hour specimens exhibited an average mass loss of slightly more than 210 g/m^2; an overall reduction in damage of 89%. The scaling damage accumulation curve for the 30 Minute specimens in Figure 5-30 demonstrates that an average of over 1000 g/m^2 of surface mass was lost within the first 10 cycles. The relative humidity in the laboratory during the time of specimen manufacture was very high at 83%. The potential effect that this high relative humidity may have had on the very poor scaling resistance of the 30 Minute specimens is discussed in Section 5.4.3.

Table 5-32: Mix 6-E Scaling Data from ASTM C672 Tests

Mix 6-E Acrylic						
Relative Humidity	Specimens	Scaled Mass at 30 Cycles (g/m^2)	Change in Scaling Amount from 30 Minutes (%)	Scaled Mass at 60 Cycles (g/m2)	Change in Scaling Amount from 30 Minutes (%)	Damage Occuring in First 15 Cycles (%)
83%	30 Minutes	1582.0	-----	1933.9	-----	64%
	4 Hours	173.0	-89%	213.8	-89%	44%
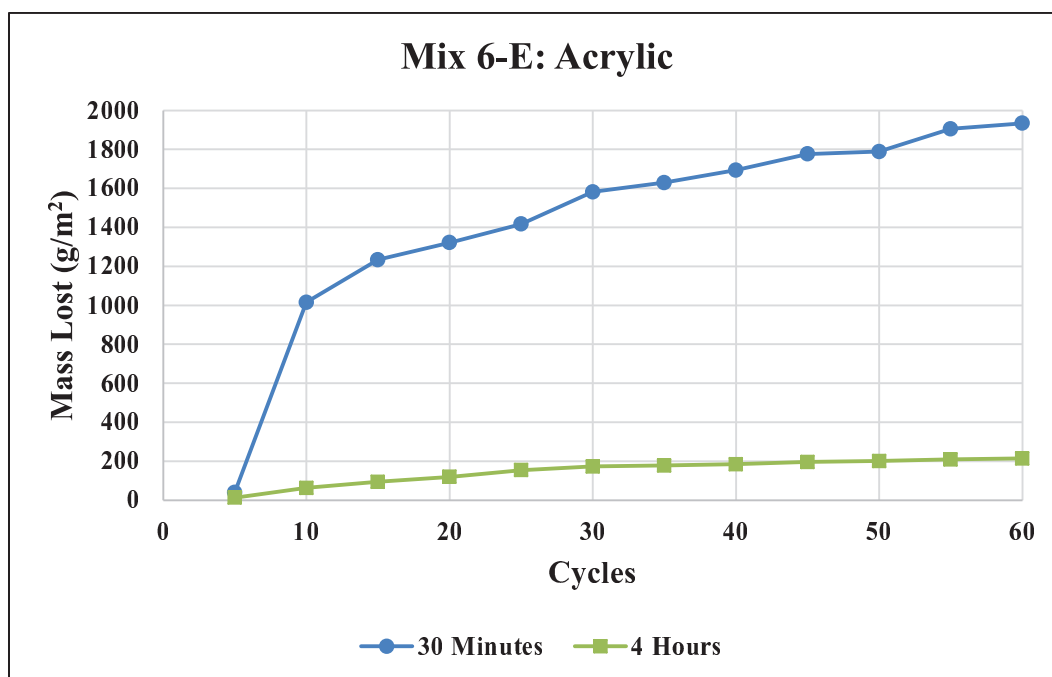
Figure 5-30: Mix 6-E Cumulative Scaling from ASTM C672 Tests

5.3 Statistical Analysis of ASTM C672 Results

The specimen testing matrix located in Appendix A was assembled with four factors: aggregate type, cementitious material, curing compound type, and application time. Application time was the primary factor. The analysis within this section was performed using the scaling data from the

full factorial population of 198 ASTM C672 specimens to observe scaling damage trends from each factor and to establish statistical significance for comparisons between application times.

Three types of statistical tests were used to analyze the data: Student's t-test for significant difference, one-factor Analysis of Variance (ANOVA), and two-factor ANOVA. For the purpose of analysis within Section 5.3.1, the samples sizes taken from the initial population size of 198 were assumed to be normally distributed. Although the full distribution of scaling data from all 198 specimens may not have strictly followed the Normal Distribution, the Central Limit Theorem allows for the assumption of normality if the sample sizes taken from the total population number 30 data points or larger (Nelson, 2003).

For the ANOVA tests within Sections 5.3.2 and 5.3.3, the samples sizes for each test were less than 30, so normality could not assumed immediately by the Central Limit Theorem. However, normality can be assumed for the purpose of the ANOVA test if the residuals of the samples means are approximately normal (Nelson, 2003). A derivation that confirms that this was true for the scaling data set used in this study is located in Appendix E. The Student's t-tests performed in Section 5.3.4 assumed unpaired samples with unequal variances being tested to identify significant differences between the sample means. The sample sizes for each test were three, corresponding to the three replicates of each mix type-curing compound-application time subset. While it is certainly ideal to have larger sample sizes, the Student's t-test has been determined to be robust enough to analyze samples sizes as small as two (de Winter, 2013).

It is important to note that the specimen testing matrix in Appendix A did not include 2 Hour specimens for all batches containing fly ash or batches coated with Acrylic. In order to perform ANOVA testing, the data sets must be balanced. Therefore, it was not possible run ANOVA tests on data sets with missing replicates of entire factors without introducing potentially inaccurate

inferences. Where fly ash or acrylic were included in the ANOVA analysis, all other 2 Hour specimens from the other treatment types were not included to prevent unbalanced data sets. Wet Room specimens were also not included in analysis where curing compound or application time were factors.

5.3.1   Analysis by Concrete Composition

The first two factors, aggregate type and cementitious material were analyzed for significance with respect to scaling resistance in this section. This included two levels of aggregate type: limestone and glacial gravel; along with three levels of cementitious material: OPC, 30% slag replacement and 30% fly ash replacement.

5.3.1.1   *Impact of Coarse Aggregate on Concrete Scaling*

A one-factor ANOVA test was performed on the total population of scaling data to compare differences between the two aggregates used within this study. A confidence level of 95% was used to assess the statistical difference between the means of the two aggregates. Of the 198 specimens evaluated in this study, 99 contained the crushed limestone and 99 contained the glacial gravel. As shown in Table 5-33, this analysis indicated that the difference in overall mean of scaling data between the two aggregates were statistically significant. Statistically significant differences imply that the two means can be compared directly. Gravel-containing specimens typically scaled approximate 30% more than Limestone-containing specimens within this project.

Table 5-33: One-Factor ANOVA Analysis of Scaling Data between Aggregates

Aggregate	Sample Sizes	Scaling Mean (g/m^2)	Statistical Significance
Limestone	99	481.6	YES
Gravel	99	627.1	

5.3.1.2 *Impact of Cementitious Material*

A single-factor ANOVA analysis was performed on the specimen scaling data to understand the impact on scaling resistance from alterations in the cementitious content of the concretes. As shown in Table 5-34 the ANOVA results showed statistically valid differences between the means of the scaling data between the three cementitious materials. Two-tailed, unequal variance Student's t-test were performed comparing the three conditions without 2 Hour specimens. The threshold level was altered in accordance with Bonferroni Correction for multiple t-tests within the same sample groups (Abdi, 2007). Statistical significance was identified between the means of OPC and slag and OPC and fly ash, but not between slag and fly ash for specimen sets not containing the 2 Hour specimen sets. An additional t-test between the OPC and slag scaling data containing their 2 Hour specimen data also found significance between the two cementitious materials, as shown in Table 5-35. Slag and fly ash-containing specimens typically scaled more than OPC-only specimens.

Table 5-34: ANOVA and t-test results of scaling data between Cementitious Materials

Single -Factor ANOVA for Cementitious Materials				t-Test	
Cementitious Material	Sample Sizes	Mean (g/m^2)	Statistical Significance	t-test Comparison	Statistical Significance
OPC	54	248.9		OPC vs Slag	YES
Slag	54	696.1	YES	OPC vs FA	YES
FlyAsh	54	643.6		Slag vs FA	NO

Table 5-35: Alternate t-test results for OPC/Slag with 2 Hour scaling data included

t-test Comparison	Sample Sizes	Scaling Mean (g/m^2)	Statistical Significance
OPC	72	285.2	YES
Slag	72	756.5	

5.3.2   Analysis by Mix Type

Two-factor ANOVA analysis to compare the scaling damage means was performed on each mix type twice: once for all three application times with Linseed/Wax/PAMS and again on only the 30 Minute and 4 Hour specimens for all four curing compounds, including Acrylic. The purpose of this was to determine for every mix type whether the curing compound, the application time, or an interaction between the curing compound and application time impacted the amount of scaling damage on that particular mix type at a statistically significant level. An interaction is where the impact of changing the level one factor such as application time on the scaling resistance of concrete is dependent upon the level of the other factor; in this case, the curing compound selection. The results are summarized in Table 5-36, where a 'YES' indicates whether the level of that factor or the interaction between the two factors has a significant effect on the amount of scaling damage at a 95% confidence level. A 'NO' in the table indicates that changing the level of a factor or the interaction between the two factors does not significantly alter the scaling damage amount.

Statistically significant interaction effects were observed between the curing compound and application time in every test except for Mix Type 1 and 2 when only the 30 Minute and 4 Hour specimens were tested. This lack of interaction in those mixes is overshadowed by the fact that the ANOVA tests at all three application times for Linseed, Wax, and PAMS did show significant interaction. The presence of interaction effects prevented overall conclusions about whether the choice of curing compound or the choice of application time was more important for determining the scaling resistance of a given mix type.

Table 5-36: Two-Factor ANOVA Results Comparing Application Time and Curing Compound Choice on Scaling Amounts on Selected Mix Types

Mix Type	Linseed/Wax/PAMS between Three Application Times				All Curing Compounds between 30 Minutes and 4 Hours			
	Curing Compound	Application Time	Interaction	Sample Size	Curing Compound	Application Time	Interaction	Sample Size
1	YES	YES	YES	27	YES	NO	NO	24
2	YES	YES	YES	27	YES	YES	NO	24
3					YES	YES	YES	24
4	YES	YES	YES	27	YES	YES	YES	24
5	YES	NO	YES	27	YES	NO	YES	24
6					YES	YES	YES	24

5.3.3   Analysis by Curing Compound-Trend of Application Times

Due to the interaction effects, the influence or lack of an influence of application time on the scaling resistance of concrete was analyzed by grouping the scaling data by curing compound. A one-factor ANOVA analysis was performed on the scaling data for each curing compound individually, with the data segregated into three different levels by application time. Unlike two-factor ANOVA analysis, balanced data sets are not required. This allowed the fly ash data sets to be included. The results from this are shown in Table 5-37.

Statistically significant differences between the means of the applications times were found in the Linseed Oil specimens and the Acrylic specimens, but not the Wax or PAMS specimens. Therefore, a direct comparison of the scaling damage means between the application times for the Linseed Oil and the Acrylic specimens across all mix types can be made at a 95% confidence level. For both Linseed Oil and Acrylic, the amount of scaling damage decreased with an increase in application time. For the Wax and PAMS formulations, the lack of statistically significant differences indicated that direct comparisons between the mean scaling amounts of the application times across

all mixes could not be made initially. Therefore, identifying significant differences on a mix-by-mix basis would be necessary to identify the impact of application time on scaling resistance for Wax and PAMS.

Table 5-37: ANOVA Analysis of Application Time on Scaling Damage for Each Curing Compound

Curing Compound	Sample Size	Application Time	Mean Scaling Amount (g/m^2)	Statistically Significant Differences
Linseed Oil	18	30 Minutes	1059.5	YES
	12	2 Hours	564.7	
	18	4 Hours	499.4	
Wax	18	30 Minutes	623.4	NO
	12	2 Hours	796.3	
	18	4 Hours	539.8	
PAMS	18	30 Minutes	457.2	NO
	12	2 Hours	747.9	
	18	4 Hours	444.2	
Acrylic	18	30 Minutes	770.3	YES
	18	4 Hours	228.0	

5.3.4 Analysis of Application Time on Scaling Damage for Each Mix Type-Curing Compound Combination

Student's t-tests were performed to determine if meaningful comparisons between application times could be made for each mix-type curing compound combination within this study. These tests were grouped by mix type. A sample size of three replicate specimens was used for each application time within each mix type-curing compound combination. The tests for significance between the means of the scaling data within this analysis assumed the means were the same as a null hypothesis, were two-tailed, and at a confidence level of 95%. If significance between the means was identified, indicated by a 'YES' within the tables, then a meaningful comparison of the scaling damage means could be made to assess if application time increased or decreased the scaling damage. If no

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significance difference was found, a comparison between the two application times could not be made confidently.

5.3.4.1 Mix Type 1: t-tests for Differences Based on Application Time

The comparisons of scaling damage results for all application times of all curing compounds on Mix Type 1 are summarized in Table 5-38. For Linseed Oil and PAMS, no statistically significant differences of scaling damage were observed between application times. For Wax, statistically significant differences were observed when comparing the 30 Minute or 4 Hour specimens to the 2 Hours specimens. Significant differences were observed between the 30 Minute and 4 Hour Acrylic specimens.

Table 5-38: t-Tests for Significance Differences between Application times for Mix Type 1 Specimens

Curing Compound	Application Time	Mean Scaling Amount (g/m^2)	Comparison	Statistically Different Means
Linseed Oil	30 Minutes	479.5	30 Min v. 2 Hr	NO
	2 Hours	446.0	30 Min v. 4 Hr	NO
	4 Hours	344.0	2 Hr v. 4 Hr	NO
Wax	30 Minutes	301.9	30 Min v. 2 Hr	YES
	2 Hours	621.6	30 Min v. 4 Hr	NO
	4 Hours	259.8	2 Hr v. 4 Hr	YES
PAMS	30 Minutes	53.9	30 Min v. 2 Hr	NO
	2 Hours	87.5	30 Min v. 4 Hr	NO
	4 Hours	133.5	2 Hr v. 4 Hr	NO
Acrylic	30 Minutes	142.1	30 Min v. 4 Hr	YES
	4 Hours	77.0		

5.3.4.2 Mix Type 2: t-tests for Differences Based on Application Time

The comparisons of scaling damage results for all application times of all curing compounds on Mix Type 2 are summarized in Table 5-39. For Acrylic and PAMS, no statistically significant differences were observed between application times. Linseed Oil 30 Minute specimens were significantly different than the 2 and 4 Hour specimens, but no significant differences were found between the 2 and 4 Hour specimen sets. For Wax, statistically significant differences were observed when comparing the 30 Minute to the 2 Hours specimens, but statistically significant differences were discovered between the 30 Minute and 2 Hour specimens when independently compared to the 4 Hour specimens.

Table 5-39: t-Tests for Significance Differences between Application times for Mix Type 2 Specimens

Curing Compound	Application Time	Mean Scaling Amount (g/m^2)	Comparison	Statistically Different Means
Linseed Oil	30 Minutes	1032.1	30 Min v. 2 Hr	YES
	2 Hours	587.4	30 Min v. 4 Hr	YES
	4 Hours	586.8	2 Hr v. 4 Hr	NO
Wax	30 Minutes	546.0	30 Min v. 2 Hr	NO
	2 Hours	818.3	30 Min v. 4 Hr	YES
	4 Hours	305.2	2 Hr v. 4 Hr	YES
PAMS	30 Minutes	867.0	30 Min v. 2 Hr	NO
	2 Hours	1238.0	30 Min v. 4 Hr	NO
	4 Hours	851.2	2 Hr v. 4 Hr	NO
Acrylic	30 Minutes	617.0	30 Min v. 4 Hr	NO
	4 Hours	454.5		

5.3.4.3 Mix Type 3: t-tests for Differences Based on Application Time

The comparisons of scaling damage results for all application times of all curing compounds on Mix Type 3 are summarized in Table 5-40. Statistically significant differences between means of

the application times were observed within the Linseed Oil, Wax, and Acrylic specimen sets, but not within the PAMS specimen sets.

Table 5-40: t-Tests for Significance Differences between Application times for Mix Type 3 Specimens

Curing Compound	Application Time	Mean Scaling Amount (g/m²)	Comparison	Statistically Different Means
Linseed Oil	30 Minutes 4 Hours	1479.4 642.7	30 Min v. 4 Hr	YES
Wax	30 Minutes 4 Hours	847.9 394.0	30 Min v. 4 Hr	YES
PAMS	30 Minutes 4 Hours	309.2 211.2	30 Min v. 4 Hr	NO
Acrylic	30 Minutes 4 Hours	734.1 130.9	30 Min v. 4 Hr	YES

5.3.4.4   Mix Type 4: t-tests for Differences Based on Application Time

The comparisons of scaling damage results for all application times of all curing compounds on Mix Type 4 are summarized in Table 5-41. Statistically significant differences in the scaling means were observed between all three application times for Linseed Oil. No statistically significant differences were observed between any application time for the Wax, PAMS, and Acrylic specimen sets. It is important to note that using the two-way t-test examines statistically significant differences in the means as being either larger or smaller than one another. As a result, Type II statistical error (Nelson, 2003), where no significant difference is detected when it might actually exist can occur in cases where one mean is distinctly larger or smaller than the other, such as the case with Acrylic. The standard deviations for the Acrylic 30 Minute and 4 Hour specimen sets were 175.4 and 8.9 g/m², respectively. Using these along with the means, it is evident that these means are distinctly different despite the lack of statistical significance. This implies that Type II error occurred within this test.

Table 5-41: t-Tests for Significance Differences between Application times for Mix Type 4 Specimens

Curing Compound	Application Time	Mean Scaling Amount (g/m²)	Comparison	Statistically Different Means
Linseed Oil	30 Minutes	609.1	30 Min v. 2 Hr	YES
	2 Hours	300.6	30 Min v. 4 Hr	YES
	4 Hours	217.1	2 Hr v. 4 Hr	YES
Wax	30 Minutes	178.9	30 Min v. 2 Hr	NO
	2 Hours	317.7	30 Min v. 4 Hr	NO
	4 Hours	147.3	2 Hr v. 4 Hr	NO
PAMS	30 Minutes	599.9	30 Min v. 2 Hr	NO
	2 Hours	591.4	30 Min v. 4 Hr	NO
	4 Hours	426.9	2 Hr v. 4 Hr	NO
Acrylic	30 Minutes	386.1	30 Min v. 4 Hr	NO
	4 Hours	48.0		

5.3.4.5 *Mix Type 5: t-tests for Significance Differences within Scaling Damage Means of Application time*

The comparisons of scaling damage results for all application times of all curing compounds on Mix Type 5 are summarized in Table 5-42. No statistically significant differences were observed between application times for Linseed Oil, Wax, or PAMS. Acrylic was the only curing compound within Mix Type 5 where scaling damage between application time sets were statistically significant. It is important to note that Type II error appeared to occur within the Linseed Oil set, similar to the Acrylic specimens from Mix Type 4. The standard deviations for the 30 Minute, 2 Hour and 4 Hour sets for Linseed Oil are 394.1, 132.8, and 166.9 g/m² respectively. The standard deviation for the 30 Minute set was large enough to prevent significance differences from being established within a two-way t-test at a 95% confidence level. Despite this, the distinct difference in scaling amounts between the 2 and 4 Hour sets versus the 30 Minute sets indicate that Type II error may have occurred.

Table 5-42: t-Tests for Significance Differences between Application times for Mix Type 5 Specimens

Curing Compound	Application Time	Mean Scaling Amount (g/m^2)	Comparison	Statistically Different Means
Linseed Oil	30 Minutes	1295.2	30 Min v. 2 Hr	NO
	2 Hours	480.2	30 Min v. 4 Hr	NO
	4 Hours	478.9	2 Hr v. 4 Hr	NO
Wax	30 Minutes	1082.1	30 Min v. 2 Hr	NO
	2 Hours	1427.4	30 Min v. 4 Hr	NO
	4 Hours	1331.4	2 Hr v. 4 Hr	NO
PAMS	30 Minutes	655.8	30 Min v. 2 Hr	NO
	2 Hours	1074.8	30 Min v. 4 Hr	NO
	4 Hours	791.3	2 Hr v. 4 Hr	NO
Acrylic	30 Minutes	808.4	30 Min v. 4 Hr	YES
	4 Hours	444.0		

5.3.4.6 Mix Type 6: t-tests for Significance Differences within Scaling Damage Means of Application time

The comparisons of scaling damage results for all application times of all curing compounds on Mix Type 6 are summarized in Table 5-43. Statistically significant differences were observed between the application times for the Linseed Oil and Acrylic specimen sets, but not in the Wax or PAMS specimen sets.

Table 5-43: t-Tests for Significance Differences between Application times for Mix Type 6 Specimens

Curing Compound	Application Time	Mean Scaling Amount (g/m^2)	Comparison	Statistically Different Means
Linseed Oil	30 Minutes	1461.6	30 Min v. 4 Hr	YES
	4 Hours	726.2		
Wax	30 Minutes	783.4	30 Min v. 4 Hr	NO
	4 Hours	801.2		
PAMS	30 Minutes	257.2	30 Min v. 4 Hr	NO
	4 Hours	251.3		
Acrylic	30 Minutes	1933.9	30 Min v. 4 Hr	YES
	4 Hours	213.8		

5.3.5 Analysis of Wet Room Specimen Data

Wet Room cured specimens from each mix type were tested to represent a theoretically ideal curing condition. Based upon a review of existing literature, it was thought that specimens cured in this manner would exhibit a baseline level of scaling damage from which to compare the performance of the curing compounds. To make these comparisons, the average scaling amounts from every curing compound-application time specimen set were normalized to the Wet Room scaling damage amounts for each mix type. The result of this normalization were damage factors by which a curing compound-application time specimen set scaled at relative to the Wet Room specimen set. The average and range of these damage factors across mix types for each curing compound-application time are presented in Table 5-44.

Table 5-44: Scaling Damage Factors of Curing Compounds Relative to Wet Room Specimens

Curing Compound	Application Time	Range of Factors		Average
		Min	Max	
Linseed Oil	30 Minutes	5	28	12
	2 Hours	2	26	9
	4 Hours	2	20	6
Wax	30 Minutes	3	18	7
	2 Hours	5	36	13
	4 Hours	2	15	5
PAMS	30 Minutes	1	10	4
	2 Hours	5	10	7
	4 Hours	1	8	4
Acrylic	30 Minutes	3	8	6
	4 Hours	1	5	2

In all specimen sets except PAMS 30 Minutes, PAMS 4 Hours, and Acrylic 4 Hours, the curing compounds at a minimum exhibited increased scaling relative to the Wet Room specimens. At a maximum, select specimen sets from Linseed Oil and Wax at all application times and PAMS at 30 Minutes and 4 Hours displayed scaling damage amounts that were at least an order of magnitude

higher than the Wet Room specimens. Averaged across all mix types, the curing compound specimens exhibited scaling damage that exceeded the Wet Room specimens from a factor of 2 within the Acrylic 4 Hour specimens to a factor of 13 within the Wax 2 Hour specimens.

5.4   Discussion of Contrary Data

A review the data in Section 5.2 revealed that from the 30 non-Wet Room subsets, approximately 21 of them had scaling patterns that followed the hypothesis that scaling resistance increased with an increase in the application time based upon magnitude alone. These included all of the Linseed Oil and Acrylic coated specimen sets. The remaining nine subsets that did not immediately fit the prediction all exhibited levels of scaling in the 2 Hour or 4 Hour specimen sets that exceeded the 30 Minutes scaling amounts. All were either coated with the Wax or the PAMS compounds, with four of them being applied to concretes containing slag. At least one of the following four factors were present in these nine outlier batches:

1. Inclusion of slag cement,

2. Wax-based curing compound,

3. Higher ambient relative humidity at the time of curing compound application,

4. High deviation in scaling losses between samples from the 1-D and 6-D mixes.

This section discusses the potential impact that these factors may have had on the scaling patterns of the nine outlier batches. For reference, Table 5-45 summarizes each affected mix and factors that may explain their outlying scaling patterns.

Table 5-45: Mixes with Outlying Scaling Data

Affected Mixes	Potential Explanations			
	Impact of Slag	Wax Curing Compound	High Relative Humidity	1-D/6-D
1-C		X		
1-D				X
2-C	X	X		
2-D	X			
4-C		X		
5-C	X	X	X	
5-D	X			
6-C		X	X	
6-D				X

5.4.1 The Impact of Slag Cement

Affected mixes: 2-C, 2-D, 5-C, 5-D

Previous research at the University of Wisconsin-Madison and at other institutions has shown that concretes made with partial replacement of OPC with Slag Cement typically have lower freeze-thaw scaling resistance than comparable concretes made with OPC. Carbonation of hydration products within the cement paste at the concrete surface has been identified as a longer term cause for a reduction in scaling resistance in slag-containing concretes. Including slag as a replacement for cement reduces the amount of calcium hydroxide (CH) during the hydration reactions shown in equations (1) and (2) from Section 3.2.This slows the growth of the hydration products in the paste, resulting in a fresh surface that exhibits increased bleeding time to set (Wainwright and Ait-Aider, 1995). Longer term, ambient carbon dioxide in the air surrounding the concrete will infiltrate the paste, and react preferentially with CH to form calcium carbonate, a product that densifies the surface over time. In slag-containing concretes that do not supply enough CH, the carbon dioxide will react

with the CSH gel by combining with the calcium in the gel, to form calcium carbonate. This results in a decalcified CSH microstructure of the paste, reducing the volume and increasing the porosity of the surface; ultimately reducing the scaling resistance of the surface layer. (Battaglia et al. 2010)

Research by Wainwright and Ait-Aider confirmed that slag cement as a replacement for OPC had the dual effect of increasing the both the rate and amount of bleeding (1995), most likely as a result of the delayed growth of hydration products (Battaglia et al. 2010). The application of a curing compound immediately reduces the moisture loss of unbound water at a concrete surface (Wang et al. 1994). However, the disassociation of a curing compound membrane may occur due to the carrier emulsion possibly having a lower density than that of the bleed water it envelopes. The precise densities of the compound emulsions used in this study could not be ascertained due to their proprietary nature.

Therefore, it is hypothesized that the inclusion of slag with the resulting differences in rate of duration of bleeding renders a less predictable situation as to the ideal time to apply curing compounds. In both the limestone-based and glacial gravel-based slag cement specimens, 2 Hour application of Wax and PAMS curing compounds exhibited the highest rates of scaling and in some of these cases the 4 Hour application scaling exceeded the 30-minute values. This suggests that bleeding in the slag cement specimens possibly peaked around 2 hours and continued through the 4 hour time. As these compounds are applied, they mix with or float on the surface of the bleed water, preventing the membrane from fully forming to ensure proper curing and deicer ingress. By applying these curing compounds at 30 Minutes, the membrane may solidify prior to being disassociated by additional bleed water unlike the 2 Hour specimens.

5.4.2   The Impact of Wax-Based Curing Compound

Affected Mixes: 1-C, 2-C, 3-C, 4-C, 5-C, 6-C

The Wax, PAMS, and Linseed Oil curing compounds used on this study were emulsions consisting of a solid portion suspended in a water carrier. Upon placement, the water carrier evaporates and deposits the solid portion, which forms the membrane. PAMS and Linseed Oil were ASTM C309 defined Type II Class B curing compounds, specifying the solids portion as organic resins with typically high molecular weights. The Wax curing compound however was defined as a Type II Class A curing compound, which doesn't specify any particular structure for the solids portion. The manufacturer specification defined the solids portion to be a wax of petroleum origin, most likely a form of paraffin wax. It was characterized by appearing to have a lower viscosity and density than the other emulsion-based curing compounds. This is most likely due to the manufacturer specifying the solids portion between 15 to 25% of the emulsion, as compared with 40 to 50% for the Linseed Oil and approximately 52.5% for the PAMS. Because all three compounds had identical spray rates; this meant that the Wax deposited less of the solid, or active component of the membrane by weight than the other two emulsions.   This may have made it more sensitive to surface conditions of the concrete.

By visual inspection, the Wax coating appeared thinner and less dense than the other emulsions. This was especially prevalent when sprayed upon the 30 Minute and 2 Hour specimens; where the coating appeared to float on the surface of the bleed water layer and very thin but noticeable cracks developed where bleed water appeared to disassociate the pigmentation. During ASTM C672 testing, the Wax layer stripped off in thousands of small flakes within the first 10-15 cycles for all specimens; indicative of a poorly bonded coating to the surface of the concrete. While the high amounts of scaling within the 2 Hour specimens relative to the other two application times may have indicated that 2 hours was insufficient in some mixes for the bleed water to dissipate.

5.4.3 The Impact of High Relative Humidity at the Time of Curing Compound Application

Affected Mixes: 5-C, 6-C

Relative humidity is the measure of the level of water vapor within a sample of air expressed as a percentage of the maximum amount that air could possibly hold at that temperature. In this study, all specimens were cast in a controlled laboratory setting where temperatures ranged between 70-74°Fahrenheit at the time of casting; so the comparisons of the relative humidities are nearly direct comparisons to the amount of water vapor within the air. When air temperature is held constant with no turbulence, the rate of evaporation decreases with an increase in the relative humidity.(Uno, 1998)

Mixes 4-E, 5-A, 5-C, 5-E, 6-A, 6-C, and 6-E were poured in times of elevated ambient relative humidities, as defined by being 60% or above measured by the laboratory hygrometer. All other mixes were poured during the heating season with relative humidities between 21% and 36%. Two of these mixes, 5-C and 6-C exhibited scaling amounts after 60 cycles within their 2 and 4 Hour application time specimens that exceeded the scaling in their 30 Minute specimens. Both of these mixes used Wax curing compound; with 5-C possessing slag. Mix 5-C did exhibit the highest amount of scaling across all three application times of all mixes except for the 30 Minute specimens from Mix 6-E.   This suggests that the detrimental effects previously discussed in Sections 5.4.1 and 5.4.2 resulted in increased bleed water at the later application times, leading to reduced scaling resistance. The other mixes poured at high relative humidities did not have additional 2 Hour specimens to analyze. It is hypothesized that due to reduced evaporation rates as a result of high relative humidities, bleed water remained on the surface of these concretes when the Wax was applied at 2 and 4 Hours for mixes 5-C and 6-C. The presence of bleed water on the surface at these application times may have induced areas of disassociated membrane, leaving the surface more susceptible to

improper curing and deicer ingress. Small, yet noticeable pinholes and cracks in the solid membrane were detected visually in these specimens after demolding. This potentially had the effect of reducing the Wax compound's ability to provide an adequate membrane, and by extension, reduced the scaling resistance of the concrete.

The very high laboratory relative humidity of 83% when Mix 6-E specimens were manufactured and coated may have reduced the evaporation of bleed water significantly, resulting in the entrapment of large amounts of bleed water when the Acrylic was applied at 30 Minutes. This entrapment of bleed water may have severely reduced the scaling resistance of these specimens, which resulted in the very high scaling damage exhibited in Figure 5-30.

5.4.4   Mix 1-D and Mix 6-D

Affected mixes: 1-D, 6-D

Both Mix 1-D and 6-D possessed scaling accumulation curves that were worthy of further analysis. Mix 1-D (OPC with PAMS) as shown in Figure 5-4, exhibited very low scaling and very similar scaling accumulation curves across all three application times. The behavior that warranted further review was that the 30 Minute specimens scaled the least and the 4 Hour specimens scaled the most. Alternatively, Mix 6-D (Fly ash with PAMS) as shown in Figure 5-29, did exhibit more total scaling in the 30 Minute specimens than the 4 Hour specimens, but only after the 4 Hour specimens had sustained a higher level of scaling for the first 55 cycles.   These relatively low scaling amounts combined with the statistical analyses shown in section 5.3.4 suggest that the PAMS specimens ability to resist scaling was not sensitive to application timing and within the application range of 30-minutes to 4-hours, the scaling results were similar.

5.5 Comparison to Results from Phase I to Determine Repeatability

A goal of this research project was to assess the repeatability of scaling data results from Phase I. It was expected that repeatability under controlled lab conditions following careful protocols with concrete manufactured with similar materials would represent a lower bound of the variability that is likely to occur in field conditions.   Major observations from Phase I included:

- Linseed Oil exhibited poor scaling resistance relative to other emulsions.

- Wax emulsion curing compound exhibited good scaling resistance in comparison with PAMS and Linseed Oil emulsion.

- Curing/sealing agents provide highly durable surfaces in relation to the emulsion-based curing compounds and that may justify their additional expense.

- There was significant variability not between specimens from of the same mix-curing compound subset, but from mix type to mix type with a given curing compound

The specimen mix matrix for Phase II in Table A-1, was assembled to reflect and expand upon these findings. Both studies used very similar materials and admixtures. Phase II used the same Wax, PAMS, Linseed Oil, and Acrylic curing compounds as Phase I, in addition to wet room cured control specimens. The mixing procedure, molds, finishing efforts, and pre-ASTM C672 specimen preparation features were used to restrict variability as much as possible. All Phase I specimens were prepared with a constant two hours between initial finishing and curing compound application. Phase II included a limited number of two hour samples to provide a linkage back to Phase I results. Fly Ash blends and specimens coated with the Acrylic sealing compound in Phase II were not

manufactured with a two hour specimen set because of limitations in the scope of the Phase II study. In both studies, three replicates of each mix type-curing compound-application time were created.

Curing compound application rates were slightly different between the two studies as the application monitoring process was more refined in Phase II. Average recorded application rates of the curing compounds between the two studies are shown in Table 5-46. The 'Target Amount' was an approximate amount of curing compound per specimen calculated from each compounds' manufacturer's recommended spray rate. The Phase II specimens average application rates were at or within 0.1 grams of the spray rate. The differences in spray rate expressed as a percentage of the target amount for Phase I Wax and PAMS were 3% less and 3% more, respectively. Relative to the overall spray rate, these differences are small. Acrylic spray rate from Phase I was 44% higher than the manufacturer's recommendation, an apparent calculation error. .

Table 5-46: Average Curing Compound Application Rates from Phases I and II

Curing Compound Spray Rates (grams/specimen)			
Curing Compound	Target Amount	Phase I	Phase II
Linseed Oil	14.9	14.9	14.8
Wax	14.9	14.5	14.9
PAMS	14.9	15.4	14.9
Acrylic	9.1	13.1	9.2

5.5.1   Comparison of Results between Phases I and II by Scaling Amounts

The scaling amounts measured from like specimens in Phase I versus Phase II were not consistently comparable. Consider the Phase I and Phase II scaling comparisons shown in Figures 5-31 through 5-35 for the Linseed Oil curing compound. Figure 5-31 shows the scaling results for the OPC-Limestone aggregate mix (Mix 1). While Phase I scaling results had the general magnitude observed in Phase II, the 2 Hour Phase I results scaled less than all Phase II application times. For

this situation, Phase I and Phase II results were obviously not repeatable. Figure 5-32 shows the scaling results for the Slag cement-Limestone aggregate mix (Mix 2). In this case, Phase I scaling results were greater than Phase II for all curing compound application times and again, Phase I and Phase II cannot be considered repeatable. Figure 5-33 shows the scaling results for the OPC-Glacial gravel aggregate mix (Mix 4). Again, Phase I scaling results are considerably higher than Phase II results for all application times. Figure 5-34 shows comparable results for the Slag Cement-Glacial gravel aggregate mix (Mix 5). In this case the Phase I results between the 30 Minute and 4 Hour application time results for Phase II but again higher than the Phase II 2 Hour results. These four examples are representative of the other results. They illustrate that while the Phase I scaling results were generally of similar in magnitude of the Phase II scaling results they were not generally repeatable such that the Phase I and Phase II data sets could be combined.

The error in Phase I of applying acrylic at higher rate than in Phase II provided an interesting evaluation of the impact of application rate versus time of application. Generally the higher application rate did result in less scaling but time of application appeared to dominate in most conditions.
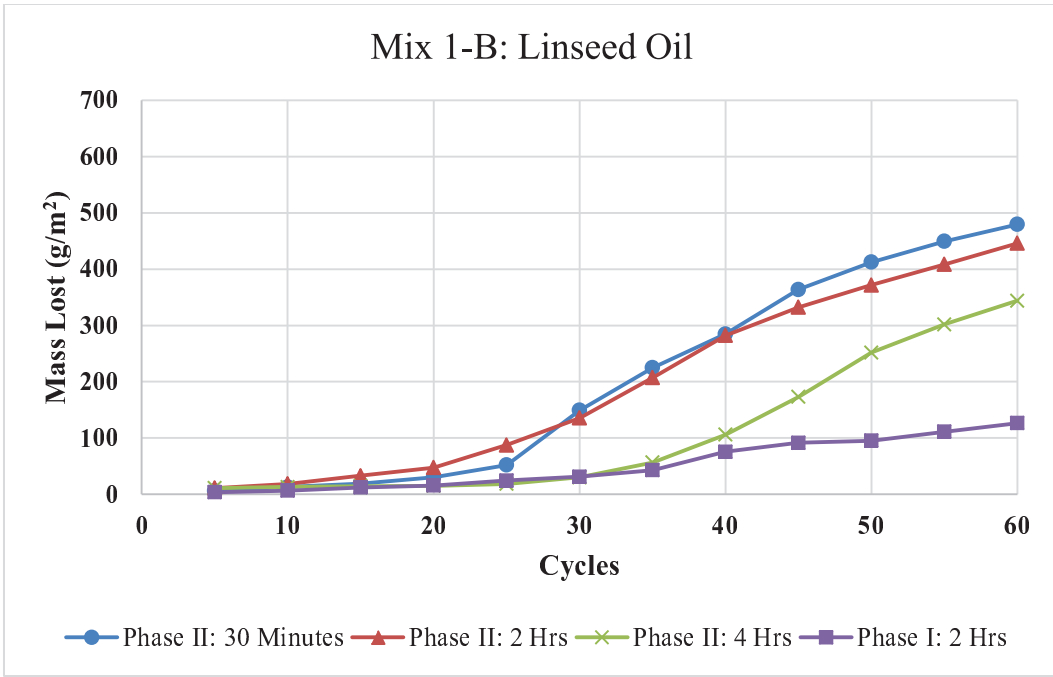
Figure 5-31: Mix 1-B Phase II Scaling Data from ASTM C672 Tests Compared to Phase I
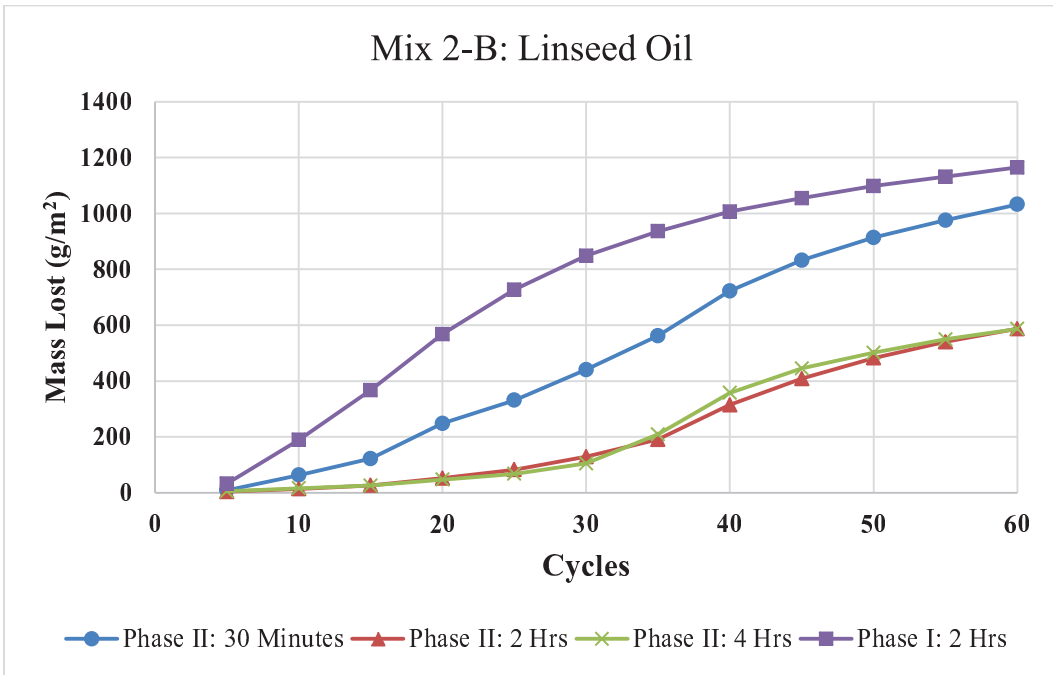
Figure 5-32: Mix2-B Phase II Scaling Data from ASTM C672 Tests Compared to Phase I

90
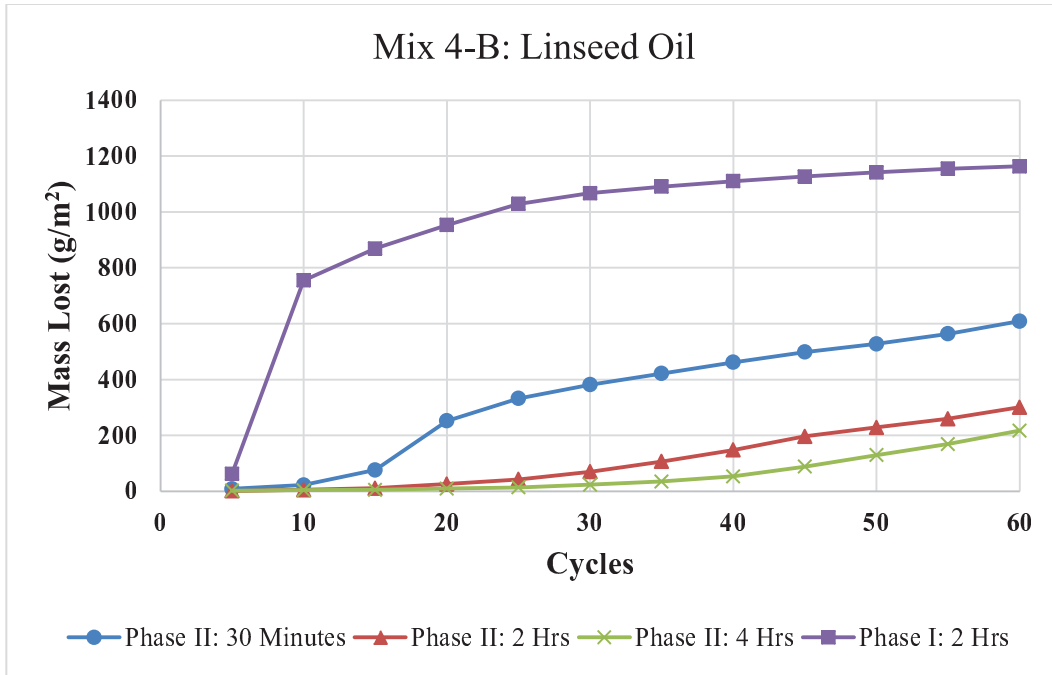
Figure 5-33 Mix 4-B Phase II Scaling Data from ASTM C672 Tests Compared to Phase I
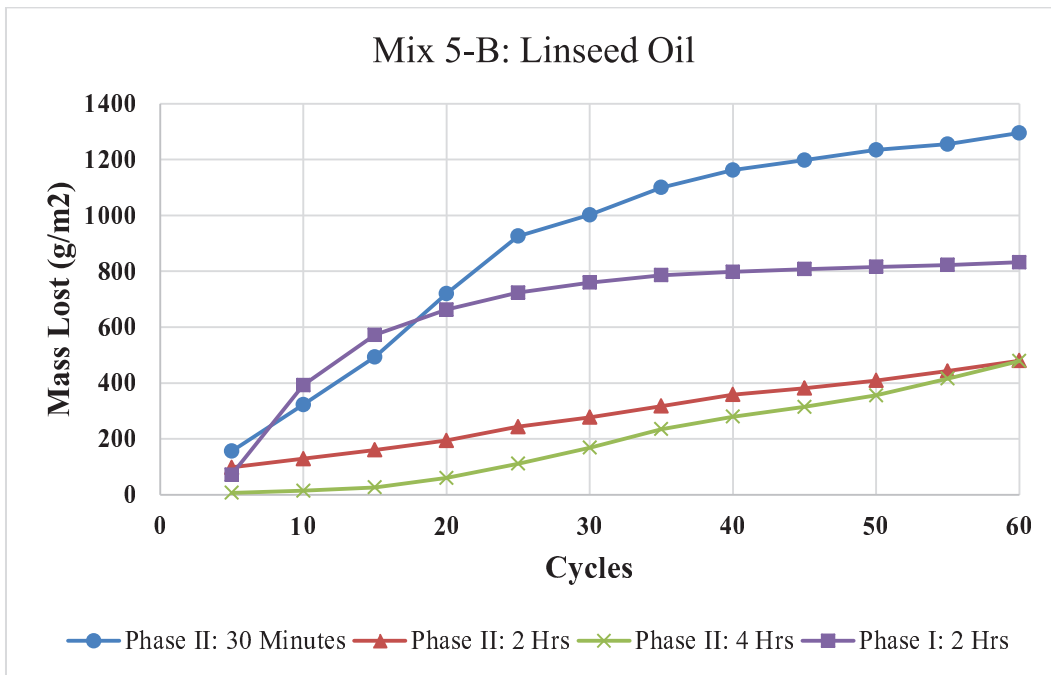
Figure 5-34 Mix 5-B Phase II Scaling Data from ASTM C672 Tests Compared to Phase I

5.5.2 Comparison of Results between Phases I and II by Ranked Curing Compound Performance

An alternative way of evaluating repeatability is the ranking of scaling data after 60 cycles on each mix type between the compounds from most resistance to least resistance. This allows for simple identification of repeatability of curing condition performance relative to the other curing conditions, even if the magnitudes of scaling might not be similar from each Phase. Wet room specimens were not included in this repeatability analysis due to their absence from Phase I's results.

Table 5-47 shows the curing compound performance of the conditions on Mix Type 1. It is important to note that the repeatability of this set cannot be inferred by ranking due to the lack of detailed application rates of the Acrylic, PAMS, and Linseed specimens from Phase I for this mix.

Table 5-47: Ranking of Curing Compound Scaling Resistances for Mix Type 1.

Mix Type 1-Limestone/OPC				
Rank	Phase II-30 Minutes	Phase I-2 Hours	Phase II-2 Hours	Phase II-4 Hours
1	PAMS	Acrylic	PAMS	Acrylic
2	Acrylic	Wax	Linseed	PAMS
3	Wax	PAMS	Wax	Wax
4	Linseed	Linseed		Linseed

The Mix Type 2 batches are compared in Table 5-48. PAMS and Linseed formulations display low repeatability, as in Phase I PAMS showed excellent resistance relative to the other compounds while Linseed showed poor resistance.

Table 5-48: Ranking of Curing Compound Scaling Resistances for Mix Type 2

Mix Type 2-Limestone/Slag				
Rank	**Phase II-30 Minutes**	**Phase I-2 Hours**	**Phase II-2 Hours**	**Phase II-4 Hours**
1	Wax	PAMS	Linseed	Wax
2	Acrylic	Acrylic	Wax	Acrylic
3	PAMS	Wax	PAMS	Linseed
4	Linseed	Linseed		PAMS

Curing compound rankings for Mix Type 3 are shown in Table 5-49. The Acrylic compound in both Phases exhibited the best or second best scaling resistance. The Linseed exhibited the worst resistance to scaling of all compounds in both studies, indicating repeatability. The PAMS and Wax did not display repeatability between the two studies based upon ranking. In summation, the Acrylic and Linseed showed repeatability between studies while the PAMS and Wax did not.

Table 5-49: Ranking of Curing Compound Scaling Resistances for Mix Type 3

Mix Type 3-Limestone/Fly Ash			
Rank	**Phase II-30 Minutes**	**Phase I-2 Hours**	**Phase II-4 Hours**
1	PAMS	Wax	Acrylic
2	Acrylic	Acrylic	PAMS
3	Wax	PAMS	Wax
4	Linseed	Linseed	Linseed

Curing compound rankings for Mix Type 4 are shown in Table 5-50. Wax, Acrylic, and PAMS all exhibited repeatability in the 2 Hour specimens for both Phases. Linseed Oil exhibited the worst scaling resistance in Phase I 2 Hour specimens of all the curing compounds, while it exhibited the best resistance among the Phase II 2 Hour specimens.

Table 5-50: Ranking of Curing Compound Scaling Resistances for Mix Type 4

Mix Type 4-Gravel/OPC				
Rank	Phase II-30 Minutes	Phase I-2 Hours	Phase II-2 Hours	Phase II-4 Hours
1	Wax	Wax	Linseed	Acrylic
2	Acrylic	Acrylic	Wax	Wax
3	PAMS	PAMS	PAMS	Linseed
4	Linseed	Linseed		PAMS

Curing compound rankings for Mix Type 5 are shown in Table 5-51. Similar to the Mix Type 4 results, the Linseed Oil exhibited the worst scaling resistance of the formulations in Phase I's 2 Hour specimens, while exhibiting the best resistance in Phase II's 2 Hour specimens. Acrylic, PAMS and Wax were ranked the same in both 2 Hour specimen sets, while the Acrylic specimens in both Phases were either the best or second best formulation for resisting scaling.

Table 5-51: Ranking of Curing Compound Scaling Resistances for Mix Type 5

Mix Type 5-Gravel/Slag				
Rank	Phase II-30 Minutes	Phase I-2 Hours	Phase II-2 Hours	Phase II-4 Hours
1	PAMS	Acrylic	Linseed	Acrylic
2	Acrylic	PAMS	PAMS	Linseed
3	Wax	Wax	Wax	PAMS
4	Linseed	Linseed		Wax

Curing compound rankings for Mix Type 6 are shown in Table 5-52. The Linseed Oil specimens from both Phases exhibited the worst or second worst scaling resistance, which can be considered repeatable. The PAMS, Acrylic, and Wax specimens from both studies exhibited no consistent order of rankings that would indicate repeatability, as each compound held three different

rankings among the three sets analyzed. Therefore, only the Linseed Oil results can be considered repeatable.

Table 5-52: Ranking of Curing Compound Scaling Resistances for Mix Type 6

Mix Type 6-Gravel/Fly Ash			
Rank	Phase II-30 Minutes	Phase I-2 Hours	Phase II-4 Hours
1	PAMS	Wax	Acrylic
2	Wax	Acrylic	PAMS
3	Linseed	PAMS	Linseed
4	Acrylic	Linseed	Wax

The results from this analysis of the repeatability based on relative ranking of scaling resistance between Phases I and II are summarized in Table 5-53. Of the five mixes that were analyzed, Acrylic was the only compound with repeatability identified four times. This shows consistent repeatability across most mixes, despite the differences in application rate between the two studies. Linseed Oil was only repeatable in Mix Types 3 and 6, which contained fly ash. While Wax was repeatable on three mix types, more conclusions about the repeatability of the Wax across all mix types cannot be argued without more analysis. Likewise, PAMS repeatability in only two mixes prevent larger conclusions from being drawn.

Table 5-53: Summary of Repeatability Rankings between Phases I and II

	Mix 1	Mix 2	Mix 3	Mix 4	Mix 5	Mix 6
Repeatable	N/A	Wax Acrylic	Linseed Acrylic	Wax PAMS Acrylic	Wax PAMS Acrylic	Linseed
Not Repeatable	N/A	Linseed PAMS	Wax PAMS	Linseed	Linseed	Wax PAMS Acrylic

5.5.3   Repeatability Results Discussion

Consistent repeatability in actual scaling amounts between like specimens in Phase I and Phase II did not generally occur. This was despite very similar materials, specimens fabricated under similar laboratory conditions, but with two different researchers. Certainly, the variabilities in actual field construction far exceed those in this study and thus it is clear there are variables at play that likely defy simple application rules.

Two unaccounted variables could have influenced the results. The first is the changes in relative humidity within the laboratory and the influence this humidity had on concrete surface moisture. Since we do not have laboratory relative humidity measurements from Phase I, analysis with that variable is not possible. There have been indications in Phase II that laboratory humidity makes a difference.

The second unaccounted variable concerns finishing practice in the ASTM C672 specimen fabrication. Each researcher generally used similar techniques and tools that conformed to the standard but precise level of effort to finish the top of the specimens was uncontrolled. As mentioned earlier approximately 12 passes were made over each specimen in Phase II. In Phase I, the minimum amount of finishing required to cover the large aggregate particles near the surface using a wooden strike-off board was the procedure but the actual number of passes with each mix type were not recorded.   To explore the sensitivity of this effect, the Phase II researcher examined the influence of different levels of finishing effort on ASTM C672 outcomes for the two OPC mix types using only the PAMS curing compound. ASTM C672 specimens were finished with 2 passes of the wood strike off board, 10, 15 and 30 passes and then after 2 Hours, treated with the PAMS curing compound. Figure 5-35 shows excellent repeatability amongst both Phase I and Phase II for OPC-Limestone aggregate-PAMS combinations and indicates that finishing had very little effect on C672 durability

and all of the results are bracketed by the 30 Minute (most scaling) and the 4 Hour (least scaling). In general more finishing produced slightly more durable surfaces. Figure 5-36 for OPC-Glacial gravel-PAMS does not show the same level of repeatability. In this case, the results somewhat sort by mix date with the 12 pass Phase II mixes being made on one date, Phase I on another date and all other Phase II mixes made on a third date. The major observed difference between these mixes was that the laboratory relative humidity was considerably higher for the 12 pass Phase II mixes while the relative humidity was low for the other Phase II mixes and is believed to have been low for the Phase I mix based on date of mixing. The Phase II specimens manufactured with different levels of finishing generally correspond to the Phase I scaling results, indicating repeatability in that case but not the other.
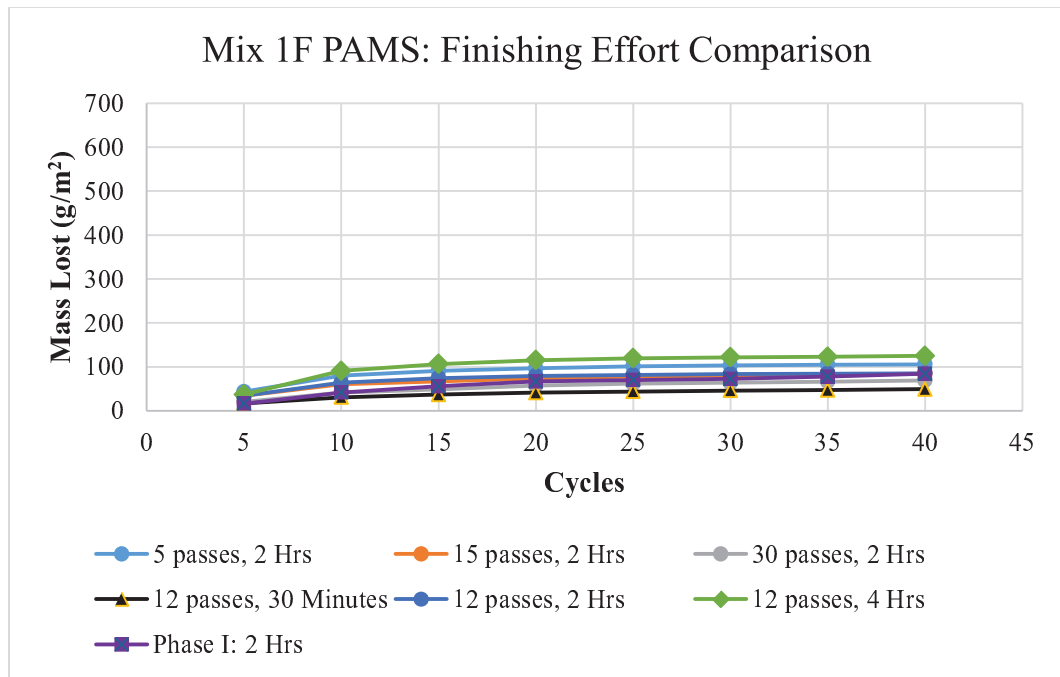
Figure 5-35 Phase II and Phase I Finishing Effort Comparison for OPC Limestone Aggregate and PAMS
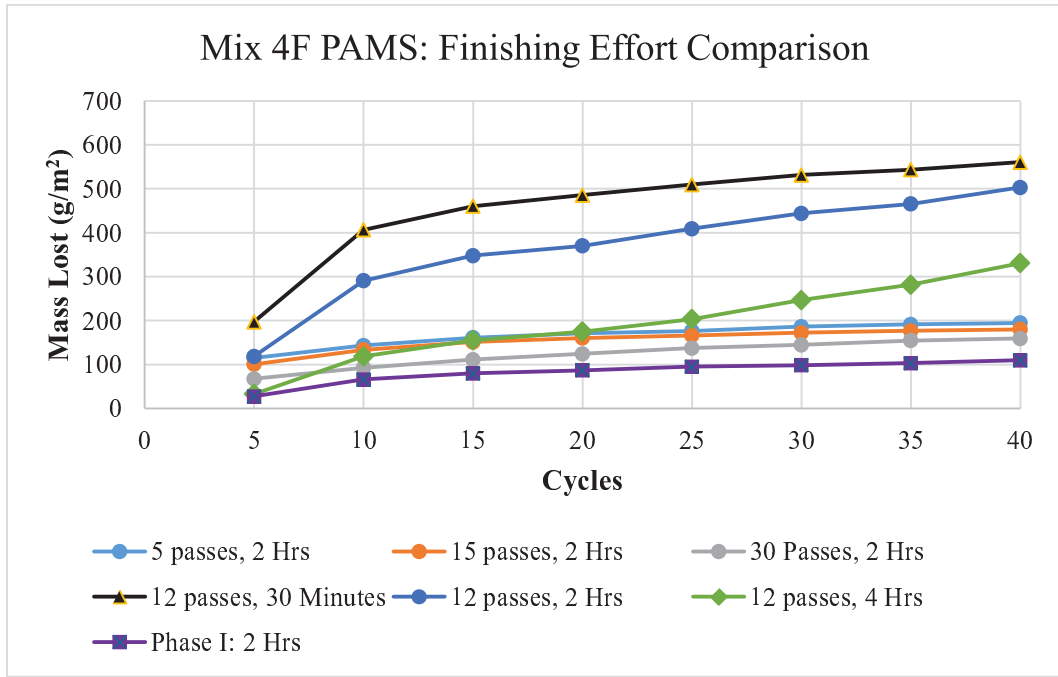
Figure 5-36 Phase II and Phase I Finishing Effort Comparison for OPC Gravel Aggregate and PAMS

Organizing the data by ranking the performance of each curing condition on each mix type reveals that there are levels of repeatability between the two studies despite some inconsistencies in scaling magnitude. By ranking the data, all curing compounds showed levels of repeatability in at least two of the mix types used, with Acrylic sealing compound displaying repeatability across all six. Linseed and Wax specimens introduced variability that brought down the level of repeatability in the ranking analysis. Overall, ranking the scaling performance of each curing compound relative to one another did display acceptable levels of repeatability between the two phases for Wax and Acrylic in particular.

5.6 ARCHERS Results and Discussion

The design of the ARCHERS logic was continually revised through the project to improve system response.

5.6.1 ARCHERS Performance and Data Analysis

Ideal performance from the ARCHERS was predicated upon notifying an operator about the operation of the fan duty cycle graphically. The presence of concrete bleed water on the surface would dictate the response of the fan speed in three distinct operational phases as shown with approximate times in Figure 5-33:

Figure 5-37: Ideal Fan Duty Cycle Performance for ARCHERS During Concrete Bleeding

Phase 1.   Bleeding (0 to around 1.5 hours): Bleed water migrates to surface of concrete, and water is present on the surface. Evaporated water is continually measured by the outlet hygrometer, which sustains the FDC at or near its upper operational limit.

Phase 2.   Evaporation (1.5 to 2.5 Hours): Concrete bleeding terminates. The remaining standing water evaporates, bringing the relative humidity measurements at the outlet down to inlet levels gradually. FDC responds accordingly, gradually reducing speed towards the lower limit.

Phase 3.   Curing (2.5 Hours and beyond): The relative humidity levels at the inlet and outlet equalize in the absence of bleed water. FDC flat lines at the lower operational limit, which notifies the operator that bleeding has ceased and application of curing compounds can begin.

This idealized performance of the FDC from the ARCHERS was not achieved in the vast majority of concrete testing situations during development and application. FDC was initially erratic and considerable research effort was expended in data filtering and smoothing. A performance level that clearly distinguished the three phases of humidity (Fig. 5-33) was not achieved. The use of relative humidity provided a measure of the capacity of the air to take on water vapor but not an indication of the amount of water vapor in the air between the two measurement points.

Empirical relationships between temperature, relative humidity, and vapor pressure exist that allow for a determination of the partial pressure water vapor if volumes and absolute pressure are assumed constant. This can be used to measure the amount of water vapor in a section of air by using the August-Roche-Magnus approximation to relate saturation vapor pressure and temperature, and convert to a partial pressure of water vapor through the definition of relative humidity (Perry & Green, 2008).

By converting the chamber and outlet relative humidity values into vapor pressures, the change in the water profile evaporating from the concrete surface over time could be quantified more directly than using the relative humidities. Table 5-55 shows the difference in the vapor pressures between the outlet and chamber hygrometers from ARCHERS.

Table 5-54: Average Difference in Vapor Pressure between Outlet and Chamber Hygrometers from ARCHERS Results, per Mix Type at Application times

Average Calculated Difference in Vapor Pressure at Application Times (hPa)			Number of Tests
Mix Type	30 Min	2 Hr	4 Hr	
1	2.41	0.44	0.13	3
2	0.63	0.50	-0.33	2
3	0.57	0.32	-0.03	2
4	1.80	1.04	0.67	4
5	3.62	2.49	1.30	5
6	2.67	2.10	1.04	4

The results in Table 5-55 show that the difference in vapor pressure between the hygrometers was larger at the start of the test and that the difference decreased over time as the bleed water evaporated. This method offers the advantage of directly detecting the changing amount of water in the air as an indicator for the presence of bleed water on the concrete surface over time, rather than the indirect way of using the FDC. Zero or negative values of this difference indicate that no additional water has evaporated, implying that bleeding has finished and that the curing compound application can begin.

5.6.2 Analysis of ARCHERS Results

ARCHERS data from two separate concrete batches were analyzed using the relative humidity-vapor pressure relationships to further demonstrate the operational potential of the ARCHERS. The shape of the FDC response curves from these batches were both poor relative to the idealized behavior, and very different from one another. In addition, the results from ARCHERS operation on Mix 5-C in Section 5.6.2.2 were chosen due to the high ambient laboratory relative humidity. ARCHERS recorded the relative humidity and temperature, and adjusted the FDC every five seconds. The purpose of analyzing these trends was to evaluate the operational capacity of the ARCHERS.

5.6.2.1 Mix 4-B

Mix 4-B was chosen for further analysis due to the shape of the FDC curve and the scaling patterns that decreased with an increase in application time. The ambient relative humidity at the time of concrete specimen manufacture for Mix 4-B was approximately 27%. This corresponded to an ambient vapor pressure of 7.2 hPa. Figure 5-38 is a plot of the Fan Duty Cycle over time for Mix 4-B, with readings registered every 5 seconds. The initial 2.5 hours of the test behaved as predicted, but the following 3.5 hours did not. The sharp peaks and valleys of the FDC curve indicate that the

factors that controlled system response were poorly tuned and overly sensitive to the input readings, resulting in an FDC curve with an erratic ringing performance. Instituting a longer running average than 5 seconds or more optimal PID gains would improve this performance. Figure 5-39 displays the partial pressure of water vapor in the ARB and at the outlet of the contactor relative to the ambient conditions. Perturbations of the vapor pressure within the ARB were observed in the outlet vapor pressure, indicating that the ARB's contribution to the FDC was not insignificant, and better instrument design would attempt to limit this. Figure 5-40 shows the difference in vapor pressures between the outlet and the ARB. This provides the best estimate for the behavior of the bleed water on the surface of the concrete over time. It is evident that over time that the vapor pressure of the outlet air is increased for the first hour of the test, as a direct result of the concrete bleed water. Over time, a nearly linear decrease in the outlet vapor pressure approaches that of the chamber, an indicator that the bleeding process has ceased. Despite performance from the FDC graph that would indicate that bleeding may not have ceased due to the FDC oscillations, the vapor pressure data reveals a more accurate picture of the behavior immediately above the concrete surface.
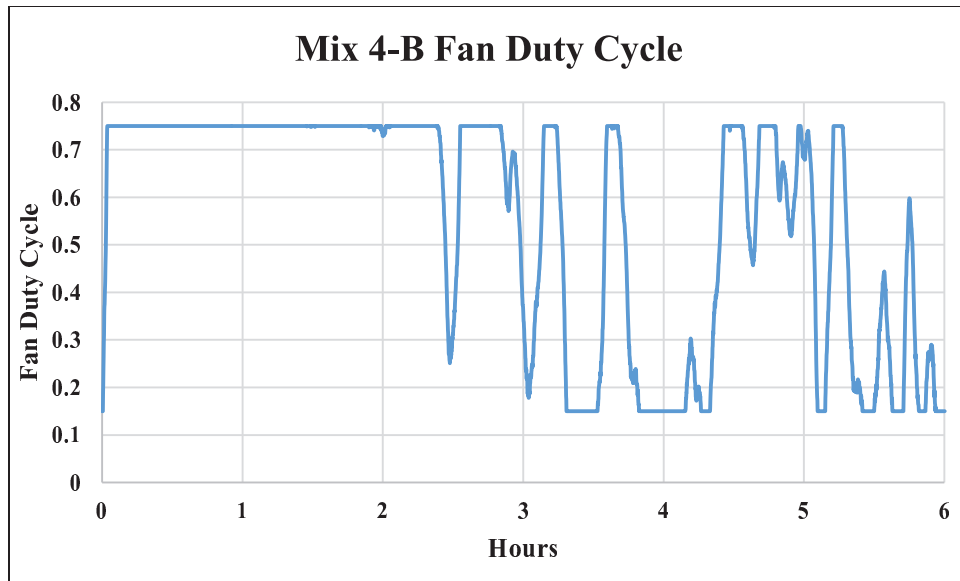
Figure 5-38: Fan Duty Cycle of ARCHERS Operation on Mix 4-B
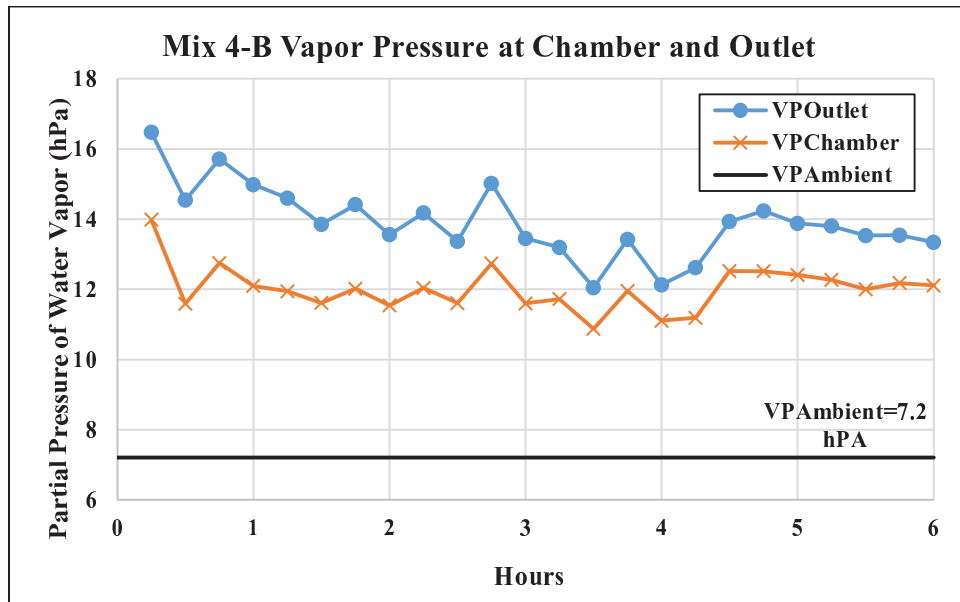
Figure 5-39: Vapor Pressure Measurements from ARCHERS on Mix 4-B
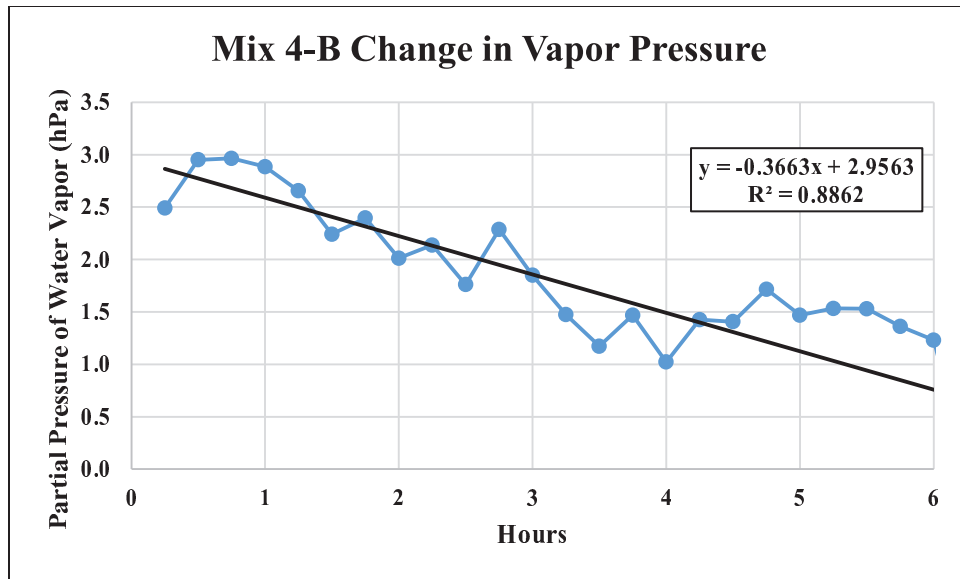
Figure 5-40: Vapor Pressure Reading Difference for Mix 4-B

5.6.2.2 *Mix 5-C: High Relative Humidity Condition*

ARCHERS' performance was heavily impacted by the ambient conditions during concrete placement. When ARCHERS was used on the mixes that were poured on days with high relative humidity, the FDC curve that was generated was similar to that in Figure 5-41, which indicated that bleeding had ended early due to low perceived differences in relative humidity. Mix 5-C was poured when the relative humidity in the testing laboratory was 68%, as compared to Mix 4-B which was at 27% for approximately the same temperature. Figure 5-39 showed that the ambient vapor pressure at the time Mix 4-B was poured indicates a relatively low 7.2 hPa; an amount that neither the chamber nor the outlet appeared to be approaching. The opposite is shown in Figure 5-42 where the ambient vapor pressure was much higher at 18.1 hPa; a level that both the chamber and the outlet appeared to approach.
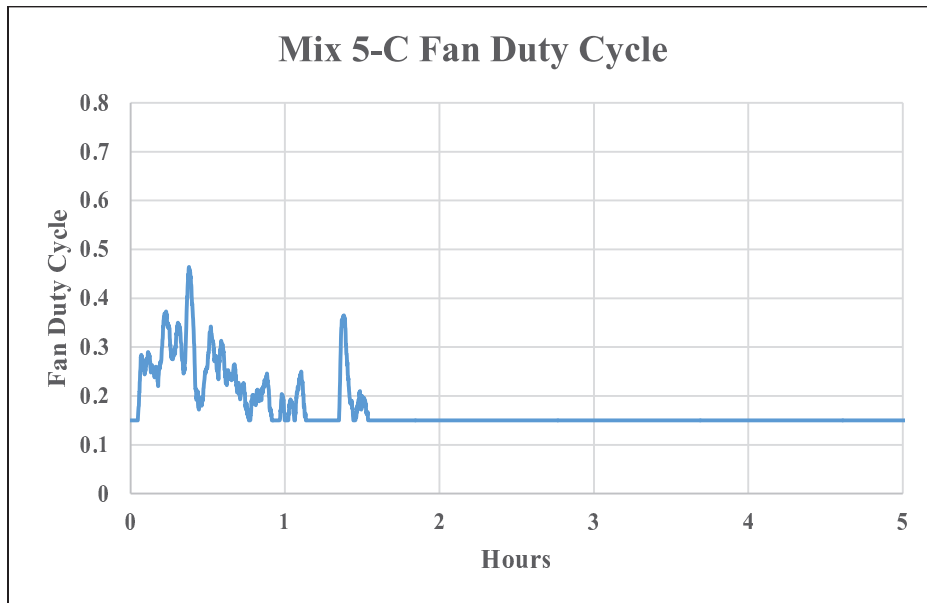
Figure 5-41: Fan Duty Cycle of ARCHERS Operation on Mix 5-C
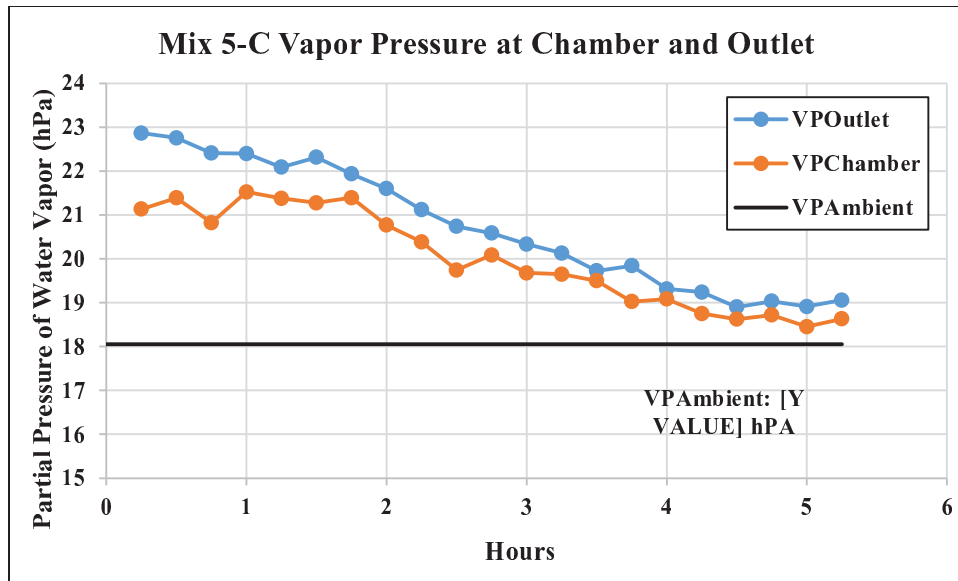
Figure 5-42: Vapor Pressure Measurements from ARCHERS on Mix 5-C

Mix 5-C displayed heavy scaling damage across all three application times, in excess of 1000 g/m². While the levels of vapor pressure at the outlet versus the ambient vapor pressure do not confirm directly the presence of bleed water due to the high relative humidity of the laboratory, the results from the ARCHERS do confirm that ambient vapor pressures for this mix, and other mixes with similar ambient relative humidities, were much higher than most other mixes. This indeed may have resulted in higher levels of scaling at the 2 and 4 Hour application times as a result of lower evaporation rates of bleed water.

5.6.3   Recommendations for ARCHERS Improvement

It is evident that the current design of the ARCHERS is not ready for laboratory or field use to indicate the dissipation of bleed water. However, the device did demonstrate a level of potential for evaluating the air environment immediately above a concrete surface that would warrant future investment towards operational improvement. The following list of recommendations identifies several strategies to build upon the current design of the ARCHERS that could result in improved operational capability:

- Changes to instrument calibration, sampling rate, system tuning, data smoothing, and other modifications would improve the performance of the ARCHERS. Digital hygrometer variability proved to be an on-going challenge to the development of a precise device. One hygrometer typically read relative humidity values 0-8% higher than the other hygrometer when subject to the same air.

- Evaluating the difference in vapor pressure as the process variable and the performance indicator would provide a better assessment of the bleed water presence on the concrete surface.

Chapter 6 **Conclusions**

This laboratory research was directed toward identifying the best time to apply curing compounds to minimize scaling of pavement concrete. Curing environments for concrete pavements should typically include:

- A barrier to limit evaporation of near surface moisture ,
- A reflective component, to limit heat buildup and evaporation of bleed water due to solar radiation.

Curing compounds are designed to provide these attributes and yet observed scaling resistance in concretes with these coatings are highly variable (Afrani and Rogers 1994). Scaling test results of concretes with curing compounds applied can additionally be complicated by the presence of supplementary cementitious materials like Ground Granulated Blast Furnace Slag and Fly Ash.

Application instructions on most curing compounds indicate that application to the concrete surface should occur following final finishing efforts and after bleeding has ceased. Despite this common instruction, factors such as ambient relative humidity, ambient temperature, wind speed, solar radiation, total mix water, admixtures, and supplementary cementitious materials among others can make it difficult to know when the bleeding process has finished. Elevated evaporation rates can lead to application times prior to cessation of bleeding, and depressed rates of evaporation due to high humidity can delay application times due to the continued presence of water despite bleeding cessation. This project was conducted in a temperature controlled, enclosed laboratory to limit other environmental concerns that affect the presence of bleed water on the surface. This allowed the study to focus on evaluating one primary variable of the bleeding process: time. Because bleeding is time dependent, three curing compound application times were chosen to indirectly evaluate the stages of bleeding: 30 Minutes after placement when bleeding was still in its nascent stage, 2 Hours to indicate

a stage where bleeding could be concluding but water might or might not be present on the surface, and finally 4 Hours where it was assumed that the bleeding process would typically be concluded had completed with no water remaining on the surface. Additionally, the 2 Hour specimens were used to compare outcomes with those from Phase I.

This research shows that when using curing compounds, scaling resistance was influenced by two factors: the choice of compound used and application time matched to the particular mix characteristics and environmental conditions of the concrete. The application time reflect the time needed for bleeding to diminish and the surface to be free of water. Although efforts were expended to develop a device based on thermohygrometric technology to predict this state, that research was not successful within the scope and time constraints of this project. The device development showed promise but would require significantly more focused development to render a practically usable device. As a result, the research was conducted with the curing compound fixed application times of 30 Minutes, 2 Hours and 4 Hours.

Based on statistical analysis of the scaling amounts associated with each condition, the shortest successful curing compound applications times were determined for each mix and these are summarized in Table 6-1. In addition, the two curing compounds and application times with the greatest degree of scaling resistance were identified as preferred and alternative options. In general, the researchers observed that later application times within the 30 minute to 4 hour window resulted in higher levels of durability.

Table 6-1: Decision Matrix for Curing Compound Application Time Providing the Highest Degree of De-Icer Scaling Resistance

Aggregate	Cement Type	Compounds Used				Preferred Compound/Time	Alternative Compound/Time
		Linseed Oil	Wax	PAMS	Acrylic		
Limestone	OPC	30 Minutes	30 Minutes	30 Minutes	30 Minutes	PAMS at 30 Minutes	Acrylic at 30 Minutes
	30% Slag	2 Hours	4 Hours	30 Minutes	30 Minutes	Wax at 4 Hours	Linseed Oil at 2 Hours
	30% Fly Ash	4 Hours	4 Hours	30 Minutes	4 Hours	Acrylic at 4 Hours	PAMS at 30 Minutes
Gravel	OPC	2 Hours	30 Minutes	30 Minutes	4 Hours	Acrylic at 4 Hours	Wax at 30 Minutes
	30% Slag	2 Hours	30 Minutes	30 Minutes	4 Hours	Acrylic at 4 Hours	Linseed Oil at 2 Hours
	30% Fly Ash	4 Hours	30 Minutes	30 Minutes	4 Hours	Acrylic at 4 Hours	PAMS at 30 Minutes

The main findings of this research are summarized as follows:

1. Curing Condition Behavior:

   a) Wet Room: Wet Room cured specimens scaled the least out of all the specimen groups by a large margin. The Curing compounds did not come close to providing the same degree of scaling resistance as achieved with wet room curing. Concrete with the Crushed Limestone coarse aggregate typically scaled less than Gravel mixtures. Mixtures containing slag and fly ash scaled significantly more than than blends containing only OPC.

   b) Linseed Oil: Application time had a strong effect on scaling resistance for Linseed Oil-coated concretes. After two hours, all mixes saw large reductions scaling damage. However, scaling resistance between the application times of 2 and 4 Hours typically did not increase by significant amounts suggesting that there was little or no benefit in waiting beyond 2 hours to apply the Linseed Oil compound.

c) Wax: 4 Hours application times provided the greatest scaling resistance with the Wax curing compound. The largest amounts of scaling damage for concretes coated with Wax were observed at 2 Hours, with only marginal differences in scaling damage between the 30 Minute and 4 Hour specimens. This suggests that at 30 minutes although bleeding was in process, it had not resulted in levels of water at the surface that inhibited the sealing characteristics of the compound. But at 2 hours sufficient moisture on the concrete surface disrupted the protective characteristics of the compound. Very poor scaling was observed in the two Wax-coated concretes poured at high relative humidities. All specimens coated with Wax lost over 90% of the distinctive white pigmentation prior to the 30 cycle mark; oftentimes within the first 10 cycles.

d) PAMS: The PAMS curing compound scaling results did not appear to be sensitive to application time. PAMS displayed marginal differences in scaling resistance between all three application times. While PAMS typically scaled less than other formulations on concrete with OPC or fly ash, higher scaling amounts across all three application times on slag concretes indicate a possible incompatibility.

e) Acrylic: Acrylic sealing compound specimens displayed large reductions in the amount of scaling damage with an increase in the application time. When applied at 4 Hours, Acrylic offered significantly higher scaling resistance compared to other formulations.

2. Scaling resistance of concrete is dependent upon the mix materials, especially if supplementary cementitious materials such as slag or fly ash are included in the mix design. The mixes containing gravel typically experienced higher amounts of scaling than the crushed limestone mixes. Slag-containing mixes typically suffered a higher total amount of

scaling than OPC concrete. The Slag results from this study point to this being most likely increased amounts of bleeding, and delayed timing of bleeding.

3. Increased levels of relative humidity at the time of specimen casting appeared to extend the time and amount of water that remains on a fresh concrete surface and thus should result in delays to the application of curing compounds.

4. Initial analysis of the results from the curing compound investigation conducted by Kropp et al. in Phase I and the results from this study did not establish a high degree of repeatability when comparing scaling magnitude alone. We believe these differences can be largely traced to the amount of water on the surface of the concrete at the time of curing compound application. Bleeding and surface water may be stochastic processes within some range of parameters but certainly will be influenced by prevailing relative humidity. It appears that in many instances the 2 hour application time did not reliable offer sufficient time to ensure that the surface was relatively free of accumulated bleed water. A review of the scaling results from both phases of the study that ranked the scaling performance of each curing condition relative to the other on individual mix types showed some levels of repeatability.

5. This study provides indirect evidence as to why higher scaling amounts were sometimes observed at earlier application times;

      The curing compounds used within this study were described by manufacturer's specification for having a specific gravity very close to that of water when in emulsion form. Therefore, it is likely that the curing compounds blended with, or were disassociated by the similarly dense bleed water; preventing a well adhered, full protective membrane from forming. Gaps in the membrane may have allowed for both premature evaporation of water needed for adequate curing and areas more susceptible to the ingress of deicing chemicals, leading to reduced scaling resistance.

Furthermore a poor bond to the concrete surface means that initial freeze-thaw action (even in the absence of traffic) will quickly remove the curing compound. We believe the variation in scaling observed amongst different curing compounds and times of compound application for the same mix can be attributed to the amount of bleed water on the surface at the time of application and the susceptibility of the membrane of that curing compound be disrupted by the bleed water.

Chapter 7 **Recommendations**

It is clear from the results of this study that a single optimal time to apply curing compounds to concrete pavements does not exist. Concrete mixes, curing compound formulations and environmental conditions at laboratory and field sites, all influence when it will be most effective from a scaling resistance perspective to apply the curing compound. In this study, time was a pseudo-indicator of the presence of bleed water on the surface and it turned out that even in the relative controlled conditions of the laboratory, it was not a particularly good indicator.   Two recommendations arise from these observations. First continue to seek and/or develop a method that assesses the surface conditions of the concrete, particular the presence of bleed water. The ARCHERS development demonstrated the complexity of this problem but also showed a promising direction. We believe the difficulties around repeatability in measuring scaling resistance are more tied to the repeatability of the bleed water condition of the concrete surface rather than minor variations in laboratory procedures.   If one makes a few logical assumptions about surface bleed water, much of the apparent lack of repeatability can be readily explained. Alternatively and in the meantime, a more empirical approach could be taken that expands upon the decision matrix in Table 6-1. To be most useful, such a decision matrix would need to expand in the dimensions of variables considered to include environmental factors in addition to times, mix characteristics and curing compound types.

Chapter 8 **References**

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Chapter 9 **Standards**

ASTM C39 (2012). *Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens.*

ASTM C117 (2013). *Standard Test Method for Materials Finer than 75-μm (No. 200) Sieve in Mineral Aggregates by Washing.*

ASTM C127 (2012). *Standard Test Method for Density, Relative Density (Specific Gravity), and Absorption of Coarse Aggregate.*

ASTM C128 (2012). *Standard Test Method for Density, Relative Density (Specific Gravity), and Absorption of Fine Aggregate.*

ASTM C136 (2006). *Standard Test Method for Sieve Analysis of Fine and Coarse Aggregates.*

ASTM C143 (2012). *Standard Test Method for Slump of Hydraulic-Cement Concrete.*

ASTM C156 (2011). *Standard Test Method for Water Loss [from a Mortar Specimen] Through Liquid Membrane-Forming Curing Compounds for Concrete.*

ASTM C192 (2013). *Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory.*

ASTM C231 (2010). *Standard Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method.*

ASTM C232 (2013). *Standard Test Method for Bleeding of Concrete.*

ASTM C309 (2011). *Standard Specification for Liquid Membrane-Forming Compounds for Curing Concrete.*

ASTM C617 (2012). *Standard Practice for Capping Cylindrical Concrete Specimens.*

ASTM C672 (2012). *Standard Test Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals.*

ASTM C1315 (2011). *Standard Specification for Liquid Membrane-Forming Compounds Having Special Properties for Curing and Sealing Concrete.*

Appendix A Specimen Matrix

Table A-1: Specimen Manufacture Mix Matrix

Materials			MFCC Application Times and Specimen Group Labels			Number of Samples
Mix/Compound Label	Components	Curing Compound	30 Minutes (α)	2 Hours (β)	4 Hours (ø)	
1-A	Limestone/OPC	Wet Room	1-A-α	-	-	3
1-B		Linseed	1-B-α	1-B-β	1-B-φ	9
1-C		Wax	1-C-α	1-C-β	1-C-φ	9
1-D		PAMS	1-D-α	1-D-β	1-D-φ	9
1-E		Acrylic	1-E-α	-	1-E-φ	6
2-A	Limestone/Slag	Wet Room	2-A-α	-	-	3
2-B		Linseed	2-B-α	2-B-β	2-B-φ	9
2-C		Wax	2-C-α	2-C-β	2-C-φ	9
2-D		PAMS	2-D-α	2-D-β	2-D-φ	9
2-E		Acrylic	2-E-α	-	2-E-φ	6
3-A	Limestone/Fly Ash	Wet Room	3-A-α	-	-	3
3-B		Linseed	3-B-α	-	3-B-φ	6
3-C		Wax	3-C-α	-	3-C-φ	6
3-D		PAMS	3-D-α	-	3-D-φ	6
3-E		Acrylic	3-E-α	-	3-E-φ	6
4-A	Gravel/OPC	Wet Room	4-A-α	-	-	3
4-B		Linseed	4-B-α	4-B-β	4-B-φ	9
4-C		Wax	4-C-α	4-C-β	4-C-φ	9
4-D		PAMS	4-D-α	4-D-β	4-D-φ	9
4-E		Acrylic	4-E-α	-	4-E-φ	6
5-A	Gravel/Slag	Wet Room	5-A-α	-	-	3
5-B		Linseed	5-B-α	5-B-β	5-B-φ	9
5-C		Wax	5-C-α	5-C-β	5-C-φ	9
5-D		PAMS	5-D-α	5-D-β	5-D-φ	9
5-E		Acrylic	5-E-α	-	5-E-φ	6
6-A	Gravel/Fly Ash	Wet Room	6-A-α	-	-	3
6-B		Linseed	6-B-α	-	6-B-φ	6
6-C		Wax	6-C-α	-	6-C-φ	6
6-D		PAMS	6-D-α	-	6-D-φ	6
6-E		Acrylic	6-E-α	-	6-E-φ	6

Appendix B Fresh Concrete Mix Data

Table B-1: Properties of Fresh Concrete Mixes

Mix ID	w/c Ratio	Batch Size (ft^3)	AEA (mL)	WRA (mL)	Slump (in)	Unit Weight (lb/ft^3)	Air Content (%)	Ambeint Relative Humidity (%)	ASTM C672 Specimens Made	Compresssive Strength (Psi)
1-A	0.40	2.7	9	200	1.25	145.4	6.5%	27%	9	6370
1-B	0.40	2.7	12	160	1.25	145.6	6.4%	21%	9	6583
1-C	0.40	2.7	8	200	1	146.2	6.0%	25%	9	6420
1-D	0.40	2.7	12	160	1.25	144.6	6.9%	21%	9	5427
1-E	Made in Same Batch as 1-A									
2-A	0.40	2.7	14	175	1	147.0	5.4%	24%	9	6487
2-B	0.40	2.7	15	175	1.5	143.8	6.9%	23%	9	6653
2-C	0.40	2.7	14	175	1.75	143.0	7.1%	31%	9	5373
2-D	0.40	2.7	13	175	1.25	146.7	6.1%	31%	9	5927
2-E	Made in Same Batch as 2-A									
3-A	0.40	2.7	7	75	2.5	145.8	6.4%	32%	9	5630
3-B	0.40	2.0	6	50	2	147.7	5.4%	29%	6	6017
3-C	Made in Same Batch as 3-A									
3-D	0.40	2.0	6	60	2.5	144.0	6.7%	27%	6	5333
3-E	0.40	2.0	6	60	3	146.6	6.1%	36%	6	6273
4-A	0.40	2.7	18	140	1.25	144.6	6.1%	35%	3	5287
4-B	0.41	2.7	12	175	2	140.9	5.8%	27%	9	5517
4-C	0.41	2.7	12	200	2.25	142.2	7.5%	25%	9	5353
4-D	0.41	2.7	12	175	1	142.6	6.4%	28%	9	5583
4-E	0.40	2.7	18	140	1.375	144.7	6.2%	76%	9	5000
5-A	0.40	2.7	15	175	1	144.2	6.3%	64%	3	5923
5-B	0.40	2.7	13	175	1	145.0	5.2%	33%	9	6223
5-C	0.40	2.7	15	160	1.25	145.0	6.0%	68%	9	6103
5-D	0.40	2.7	14	200	2	145.0	6.4%	26%	9	6317
5-E	0.40	2.7	13	160	1.5	145.2	5.3%	75%	6	6080
6-A	0.40	2.7	8	80	2.75	144.7	6.4%	83%	9	5603
6-B	0.40	2.0	6	55	2.5	145.4	5.6%	31%	6	6363
6-C	0.40	2.0	8	70	2.25	144.9	5.8%	65%	6	5700
6-D	0.40	2.0	6	65	3	145.4	5.9%	30%	6	5603
6-E	Made in Same Batch as 6-A									

Appendix C ASTM C672 Scaling Mass Loss Data

Table C-1: Scaling Mass Loss Data for Mix Type 1 Specimens

Mix Type 1-Scaling Damage Accumulation per Cycle (g/m^2)														
Compound	Application Time	Label	Cycles												
			5	10	15	20	25	30	35	40	45	50	55	60
Wet Room	----	1-A	0.0	0.7	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.6	10.5	17.1
Linseed Oil	30 Minutes	1-B-α	5.9	13.2	19.1	30.3	52.0	149.3	225.0	284.8	363.8	412.4	449.3	479.5
	2 Hours	1-B-β	11.2	18.4	32.9	47.4	87.5	135.5	207.2	282.2	332.2	371.7	408.5	446.0
	4 Hours	1-B-φ	11.2	13.2	14.5	15.1	18.4	30.3	56.6	105.9	173.0	251.9	301.9	344.0
Wax	30 Minutes	1-C-α	26.3	39.5	44.7	48.0	51.3	53.9	55.3	57.2	59.2	112.5	246.0	301.9
	2 Hours	1-C-β	19.7	52.0	58.5	69.7	88.1	105.2	120.4	129.6	145.4	259.2	491.4	621.6
	4 Hours	1-C-φ	3.3	22.4	34.9	44.1	51.3	56.6	60.5	63.8	65.1	88.8	173.7	259.8
PAMS	30 Minutes	1-D-α	17.1	30.9	36.8	41.4	44.1	46.0	47.4	49.3	50.7	53.3	53.9	53.9
	2 Hours	1-D-β	34.2	63.8	74.3	79.6	81.6	83.5	84.9	85.5	86.8	86.8	86.8	87.5
	4 Hours	1-D-φ	36.8	90.8	106.6	115.1	119.7	121.7	123.0	125.0	127.6	130.2	131.6	133.5
Acrylic	30 Minutes	1-E-α	68.4	73.7	80.3	87.5	93.4	100.6	108.5	115.1	121.0	129.6	136.2	142.1
	4 Hours	1-E-φ	16.4	29.6	34.9	41.4	44.7	48.0	52.6	56.6	63.1	69.1	73.7	77.0

Table C-2: Scaling Mass Loss Data for Mix Type 2 Specimens

Mix Type 2-Scaling Damage Accumulation per Cycle (g/m^2)														
Compound	Application Time	Label	Cycles												
			5	10	15	20	25	30	35	40	45	50	55	60
Wet Room	----	2-A	24.3	61.2	89.5	101.3	110.5	121.0	128.3	136.8	153.9	162.5	171.0	180.9
Linseed Oil	30 Minutes	2-B-α	9.2	63.1	121.7	248.0	330.9	440.7	561.8	722.3	832.1	913.7	975.5	1032.1
	2 Hours	2-B-β	3.9	13.8	26.3	52.0	82.2	128.9	190.8	314.4	408.5	482.2	540.1	587.4
	4 Hours	2-B-φ	5.3	15.8	25.7	46.7	67.8	104.6	209.2	357.2	445.3	501.2	549.3	586.8
Wax	30 Minutes	2-C-α	274.3	308.5	336.1	346.0	352.6	363.1	376.3	392.0	415.1	450.6	499.9	546.0
	2 Hours	2-C-β	297.3	342.7	367.1	379.6	392.7	416.4	465.1	517.0	581.5	658.5	744.0	818.3
	4 Hours	2-C-φ	121.7	151.3	158.5	164.5	168.4	178.3	188.8	201.3	219.0	245.4	275.0	305.2
PAMS	30 Minutes	2-D-α	487.4	641.4	687.4	717.7	727.5	740.0	754.5	774.9	793.3	820.3	842.0	867.0
	2 Hours	2-D-β	720.9	970.9	1014.3	1036.7	1049.2	1063.0	1091.3	1122.9	1158.4	1188.0	1218.2	1238.0
	4 Hours	2-D-φ	508.5	691.3	751.2	771.6	784.8	790.0	794.6	803.2	813.7	824.2	836.7	851.2
Acrylic	30 Minutes	2-E-α	127.0	371.7	451.3	478.2	507.2	528.9	542.0	554.5	567.7	584.1	597.9	617.0
	4 Hours	2-E-φ	67.1	235.5	287.5	303.2	330.9	352.6	367.7	382.8	397.3	413.1	431.5	454.5

Table C-3: Scaling Mass Loss Data for Mix Type 3 Specimens

Mix Type 3-Scaling Damage Accumulation per Cycle (g/m²)														
Compound	Application Time	Label	Cycles											
			5	10	15	20	25	30	35	40	45	50	55	60
Wet Room	----	3-A	5.3	30.3	65.8	75.6	80.3	81.6	84.2	86.8	86.8	88.1	88.1	94.7
Linseed Oil	30 Minutes	3-B-α	28.3	695.3	1003.8	1082.1	1116.9	1151.2	1195.2	1241.3	1294.6	1355.7	1418.2	1479.4
	4 Hours	3-B-φ	4.6	15.8	46.0	108.5	180.9	268.4	338.1	398.0	462.4	534.1	599.9	642.7
Wax	30 Minutes	3-C-α	76.3	119.7	134.8	150.0	183.5	225.6	283.5	371.0	452.6	607.2	718.3	847.9
	4 Hours	3-C-φ	55.3	118.4	128.9	138.1	148.0	157.9	167.7	197.3	236.2	296.0	338.1	394.0
PAMS	30 Minutes	3-D-α	43.4	67.1	81.6	93.4	104.6	120.4	137.5	160.5	189.4	232.9	271.7	309.2
	4 Hours	3-D-φ	69.1	88.1	95.4	102.6	109.2	113.8	122.4	128.3	141.4	169.7	195.4	211.2
Acrylic	30 Minutes	3-E-α	151.3	547.9	609.1	634.1	652.5	671.0	682.8	697.9	715.7	727.5	730.2	734.1
	4 Hours	3-E-φ	41.4	85.5	93.4	102.0	105.9	109.9	113.1	119.1	127.6	130.2	130.9	130.9

Table C-4: Scaling Mass Loss Data for Mix Type 4 Specimens

Mix Type 4-Scaling Damage Accumulation per Cycle (g/m²)														
Compound	Application Time	Label	Cycles											
			5	10	15	20	25	30	35	40	45	50	55	60
Wet Room	----	4-A	3.9	8.6	15.1	23.0	30.3	37.5	41.4	44.7	48.0	51.3	53.9	57.2
Linseed Oil	30 Minutes	4-B-α	8.6	22.4	76.3	251.9	332.2	381.5	421.7	461.8	498.6	527.6	563.7	609.1
	2 Hours	4-B-β	1.3	5.3	10.5	25.7	42.1	69.7	106.6	148.0	196.7	228.3	259.2	300.6
	4 Hours	4-B-φ	2.0	3.9	5.3	9.9	13.8	23.7	34.9	53.3	88.1	129.6	169.1	217.1
Wax	30 Minutes	4-C-α	46.0	102.6	118.4	122.4	127.0	139.5	153.3	159.2	168.4	171.0	176.9	178.9
	2 Hours	4-C-β	93.4	196.7	230.9	244.7	251.9	260.5	269.7	283.5	293.4	300.0	306.5	317.7
	4 Hours	4-C-φ	11.2	32.9	56.6	68.4	81.6	91.4	103.3	114.5	126.3	134.8	140.1	147.3
PAMS	30 Minutes	4-D-α	197.3	406.5	460.5	485.5	509.8	531.5	543.3	560.4	570.3	580.8	591.4	599.9
	2 Hours	4-D-β	118.4	290.7	348.0	370.3	409.2	444.0	465.7	503.2	525.6	547.9	569.0	591.4
	4 Hours	4-D-φ	32.9	118.4	155.2	175.0	203.3	246.7	281.5	330.9	355.9	382.2	409.8	426.9
Acrylic	30 Minutes	4-E-α	128.9	203.9	225.0	241.4	262.5	277.6	286.8	298.0	322.3	338.1	372.3	386.1
	4 Hours	4-E-φ	7.9	17.8	23.0	27.6	32.9	35.5	37.5	39.5	42.8	44.1	46.0	48.0

Table C-5: Scaling Mass Loss Data for Mix Type 5 Specimens

Mix Type 5-Scaling Damage Accumulation per Cycle (g/m^2)														
Compound	Application Time	Label	Cycles											
			5	10	15	20	25	30	35	40	45	50	55	60
Wet Room	----	5-A	19.1	44.1	57.9	66.4	77.0	90.1	97.4	111.2	125.0	140.8	173.7	202.6
Linseed Oil	30 Minutes	5-B-α	155.9	322.3	493.4	720.3	925.5	1001.8	1099.8	1162.3	1197.9	1234.7	1255.1	1295.2
	2 Hours	5-B-β	98.0	128.9	160.5	194.1	243.4	276.9	317.1	358.5	381.5	409.2	442.7	480.2
	4 Hours	5-B-φ	6.6	14.5	26.3	59.9	111.2	168.4	234.8	279.6	314.4	355.9	415.7	478.9
Wax	30 Minutes	5-C-α	436.8	755.2	814.4	845.9	865.0	881.5	900.5	910.4	922.9	959.1	1007.8	1082.1
	2 Hours	5-C-β	668.3	928.2	991.3	1030.1	1059.7	1078.8	1105.1	1132.1	1150.5	1203.1	1285.3	1427.4
	4 Hours	5-C-φ	483.5	814.4	915.0	949.2	987.4	1003.8	1035.4	1070.2	1092.6	1161.0	1262.3	1331.4
PAMS	30 Minutes	5-D-α	150.6	403.2	502.6	536.8	561.1	578.9	597.9	612.4	622.3	630.2	646.0	655.8
	2 Hours	5-D-β	238.1	756.5	878.2	919.6	940.7	959.1	974.9	991.3	1003.8	1020.2	1054.5	1074.8
	4 Hours	5-D-φ	194.1	565.1	646.6	668.3	680.2	691.3	709.1	720.3	732.1	745.3	770.9	791.3
Acrylic	30 Minutes	5-E-α	172.3	416.4	465.7	500.6	524.3	536.1	563.7	605.8	642.7	663.7	713.7	808.4
	4 Hours	5-E-φ	110.5	276.9	323.0	353.2	372.3	380.9	393.4	405.2	413.1	427.6	436.1	444.0

Table C-6: Scaling Mass Loss Data for Mix Type 6 Specimens

Mix Type 6-Scaling Damage Accumulation per Cycle (g/m^2)														
Compound	Application Time	Label	Cycles											
			5	10	15	20	25	30	35	40	45	50	55	60
Wet Room	----	6-A	5.3	42.1	67.1	83.5	109.9	125.6	134.8	150.6	180.9	213.8	280.9	312.5
Linseed Oil	30 Minutes	6-B-α	13.2	291.4	531.5	622.9	761.7	854.5	949.2	1014.3	1112.3	1208.4	1345.2	1461.6
	4 Hours	6-B-φ	1.3	2.6	25.0	33.5	111.2	176.3	234.2	313.1	490.7	576.9	672.3	726.2
Wax	30 Minutes	6-C-α	50.0	229.6	291.4	314.4	350.0	380.2	391.4	402.6	422.3	465.7	560.4	783.4
	4 Hours	6-C-φ	37.5	298.0	353.2	380.2	449.9	506.5	517.0	532.2	544.0	567.7	609.8	801.2
PAMS	30 Minutes	6-D-α	38.8	77.6	94.1	128.9	148.0	164.5	177.6	183.5	190.8	217.1	236.8	257.2
	4 Hours	6-D-φ	49.3	137.5	159.8	201.9	221.7	229.6	235.5	236.8	238.1	244.7	247.3	251.3
Acrylic	30 Minutes	6-E-α	39.5	1015.6	1234.0	1321.5	1416.9	1582.0	1629.4	1693.2	1776.7	1788.6	1905.6	1933.9
	4 Hours	6-E-φ	13.2	63.1	94.1	119.7	153.9	173.0	178.3	184.8	196.0	200.6	209.2	213.8

Appendix D Mix Designs

Table D-1: Mix Type 1 Designs

Mix Type 1-Limestone/OPC					
Mix	Volume (ft^3)	Cement (lbs)	Fine Aggregate (lbs)	Coarse Aggregate (lbs)	Water (lbs)
Butter	0.66	13.81	31.22	46.83	6.29
Batch 1: 9 Specimens	2.70	56.50	127.71	191.56	25.73

Table D-2: Mix Type 2 Designs

Mix Type 2-Limestone/Slag						
Mix	Volume (ft^3)	Cement (lbs)	Slag Cement (lbs)	Fine Aggregate (lbs)	Coarse Aggregate (lbs)	Water (lbs)
Butter	0.66	9.66	4.16	31.02	46.53	6.28
Batch 1: 9 Specimens	2.70	39.50	17.00	126.89	190.33	25.71

Table D-3: Mix Type 3 Designs

Mix Type 3-Limestone/Fly Ash						
Mix	Volume (ft^3)	Cement (lbs)	Fly Ash (lbs)	Fine Aggregate (lbs)	Coarse Aggregate (lbs)	Water (lbs)
Butter	0.66	9.66	4.16	31.02	46.23	6.28
Batch 1: 6 Specimens	2.00	29.26	12.59	93.38	140.08	19.03
Batch 2: 9 Specimens	2.7	39.5	17	126.07	189.1	25.69

Table D-4: Mix Type 4 Designs

Mix	Volume (ft³)	Cement (lbs)	Fine Aggregate (lbs)	Coarse Aggregate (lbs)	Water (lbs)
Mix Type 4-Gravel/OPC					
Butter	0.66	13.81	30.85	46.27	6.02
Batch 1: 9 Specimens	2.70	56.50	126.19	189.28	24.62
Batch 1 with 0.41 w/c	2.7	56.50	126.19	189.28	25.44

Table D-5: Mix Type 5 Designs

Mix	Volume (ft³)	Cement (lbs)	Slag Cement (lbs)	Fine Aggregate (lbs)	Coarse Aggregate (lbs)	Water (lbs)
Mix Type 5-Gravel/Slag						
Butter	0.66	9.66	4.16	30.65	45.97	6.01
Batch 1: 9 Specimens	2.70	39.50	17.00	125.38	188.07	24.60

**Table D-6: Mix Type 6 Designs**

Mix	Volume (ft³)	Cement (lbs)	Fly Ash (lbs)	Fine Aggregate (lbs)	Coarse Aggregate (lbs)	Water (lbs)
Mix Type 6-Gravel/Fly Ash						
Butter	0.66	9.66	4.16	30.45	45.68	6.01
Batch 1: 6 Specimens	2.00	29.26	12.59	92.27	138.41	18.22
Batch 2: 9 Specimens	2.7	39.5	17	124.57	186.85	24.59

Appendix E Normality Assumption Derivation by Residuals

Normality can be assumed within samples of a larger population if the residuals of sample means are determined to be approximately normally distributed. A residual is the difference between a collected data point and the sample mean. The collected data points were the scaled damage amounts from individual ASTM C672 specimens, and the sample means were the average mass loss amounts after 60 cycles for a mix type-curing compound-application time set. These sample means were the average of three replicate specimens, so each sample mean had three residuals. A total of 198 residuals were calculated, corresponding to the 198 specimens within this study.

Figure E-1 is the histogram of the frequencies of the residuals using a bin size of approximately 5 g/m^2. The plot of the normal distribution scaled to the frequency data is superimposed upon the histogram. Because the residual frequency data appears to fit normal distribution, it is safe to assume that the data used to calculate the samples mean are normally distributed.
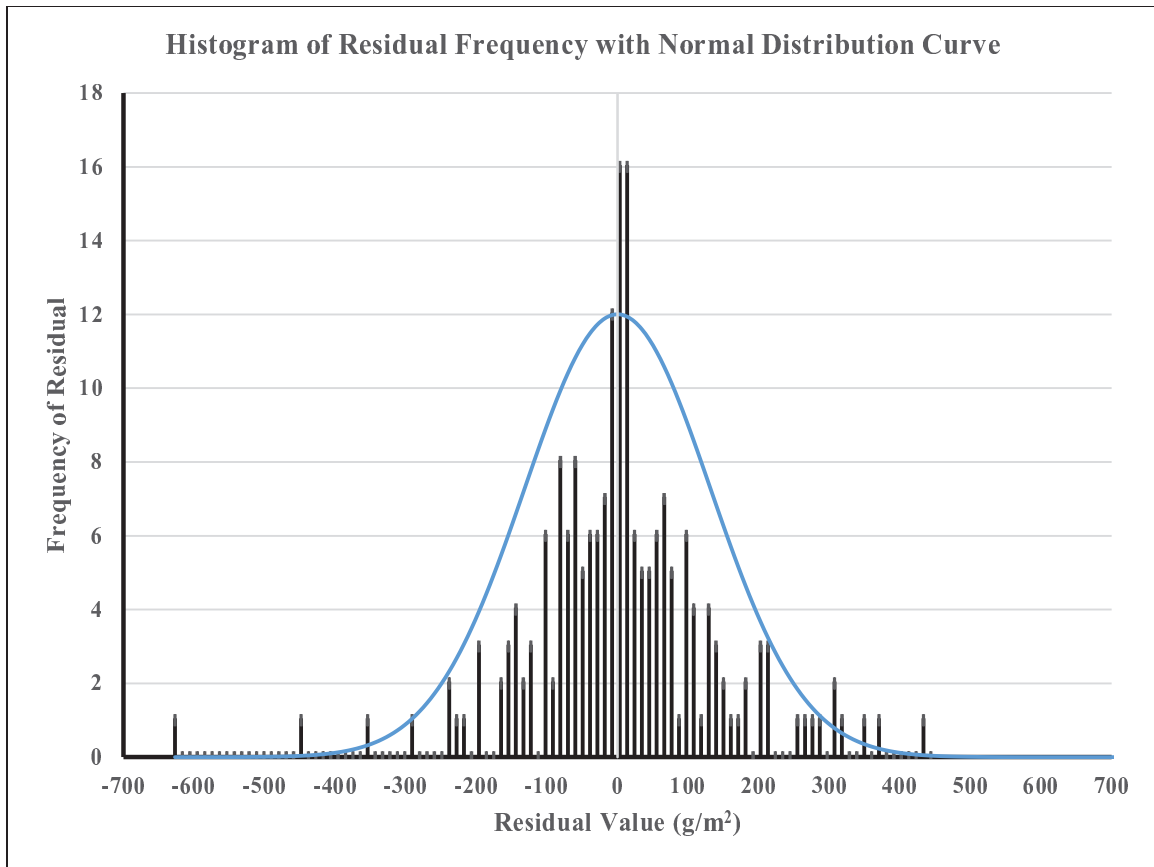
Figure E-1: Histogram of Residual Frequency with Superimposed Normal Distribution

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