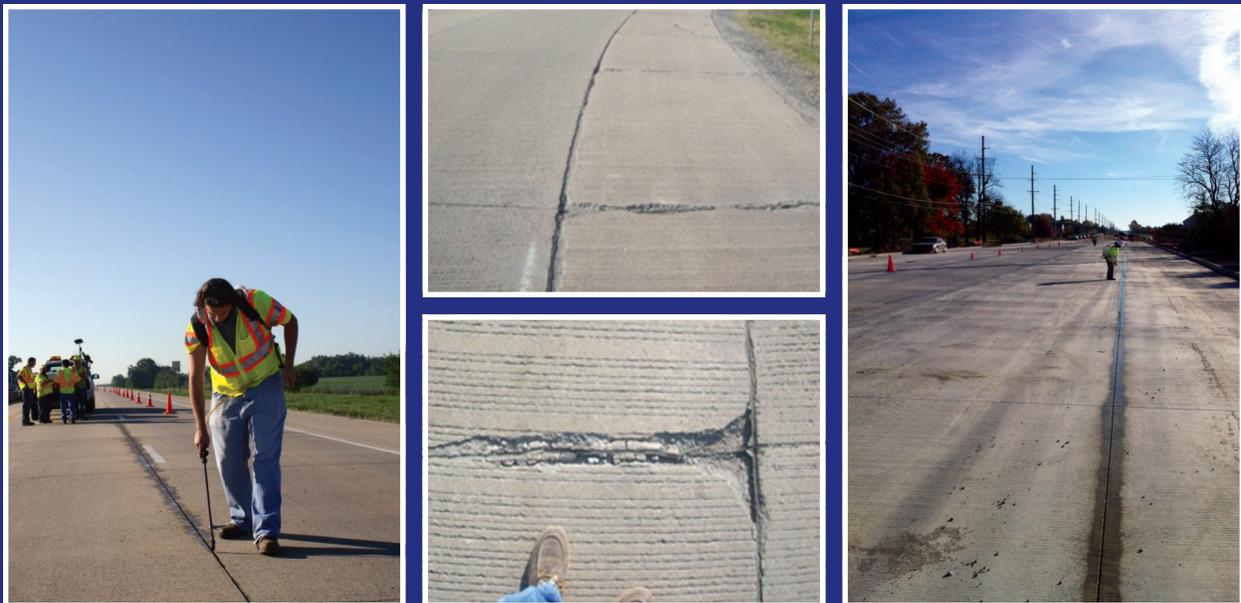


JOINT TRANSPORTATION RESEARCH PROGRAM

INDIANA DEPARTMENT OF TRANSPORTATION
AND PURDUE UNIVERSITY



Evaluation of Sealers and Waterproofers for Extending the Life Cycle of Concrete



**Andrew Wiese, Yaghoob Farnam, Wesley Jones,
Paul Imbrock, Bernie Tao, W. Jason Weiss**

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16. Abstract <p>Concrete pavements represent a large portion of the transportation infrastructure. While the vast majority of concrete pavements provide excellent long-term performance, a portion of these pavements have recently shown premature joint deterioration. Substantial interest has developed in understanding why premature joint deterioration is being observed in jointed portland cement concrete pavements (PCCP). While some have attributed this damage to insufficient air void systems, poor mixture design, or chemical reaction between the salt and the paste, it is the hypothesis of this work that a component of this damage can be attributed to fluid absorption at the joints and chemical reactions between the salt and chemistry of the matrix. This paper discusses the role of soy methyl ester - polystyrene blends (SME-PS) as a potential method to extend the service life of concrete pavements by limiting the ingress of salt solutions. The report discusses field application of the SME-PS blends for field investigation in Lafayette and Fishers. Low temperature-differential scanning calorimetry (LT-DSC) techniques identified noticeable differences between plain mortar samples and mortar treated with SME-PS. The report also discusses the development of a test to assess chloride solution ingress during temperature cycling. The aim of this work is to provide background on some aspects that can lead to joint deterioration and provide early documentation showing that sealers may help to reduce the impact of deicers on joint damage, thereby extending the life of the concrete pavement. It should be noted that these sites as well as others are still ongoing and should be monitored for long term performance. Application procedure for SME-PS should follow manufacturer's recommendation.</p>			
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EXECUTIVE SUMMARY

EVALUATION OF SEALERS AND WATERPROOFERS FOR EXTENDING THE LIFE CYCLE OF CONCRETE

Introduction

Concrete pavements represent a large portion of the transportation infrastructure. While the vast majority of concrete pavements provide excellent long-term performance, a portion of these pavements have recently shown premature joint deterioration. Substantial interest has developed in understanding why premature joint deterioration is being observed in jointed portland cement concrete pavements (PCCP). While some have attributed this damage to insufficient air void systems, poor mixture design, or chemical reaction between the salt and the paste, it is the hypothesis of this work that a component of this damage can be attributed to fluid absorption at the joints and chemical reactions between the salt and chemistry of the matrix. This discusses the role of soy methyl esters-polystyrene blends (SME-PS) as a potential method to extend the service life of concrete pavements by limiting the ingress of salt solutions.

The report discusses field application of the SME-PS blends for field investigation in Lafayette and Fishers. Low temperature-differential scanning calorimetry (LT-DSC) techniques identified noticeable differences between plain mortar samples and mortar treated with SME-PS. The report also discusses the development of a test to assess chloride solution ingress during temperature cycling.

The aim of this work is to provide background on some aspects that can lead to joint deterioration and to provide early documentation showing that sealers may help to reduce the impact of deicers on joint damage, thereby extending the life of the concrete pavement. It should be noted that these sites as well as others are still ongoing and should be monitored for long-term performance.

Major Findings

- SME-PS has been found to reduce salt ingress and freeze-thaw damage.
- Work was done using DSC techniques to determine the amount of reaction occurring during temperature change; e.g., freeze-thaw and chemical reaction events.
- Field applications of SME-PS show that salts do not penetrate as far when compared with unsealed joints verified by titration tests and visual chloride penetration tests.
- Joint details are an important part of pavement durability, and saw-cut depths can affect the service life of pavements. Within this study is an observed joint that was sawn to only one inch, and cracking occurring away from the joint and has been growing ever since.

Suggestions for Implementation

- It is recommended that the design of longitudinal and transverse joints in portland cement concrete pavements in Indiana be reconsidered. When considering only the durability of the concrete joint, it appears that removing the conventional sealant and sealing the concrete with a penetrating sealer like SME-PS may extend the life of the joint. The redesign of the joint, however, also considers the potential for incompressible materials to enter the joint. Further, the redesign of the joint requires that the subgrade be able to function properly with additional fluid that may come.
- Quality control when sawing joints is important. The saw cut should be approximately one third the depth of the pavement in order to ensure cracking occurs within the saw cut if stresses are high enough in the pavement to generate a crack.

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1. INTRODUCTION AND OVERVIEW

Concrete pavements represent a large portion of the transportation infrastructure. While many of these pavements provide excellent long-term performance, a portion of pavements in the Midwestern United States have recently shown premature joint deterioration (Arribas-Colón, Radliński, Olek, & Whiting, 2012; Leech et al., 2008; Harris, Farnam, Spragg, Imbrock, & Weiss, 2015; Jain, Janusz, Olek, & Józwiak-Niedźwiedzka, 2011; Jitendra, Olek, Janusz, & Józwiak-Niedźwiedzka, 2012; Weiss, 2014; Olek, Radliński, & del Mar Arribas, 2007; Olek & Weiss, 2014; Radliński et al., 2008, Radliński, Olek, Zhang, & Peterson, 2010; Rangaraju, Sompura, & Olek, 2006; Sutter, Van Dam, Peterson, & Johnston, 2006; Weiss, Abraham, & Nantung, 2007). This joint deterioration is problematic because it compromises the performance and potential service life of an otherwise healthy pavement.

Figure 1.1 provides photographs showing the typical pavement joint damage that is being discussed in this report (Golias et al., 2012; Li, Pour-Ghaz, Castro, & Weiss, 2012). This type of damage is frequently seen as one of two types of damage. The first type of damage consists of small cracks that progressively spall off; these cracks are parallel to the saw-cut face at fractions of an inch from the saw-cut face. The second type of damage consists of the development of cracking parallel to the joint or spalling; the damage begins with a hollowed out region at the bottom of the saw cut,

resulting in a crack to the surface of pavement that is typically four to six inches from the edge of the joint. Unfortunately, damage is not frequently observed at the surface of the pavement until a significant amount of damage has occurred inside the joint. During field inspections it has been observed that where the joints are damaged, the sealant is damaged and the joint contains a significant amount of standing water (or fluid), as shown in Figure 1.2. The pavements in Figure 1.2 were examined in 2005. The material in the pavement exhibited a complete destruction of the matrix while the aggregate remained intact. It should be noted that it had not rained for nearly two weeks; however, fluid was standing in the joint. It is important to note that while this damage is more likely to occur in pavements with poor air entrainment, damage occurs in both poorly air entrained and properly air entrained concrete.

Figure 1.3 illustrates a proposed series of mechanisms that appear to be partially responsible for this damage (Weiss & Nantung, 2007). In Figure 1.3, a 1-D construction joint is illustrated that is typical of that used by the Indiana DOT. The joint is created by saw-cutting the pavement shortly after placement with a single cut that is approximately 1/3 the depth of the pavement. The intention of this saw-cut is to provide stress relief, which can allow cracks to form at these locations. A previous report provided details on how to best place and time these saw-cuts to minimize the potential for random cracking (Raoufi, Weiss, &



Figure 1.1 Field observation showing damage in pavement joints.



Figure 1.2 Field observation showing damage in pavement joints containing water.

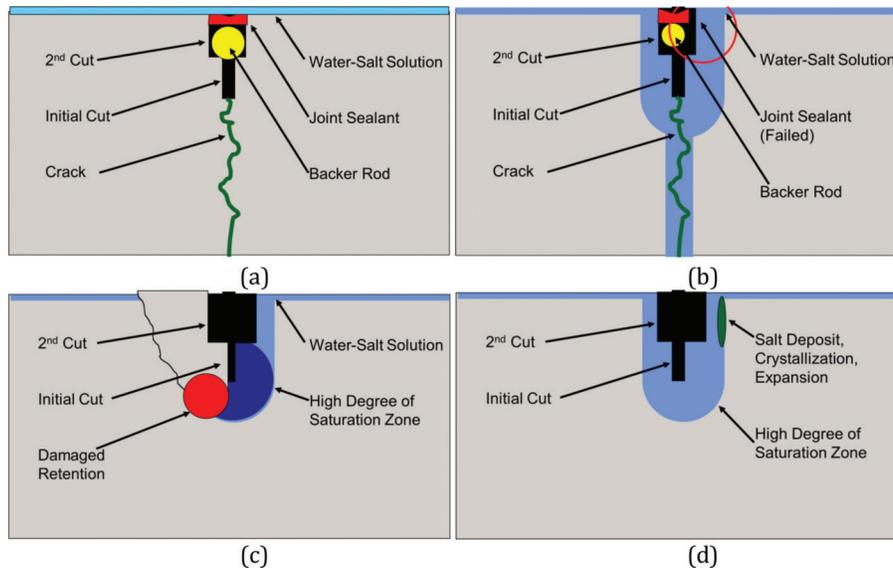


Figure 1.3 Potential degradation hypothesis: (a) an ideal joint, (b) a joint with damaged joint sealant that is cracked, (c) a joint with damaged joint sealant that is uncracked, and (d) a joint illustrating potential salt crystallization locations.

Nantung, 2008). The pavement would then be expected to crack at the joint (though this crack does not always occur, as explained in the following paragraphs). After several days (to weeks) the saw cut is cut again to widen the cut, which provides a notch at the top of the joint for the placement of backer rod and a joint sealant. This second cut also allows minor raveling to be removed and attempts to provide a more uniform joint width since some of the opening has already occurred. The joints are then cleaned, dried, and a non-absorptive backer rod is placed in the joint and a joint sealant is then placed. If ideal, this constructed sealed joint will keep water and deicing salt out of joints; however, invariably the sealer pulls away from one of the walls or cracks as shown in Figure 1.3 (b). It is proposed that this allows water and deicing salt to enter the joint and be absorbed by the faces of the saw cut. While the fluid enters the hollow section created by the saw-cuts, the fluid likely drains through the crack at the base of the saw cut into the open graded base. This will increase the degree of saturation along the walls of the saw-cut and along the walls of the crack. This changes dramatically, however, it will not occur if the crack does not form below the saw-cut (as shown in Figure 1.3 (c)). Without a crack, the fluid pools in the joint is absorbed by the concrete and becomes increasingly concentrated (with respect to deicing salt) over time. This can develop a zone with a high degree of saturation. This high level of saturation can result in freezing damage and/or in chemical reaction in some systems (e.g., the formation of Friedel salt that can infill air voids (Arribas-Colón et al., 2012; Bu & Weiss, 2014; Farnam, Bentz, Hampton, & Weiss, 2014; Farnam, Bentz, Sakulich, Flynn, & Weiss, 2014; Farnam, Wiese, Bentz, Davis, & Weiss, 2015; Jain et al., 2011; Jitendra et al., 2012; Olek et al., 2007; Radliński et al., 2008; Radliński et al., 2010), the production of oxychloride (Farnam,

Dick, et al., 2015; Farnam, Wiese, et al., 2015; Hooton, Mesisc, & Beal, 1993), or the replacement of calcium in calcium silicate hydrate (Sutter et al., 2006)). While this mechanism would explain the ‘soft zone’ frequently observed at the base of the saw-cut, an alternative damage pattern is observed with the side of the saw-cut flaking off in layers that are fractions of an inch thick. At the current time it seems that this may be due in part to freezing and thawing or due to salt crystallization pressure (Scherer, 1999); however, additional work is needed to examine this type of failure.

The previous section discussed how fluid can sit in the saw-cut if the joint sealant fails and the concrete pavement has not cracked. It has been hypothesized (Weiss et al., 2007) that the joints may be less likely to crack if pavements gain strength more rapidly, if the joints become more closely spaced, or if the pavements are thicker. In all these cases, the factors that drive the cracking are reduced. The width of the cracks that form are controlled by the presence and amount of steel tie bars in the longitudinal joint that may hold sections together while the width of the transverse joints is dependent on the number of joints that crack and the alignment of the dowel bars. It should also be noted that if the joints are sawed late micro-cracking can occur at the base of the saw-cut and this micro-cracking (if present) would enable faster water absorption, resulting in a higher degree of saturation (Yang, Weiss, & Olek, 2006).

It is important in this discussion to remember that the fluid in the joint is not water. It is a solution that contains deicing salts since deicing salts are used to depress the freezing point of the water on the concrete surface. Although the addition of deicing salts on the surface of concrete elements are effective for melting the ice and increasing the safety of infrastructure, it may also be partially responsible for the issues that develop

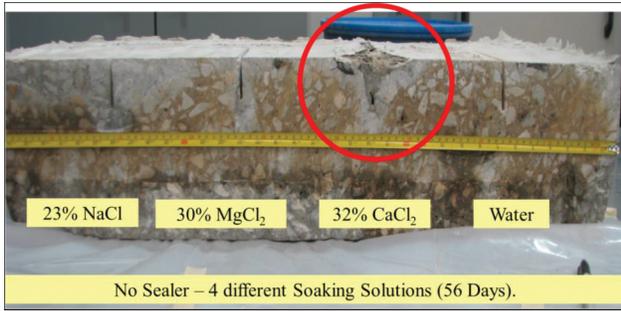


Figure 1.4 Damage development due to drying and wetting with deicing salts.

at the joints in the pavement. When concrete that contains a deicing salt is exposed to freezing and thawing, a variety of complex damage mechanisms occur (1–8). Ice can form inside the concrete pore structure (1); osmotic pressure can develop due to partial freezing of solutions in capillaries (3); and large-scale migration of unfrozen water from small pores to large cavities filled by frozen water (5) and crystallization of salt can occur.

For example, Figure 1.4 illustrates the behavior of salts under only drying and wetting and it can be noticed that even in cases where freezing may not be occurring, damage can develop at the joint. At the current time this appears to be consistent with salt crystallization during drying and wetting however this mechanism has not been studied in great detail for pavement joints.

It has been proposed (Coates, Mohtar, Tao, & Weiss, 2009; Golias, 2010) that one way to reduce the increase in the degree of saturation or the ingress of chloride ions is to seal the faces of the cut concrete surface to help repel the fluid that may sit in the joints. While the concept sounds positive, there are some results that suggest that sealers may not be effective to reduce damage (Arribas-Colón et al., 2012). As a result, field trials were performed in this work to examine if sealers have potential to improve joint durability. If sealers are a positive approach, guidelines would be needed for their use as well as test methods that may evaluate these materials and these guidelines are provided.

This report was originally developed as a part of a pooled fund study conducted by the National Concrete Consortium and SPR-3523 for concrete pavements. This work includes data from that report for completeness however it also includes preliminary work from field measurement and observations of the first three years of sealer performance.

2. USE OF SOY METHYL ESTER–POLYSTYRENE (SME-PS) AS A CONCRETE SEALANT

2.1 Background on SME

The previous sections of this report have shown that the ingress of water and salt contribute to the deterioration of the joints in concrete pavements. It was discussed

that there is potential to use a concrete ‘sealer’ to block the pores and to reduce water and salt ingress. One such ‘concrete sealer’ that has been investigated is soy methyl ester–polystyrene (SME-PS).

Plant oils such as soybean oil are made up of long-chain fatty acids containing 14 to 20 carbon atoms. The oil can be converted into a methyl group through transesterification. The transesterification process converts methanol and the plant oil (triacylglycerides) into methyl esters. Methyl esters are biodegradable and nontoxic. In addition the SME is a good solvent for synthetic polymers such as polystyrene (PS) and polyvinyl chloride (PVC) (Coates et al., 2009).

The following section provides a review of results from tests where SME-PS was applied to concrete and the influence of the SME-PS was assessed. Golias (2010) compared SME-PS with two silane sealants—a solvent-based alkyalkoxysilane sealer (SBS) and a water-based alkyalkoxysilane penetrating sealer (WBS). The SBS was a solution of silane dissolved in isopropanol that had greater than 50% active ingredients. The WBS was an emulsion of silane in water that contained 40% silane (Golias, 2010). This comparison was made since these are sealants that are frequently used.

2.2 SME as a Concrete Sealant

2.2.1 Fresh Properties

SME-PS was topically applied and admixed with fresh concrete to test its ability to retard the evaporation of water. SME-PS was topically applied to fresh concrete with 0.018 g/cm^2 (5% polystyrene) and samples were maintained at $23 \pm 1^\circ\text{C}$, $50 \pm 4\%$ RH and their masses were continuously monitored. As shown in Figure 2.1, the samples topically treated with SME-PS had a reduction in mass loss by roughly one-third at about 20 hours.

2.2.2 Hardened Properties

The compressive strength was determined for plain samples as well as for samples admixed with a dosage of 1.25% SME-PS and different polystyrene contents. Figure 2.2 shows the compressive strength versus polystyrene content at each of the 3 ages tested.

The largest reduction in compressive strength was 18% for the SME-PS with 5% polystyrene at 28 days. The reductions in compressive strength are noticeable however this can frequently be overcome in design.

Drying shrinkage tests were also performed on plain samples as well as samples with SME-PS with 1% and 20% polystyrene. It was observed that upon drying, the treated samples had less mass loss. The sample with 1% PS had a mass loss roughly 10% less than the plain sample. It was also observed that there was no substantial effect (either positive or negative) on shrinkage (Coates et al., 2009).

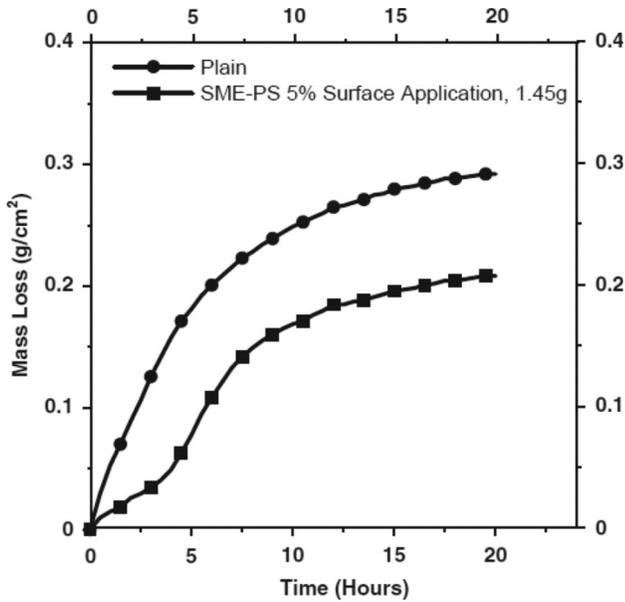


Figure 2.1 Evaporation of water over time in plain sample and sample topically treated with SME-PS.

For fluid transport, samples admixed with SME-PS showed significant improvements with regards to absorption and ion diffusion. Both the SME and the polystyrene fill the pores and thus reduce water absorption. The treated samples dosed with 1.25% SME-PS had a 91% reduction in absorption after 8 days while the treated samples dosed with 2.5% SME-PS showed a reduction by 94% over the same time period. With regards to chloride ion diffusion, the depth of the treated samples dosed with 2.5% SME-PS was, on average, reduced by 68%. Thus, the use of SME-PS as an admixture can slow chloride ingress (Coates et al., 2009).

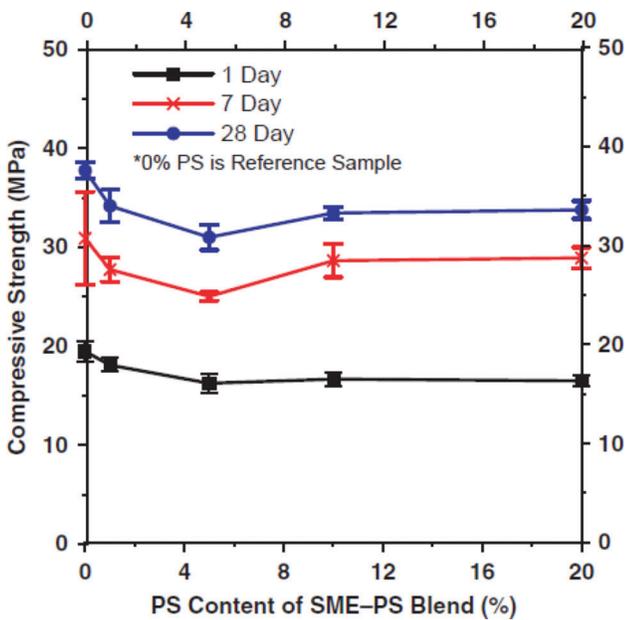


Figure 2.2 Compressive strength versus polystyrene content.

With regards to the topical application of SME-PS, it was observed that the SME-PS was able to penetrate the samples fairly quickly. Over a 5 hour period, the SME-PS with 5% PS was able to penetrate roughly 4 mm. For SME-PS application rates between 0.02 g/cm^2 and 0.036 g/cm^2 , the SME-PS was able to reduce water absorption by 85% to 93% after 12 days (Coates et al., 2009).

The ability of SME-PS to penetrate concrete depends on a few different factors. One such factor is the moisture content of the concrete. Samples were conditioned at $23 \pm 1^\circ\text{C}$ at different relative humidities for 18 months. Roughly 20 g of SME-PS were then ponded on the samples for 48 hours. Figure 2.3 shows the water and SME-PS absorption after conditioning at different relative humidities. It clearly shows that the SME-PS is highly dependent on the moisture content (Golias, 2010) however it is worth noting that a greater penetration depth is observed in a dry concrete. This implies that when the SME-PS is being investigated for use as a sealer it is better to dry the surface pores.

Another such factor that affects the penetration of SME-PS is the length of the polystyrene chain. In this experiment, 5 g of SME-PS was ponded for 48 hours. One sample had pure SME while other samples had 5% polystyrene, each with different molecular weights (or chain lengths). As shown by the images in Figure 2.4, the penetration depth of the SME-PS decreases as the chain length of the polystyrene increases. Waste polystyrene is preferred due to its availability, its ability to maintain the SME at the surface however the chain is not so long that it greatly reduces absorption.

Time is the final factor that affects the SME-PS penetrability. Samples were submerged in SME-PS.

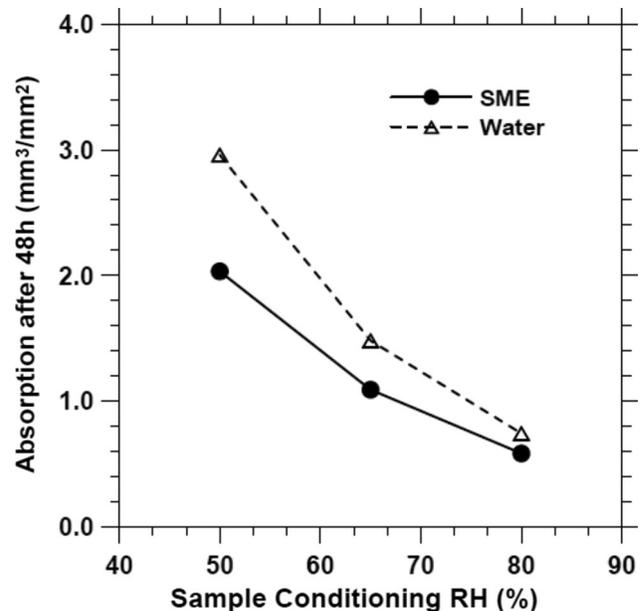


Figure 2.3 SME-PS and water absorption after 48 hours versus conditioning.

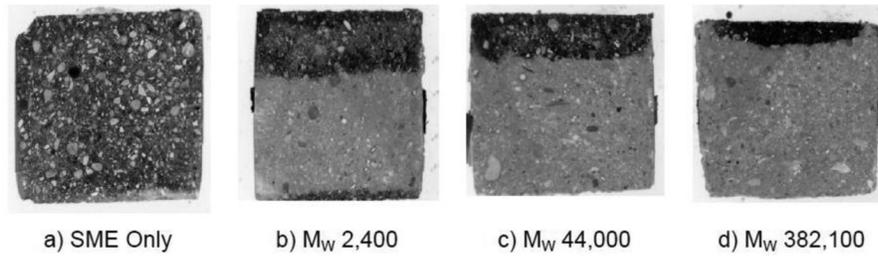


Figure 2.4 Penetration depth of SME-PS in black with varying PS chain lengths.

At different time intervals, the samples were removed from the SME-PS and cut open to determine the penetration depth. Figure 2.5 shows the plot of the penetration depth over time (Golias, 2010). Again, the main factor is that the longer the SME-PS is allowed to absorb the more SME is absorbed and the deeper it can penetrate.

For water absorption, two doses of SME-PS were used. For Dose 1, samples were submerged in SME-PS for 6 hours while for Dose 2, samples were submerged in SME-PS for 24 hours. SBS and WBS were also tested. As is evident in Figure 2.6, the sealants significantly reduced the amount of absorbed water.

The SME-PS was able to reduce the water absorbed by 88% at 7 days while the silane sealants saw a 95% reduction in absorption. Over time, the effectiveness of the sealants decreases. The SME-PS saw a 77% reduction after 28 days while the silane saw a 90% reduction during the same time period (Golias, 2010).

In cold weather, SME-PS does not behave the same way as water, which is considered to be a single-phase liquid. SME-PS is made up of different fatty acid methyl esters (FAME), each of which has a different temperature at which phase changes occur. The first

critical temperature is the cloud point whereby FAMES start to lose solubility and form conglomerations of waxy crystals, which will come out of the solution. For pure SME, the cloud point is typically 0°C while for SME-PS with 5 and 10 percent PS, the cloud point was 5°C. The pour point is the next critical temperature, which is at a lower temperature than the cloud point. At the pour point, the SME-PS turns into a gel-like substance, which typically occurs at -4°C. This is advantageous for concrete because it will clog the pores and help reduce fluid ingress (Golias, 2010). This has two implications as it relates to the use of the SME-PS as a sealer. First, the SME-PS is best applied in warmer weather and should not be applied when the temperature is approaching the cloud point. Second, the SME-PS is not expected to solidify and crack when it would freeze in practice. Rather the SME-PS would gel, then re-liquefy as the temperature heated allowing some redistribution and healing of the sealer.

A series of samples with different sealers was tested during freezing and thawing using ASTM C 666. After 75 cycles of freezing and thawing, both untreated concrete and concrete treated with SBS began to spall. After 100 cycles, both samples had the same amount of damage. It was observed that samples treated with a small dose of SME-PS did not show damage as shown by Figure 2.7 and Figure 2.8. Even when the SME-PS treated samples were damaged, the damage was still considerably less than the damage observed in the untreated samples (Golias, 2010).

When applied to concrete, silanes form a protective membrane. During freezing, silane sealants are vulnerable to thermal contraction, which can cause the sealant to crack. On the other hand, SME-PS absorbs into the pores and remains flexible after freezing.

While all the sealers were effective at reducing water absorption at 23°C, the same behavior was not observed at cooler temperatures. After 7 freeze-thaw cycles, SBS was the most susceptible to freeze-thaw. During freezing, SBS absorbed 85% of the volume of water that the untreated concrete had absorbed. SME-PS, on the other hand, was the least susceptible to changes caused by freezing—only losing roughly 20% of its effectiveness (Golias, 2010). As a result of this observation it appears that a testing procedure is needed to evaluate how the sealer performs when they are both exposed to water and undergoing thermal cycling.

The ability of SME-PS and the silanes to reduce the penetration of different chlorides was also examined.

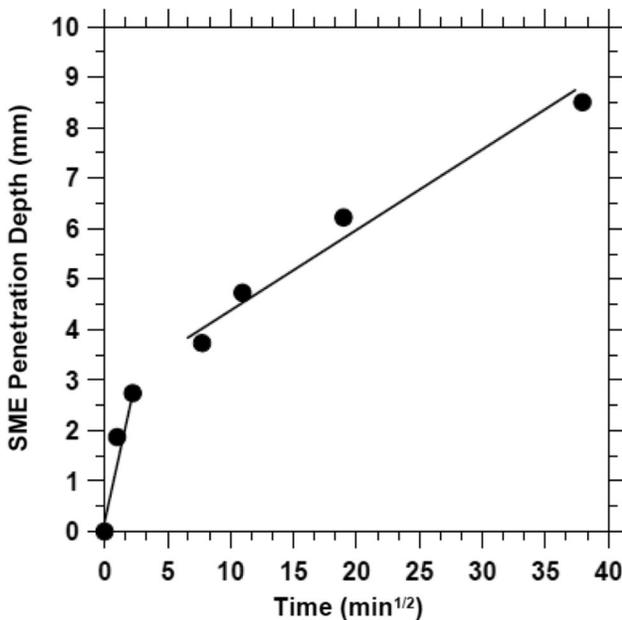


Figure 2.5 SME-PS penetration over time.

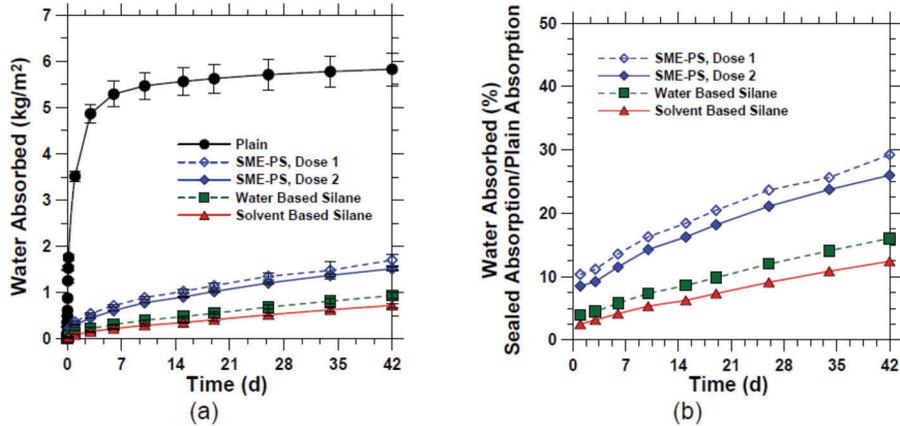


Figure 2.6 Water absorption where (a) is the amount absorbed and (b) is the percent reduction in absorption.

The solutions used were 32% calcium chloride (CaCl₂), 23% sodium chloride (NaCl), and 30% magnesium chloride (MgCl₂). After 21 and 42 days of ponding, samples were cut open and sprayed with 0.1M silver nitrate (AgNO₃). Photographs were taken of the samples and imported to determine the penetration depths. Figure 2.9 shows the penetration depths for the different salt solutions where (a) is sodium chloride, (b) is magnesium chloride, and (c) is calcium chloride.

The SBS was the most effective in limiting salt ingress—it was able to eliminate the chlorides for MgCl₂ and CaCl₂ and was able to reduce the depth by 80% at 42 days for NaCl. The effectiveness of the SME-PS was related to its dosage rate. The reduction in depth for the larger dosage of SME-PS was roughly 10% greater than the reduction from the smaller dosage rate (Golias, 2010).

With regards to Alkali-Silica Reaction (ASR), Golias (2010) observed that the sealants were able to postpone the expansion. For dosages of 5 minutes, 2 hours, and 24 hours, the SME-PS was able to postpone the expansion by 1 day, 1 week, and 3 weeks respectively. The SBS sealant was able to postpone the expansion by roughly 6 weeks. It was further observed that despite the fact that doses 1 and 2 of SME-PS were able to delay the expansion, at later ages, those 2 samples actually had more expansion than the plain sample. This is probably due to the conditioning method prescribed in ASTM C1260. After the 24 hours of

saturation in water, the plain samples absorbed 5 times more water than the treated samples. Thus, the plain samples, during testing, absorbed less of the highly alkaline sodium hydroxide. The sealed samples, on the other hand, absorbed more fluid during the test, resulting in an exaggerated expansion (Golias, 2010).

2.3 SME-PS Application Instructions and Concerns

2.3.1 Introduction

A significant interest has been shown by various groups in using SME-PS as a treatment for concrete pavement joints. The goal of this treatment is to reduce premature pavement joint deterioration by minimizing the ingress of fluids such as aqueous salt solutions that result from roadway deicing salts. In order to accomplish this goal, SME-PS must be absorbed into the pores of the pavement joint at the time of application. This will minimize the fluid from within the joint from entering the surrounding concrete. Some care must be taken in the application and preparation process in order to ensure the sealant absorbs into the desired areas. The application process is quite simple, but each step of the process is necessary in order to ensure the availability of SME in all the pores in the locations that require sealing.

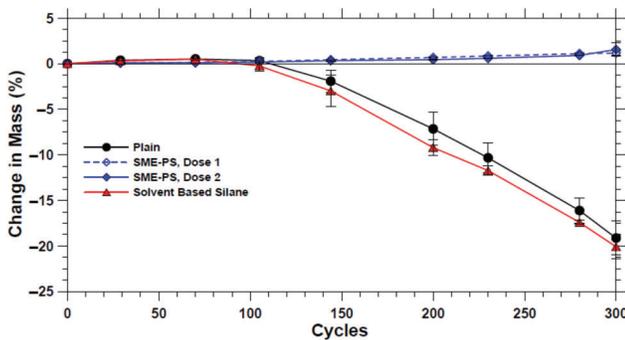


Figure 2.7 Mass change due to freezing and thawing.

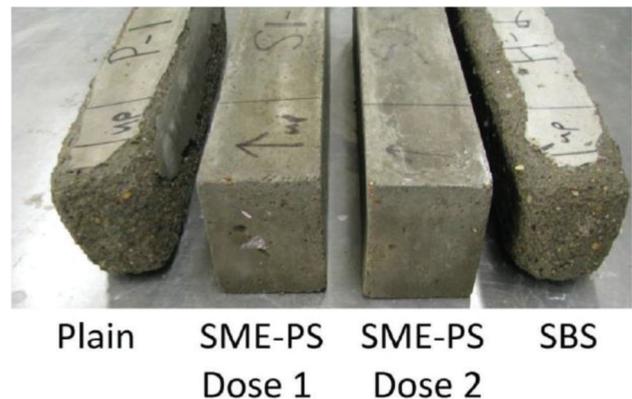


Figure 2.8 Samples after 280 cycles of freeze-thaw.

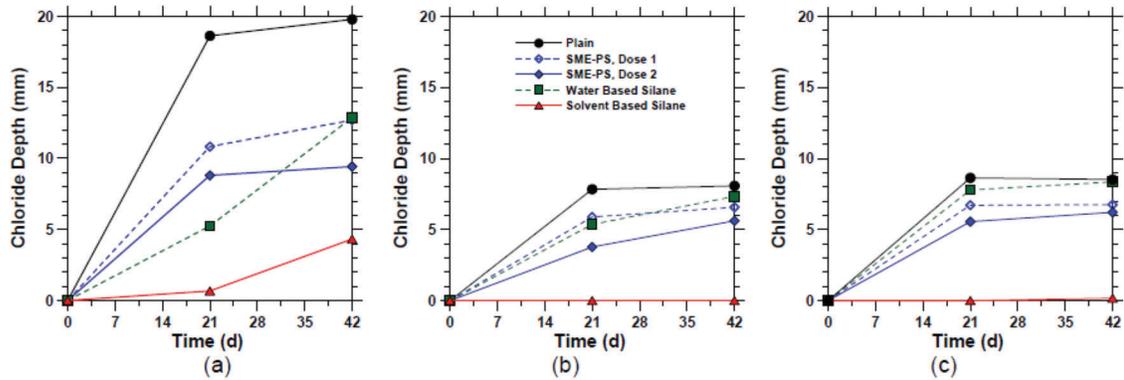


Figure 2.9 Chloride penetration depth for (a) sodium chloride, (b) magnesium chloride, and (c) calcium chloride.

2.3.2 Precautions

SME-PS is a solvent, which brings a few concerns with its use in the construction industry. This property can cause issues when SME-PS is being used in an area near asphalt or, less so, near paints. When exposed directly to asphalt and given a period of time the SME-PS will soften the asphalt, potentially up to a point where it can be removed with mechanical abrasion. This property has created both negative and positive situations. Although it can cause unwanted damage to asphalt that lies in areas near concrete pavements that are being treated, it can be beneficial when removing ineffective areas of asphalt overlay that had been used in an attempt to seal the joints or cracks.

In locations where the removal of asphalt is desired, a three-stage process could be quite effective and require minimal labor. A first, light coating can be used to soften the asphalt seal. After a short period of time (less than one hour) some form of mechanical abrasion can be used to remove a portion of the seal and fully expose the joint. Once the joint is exposed, a second coat of SME-PS (applied as directed in the following sections) can be used to fully seal the joint. The only sided effect of this process (as opposed to traditional treatment of an already open joint) is that residual, softened asphalt can cause a slight discoloration in the SME-PS treated areas.

Up to this point the solvent properties have been of a lesser concern when used near a painted section. Although SME-PS has the potential to dissolve some paints, it has not been an issue in any field trials thus far. Laboratory investigation has shown that roadway paints can be removed with SME-PS but require an extended exposure to the sealant (see Figure 2.10).

In one trial, paint stripes were exposed to significant amounts of SME-PS mopped onto the surface, as well as a significant amount of mechanical abrasion that was used to remove asphalt, yet the paint stripes remained unaffected by the process. Results of an asphalt removal project are shown in Figure 2.11. Asphalt had been mistakenly spilled onto a section of Kalberer Road on the north side of West Lafayette, IN. SME and absorptive pads were used to clean up the main

portion of the spill. It is evident that this process had very little impact on the road paint.

The solvent properties of SME-PS can also cause issues with storage and equipment if not handled appropriately. This issue is easily mitigated by the selection of appropriate plastics for storage and application equipment. Polycarbonate (PC), polystyrene (PS), and acrylonitrile butadiene styrene (ABS) plastics should be avoided. This includes storage containers, sprayers, hoses, gaskets, and nozzles that will, at any point throughout the application process, come into contact with SME-PS.

2.3.3 Preparation

There are two main concerns that should be addressed in preparation for SME-PS application in pavement joints. The pores around the joint must be empty and they must be accessible. Ensuring accessibility is simple; remove any materials that may have entered the joint, wash away any slurry that may remain in the joint from saw-cutting, making sure that the inside faces of the joint are clean and exposed. It is recommended that an air



Figure 2.10 Tests of compatibility of SME-PS with INDOT road paint under normal exposure (left) and extreme exposure (right).



Figure 2.11 Asphalt spill on Kalberer Road before and after removal using SME-PS.

compressor be used to blow air through the joint before application. The intent of this air current is to blow water out of the joint and minimize the degree of saturation in the pores near the surface of the joint.

Application of the SME-PS should not occur during or 24 hours after the most recent rainfall. This ensures the pores of the concrete are open and available to absorb the applied SME-PS. Similarly it is advised that application not occur when the temperature is below the dew point, as this will allow water to condense within the pores. If the temperature falls below the dew point on the day of application it is recommended that the area be retreated with compressed air, in order to accelerate vaporization of any water that may have condensed, prior to application. The consequences of a shortened rain free period after application will be discussed in the following section.

2.3.4 Application

The application of SME-PS is a simple process. The field applications that will be discussed in the following section (section 2.4) have been performed with the use of a 4 gallon, RL Pro Flo-Master backpack sprayer with a fanning spray tip (shown in Figure 2.12). The objective of the application is to coat the inside faces of the joint and 2 in. (50.8 mm) inches at the surface on either side of the joint opening. For the best results, enough sealant should be applied so as to create a small reservoir at the base of the joint that can be absorbed into that critical area over the following hours. For a typical joint, assuming a 4 in. (101.6 mm) depth, the application rate is 160 linear feet (48.8 m) per gallon. SME-PS application procedure should follow manufacturer's recommendation.

The excess sealant will settle in the lowest locations. These locations will see a longer exposure time to SME-PS, and consequently a deeper penetration of the sealant. This is beneficial to the joint network because these locations are those that will also see similar

settlement and the highest exposure to the deleterious fluids that lead to deterioration. If rain occurs within 24 hours after application much of this reservoir may be cleaned out of the joints before it has time to be absorbed into the concrete. This will leave these areas more vulnerable to fluid ingress. If rain occurs within the first three hours after application retreatment is recommended. In this case, much of the sealant on the joint faces and at the surface may be washed away before being fully absorbed into the surface.

2.3.5 Traffic Considerations

The application of SME-PS could create a slick surface until it can be absorbed into the surface. For new construction projects, there is minimal concern for traffic as the sealant can be applied and absorbed well



Figure 2.12 RL Pro Flo-Master backpack sprayer used in field application of SME-PS.

before the project is open to traffic. Experience in field trials thus far has shown this to be of minimal concern. However, in order to avoid liability issues it is recommended that traffic be diverted from the joint for three hours following the application and that field personnel examine when they feel comfortable opening the pavement to the travelling public.

2.3.6 Potential Inclusion of Scent and Application Indicators

It has been discussed that many DOTs desire the inclusion of an indicator in order for inspectors to verify where sealant has been applied. The solvent properties SME-PS provide a simple solution for indicating sealed locations. Pigments can be dissolved into the sealant to show where the material has been applied. The color can be controlled by the particle size of the chosen pigment. If the particle size is slightly bigger than the size of the surface pores of the concrete, than the pigment will be screened at the surface and will be washed off with subsequent rainfalls. If the particle size is slightly smaller it will remain in the sealant and provide a long term discoloration. Scents have also been added to the SME-PS.

2.4 SME-PS Field Applications in Central Indiana

Several field trials of SME-PS have been initiated throughout central Indiana. These trials have been documented by recording videos of all treated joints for later reference so that any areas of damage can be monitored over time. Conditions on the day of application were also recorded including minimum, maximum, and mean temperatures, dew point, and wind speeds. As was mentioned in the previous section, the application was conducted with the use of an RL-Pro Flowmaster backpack sprayer. In each case application was conducted with four walking passes along the joints, two in each direction, holding the nozzle of the sprayer about an inch (25 mm) above the joint opening as demonstrated in Figure 2.13. All sealant used in field trials contained 2% PS by mass.

2.4.1 US-231 in Lafayette, IN

The first field trial of SME-PS is a small section of southbound US-231. This section of highway was 12 years old at the time of application and was originally placed in the summer of 1999. The test section lies between the intersections with W 400 S and W 500 S and consists of twelve slabs with the previously installed backer rod and silicon sealant removed. These slabs were divided into two sections of six slabs with one slab in between them with the backer rod and sealant intact. The northern six slabs were treated with SME-PS following the procedures recommended in section 2.3. The southern six slabs were left untreated as a control section. The test area is shown on the map in Figure 2.14.

The application of SME-PS in the joints on US-231 was conducted on the morning of August 6, 2011 by



Figure 2.13 Demonstration of field application of SME-PS, on US-231 in Lafayette, IN.

INDOT maintenance personnel under the supervision of INDOT officials and researchers along with Purdue researchers. Application procedure was as per the manufacturer's specification. On the day of application the minimum temperature was 71.6°F (22.0°C), the maximum 89.1°F (31.7°C), and the mean 80.2°F (26.7°C). The mean dew point was 72°F (22.2°C), at the beginning of application the temperature was above this point. Wind speed was also recorded, as it can influence the moisture condition of the concrete. The mean wind speed was 4.0 mph (1.79 m/s), with a maximum sustained speed of 11.1 mph (4.96 m/s) and a maximum gust of 20.7 mph (9.25 m/s) (The Old Farmer's Almanac, n.d.).

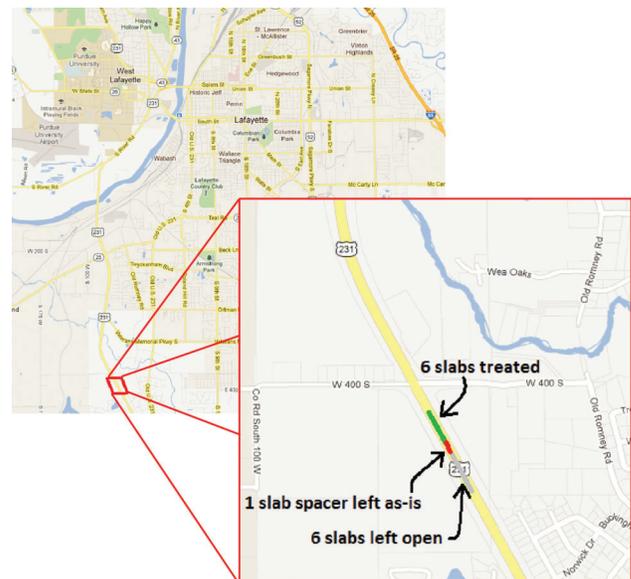


Figure 2.14 Location of the US-231 field trial section.



Figure 2.15 Treatment of the median barrier at the SME-PS test section on US-231.

For this application only the longitudinal joint between the two lanes of traffic was treated. During application the two lanes of traffic were reduced to a single lane and moved to the shoulder. The traffic pattern for application was established at 9:00am and returned to regular two-lane traffic flow at 1:00pm, approximately three hours after the application was complete as per manufacturer’s recommendation.

During application it was noticed that the median barrier through this section of highway sits in a low spot, with the pavement on either side draining towards the barrier. This is likely to cause a high exposure to deicing salts during the winter months. The base of this barrier was treated with SME-PS as shown in Figure 2.15. The intent of this treatment was to provide an approximation of the exposure seen by vertical surfaces near salt water, such as bridge piers or abutments.

In January of 2013 the section was revisited and each area was documented with pictures and videos of the treated areas. These were compared to those taken on the day of application in order to determine if any new damage has become visible in either the joint or the barrier. In a side-by-side comparison, no new damage was found to have occurred in the 18 months between the time of application and the reevaluation. While data collected to this point has been rather qualitative, quantitative data will be taken for an analysis of long-term performance. Samples of the area will be taken either by coring and then subsequently grinding the cores into powdered samples of each millimeter throughout the depth of the core. These powders will be titrated in order to determine a profile of chloride content throughout the depth of the sample. Alternatively, a dry drill may be used to collect powdered samples in increments from the joint and similarly titrated to obtain a chloride content profile.

2.4.2 126th Street Fishers, IN—East of I-69

The second test section, and first where SME-PS has been applied to a newly constructed pavement, was on 126th street in Fishers, IN beginning to the east of the overpass at I-69. This section of roadway consists of two lanes in each direction, with turn lanes and uncurbed median in several locations throughout the test area. All of the longitudinal joints, over 3000 linear

feet, in this section of pavement were treated, with the exception of the first 200 feet of joint east of the overpass bridge which was left untreated as a control section. At the request of INDOT/FHWA the transverse joints were sealed with traditional methods of backer rod and silicon sealant after the application of SME-PS was completed in the longitudinal joints. Treatment was performed by Berns Construction and began immediately east of the control section and continued to Raiders Blvd. A map of the location can be seen in Figure 2.16.

Sealing of this section occurred on October 25, 2011 with the coordination of BFS Engineering and Berns Construction who were managing the construction project. Application was conducted by members of the Berns construction team per manufacturer’s specification as shown in Figure 2.17. After application,

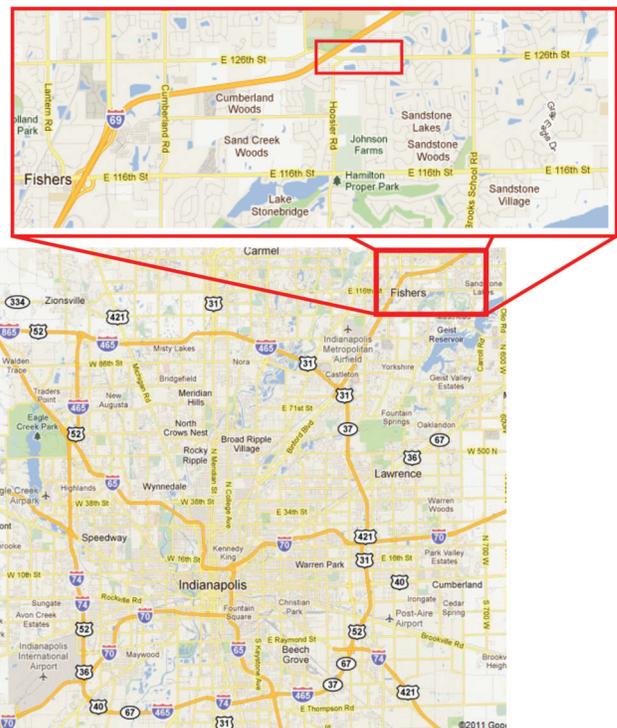


Figure 2.16 Location of the second SME-PS trial location, on 126th Street in Fishers, IN, east of I-69.



Figure 2.17 Application of SME-PS to 126th street in Fishers, IN being performed by members of Berns Construction.

contractors said this application method was simpler and faster than current sealing practices. All of the application procedures and recommendations from section 2.3 were maintained throughout this project as per manufacturer’s recommendation.

Since this application occurred later in the construction season, temperature and dew point were monitored closely through the morning of application. The minimum temperature was 43.0°F (6.1°C), the maximum 73.0°F (22.8°C), and the mean 55.6°F (13.1°C). The mean dew point was 38.9°F (3.8°C). The mean

wind speed for the day was 7.1 mph (3.17 m/s), with maximum sustained wind of 15.0 mph (6.70 m/s), and a maximum gust of 25.3 mph (11.31 m/s). All treated joints were videotaped for later reference in order to document damage from sawing so that it can be differentiated from any other damage that may occur.

2.4.3 126th Street Fishers, IN—West of I-69

The construction of 126th Street continued the following summer with the replacement of the portion west of the overpass at I-69. This test area begins at the intersection of Reynolds Drive and ends at the intersection with Cumberland Road. In this section all longitudinal and transverse joints were treated with SME-PS. The only exception being all of the joints in the last two slabs at the east end of this test section, commencing at the intersection with Cumberland Road. This test section is shown on the map in Figure 2.18. All treated joints were documented with videos for future reference.

Berns Construction performed application as per the manufacturer’s recommendation throughout this test section in coordination with BFS Engineering, similarly to the section East of I-69. Application occurred on September 25, 2012. On the day of application the minimum temperature was 39.0°F (3.9°C), the maximum 66.9°F (19.4°C), and the mean 57.7°F (14.3°C). The mean dew point was 49.1°F (9.5°C), application began slightly later in the day than other trials to ensure that the temperature was above the dew point. The mean wind speed for the day was 7.8 mph (3.5 m/s) with sustained winds of 11.0 mph (4.9 m/s).

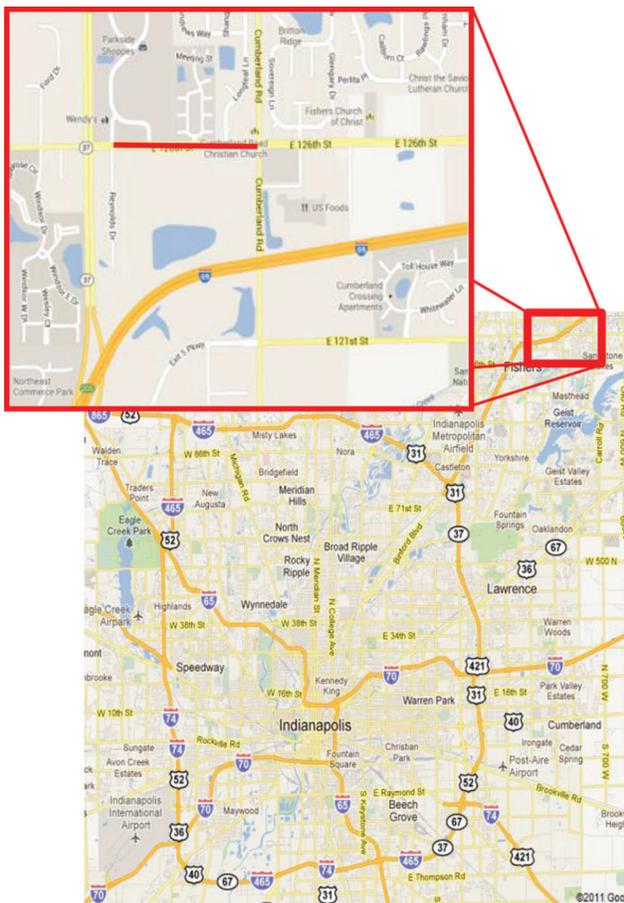


Figure 2.18 Location of 126th street field trial, west of I-69 in Fishers, IN.

3. ASSESSMENT OF FIELD PERFORMANCE OF SOY METHYL ESTER–POLYSTYRENE AS A CONCRETE SEALANT

3.1 US-231 in Lafayette, Indiana

Samples were taken from US-231 just south of the intersection of W 400 S and US-231 in south Lafayette. Three cores were taken November 26, 2014 at approximately 9:00am. At the time, the pavement was approximately 15 years old. Three cores were taken from different sections A, B, and C along the longitudinal

joint corresponding to the joint sealer application: SME-PS (A), filled with typical backer rod and sealant (B), and left open with no sealant (C) as seen in Figure 3.1. The application of these three experimental sections began on August 6, 2011 corresponding to a testing time of three years.

The objective of this research was to understand the behavior of SME-PS used as a topical sealant on a US highway exposed to winter conditions. The three different test sections were spaced next to each other to minimize the difference in exposure to weather, traffic, and salt application.

The SME-PS core was taken from slab A6 two feet north of the intersection of slab A6 and B, Figure 3.2. The as-is (b) core was taken from slab B, two feet north of the intersection of slab B and slab C1. The core taken from the open section was from the slab C1 two feet north of the intersection of the slab C1 and C2.

Pictures were taken along sections of the pavement. Special consideration was given to locations along the joint where damage was seen. Along section A, the SME-PS treated section, there was change in the one of

the joint intersections between panel A4 and A5 as seen in Figure 3.3 (a) and (b). In Figure 3.3 (c–f), the untreated joints are also showing signs of deterioration.

Cores taken from section A and C were in two pieces when taken out of the pavement. One side of the joint was split into two pieces and sprayed with AgNO_3 to see the ingress of chlorides as seen in Figure 3.4. After the silver nitrate reacted with the chlorides a sharpie outlined the extent of chloride ingress. From Figure 3.4, the treatment of SME-PS appears to be reducing the ingress of chlorides when compared to the slab that was left untreated.

The other side of the core was tested for chloride ingress by the grinding and titration method. Powder was obtained by grinding the bottom of the surface of the saw cut with a diamond core bit (see Figure 3.5 and Figure 3.6). Taking titration measurements, thus obtaining a chloride profile perpendicular to the saw cut, allowed understanding of how the chlorides moved horizontally into the concrete from the side of the joint. The powder was taken at the depth, D, where the saw cut made the bottom of the joint. The measurement of distance from the vertical surface of the bottom of the

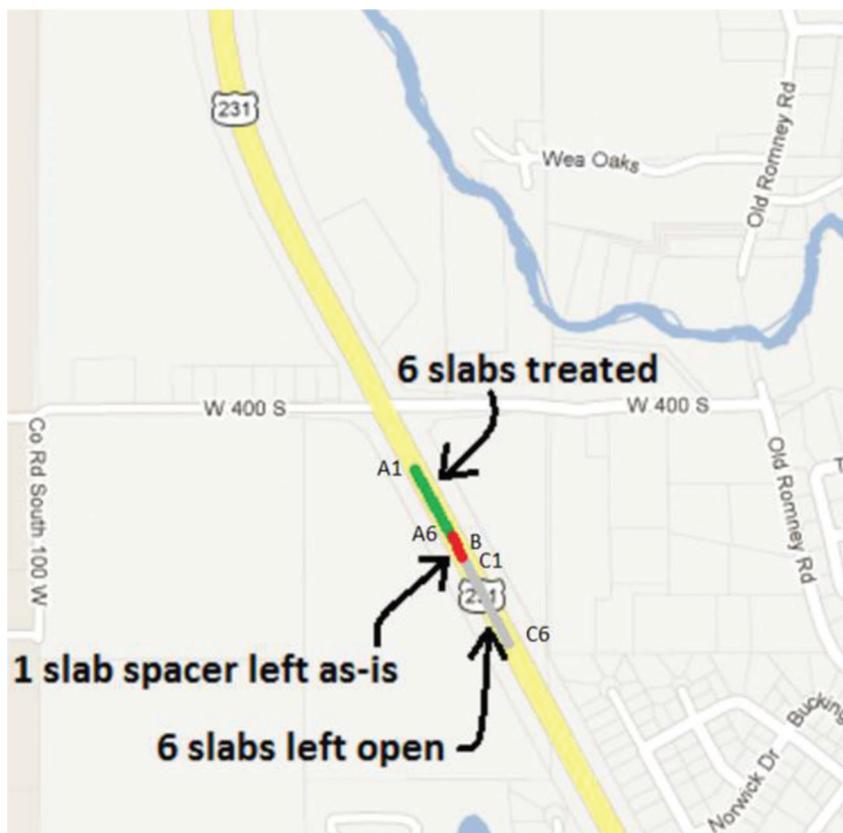


Figure 3.1 Sampling location overview for US-231.

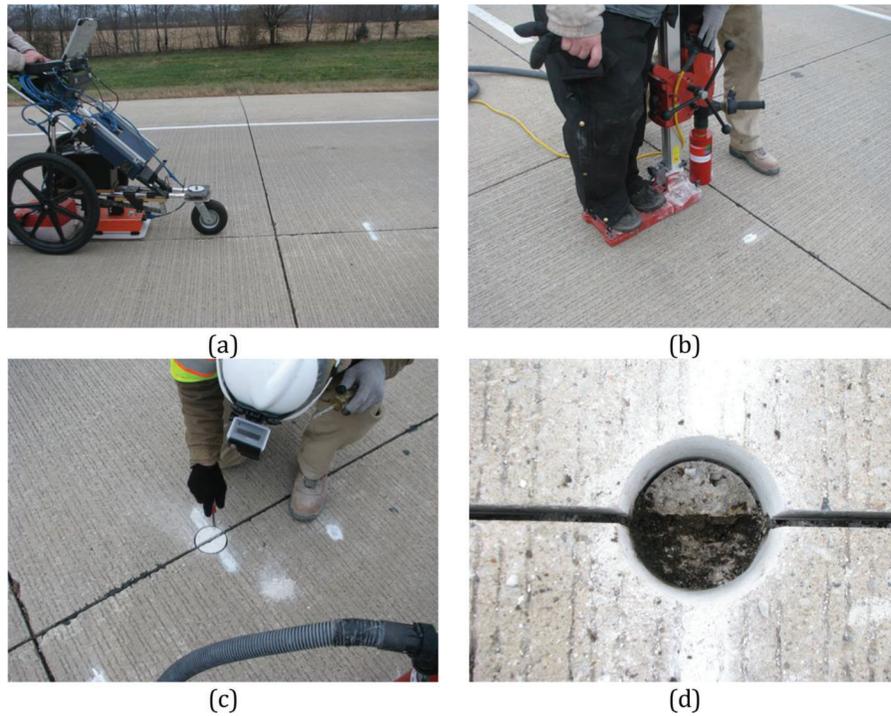


Figure 3.2 Coring process (typical for all coring locations): (a) location for core marked, (b) core drill drilling pavement at joint, (c) removing core from road, and (d) joint with core removed. Not seen is the core being filled in with hot mix asphalt.

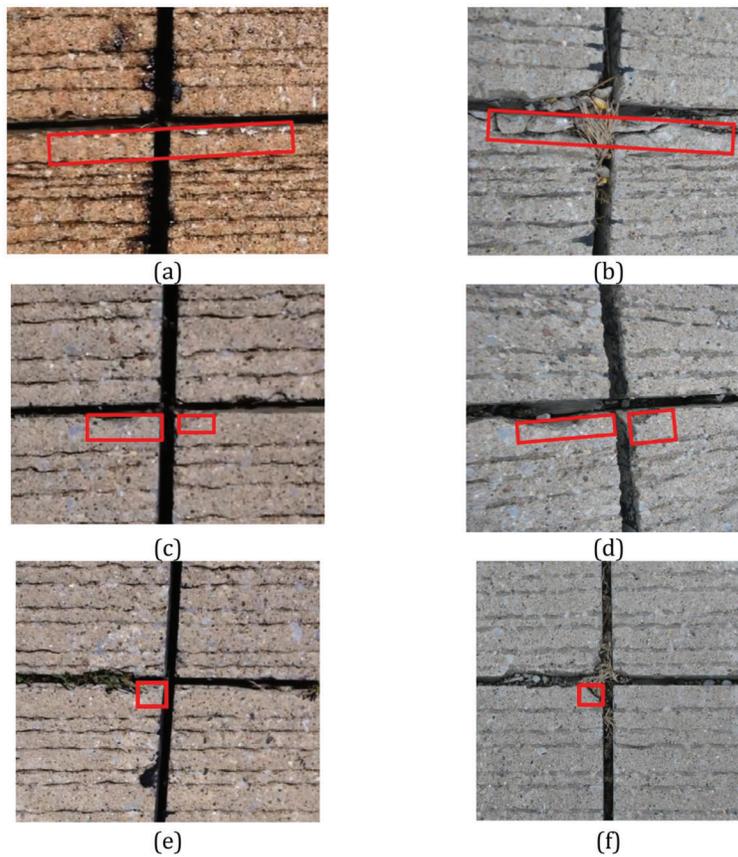


Figure 3.3 Before and after pictures of damaged locations: (a) slab A4-A5 before, (b) slab A4-A5 after, (c) slab C1-C2 before, (d) slab C1-C2 after, (e) slab C3-C4 before, and (f) slab C3-C4 after.

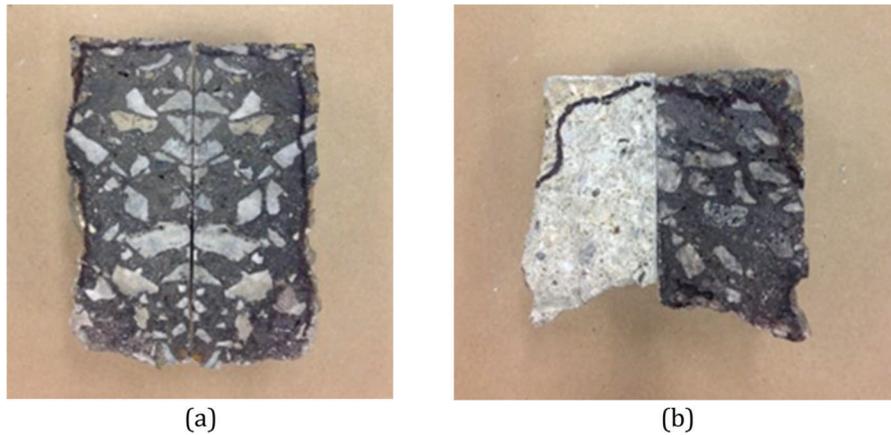


Figure 3.4 Cores split in half with silver nitrate applied. Sharpie mark indicates approximate depth of chloride penetration: (a) SME, (b) control.

saw joint is recorded in millimeters from the surface of the saw joint into the concrete core. For the three cores taken at the US-231 site this saw-cut depth, D , was approximately two inches. The distance from saw joint surface grounded was nearly 15mm.

As seen in Figure 3.7, when the joint is untreated, the amount of chlorides in the pavement at the first few millimeters up to about 12 mm are doubled. The SME sample reduced the amount of chlorides by about 40% compared to the untreated sample while it was allowing 20% more chlorides into the concrete compared to the backer rod and filler. This increase is expected since the control section has a physical barrier keeping water out compared to the SME section which is reducing the amount of water and deicers that can be absorbed into the pores of the paste. As the SME continues to penetrate into the concrete some of the larger capillary voids and air voids may not be completely filled with SME. This leaves open space and an opportunity for deicers to enter the pavement. Two things should be

emphasized: (1) the SME is working as a sealer so far, and (2) the difference in chloride content between the control and SME samples may not mean the SME is not performing as well.

One possibility why the background chlorides are so high is that there could have been additional chlorides added into the concrete at the time of mixing to accelerate the rate of reaction and to reduce the curing time. To try and test these hypotheses, the portion of the cores furthest away from the original pavement and joint surfaces were ground and titrated so as to not be affected by environmental chloride ingress. Similar results were found when compared to the chloride contents seen at depths past 11 mm. While no records are available the high chloride background may suggest the use of chloride accelerators used when this concrete was placed.

One expectation from a chloride profile is the chloride percentage increasing as the distance from the surface decreases. However, less chlorides are seen near the surface than there are a few millimeters away from the surface. This may be due to diffusion of chlorides across the surface of the pavement which have closer proximity to the open environment. Depending on the concentration gradient across the pavement surface, chlorides may be inclined to enter or exit the pores at the first few millimeters. Since the cores had not been taken after the summer season the joints had not been exposed to deicers and rainwater could have washed salt out of the joint and caused the chlorides at the surface to diffuse back out of the cement explaining the drop in chlorides at the surface of the joint face.

A limitation to account for in this test is the type of chloride that is being measured. This testing method is unable to distinguish between bound chlorides and free chlorides. It is hypothesized that the section coated with SME is protected from deicers even though chlorides are entering the pavement. This is because the SME which is coated on the wall is hydrophobic and keeping the free chlorides from reacting with the cementitious system. This explains the difference in chloride content between the SME and the other samples. While the

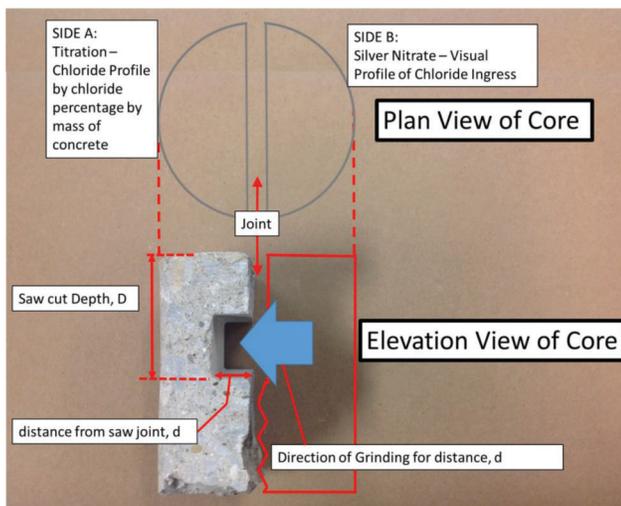


Figure 3.5 Elevation and plan view of core; one side of core used for titration, one side visually inspected for chloride ingress.

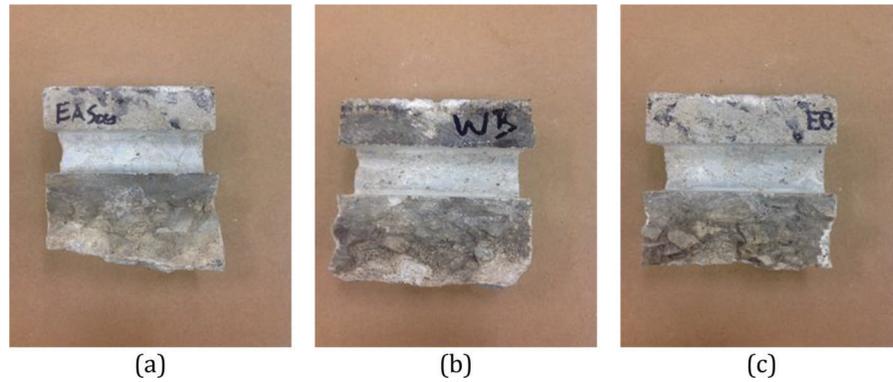


Figure 3.6 Titration side of 3 samples taken from US-231: (a) soy, (b) as-is, (c) control.

SME does not completely keep water and deicers out it does restrict the ingress of chlorides compared to the unsealed section. This may indicate that even though the SME section has a higher chloride content than the control section, the chlorides are not reacting with the cementitious materials (Figure 3.7 and Figure 3.8).

3.2 126th Street in Fishers, Indiana

In total nine cores were taken from 126th street in Fishers (Figure 3.9). The sampling was spread between two sampling dates. During the first trip on December 9, 2014, five cores were taken and on a second trip on March 17, 2015, four more samples were taken. New videos were taken at both locations however in the previous documentation, there were few usable pictures or videos taken of the I-69 overpass. New video and photo records from the west bound lane just west of the overpass were taken and the location recorded for future site visits.

The pavement was approximately 3 years old on the west side of the I-69 overpass and approximately 4 years old on the east side at the time of sampling in December. Two cores were taken from the section of pavement on the west bound side of 126th street between Enterprise Drive and Parkside Drive. One core was taken from the longitudinal joint and one from the transverse joint to measure the difference in chloride ingress between the two SME-treated joints of differing joint orientations. The second and third core was extracted from a section of concrete on the west bound side of 126th street along the longitudinal joint a few slabs west of the I-69 overpass and across the bridge from the control section. The third control sample was unable to be titrated since the depth of the saw cut was

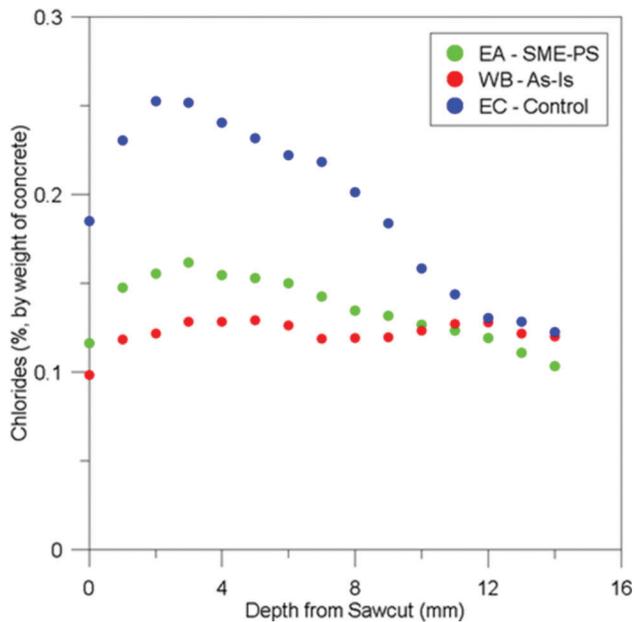


Figure 3.7 Chloride profile data obtained from titration machine.

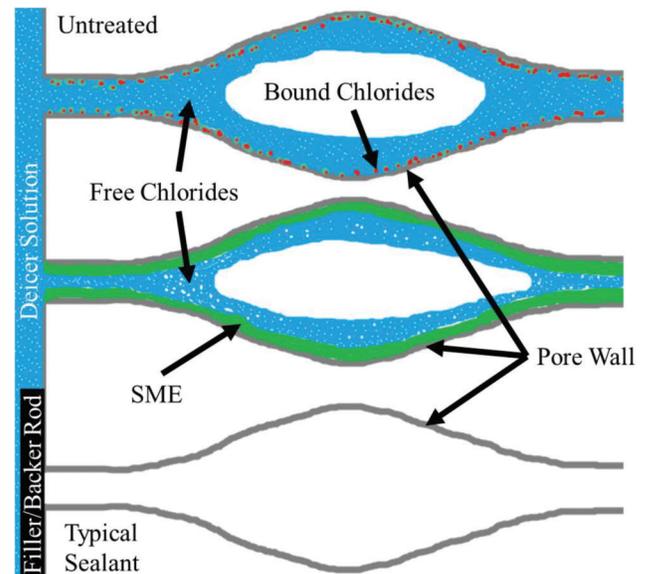
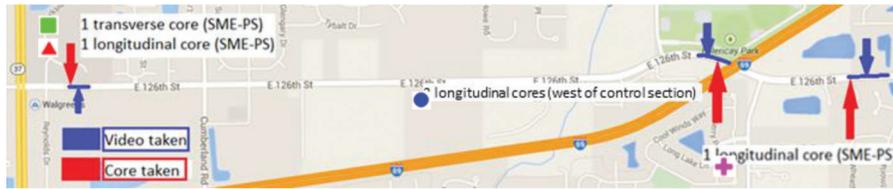


Figure 3.8 Simplified model showing the difference between an untreated sample, an SME treated sample, and a sample with a typical sealant like backer rod and filler. Untreated sample allows water and chlorides to enter pore (some of the free chlorides become bonded to the pore wall as they react with the pore wall). SME sample restricts full flow of deicer solution and keeps free chlorides from reacting with cementitious pore wall. Typical sealant keeps all water out assuming the sealer is untarnished and working properly.



(a)



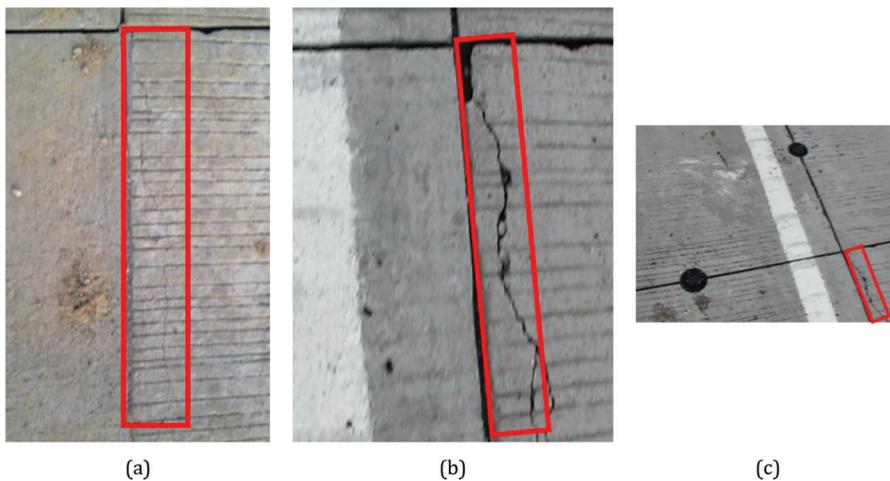
(b)

Figure 3.9 Location of samples taken on (a) December 9, 2014, and (b) March 17, 2015.

too shallow (Figure 3.11 (c)). The fifth SME core was taken in the median along the longitudinal joint in between the median and the east bound lane east of the I-69 overpass. The core was one slab east of the curbed median and two feet east from the intersection of transverse and longitudinal joint. The pavement where the cores were taken was undamaged except for one section shown in Figure 3.10.

At this test section a crack has formed that is parallel to the longitudinal joint. It was visible approximately three years ago and when compared with the most recent picture, further growth can be seen. One of the possible reasons to explain why the crack has grown are

the inconsistent saw-cut depths. As seen in Figure 3.11 there is a range from half an inch to three inches from the five samples taken from the field. A change in saw joint depth can cause problems in the pavement by not localizing the crack into the saw joint which increases the chance that the crack forms elsewhere as seen in Figure 3.10. In the case of crack running alongside the longitudinal crack, this is most likely attributed to the fact that the saw cut was only one inch deep Figure 3.11 (b). This lack of depth may attribute to the crack and its growth seen in Figure 3.10. There is no crack parallel to the transverse joint and this may be due to the saw-cut depth being adequate as seen in Figure 3.11 (a).



(a)

(b)

(c)

Figure 3.10 First location of longitudinal joint sampling of SME-PS samples: (a) 1 slab west of sampling location on the day of SME-PS application, (b) 1 slab west of sampling location on the day of coring, (c) overview of sampling location and visible crack.

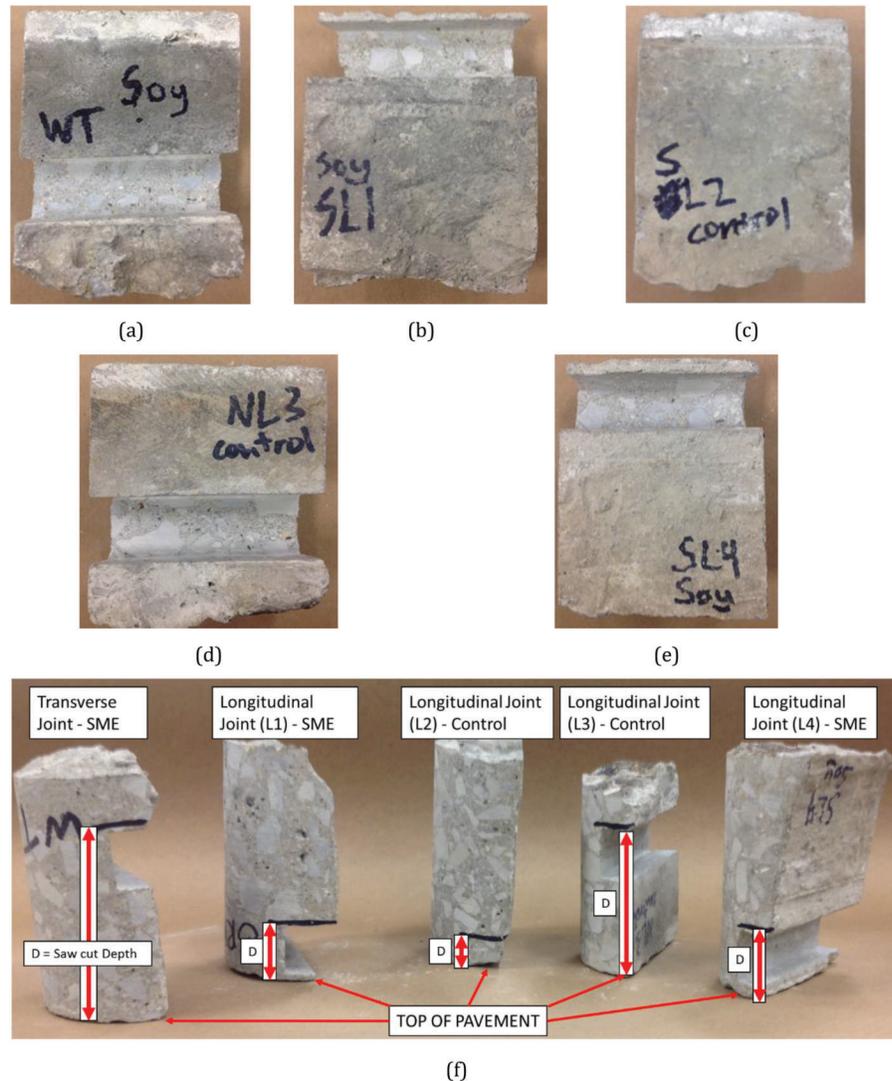


Figure 3.11 Titration side of 3 samples from Fishers: (a) transverse SME joint; (b) longitudinal SME joint, (c) longitudinal control joint; (d) unusable longitudinal joint (L3)—control; (e) longitudinal SME joint; and (f) titration side of 5 samples taken from Fishers in order from left to right—T, L1, L2, L3, L4, L5. Note: control depth of saw cut varies.

The varying depths of saw cuts and miscommunication about the location of the control section prompted another site visit to Fishers on March 17, 2015. In total, four cores were taken in the second visit. Two cores were taken from the correct control zone on the east side of I-69 in the westbound direction. One longitudinal core was collected near the bridge and one 5 slabs further from the bridge. The bridge was expected to have higher chloride exposure so the distance away from the bridge can be taken into account when interpreting results from titration tests. Further east on the same side of the road 2 more cores were taken in front of the church to the north. One core taken was of the longitudinal joint sealed with SME and the other a transverse joint sealed with backer rod and filler. Titration test were run on the four samples and data is presented in Figure 3.12. The most apparent difference between the two different site visits is the magnitude of chlorides. Since the locations of

the cores shifted the chloride contents are not expected to be the same. Also the cores taken in March were exposed to an additional deicers over the winter which would explain why the chloride content has increased. A positive to note is the SME-PS sample and conventional sealer sample were taken from the same location had similar chloride contents indicating the SME-PS performs as well as the conventional sealer. Untreated slabs were higher in chlorides than treated slabs which was expected.

In Figure 3.12 (a), the two SME samples denoted by a green square and red triangle are two cores from the same slab but at different joints, the first in the transverse joint and the second in the longitudinal joint. The sample with a one-inch saw cut has higher chlorides than the sample adjacent to it likely because the chlorides from the surface are being measured rather than only the chlorides coming in from the horizontal direction.

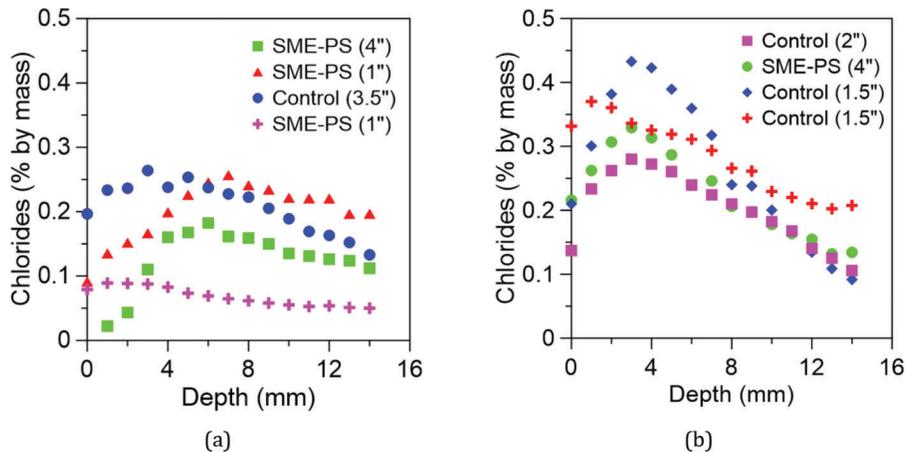


Figure 3.12 Chloride profile results (depth from top surface in parenthesis) taken in (a) December 9, 2014, and (b) March 17, 2015.

Cracks may be forming elsewhere in the pavement as seen in Figure 3.10 and therefore the chlorides may be penetrating into the concrete faster than if they would have diffused through the paste. In Figure 3.12 (a), the control sample is expected to have higher chlorides since it is next to a bridge which sees higher salt exposure. The SME sample denoted by the pink cross in Figure 3.12 (a) is the lowest of the four samples tested from the first sampling likely due to the fact that the core was taken from the passing median where less salt is applied compared to the roadway. Similar to the titration data from US-231, the chloride profile increases for the first few millimeters and then dropping off instead of constantly decreasing. This might be due to the total rain-water in 2014 washing away and diffusing chloride ions in the pavement joint near the surface.

In the second visit the chloride contents all increased. This is because the pavement had been through another winter and subsequent exposure to deicers. Additionally the location of the cores taken in the field on the second trip in March was from the east side of the I-69 overpass and were one year older than the samples on the east side. The two samples in Figure 3.12 (b), the blue diamond and red cross, were taken in the control section near the bridge. Both samples show similar chloride contents but the red cross sample has a more shallow slope to its maximum chloride content at around 1mm. The slope is assumed to be affected by the free chlorides ability to diffuse to the surrounding areas. This idea is supported because the area where the red cross sample was taken was from a higher elevation compared to the blue-diamond sample. Since the sample was at a higher elevation the amount of rain run off was less than the sample downhill. At locations where there is more rain water passing through the joint it is more likely that chlorides could diffuse back out of the concrete. This would explain why at the beginning of the chloride profile, the sample at the higher elevation shows higher amounts of chlorides. On the other hand the blue diamond sample lower in elevation shows a decrease in chlorides as the profile nears the pavement surface. This

indicates that chlorides are diffusing back into solution and leaving the concrete.

Continuing analysis of Figure 3.12 (b), the pink square control sample taken from the transverse joint and the green circle SME longitudinal joint sample were taken from the same slab but from different joint locations. The SME sample shows at most a 20% increase in chlorides. This can be attributed to the SME leaving space in the pores allowing deicer solution to enter concrete surface as was seen in the US-231 cores. Additionally the SME sample is at a depth of 4in while the control sample is at a depth of 2in. Water and deicer solution could and keep the joint which had a deeper saw cut than the adjacent sample in a moist, chloride-rich environment where more chlorides would diffuse into the concrete.

It is important to emphasize that this test method may not be the best in assessing the performance of sealers. It is useful in determining chloride content as long as the impact of the two different sealing techniques on the results is accounted for. As was said for the US-231 samples, there is a 20% increase in chloride ingress seen in some SME samples compared to control samples and this is likely due to more of the deicer solution allowed to enter the pores compared with the control that keeps the chlorides out by means of a physical barrier. This increase in chloride content may not necessarily disqualify SME from use. The chlorides may not react with the cementitious system if the pore walls are coated with SME and therefore no damage due to chemical reaction would be seen indicating the sealer is doing the job of protecting the pavement. In the future, it is recommended that samples continue to be taken to compare SME and the control to determine when reapplication is necessary.

4. EVALUATION OF PENETRATING SEALANTS USING SORPTION TEST WITH THERMAL CYCLING

4.1 Introduction and Objectives

While many have looked at the ingress of chlorides due to its potential to cause corrosion to the embedded

reinforcement steel (Pfeifer & Scali, 1981), this work examines the penetration of chlorides due to the potential interaction with chemical species in the concrete with respect to the potential for deterioration. One method that has been found effective in reducing the amount of chloride ingress into the concrete matrix is the use of sealers, pore blocker, and water repelling materials. The use of these materials may be able to improve the service life of concrete pavements.

Concrete sealants can be used on freshly placed concrete or on concrete already in service. One question that arises however is how to test the performance of these sealants. There are current practices to determine the sealants effectiveness in reducing water ingress or chloride ingress at room temperature. However, it is not clear what happens when the sealant is exposed to freezing and thawing.

The objective of this study is to develop and evaluate a testing methodology to assess the impact of sealers, pore blockers, and water repelling materials for the ingress of fluids containing deicing salts. A proposed testing protocol is presented, which evaluates the sorption as the temperature is varied. This project examines various topical, penetrating, concrete sealants in order to investigate depth of penetration of the sealant into the concrete matrix and how this relates to the sealants ability to reduce chloride ingress through temperature cycling.

Research will compare different sealants to determine the depth of penetration into the concrete surface and to investigate if this is directly related to the sealants ability to reduce chlorides during freezing and thawing exposure. The further the sealant penetrates into the concrete, the more effective the sealant should be at reducing chloride ingress.

4.2 Experimental Approach

An experimental method to evaluate the effectiveness of sealants, pore blockers, and water repellants during thermal cycling is the focus of this study. A test was developed to closely replicate absorption in concrete that is exposed to fluids containing salts, such as deicing solutions. For this study it is of interest to look at concrete exposure to salt solutions in a freezing and thawing environment.

Figure 4.1 shows the experimental setup. This is very similar to ASTM C1585 (2011), however, unlike the current standard where absorption is performed on a sample in room temperature water, this test exposes the sample to chlorides and freeze-thaw conditions. The test uses a cold-plate device to induce cooling from the bottom face of the concrete specimen. Note that the cooling will take place in a one dimensional heat flow, or “bottom-up” fluid flow through the concrete. This would be a similar situation to concrete pavement, which has a single face exposed to deicing salts, although gravity would be acting in the normal downwards direction. It has been shown gravitational effects on fluid transport in the capillary pores, provided large cracks are not present,

can be neglected when compared to the suction effects of the pore structure (Yang, Weiss, & Olek, 2004).

Samples are placed in a chloride solution, similar to the water used in absorption testing. Concrete specimens are placed in a container that is thermally conductive and resistant to corrosion. For this experiment, a galvanized steel, open-faced, box was used to contain the solution and sample. The box was covered with a plastic wrapping to minimize evaporation. A thermal pad (Thermacool TC3000) was placed in-between the cold plate surface and the container to effectively transfer heat from the cold plate to the container. A further discussion of thermal transfer for this setup can be found in Section 4.4.1. The samples should be placed such that they are not in direct contact with the container. This is to allow for absorption of the chloride solution throughout the exposed face of the concrete specimen. The system should be insulated such that temperature loss is a minimum. The container is to then be filled with the desired solution reaching no more than 0.15in [3mm] above the sample face. The deicing solution will not freeze during the testing and will serve as a thermal conductor from the container to the sample.

4.3 Materials and Specimen Geometry

4.3.1 Mixture Design

All samples used in this study were of the same mixture. The samples were a part of a large-scale cast using a commercial concrete mixture from a ready-mix concrete truck. The concrete was prepared with a $w/c = 0.42$. The fresh properties were tested in accordance with ASTM C192, ASTM C143 (2012), and ASTM C231 (2010). The slump was 4" and the air content 6.2%. The coarse aggregate has an absorption of 0.58%, specific gravity at SSD of 2.77, and is AP#8 aggregate from U.S. Aggregates in Delphi, IN. The fine aggregate has an absorption of 1.33%, specific gravity at SSD of 2.65, and



Figure 4.1 Schematic of experimental setup for freeze-thaw absorption with chloride solution.

is #23 Swisher Road from Vulcan Aggregates in West Lafayette, IN. The mixture proportions are shown in Table 4.1. The batching ticket can be found in the appendix of this report.

4.3.2 Specimen Geometry and Preparation

Approximately 30, 4" x 8" concrete cylinders were cast in accordance with ASTM C192 (2012) to be used as test specimens for this study. At 24 hours the specimens were removed from their molds and placed in plastic bags, to create a sealed condition. When the appropriate time for the specimens to be tested came, the cylinders were cut into 3-inch tall samples. The top and bottom inch of the cylinder were removed. For initial evaluation of chloride penetration, the samples have a height of approximately 3 inches. This is for preliminary experiments, but will be reduced to 2-inch samples for the final testing protocol to be consistent with ASTM C1585 (2011) sample geometry. A height of 3 inches was chosen for preliminary testing to allow for potentially high absorption in the sample.

The circumference and one end of the samples were coated with a two-part epoxy (a similar cohesive moisture barrier would also be acceptable), leaving one face uncoated. This is to only allow fluid penetration through the desired face of the specimen. A plastic covering should be used at the top surface in future testing to eliminate potential pressure buildup at the epoxied face of the sample. However, for the purposes of this testing that was not done in order to eliminate any possibility for the chloride solution collection and movement at the top surface. Application of the cohesive moisture should be according the manufacturers specifications, or enough to prevent any moisture from entering/exiting the sample.

After the application of epoxy, the samples were placed in a $23 \pm 1^\circ\text{C}$, $50 \pm 1\%$ RH environment until the sealant was applied, as applicable. Sample conditioning will be further discussed in Section 4.5.1. The sealants will then be applied to the un-epoxied face of the half-cylinders at the manufactures recommended rate of application. The samples should be left to dry after the application of sealant until there is no longer visible moisture from the sealant on the surface of the sample. Sealant application will be further discussed in Section 4.5.2.

TABLE 4.1
Design quantities of materials used in concrete test specimens.

Material	Design Quantity	Units
#8 Coarse Agg., SSD	1800	lb/yd ³
#23 Fine Agg., SSD	1240	lb/yd ³
Cement	658	lb/yd ³
Water	270	lb/yd ³
Water Reducer	2.0	oz/100 lb Cement
Air Entrainer	1.2	oz/100 lb Cement
Retarder	2.0	oz/100 lb Cement

4.4 Preliminary Testing Results

In order to develop a testing protocol for evaluating sealants, pore blockers, and water repellents, a series of preliminary experiments must be conducted to determine appropriate parameters for testing. Variables such as temperature, number of cycles, concentration of solution, and rate of heating/cooling have been evaluated. It is desired to find a simple, practical, and effective way to test sealants. To do this, each test is evaluated on the basis of chloride concentration and depth of penetration of the chlorides. Each previously mentioned parameter is determined for the standard protocol based on the worst condition experienced by the concrete sample, i.e., the higher concentration/further penetration into the concrete.

After each round of testing the samples were split in half, where one half of the split samples was sprayed with silver nitrate (AgNO_3) for visual evidence and the other half was titrated to give chloride concentration throughout the depth of the sample. Titration was completed according to ASTM C1556 (2011) using surface grinding at depths of every 2mm, as seen in Figure 4.2.

Comparative samples, i.e., sealed vs. unsealed, were used in some of the preliminary testing. All sealed samples for preliminary testing used Soy Methyl-Ester blended with 2% Polystyrene by mass (SME-PS), as was discussed in Chapter 5.

It should be noted that various conditioning times were used for the sets of samples in preliminary testing. With that being said, the results of samples in each preliminary test should only be compared to results of samples made from the same material and tested under



Figure 4.2 Surface grinding used to obtain powder for titration.

a given set of conditions, as results from one test will not correlate with that of another.

4.4.1 Temperature Testing/Cycle Length Determination

As in any freezing test, it is necessary to monitor the temperature throughout the test specimen. In order to represent pavements that are exposed to high concentrations of salt solution in a freezing and thawing environment, a condition must be replicated in which the capillary pore water freezes, but the chloride solution does not freeze. It has been shown that capillary water freezes around -3°C (Rønning, 2001); however it was -8°C for the materials used here (Li et al., 2012) potentially due to the composition of the material or due to undercooling.

In order to monitor internal temperature throughout the sample, guarded thermocouple wiring was cast at known depths inside a sample. The sample was then cut and a temperature profile was developed for the sample. Temperature was also monitored throughout material where losses would occur. Figure 4.3 shows a schematic for the section view of the test setup, as well as locations of each thermocouple wire.

Figure 4.4 (a) shows the input program used to control the cold plate, while Figure 4.4 (b) shows the real-time temperature change throughout the sample (as noted above). It is desired to have the center of the sample reach at least -3°C (-8°C was selected in this case since the freezing temperature of capillary water varies and Li et al. (2012) measured damage at -8°C) before commencing the thawing phase. From this data it can be concluded that in order to properly freeze the capillary water, while maintaining a liquid solution, the sample should be in a frozen state for at least 4 hours. In order to properly thaw the sample, a temperature of 10°C should be maintained for approximately 30 minutes.

4.4.2 Rate of Temperature Change

The next parameter of interest, after determining the duration of the freeze and thaw period, is the rate at which the temperature change during the cycle should occur. To investigate this parameter, two tests were run,

in which the only variable was the rate of freezing. Figure 4.5 shows the two different freezing rates at which the samples were tested. Figure 4.5 (a) shows the test in which the freezing rate was “immediate”, or quickly loaded. Figure 4.5 (b) shows the test in which the temperature drop took place gradually over the duration of the 4 hours, or slowly loaded.

For the rate of loading test, it was necessary to compare both sealed and unsealed samples at each of the loading rates. Alongside this test, specimens (both sealed and unsealed) were placed in a container with room temperature chloride solution and set aside until the completion of the cycling tests. This was used to look at the effects of altering solution properties.

Results. A plot of the chloride concentration vs. depth for the loading rate test can be seen in Figure 4.6 (a) and further analyzed in Figure 4.6 (b) and (c). It can be seen in both the sealed and unsealed samples, Figure 4.6 (c), that the chloride concentration is higher when the samples loaded at a slower rate. It is also interesting to note that the samples that were cyclically loaded had further chloride penetration than those that were absorbing solution at a constant temperature.

It can be seen in Figure 4.6 (b) that the samples in a room temperature environment do not contain as many chlorides as the cycled samples. This seems counter-intuitive since as discussed in Chapter 2 as the temperature decreases the viscosity (resistance to flow) of the solution increases and the diffusion coefficient would be decreased. What is aiding in the absorption of chlorides during the freezing and thawing cycles?

The internal temperature of the sample drops to a point in which freezing of capillary water occurs. At this temperature the chloride solution has not frozen and an osmotic pressure draws the chloride solution (still liquidous) further into the sample. This is part of an ongoing study being conducted by Wes Jones, Dale Bentz, and Jason Weiss.

4.4.3 Cycle Testing

Now that the cycles’ length, minimum and maximum temperature of the cycles, and rate at which the

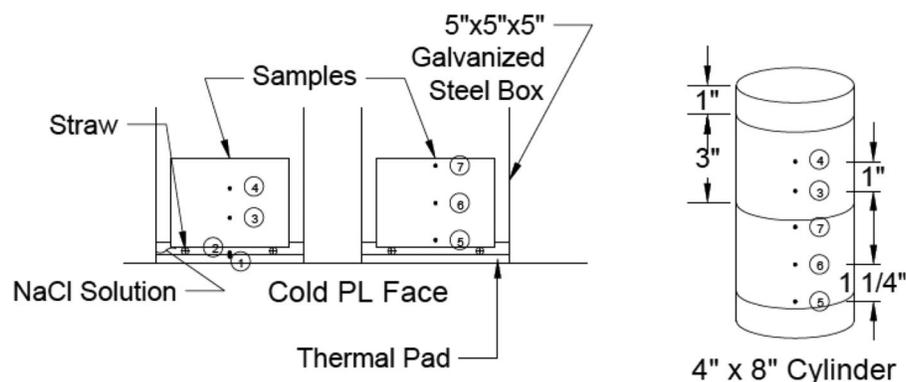


Figure 4.3 Section view of the test setup (left) and the locations of the thermocouple wiring in the cylinder (right).

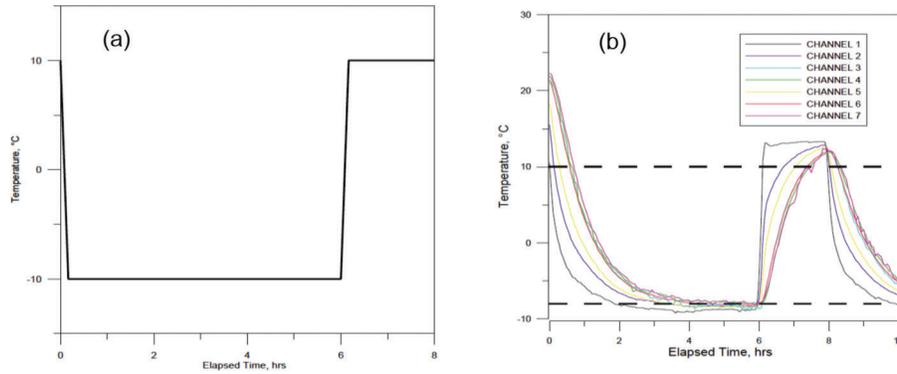


Figure 4.4 (a) Cycling of cold plate for temperature testing and (b) temperature output used to determine cycle lengths.

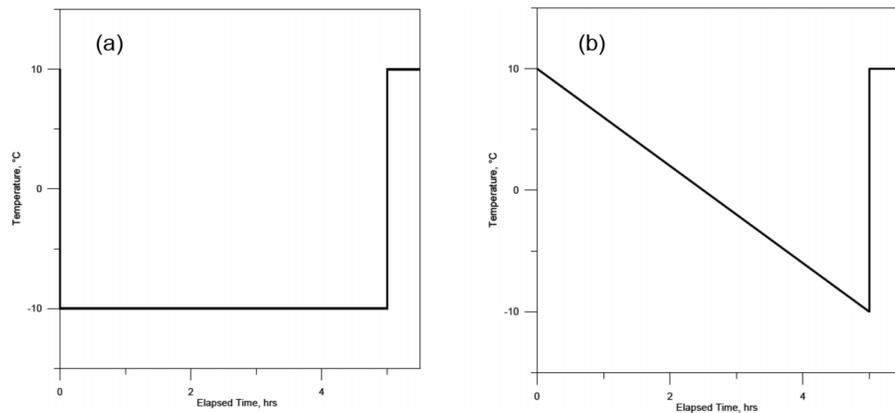


Figure 4.5 Cycling for determination of the freezing rate (a) shows “quick” loading and (b) shows “slow” loading.

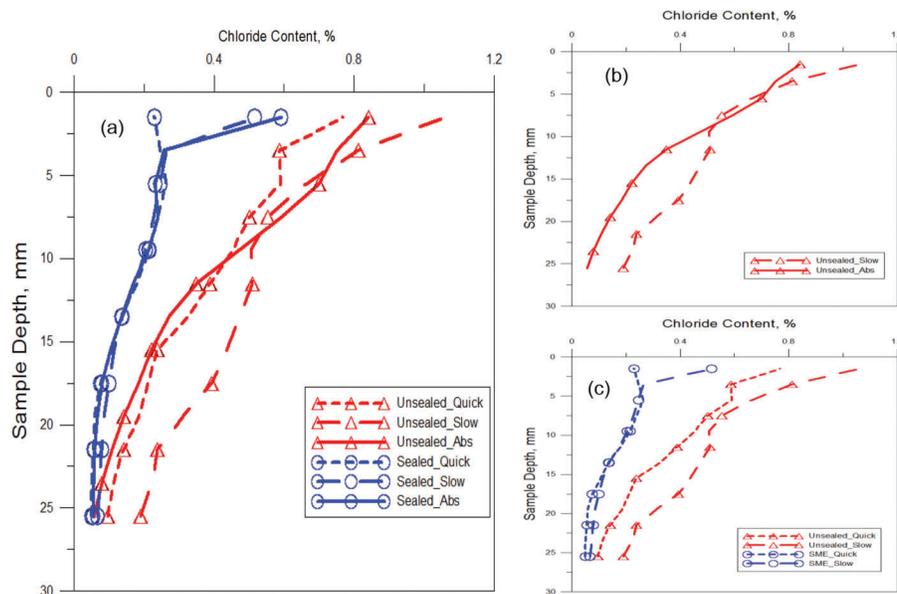


Figure 4.6 (a) Compares chloride concentration for sealed and unsealed samples at different loading rates, (b) demonstrates chloride pumping, and (c) is evidence that “slow” loading causes a higher concentration of chlorides further into the sample than quick loading.

temperatures are changing have been determined, it is desired to know how many cycles the samples should run through. In order to do so, sealed samples with a high degree of saturation were tested at 10, 20, 30, 40, 50, 60 and 90 cycles. Only sealed samples were chosen because it was desired to know if any breakdown of the “barrier” provided by the sealant would occur. A high degree of saturation was used to see if there was a drastic change in chloride concentration as the number of cycles increased.

Through visual evidence it was noticed that no breakdown of the sealant occurred. This is concluded since maximum chloride penetration in each sample, regardless the number of cycles was approximately the same depth. It is also interesting to note than no scaling of the sample face was visually noticed, even at 90 cycles (i.e., no flaking of concrete was found in the bottom of the pans, as would happen in an ASTM C666—Freeze and Thaw test). For practicality purposes, 35 cycles per test was chosen. Running 35 cycles at 5 hours a cycle will allow a single week of testing.

4.4.4 Solution Testing

This round of testing was used to determine the concentration of sodium chloride to be used in the standard testing procedure. The plot seen in Figure 4.7 shows that as the concentration of the solution increases, the chloride content throughout the sample increases. Although a lower concentration could theoretically penetrate further into the sample due to slightly lower viscosity, this was not seen here. For the purposes of our testing, a nearly saturated sodium chloride solution was used. This is not to say that other chloride solutions and concentrations couldn't be considered, however further

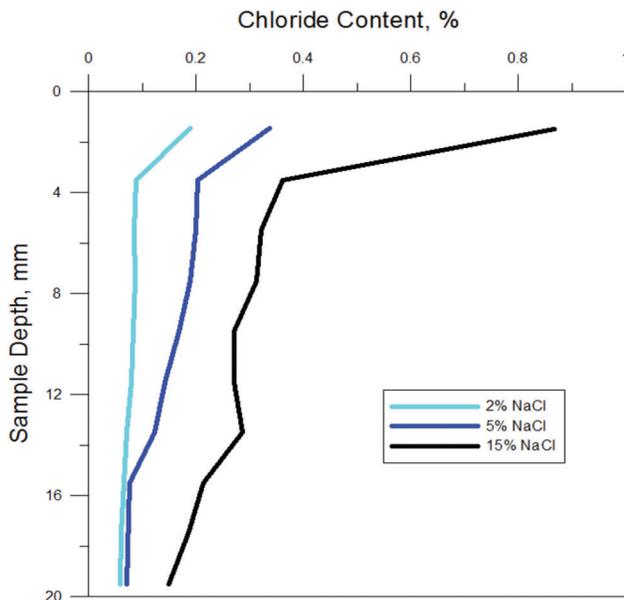


Figure 4.7 Chloride profile for solutions containing 2%, 5%, and 15% NaCl by mass.

studies may need to be done on keeping the desired concentration throughout the test, i.e., how often to flush the system. It should also be noted that a concentration that allows the chloride solution to freeze should not be permitted.

4.5 Protocol for Sealant Testing

4.5.1 Sample Conditioning

Sample conditioning plays a major role in the absorption and rate of absorption in unsaturated hydraulic cement concretes (Castro, Bentz, & Weiss, 2011). Absorption in concrete is generally described as the ability to take in water by means of capillary suction. A large fraction of concrete in service is only partly saturated and the initial ingress of water and dissolved salts is influenced, at least in part, by capillary absorption (Hearn, Hooton, & Mills, 1994). Absorption has been used as an important factor for quantifying the durability of cementitious systems (Fagerlund, 1996; Hearn et al., 1994; Hooton et al., 1993; Neithalath, 2006; Parrot, 1992). Therefore, conditioning must be given proper focus in this study.

4.5.2 Uniform Application/Application Rate of Sealant

Obtaining a uniformly consistent application of sealers, pore blockers, and water repellants is a necessity when trying to compare one manufacturer's product to another. In order accomplish uniform consistency a “flat-board” device in which the concrete samples could sit flush was created. This can be seen in Figure 4.8 (polystyrene is shown here; however, since some of the sealants are solvents, a non-degradable base is a better choice).

Application rate plays a role in the effectiveness of the sealant. Aside from the use of the flat-board, a light sprayer was used to apply each of the sealants. Each sealer was applied using an application rate of 125 ft²/gal, which fell within each manufacturer's recommendation for concrete applications.

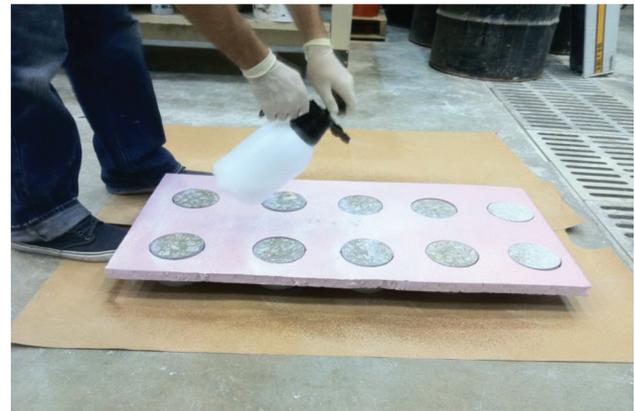


Figure 4.8 Uniform application of sealants to cylindrical concrete samples.

4.5.3 Setup/Cycling

Conditioning for this portion of the sealant testing was conducted by obtaining mass equilibrium in an over-dry condition. Samples were placed in a Shel Lab vacuum oven at 685 mm Hg at $50 \pm 1^\circ\text{C}$ to minimize damaging the microstructure. Sealant application in such a = dry state is not recommended for future testing as it creates an unrealistic concrete surface for which the sealant is applied.

After gathering data from the preliminary testing a protocol for testing various sealants has been developed. When the samples have been conditioned and sealed (this will vary based on what is being examined) the testing procedure will use cycling similar to that shown in Figure 4.5 (a), with $4\frac{1}{4}$ hours freezing at $-10 \pm 2^\circ\text{C}$ and $\frac{3}{4}$ hours thawing at $+10 \pm 2^\circ\text{C}$. The solution used for sealant testing will be a 23% NaCl (sodium chloride) by mass. Specimen dimensions are 2" in height by 4" in diameter. The samples will go through 35 cycles, which will take approximately 1 week per test. Quantitative measurements consist of massing the samples at cycles 1, 2, 5, and every 5 cycles until the conclusion of testing. Also, layered grinding and chloride titration will be completed to create a chloride profile.

4.6 Sealant Testing Results

4.6.1 Sealants Used

The use of silane, siloxane, and silicon sealants are common in today's construction industry (Wehrle, 2010). It has been shown that silicones are not an effective sealant in reducing both chloride and water absorption (Klieger & Perenchio, 1963; Ryell & Chonjacki, 1970) and the only approved siloxane sealant on the Indiana Department of Transportations (INDOT) approved sealant list is no longer in production due to high volatile organic compound (VOC) content.

The sealants chosen for testing in this part of the study were two silane-based sealants, with differing percentages of alkylalkoxysilane (20% and 100%), along with soy methyl ester (SME) containing 5% polystyrene. The product data sheets for the silane sealants can be found in the appendix.

4.6.2 Penetration Depth

It was desired to test the penetration depth of each sealant. To do this, the effectiveness of the sealant will be investigated at depths of 0, 1, 2, and 5 millimeters below the surface to which the sealant was applied. The surfaces were removed to the desired depth after the application of the sealant and before testing. It is important that the sample top is flush when the sealant is applied and when the top surface is being removed.

4.6.3 Evaluation of Sealants

Figure 4.9 shows four plots, (a), (b), and (c) represent the chloride content of each of the tested sealants in the

setup described in Section 4.5.3. Figure 4.9 (d) is a chloride profile summary showing all three sealants without any top surface removed. Top surfaces were removed at 1mm, 2mm, and 5mm before testing, as seen in the three figures. The plots labeled '0mm' are samples in which no surface was removed. The plots labeled 'ABS' are samples in which no surface was removed and were placed in a room temperature chloride solution for the duration of the cycles. The chloride profile for each sample starts at the surface exposed to chloride, not at the original surface that was removed.

4.6.4 Discussion of Results

The "tails" seen at the top of the chloride profile plots represent sealant present in the powder for titration at that depth. The reason many chlorides are not present at the surfaces is that much of the pore volume is filled by the sealant and not chloride solution. The reason for the plot lines not reaching 0% chlorides can be attributed to background chlorides that are present in the sample before testing. Plot lines that do not approach zero are due to samples that have absorbed chlorides through the entire depth.

Also, it should be noted that the previously mentioned phenomenon of chloride pumping is not noticed in these samples (see unsealed absorption vs cycled plots) because capillary water is not present in these samples. It may seem apparent that chloride pumping is happening in the sealed samples, but this is simply due to the effect of freezing and thawing on the sample.

From the plots in Figure 4.9 (a), it can be concluded that the 100% silane sealant performs the best of the three sealants tested. Since the red line indicating 5mm from the face of the sealants application has prevented fewer chlorides than the unsealed sample, the 100% silane sealant is effective at preventing chlorides at a depth of 5mm. The plot showing the 20% silane sealant, Figure 4.9 (b), indicates that the sealant is more or less ineffective at a depth of 2mm and beyond, although there is a "tail" present on the 2mm sample (meaning there is still a sealant present at this depth).

It can be seen that both the 20% and 100% silane sealants perform better than SME-PS, Figure 4.9 (c). Since these samples are oven dry, it has been noticed that at the low dosage rate the SME-PS was too easily absorbed. The SME-PS appears to require a higher dosage rate or some amount of capillary water to keep the sealant near the top surface of the sample. Since oven-dry conditions were used, SME-PS was absorbed into the sample instead of blocking the pores at the surface. Traces of SME-PS were found in the sample, through titration as notice in the "tail" of the chloride profile, at depths of up to 11mm. This indicates that the sealant was not held at the surface.

Figure 4.9 (d) shows a plot of all three sealants with the originally sealed surface. It can easily be determined which sealants performed best for this particular test. All three sealants prevented chlorides from being absorbed into the sample.

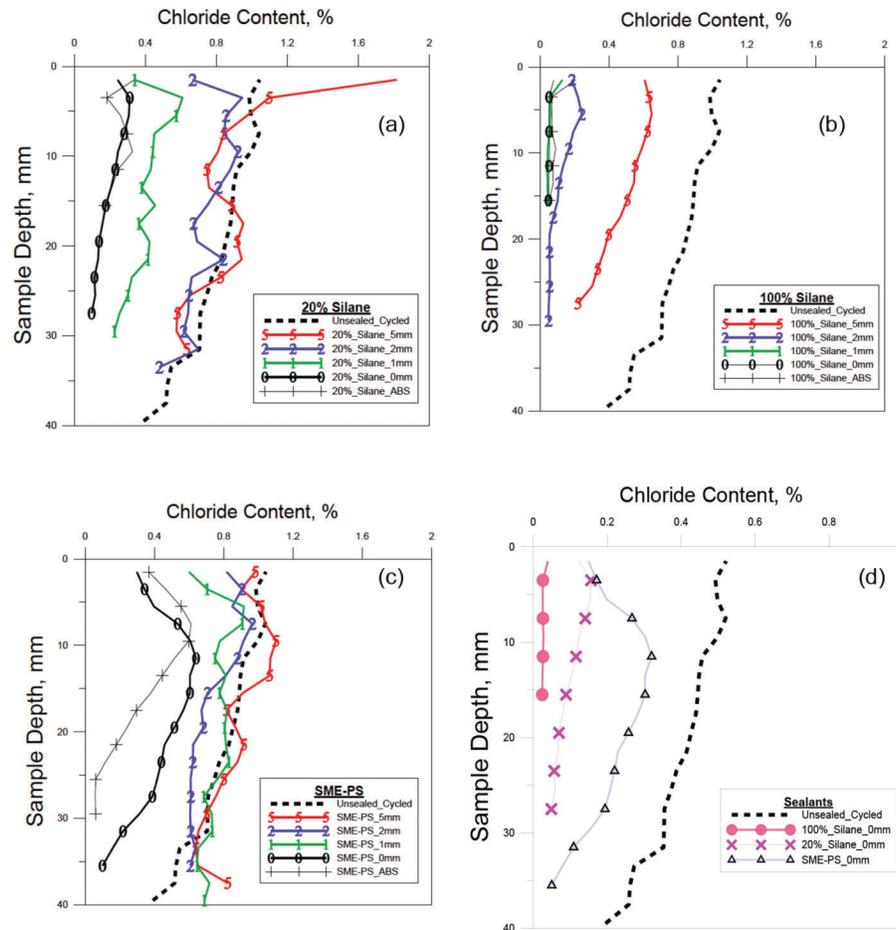


Figure 4.9 (a) Chloride profile for the 20% silane sealant used in this study, (b) chloride profile for the 100% silane sealant used in this study, (c) chloride profile for SME-PS (5% by mass) sealant used in this study, and (d) summary showing all three sealants without removal of the top surface.

4.7 Summary and Conclusions

Premature deterioration of concrete pavement joints is an issue of concern. One method that has been found effective in reducing the amount of chloride ingress into the concrete matrix is the use of sealers, pore blocker, and water repelling materials. The objective of this study was to develop a testing procedure to assess the impact of sealers, pore blockers, and water repelling materials to delay or prevent chloride solution ingress. Preliminary testing has been performed to develop a methodology, which closely replicates concrete's field exposure to fluids containing salts. Chloride pumping has been found to expedite the absorption of chlorides in concrete that are exposed to freezing conditions. Uniform application is important when comparing various sealants. Sample conditioning cannot only have a large effect on the absorption/absorption rate of concrete, but also on the application of the sealant. More testing should be done to apply this approach to samples that are conditioned to more closely represent the moisture conditions from the field. It is important

to follow manufacturer's recommendation for SME application.

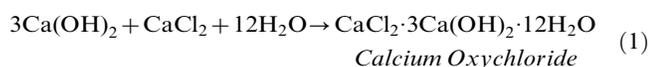
5. LOW TEMPERATURE-DIFFERENTIAL SCANNING CALORIMETRY TESTS ON PLAIN AND SME-PS-TREATED MORTAR SAMPLES

5.1 Introduction

Deicing salts can chemically and physically interact with ordinary portland cement (OPC) based materials resulting in damage in concrete elements (Farnam, Todak, Spragg, & Weiss, 2015; Farnam, Wiese, et al., 2015; Farnam, Bentz, Hampton, et al., 2014; Farnam, Bentz, Sakulich, et al., 2014; Farnam, Dick, et al., 2015; Qian, Farnam, & Weiss, 2014). The physical effects of deicers on concrete occur during the temperature reduction associated with the freeze-thaw cycle where the freezing creates hydraulic pressure (Beaudoin & MacInnis, 1974; Powers, 1945), osmotic pressure (Mehta, 1980; Mehta & Monteiro, 2005; Scherer, 1999; Valenza & Scherer, 2007), crystallization pressure (Scherer, 1999, 2004), and can

result in development of stress, cracking and spalling. The chemical effects include the detrimental reactions between the deicers and the cement paste (Colleparidi, Coppola, & Pistolesi, 1994; Farnam, Todak, et al., 2015; Farnam, Wiese, et al., 2015; Farnam, Bentz, Hampton, et al., 2014; Farnam, Bentz, Sakulich, et al., 2014; Farnam, Dick, et al., 2015; Lawrence, 1984; Peterson, Julio-Betancourt, Sutter, Hooton, & Johnston, 2013; Qian, Farnam, & Weiss, 2014; Sutter et al. 2006; Sutter et al., 2008), increase in the aggregate-cement reactions (Dubberke & Marks, 1985), or corrosion when reinforcing steel is present in the concrete (Shi et al., 2010; Wallbank, 1989).

In systems containing calcium hydroxide, the formation of calcium oxychloride have been reported very destructive (Farnam, Dick, et al., 2015; Sutter et al., 2008). It derives from the reaction between calcium chloride and calcium hydroxide (portlandite) in presence of water as shown in Equation 1. Calcium oxychloride formation generally results in a large volume structure which is believed very deleterious for the integrity of the cementitious materials. The stability of calcium oxychloride appears very easily altered by changes in temperature and moisture content.



Recent studies by Farnam, Dick, et al. (2015) have suggested that the formation of calcium oxychloride is a chemical phase transition that can be characterized using low temperature differential scanning calorimetry (LT-DSC). Results obtained with these test methods showed that the temperature at which calcium oxychloride forms in a conventional mortar resulted above 0° C and is primarily dependent on the calcium chloride concentration. At high salt concentrations (>15%) the damage caused by the formation of calcium oxychloride is considerable and occurs even if concrete does not experience freeze-thaw cycles (Farnam, Dick, et al., 2015). SME may be beneficial in mitigating damage due to calcium oxychloride formation. It is hypothesized that SME can seal the concrete preventing CaCl₂ solution in concrete. Therefore, there may be no CaCl₂ in pore solution that can react with calcium hydroxide and create damage in OPC based materials.

5.2 Sample Preparation and Testing Procedure

Two types of sample (cylinders 5 ± 1 mm in diameter and 2.5 ± 0.5 mm in height) were prepared: (1) plain mortar sample, (2) mortar sample immersed in 2% SME for 1 hr. 29.8% CaCl₂ was chosen because it is known as a very destructive deicing salt to cementitious binder. They were then immersed in 29.8% CaCl₂ solution for 2 hr and tested after 0, 1, and 7 days while the samples were sealed inside a hermetic stainless pan. A low temperature—differential scanning calorimeter (LT-DSC) was used to perform a calorimetry study to assess the deleterious phases that form when CaCl₂ and

binders react at different temperatures (Farnam, Bentz, Hampton, et al., 2014; Villani, Farnam, Washington, Jain, & Weiss, in press). The LT-DSC has a temperature accuracy of ± 0.1°C and calorimetric precision of 0.05%. During the test the temperature cycles ranged from 25°C to -90°C in the cooling path and from -90°C to 70°C in the heating ramp at a rate of a 4°C/min. The heating ramp was used to determine the endothermic behaviors associate with phase changes in the mortar samples exposed to CaCl₂ solution.

5.3 Results

In a plain mortar sample without SME treatment, it is expected that CaCl₂ reacts with calcium hydroxide from the binder and produce calcium oxychloride which is expansive and can cause degradation in concrete. Figure 2.19 shows the LT-DSC result for samples exposed to 29.8% CaCl₂ solutions. Three endothermic peaks can be seen for samples saturated with CaCl₂ solution associated with (1) eutectic solid melting at ~-50.8°C, (2) ice melting at a temperature between -50.8°C and 0°C, (3) calcium oxychloride melting (shown as Ca-Oxy in Figure 5.1) at a temperature above 0°C.

While samples with SME showed no peak corresponded to calcium oxychloride formation, a considerable amount of calcium oxychloride was seen for plain mortar. This is mainly due to the fact that SME can seal mortar and prevent CaCl₂ penetration into the mortar sample. Therefore, no reaction can occur since there may be no CaCl₂ solution in mortar sample with SME. SME was very effective as a sealant to prevent calcium oxychloride formation and can substantially decrease the damage development in concrete pavement exposed to CaCl₂ deicing salt if it is applied carefully on the surface of the pavement. While longer term tests are warranted, this is a very positive result suggesting the benefit of the SME-PS sealer on mitigating the potential reaction by keeping water (and salt water) from entering the cement paste at a rapid rate and reacting with the calcium hydroxide to result in the formation of an expansive and damaging calcium oxychloride.

5.4 Summary and Conclusions

Some concrete pavements in several Midwestern states have shown premature deterioration at the joints. It has been proposed that this can be attributed to two primary factors: increased fluid saturation and a chemical reaction that occurs between deicing salts and the cement matrix. This chapter uses a low temperature—differential scanning calorimeter (LT-DSC) to quantify the reaction that occurs to form calcium oxychloride. Calcium oxychloride is very destructive to the concrete pavement. SME-PS sealers was observed to be very effective in reducing the interaction between CaCl₂ deicing salt and cementitious matrix to form calcium oxychloride.

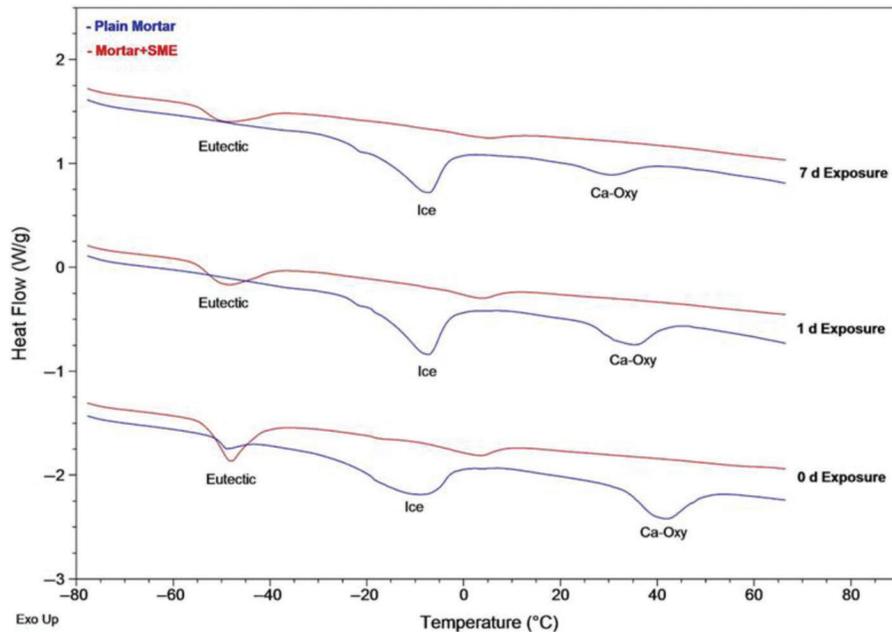


Figure 5.1 Low temperature differential scanning calorimetry (LT-DSC) for plain mortar sample and mortar sample with SME at different exposure times to 29.8% CaCl₂ solution (calcium oxychloride is shown as Ca-Oxy in the figure).

SME-PS can also be beneficial in reducing the fluid saturation in concrete pavements.

6. SUMMARY

Concrete pavements represent a large portion of the transportation infrastructure. While the vast majority of concrete pavements provide excellent long-term performance, a portion of these pavements have recently shown premature joint deterioration. Substantial interest has developed in understanding why premature joint deterioration is being observed in jointed portland cement concrete pavements (PCCP) and developing methods to mitigate this damage. It is the hypothesis of this work that the use of soy methyl ester-polystyrene blends (SME-PS) can limit water and salt ingress thereby reducing damage due to classic freeze-thaw damage or due to salt-matrix interaction.

This report discusses the potential of SME-PS seal the concrete surface and extend the service life of concrete pavements by limiting the ingress of salt solutions. SME-PS has been found to reduce salt ingress and freeze-thaw damage in the laboratory when properly applied. It is important to follow the manufacturer's specification when using SME-PS as a concrete sealer. The report also discusses field application of the SME-PS and the performance of this application after 3 to 4 years. The results from the field application of SME-PS were evaluated using samples taken from two locations, US-231 just south of Lafayette, and 126th Street in Fishers. Cores treated with SME-PS were compared with untreated sections and sections sealed with the typical filler and backer rod. The SME-PS appeared to work well in reducing the chloride ingress.

A 20% difference was observed between samples that the joint was sealed with a full depth conventional joint sealer and the joints where the concrete was sealed with SME-PS however this is hypothesized to not cause damage to the concrete since the inner walls of the pores in the paste are coated with SME. The sections where the concrete joint was left exposed without any treatment (i.e., no joint sealant or concrete sealant) showed substantially more chloride ingress than the conventional joint sealer or the concrete sealer.

The report also included data from LT-DSC experiments comparing the results of SME-PS-treated mortar with plain mortar. In a plain mortar sample without SME treatment, the CaCl₂ reacts with calcium hydroxide from the binder and produce calcium oxychloride which is expansive and can cause degradation in concrete. The samples with SME showed no peak corresponded to calcium oxychloride formation despite a considerable amount of calcium oxychloride being observed for plain mortar. This is mainly due to the fact that SME can limit the penetration of CaCl₂ solution in the mortar sample. By reducing the penetration of the CaCl₂ solution in mortar sample with SME the potential reaction with calcium hydroxide to form calcium oxychloride formation is reduced. While longer term tests are warranted, this is a very positive result suggesting the benefit of the SME-PS sealer on mitigating the potential reaction by keeping water (and salt water) from entering the cement paste at a rapid rate and reacting with the calcium hydroxide to result in the formation of an expansive and damaging calcium oxychloride. While still preliminary, this approach has the ability to shed light on how these sealers may influence the performance at joints, especially when the temperature is cycled.

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About the Joint Transportation Research Program (JTRP)

On March 11, 1937, the Indiana Legislature passed an act which authorized the Indiana State Highway Commission to cooperate with and assist Purdue University in developing the best methods of improving and maintaining the highways of the state and the respective counties thereof. That collaborative effort was called the Joint Highway Research Project (JHRP). In 1997 the collaborative venture was renamed as the Joint Transportation Research Program (JTRP) to reflect the state and national efforts to integrate the management and operation of various transportation modes.

The first studies of JHRP were concerned with Test Road No. 1—evaluation of the weathering characteristics of stabilized materials. After World War II, the JHRP program grew substantially and was regularly producing technical reports. Over 1,500 technical reports are now available, published as part of the JHRP and subsequently JTRP collaborative venture between Purdue University and what is now the Indiana Department of Transportation.

Free online access to all reports is provided through a unique collaboration between JTRP and Purdue Libraries. These are available at: <http://docs.lib.purdue.edu/jtrp>

Further information about JTRP and its current research program is available at: <http://www.purdue.edu/jtrp>

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