Evaluation of Penetrating Sealers for Concrete

Final Report
June 2022

National Concrete Pavement Technology Center

IOWA STATE UNIVERSITY
Institute for Transportation

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16. Abstract

The main factors negatively impacting concrete durability in Iowa are governed by moisture saturation and deleterious chloride ion reactions from deicing salts. Penetrating sealers are a class of materials that show promise for increasing concrete durability by reducing moisture and chloride ion penetration. Specifically, extending time to critical saturation reduces freeze-thaw exposure conditions, and limiting chloride ingress reduces the potential for oxychloride formation.

Numerous products exist in the sealer marketplace with a range of performance claims. Sealers can be categorized based on their chemical structure, functional type, amount of active chemical, carrier solution, and action mechanism, and they vary widely in performance. In addition, the parameters to define satisfactory performance are not agreed upon.

Although penetrating sealers show great promise for improving concrete durability, significant questions need to be answered before departments of transportation (DOTs) and other concrete pavement owners can be assured of a positive cost-benefit. Lack of clear performance guidelines hinder potential beneficial use given that design engineers, contractors, and owner agencies are unable to evaluate or compare performance of the available products.

The researchers reviewed a number of test methods for assessing penetrating sealants using a wide range of available products under several failure modes. Guidance was developed on how the action of the sealant should be tied to the potential failure mechanism, and which tests should be conducted on new products to ensure that they meet the needs.

This guidance can be used by agencies when reviewing the products available to them for a range of applications.

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EVALUATION OF PENETRATING SEALERS FOR CONCRETE

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

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EXECUTIVE SUMMARY

The use of sealers can be considered an effective means for enhancing the longevity of concrete slabs. However, with the growing interest in the use of concrete sealers, the number of sealer manufacturers, types of sealers, and commercial products available has increased. This has resulted in issues for owner agencies that lack the robust tools to evaluate sealer products.

The evidence is clear that sealers can help improve the longevity of concrete surfaces, and that they can, to an extent, compensate for mixtures at risk for premature deterioration.

A limited number of chemical families are available, and they act in different ways on the concrete mixtures. Owner agencies need to tie the action of the product with the performance needed.

The results of this research include a suite of test methods that are recommended for assessing sealant products to mitigate likely concrete durability distress mechanisms. This guidance can be used by agencies when reviewing the products available to them for a range of applications.

INTRODUCTION

Problem Statement

The main factors negatively impacting concrete joint durability in Iowa are governed by moisture saturation and deleterious chloride ion reactions from de-icing salts (Taylor et al. 2016, Weiss et al. 2016). Penetrating sealers are a class of materials that show promise for increasing concrete durability by reducing moisture and chloride ion penetration. More specifically, extending the time to critical saturation reduces freeze-thaw exposure conditions, and limiting chloride ingress reduces the potential for oxychloride formation.

Numerous products exist in the sealer marketplace with a range of performance claims. Lack of clear performance guidelines hinder potential beneficial use of the products because design engineers, contractors, and owner agencies aren't able to evaluate or compare the products. However, sealers can be categorized based on their chemical structure, functional type, amount of active chemical, the carrier solution, and the action mechanism. In addition, the parameters to define satisfactory performance are not agreed upon. Therefore, this research was undertaken to help address the need to establish a suite of performance criteria to define acceptability of a given product and a protocol for agencies to use in evaluating a product submitted for approval.

Objectives

Although penetrating sealers show great promise for improving joint and concrete durability, significant questions must be answered before departments of transportation (DOTs) and other concrete pavement owners can be assured of a positive cost-benefit. The objectives of this research included the following:

- Evaluate recommended protocols and standards for testing the penetrating sealers with a focus on characteristics affecting the joints subjected to chemical attacks and freeze-thaw actions

- Select, modify, or update the most appropriate testing protocols to reflect the performance of penetrating sealers

- Examine the selected sealer performance in the laboratory and extend the findings to field implementation projects in urban and/or rural areas where joint performance can be monitored long term

- Develop guidelines and protocols for investigating the short- and long-term performance of the sealers used to enhance joint durability

Background

Durability of Concrete Pavements

Concrete is a critical component in providing a reliable transportation infrastructure. Concrete pavements have a large amount of exposed surface area, which accentuates durability-related distresses, compared to other structural members. In cold regions, the distress that stems from cycles of frost action is most pronounced at the sawn joints (Arribas-Colon et al. 2012, Sutter et al. 2006, Panchmatia et al. 2014). Deterioration associated with concrete joints consumes a disproportionate amount of time, effort, and cost in maintenance and preservation activities.

Concrete pavement owners extend pavement life through a combination of preventive maintenance and localized repair prior to end-of-life activities. The use of sealers as surface treatments has the potential to be a useful methodology in this work because sealers can reduce moisture and chloride ion penetration (de Vries and Polder 1997, Basheer and Cleland 2006, Freitag and Bruce 2010, Vipulanandan et al. 2011, Christodoulou et al. 2013, Liu and Hansen 2016).

Concrete is a porous material in which the size, distribution, and connectivity of the pores largely depend on the mixture ingredients and proportions, including the type and content of the binder, the water-to-cementitious materials (w/cm) ratio, the quality of consolidation, and the degree of hydration. Interconnected voids in the paste micro-structure can be penetrated by moisture, gas, and ionic solutions, potentially promoting corrosion of embedded steel, the alkali-silica reaction, sulfate attack, freeze-thaw (F-T) damage, and scaling. The amount and rate of damage can therefore be reduced by reducing moisture, gas, and ion penetration by reducing the number and magnitude of interconnected voids (Kosmatka and Wilson 2016).

Use of supplementary cementitious materials (SCMs), high-quality aggregates, water-reducing agents (WRAs), and air entrainment are all considered able to improve long-term durability of concrete in harsh exposure environments and to enhance the service life of the concrete elements. However, such approaches may not be sufficient for the most severe exposure situations. Likewise, existing pavements that are underperforming may need treatment to extend their lifetimes.

Pavement joints are the most common location for premature failure, even in cases where the bulk of the pavement exhibits sufficient durability against frost action and deicing salt scaling. Joints in a rigid pavement can act as local reservoirs, trapping deicing salts and moisture. Exposing the interfacial transition zone (ITZ) by sawing also accelerates attack mechanisms due to the high porosity and permeability of the zone (Maso 1996, Cwirzen and Penttala 2005, Fu et al. 2020). Due to improper drainage or inactivated joints, water and contaminants can be trapped in the kerf, accelerating saturation and chemical attack (Fagerlund 1973, 2004). Whether deterioration is caused purely by water or dissolved salts, keeping these materials out of concrete is necessary for enhanced durability.

A research report by the National Concrete Pavement Technology (CP Tech) Center at Iowa State University (ISU) sought to understand the mechanisms behind premature joint deterioration and, based on this understanding, develop training materials and guidance documents to help practitioners reduce the risk of further distress and provide guidelines for repair techniques (Taylor et al. 2016). This work included activities at Purdue University and Michigan Technological University. The following physical and chemical mechanisms are among the ones that cause joint deterioration (Spragg et al. 2011, Sutter et al. 2008, Taylor et al. 2012, Taylor et al. 2016, Weiss et al. 2016):

- **Paste deterioration due to chemical attack**: Formation of expansive oxychloride, which consequently causes cracking at regular intervals parallel to the saw cut and/or the loss of paste, thus leaving clean aggregates in the voids.
- **Saturated frost damage**: The expansion of water in the saturated capillaries of the concrete as it freezes causes cracking. Cycles of F-T promote crack propagation allowing more water to penetrate, and, as a result, the concrete incrementally deteriorates. Concretes that are highly saturated are prone to accelerated damage.
- **Mechanical damage**: Joint damage can occur from stresses caused by incompressible materials (e.g., sand, rocks, other debris) trapped in the joint. Raveling of a saw cut may also be caused by aggregate particles being dislodged during sawing, typically because the concrete strength is too low when sawing.
- **Durability cracking**: Expansive freezing of water trapped inside some types of aggregate particles leads to damage that normally starts near joints and forms a characteristic D-shaped crack pattern.
- **Early-age drying damage**: High evaporation rates soon after placement result in large differences in moisture content through the depth of the concrete slab. These differences may lead to stresses great enough to cause fine horizontal cracks and delamination. In areas where the horizontal cracks intersect with vertical cracks or joints, concrete material can break free and “flat bottom,” or delamination spalling can occur.

The following solutions have been proposed to reduce the risk of concrete pavement distress:

- Lower the w/cm ratio (i.e., to about 0.40 to 0.42) to reduce permeability
- Use SCMs to maintain high Si/Ca ratio that is more resistant to oxychloride formation
- Optimize the construction techniques and enhance the stability of the air-void system to secure an adequate air-void system behind the paver
- Provide an adequate drainage system beneath the concrete
- Limit the use of aggressive salts, especially magnesium and calcium salts, to times and temperatures (typically < 15°F) when necessary for safety
- Use penetrating sealers to slow water and salt penetration into the microstructure

A challenge with the last solution listed is the lack of guidance available on which products to use. The aim of the work reported here is to help provide that guidance.

The joint deterioration mechanisms outlined above are primarily influenced by the penetration of fluids into the paste. The ingress of moisture and ions responsible for concrete deterioration occurs through three main mechanisms: diffusion, permeation, and capillary suction (Kosmatka and Wilson 2016). While acting together under natural conditions, capillary suction tends to be the governing mechanism in many cases (Christodoulou et al. 2013). Sealers and hydrophobic pore liners may therefore be effective methods to reduce moisture transport into the concrete by increasing the time to reach critical saturation (Pfeifer and Scali 1981). Moreover, the sealers can reduce the rate of chloride ion penetration into the concrete, thus reducing the risk of chemical attack, such as the formation of oxychloride.

The challenges are to prove that a given product is effective and stable and to provide guidelines on when and where the products should be applied. The sealer families listed in Table 1 are among those most commonly used ones.

Table 1. Most popular sealer families and their mechanisms of action

Sealer Family	Mechanism of Action	Description
Silanes, siloxane, and siliconates	Water repellent	Silicon based, react with hydration products
Epoxies	Pore blocking or barrier coat[1]	Thermoset polymers
Gum resins and mineral gums	Pore blocking	Synthetic or natural viscous hydrocarbons
Linseed oil	Pore blocking	Vegetable oil
Stearates	Water repellent	Soaps or metallic salts from fatty acids
Acrylics	Pore blocking or barrier coat[2]	Polymers or copolymers of acrylic acid
Silicates and fluosilicates	Pore blocking	Silicon based with no organofunctional group
Urethanes and polyurethanes	Pore blocking or barrier coat[3]	Reactive resins
Polyesters	Pore blocking or barrier coat[3]	Synthetic resins
Chlorinated rubber	Pore blocking or barrier coat[4]	Chlorinated polyisoprene
Silicones	Water repellent	Silicon based with two organofunctional groups
Vinyls	Pore blocking or barrier coat[3]	Polymers of acrylic and methacrylic acid

[1] Acting as pore blocker when less than 50% active ingredient
[2] Pore blocking if solvent based; barrier coating if water based or high molecular weight
[3] Pore blocking if diluted and as barrier coat if not diluted
[4] Mainly barrier, but can be pore blocker if too diluted
Table information source: Cady 1994

Concrete sealers can be divided into three main categories based on their mechanism of action as follows:

- **Water repellents:** Penetrate into the concrete pores and coat the pore walls, rendering them hydrophobic. In turn, water and any ions it contains cannot penetrate the pores but gases and vapors can.
- **Pore blockers:** Have a viscosity sufficiently low to allow penetration into the concrete pores, sealing them while leaving little or no measurable coating on the exterior surface of the concrete.

- **Barrier coatings:** Too viscous to penetrate inside the concrete pores but form a thick barrier layer on the surface that blocks the pores. Due to the potential effects on pavement skid resistance and the fact that these easily wear off under traffic, barrier coatings were not considered within the scope of this research.

Relatively little work has been reported on the efficacy of sealants to reduce joint deterioration directly (Wiese et al. 2015), with most laboratory tests aiming to measure their influence on the following (Jones et al. 2013):

- Ion penetration
- Fluid permeability
- Water sorption
- Surface abrasion and skid resistance
- F-T resistance
- Microstructural phase changes

In addition, penetrating sealers evaluated in the laboratory are usually tested under accelerated conditions, while the efficacy of sealers applied in the field is only expected to yield results after several years (Sutter et al. 2016). To validate any testing protocol, a field investigation program is also needed to compare the results observed in the field with those from an accelerated laboratory program.

Evaluating Performance of Penetrating Sealers

There is no unique definition on how to classify penetrating sealers. Some researchers consider both water repellents and pore blockers to be penetrating sealers (Cady 1994), while other researchers consider only water repellents to be penetrants. When applied to concrete pavements, penetrating sealers generally refer to products with the ability to block moisture ingress but do not conform to the moisture loss requirements used to classify curing compounds in the industry. Most of the products marketed as penetrating sealers fall under the category of silanes, siloxanes, silicates, and siliconates (The Concrete Sealer Guy 2016).

A research study sponsored by the South Dakota Department of Transportation (SDDOT) evaluated two types of sealers for enhancing concrete pavement joint durability. The investigated sealers were a siloxane-based product and a silane-based product. The results obtained from laboratory investigation indicated the efficiency of the incorporated materials in reducing the moisture transport and the degree of damage caused by de-icing chemicals (Sutter et al. 2008).

A research project sponsored by the Iowa DOT also investigated the effect of silane- and siloxane-based sealers on chloride ion ingress into joints of existing pavements. Results obtained by the researchers indicated no significant difference between the properties of the treated and reference joints during the two-year study window (Sutter et al. 2016). Likewise, soybean oil has been shown to reduce absorption and improve deicer scaling resistance without influencing chloride ion penetration (Kevern 2010).

In research sponsored by the Oklahoma DOT (ODOT), it was observed that the performance of penetrating sealers is a time dependent phenomenon. Results obtained from investigating more than 60 bridge decks indicated no failure in silane-based sealers during the first 12 years, although almost 50% of the sealers failed after 18 years of service (Moradllo et al. 2016).

While the vast majority of penetrating sealers used for concrete are silicone-based, other products included in the previous Table 1 have shown good performance.

Another research project, which was sponsored by the Indiana DOT (INDOT) in collaboration with Purdue University investigated the effect of soy methyl ester-polystyrene (SME-PS) sealers compared to water-based and solvent-based silane sealers (Weise et al. 2015). The study concluded the use of SME-PS sealers have beneficial effects in reducing moisture transport into concrete, as well as in reducing the ingress of chloride ions sourced from de-icing salts.

Research Plan

The work for this project was undertaken in the following three phases after the literature review was completed:

- Effect of sealers on concrete durability (a wide suite of tests was conducted on a single concrete mixture treated with a range of different products)

- Effect of sealers with different modes of action on the durability of concrete susceptible to selected durability distresses

- Field applications

The Literature Review that the researchers conducted at the beginning of this project helped define the basis of this investigation. That review has been published as a standalone document on the research project page here: https://cptechcenter.org/research/in-progress/evaluation-of-penetrating-sealers-for-concrete/.

PHASE 1: EFFECT OF SEALERS ON CONCRETE DURABILITY

The objective of this phase was to investigate the effects of different sealer types as post-construction treatments applied to the vertically sawn faces of highway concrete specimens to mimic real practices in the field. Performance and durability were assessed via laboratory test procedures to investigate enhancing extra protection against cold, harsh weather conditions. A suite of tests was conducted to identify which tests provided information about the efficacy of the products being tested.

Mixture and Samples

A single concrete mixture was used based on a mixture appropriate for DOT pavement construction in Iowa. The concrete mixture had a w/cm ratio of 0.45, target slump of 4.5 in., and target air content of 6%. A binary cementitious system was employed with 70% by mass Type I portland cement and 30% Class C fly ash. Well-graded river sand was used as the fine aggregate, and crushed limestone with a nominal maximum size of 1 in. was used as the coarse aggregate. The mixture contained the following:

- 424 lb/yd^3 cement
- 141 lb/yd^3 fly ash
- 191 lb/yd^3 water
- 1,389 lb/yd^3 sand
- 1,841 lb/yd^3 coarse aggregate

The specimens that were prepared included the following:

- 6 × 12 in. cylinders for chloride ponding
- 3 × 4 × 16 in. prisms for accelerated F-T testing
- 4 × 8 in. cylinders for compressive strength, air permeability, and water absorption

All specimens were covered with plastic sheets and wet burlap for 24 hrs after casting. The samples were then demolded and sawn (where relevant) before being placed in an environmental chamber set at 72°F and 50% RH.

Table 2 summarizes the sealer compositions and labeling used.

Table 2. Identification and chemical composition of the sealers

ID	Chemical composition	Type
LS	Water based, contains silicic acid, lithium salt, potassium methylsiliconate	Surface densifier
SC	Colloidal silica solution	Surface densifier
40% Silane	Water based, contains reactive alkyltrialkoxysilane	Pore liner, hydrophobic
SoTa	Water based, contains sodium tartrate, potassium methyl siliconate	Pore blocker/liner, hydrophobic
Acrylic	Water based acrylic polymer	Barrier coating
SME	Soy methyl ester-polystyrene	Pore blocker, hydrophobic

Prior to sealer application, all of the specimens were cleaned with high pressure air to remove dust and small particles, and the sides of the specimens were taped (as shown in Figure 1 left) to prevent overspray.

Figure 1. Application of sealer (left) and curing specimens vertically (right)

The sealers were then applied on vertically oriented sawn surfaces (to mimic saw-cut faces) at 7 days of age before the specimens were then returned to the environment chamber (Figure 1 right) for 28 days. The sealers were applied at a minimum of 200 ft^2/gal with 50% or more observed to be draining off.

Experimental Methods

The following concrete mixture properties were measured:

- Slump (ASTM C143)
- Fresh and hardened air content (ASTM C231 and C457)
- Super Air Meter (SAM) number (AASHTO TP 118)

The following test methods were conducted on the control (untreated) and sealed samples.

Depth of Penetration

Cylinder samples were fractured similar to a splitting tensile strength test to expose the internal concrete without wet sawing, which could emulsify and migrate the sealers. Fractured samples were then placed in a fabric dye solution following the protocol presented by Moradllo et al. (2016).

Wettability

A 20-µL drop of deionized water was applied to each specimen, and an image was captured after 30 seconds. Images were processed, and the contact angle was determined using ImageJ software. The contact angle was measured only on cementitious paste, avoiding coarse aggregate, and a minimum of 1 in. away from each sample edge. Results represented an average of three random locations per treatment.

The contact angle results were classified as follows:

- Hydrophilic: ϴ < 30°
- Low-hydrophobic: 30° < ϴ < 90°
- Hydrophobic: 90° < ϴ < 120°
- Over-hydrophobic: 120° < ϴ < 150°
- Super-hydrophobic: ϴ > 150°

Air Permeability

Sealed discs measuring 4 in. (100 mm) in diameter and 2 in. (50 mm) in thickness were employed to determine the air permeability according to a University of Cape Town (UCT) method, as discussed by Alexander et al. (2009). The test involved applying pressurized air to the concrete discs for six hrs. Pressure decay was monitored during the test period, and the rate of decay was employed to determine diffusion parameters.

According to this test method, an increasing air permeability index (API) represents lower air permeability. The following interpretations were applied to the results:

- API>10.0 Excellent
- 9.5<API<10.0 Good
- 9.0<API<9.5 Poor
- API<9.0 Very poor

Reducing air permeability in concrete might be associated with reducing carbonation, which may depassivate protective layers in reinforced concrete and thereby accelerate steel reinforcement corrosion. Secondly, water vapor movement out of the concrete is important to allow drying.

This study focuses on the durability of concrete joints (rather than corrosion of steel reinforcement). Once relative humidity drops below 100%, water evaporates from larger pores reducing saturation of the concrete element, thereby reducing the risk of F-T distress.

Water Absorption/Degree of Saturation

One-dimensional absorption of plain water was determined according to the procedure in ASTM C1585 *Standard Test Method for Measurement of Rate of Absorption of Water by Hydraulic-Cement Concretes*, which was performed in triplicate. Measurements were taken up to 28 days to determine initial and secondary absorption.

Following completion of the absorption test, the specimens were transferred to a desiccator connected to a vacuum pump. Deionized water was used to submerge the specimens while the pump ran at 50 mm Hg (6,650 Pa) for 24 hrs. The specimens were then removed and brought to a saturated surface dry (SSD) condition before weighing them. The samples were then dried, and the degree of saturation was determined using equation (1).

$$degree\ of\ saturation\ (DOS) = \frac{M_t - M_{dry}}{M_{sat} - M_{dry}} \times 100 \quad \textbf{(1)}$$

where M_t is the mass of the specimen at time *t*, M_{dry} is the mass of the specimen's prior contact with water, and M_{sat} is the weight of the saturated specimen.

To simulate the drying process at the surface after the absorption rate test, the specimens were kept in same exposure condition, but face up this time to measure the desaturation rate in a controlled environmental chamber (72°F, 50% RH), as shown in Figure 2.

Figure 2. Desaturation of specimens in environmental chamber

In a similar manner to absorption rate measurement, the weights of the specimens were recorded to determine the rate of water evaporation from the exposed surfaces for 15 days.

Following the absorption/desorption test, the specimens were immersed in saturated calcium hydroxide to determine the degree of saturation. Specimens were still wrapped with aluminum tape while the plastic sheet and rubber bands were removed to expose both faces of the concrete discs to water. After 30 days of immersion, the weights of the specimens were recorded, and the specimens were secured with plastic sheets and rubber bands once more to measure their desaturation rates from a saturated condition. The instantaneous degree of saturation of the specimens was determined using the previous equation (1).

The sensitivity to degradation by ultraviolent (UV) light was determined by evaluating absorption before and after UV exposure. UV exposure was performed in an accelerated weathering tester (Q-Lab QUV) typically used for external sealants and coatings for the building and infrastructure industries.

Samples were exposed to 11 months (8,000 hrs) of continuous testing at 0.60 watt per square meter per nanometer ($W/m^2/nm$) irradiance or 188 W/m^2 at a wavelength of 313 nm. To better simulate summer environmental conditions in Iowa and further accelerate deterioration, the testing chamber temperature was kept at 45°C with heated water keeping the relative humidity above 50%.

A direct correlation between the number of hours under UV testing and actual field exposure is not possible because the latitude, cloud cover, locations of clouds, and spectrum of wavelengths hitting the earth all vary between locations. Exposure-time ranges for coatings and sealants fading using the ASTM D1148 *Standard Test Method for Rubber Deterioration-Heat and Ultraviolet Light Discoloration of Light-Colored Surfaces* last 96 hrs at up to 40 W/m^2. Other test methods, including ASTM C1184 *Artificial Weathering Exposure Testing of Structure Sealants,* utilize a minimum test of 5,000 hrs.

Freezing-Thawing

F-T performance was assessed using a modified ASTM C666 *Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing* variant A as reported in National Cooperative Highway Research Program (NCHRP) Report 244 (Pfeifer and Scali 1981) where freezing fluid is introduced one-dimensionally, and samples undergo two cycles per day. In this test, concrete is dried and re-saturated before testing and can be tested at any time (unlike the ASTM C666 procedure). Prior to testing, the specimens were conditioned one-dimensionally in saturated magnesium chloride (54%) for seven days using a procedure similar to ASTM C1585. A sample under testing is illustrated in Figure 3.

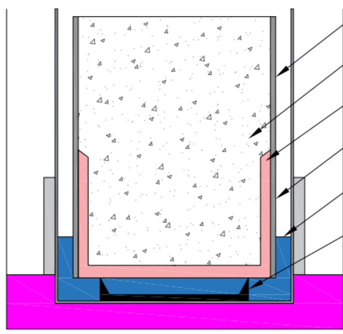


Figure 3. F-T test setup

The specimens were exposed to a 3% magnesium chloride solution that was used as the testing fluid. Relative dynamic modulus (RDM) and specimen weight were measured after each set of 14 cycles. The testing fluid was changed after each cycle set.

Chloride Penetration

Chloride penetration was measured using the standard AASHTO T 259 *Standard Method of Test for Resistance of Concrete to Chloride Ion Penetration* 91-day ponding test. Average chloride content was measured at two depths from the surface, 0.0787–0.5 in. and 0.5–1.0 in., following the AASHTO T 260 *Standard Method of Test for Sampling and Testing for Chloride Ion in Concrete and Concrete Raw Materials* procedure, followed by titration according to ASTM C1152 *Standard Test Method for Acid-Soluble Chloride in Mortar and Concrete*.

Low Temperature Differential Scanning Calorimetry (LT-DSC)

The potential for CaOXY formation in samples exposed to a 20% $CaCl_2$ salt solution at 4°C for 1 year was quantified using a differential scanning calorimeter (DSC 25) equipped with a low-temperature kit. Powder samples were crushed and sieved through a 75-µ sieve and then tested in accordance with AASHTO T 365 *Standard Method of Test for Quantifying Calcium Oxychloride Formation Potential of Cementitious Pastes Exposed to Deicing Salts*.

Results and Discussion

The slump of the fresh concrete was 3 in., while fresh and hardened air contents were 7.5% and 6%, respectively. Both were within the ACI 318 *Building Code Requirements for Reinforced Concrete* recommended range for concrete exposed to F-T cycles and salt application. The SAM number was 0.22.

Depth of Penetration

No measurable depth of penetration was observed for any of the treatments. Previous results show very small (< 5mm with most < 1 mm) depths of penetration for horizontal applications (Xiao et al. 2020), so the inability to observe penetration vertically was not surprising.

Wettability

Table 3 presents the water contact angles and classifications for concrete treated with penetrating sealers.

Table 3. Contact angle measurement on concrete mixtures

Treatment	Angle	Classification
Control	NA	Hydrophilic
LS	76°	Lower-hydrophilic
SC	74°	Lower-hydrophilic
40% Silane	105°	Hydrophobic
SoTa	120°	Hydrophobic
Acrylic	83°	Lower-hydrophilic
SME	100°	Hydrophobic

Most sealers, and particularly organic and hydrophobic sealers, possess water repellent properties. An increasing contact angle is a strong indicator that a sealer is reducing molecular attraction between water and the pores. In other words, sealers reduce the potential of capillary suction, and, with increasing water contact angle, a lower water absorption rate can be expected (Dang et al. 2014, Adil et al. 2021).

As anticipated, the untreated control water immediately flattened on the paste without exhibiting any contact angle, showing that the untreated control specimen was inherently hydrophilic. However, as shown in Figure 4, the sealer treatments demonstrated a substantial increase in their water contact angles, which would be associated with refined pore sizes in the paste matrix.

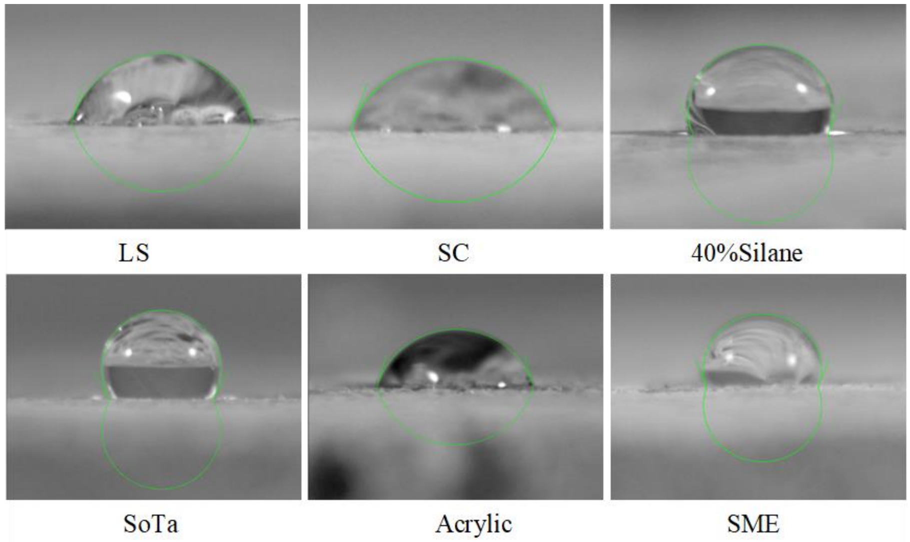


Figure 4. Contact angle measurements using ImageJ

The lower contact angle of LS and SC was expected given that surface densifiers do not inherently possess hydrophobic properties.

Air Permeability

Air permeability results are shown in Figure 5.

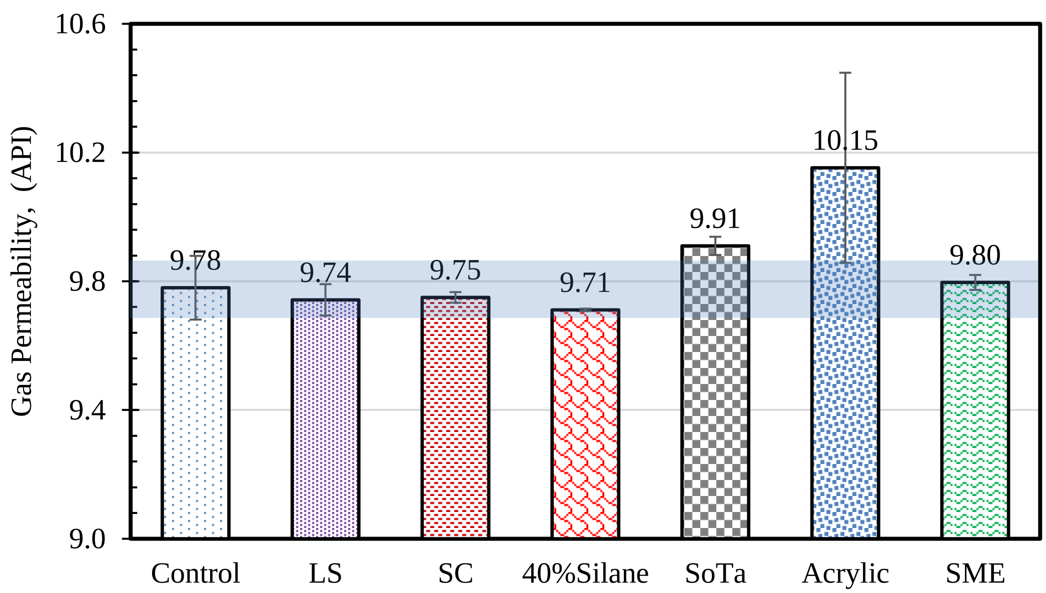


Figure 5. Air permeability results

LS, SC, and silane exhibited pore-lining behavior and did not influence air permeability. SoTa and SME-PS are pore blockers and did reduce air permeability. The acrylic is a surface coating and provided the greatest reduction in air permeability. Thus, once the concrete element is not in contact with water and %RH is lower than 100%, the vapor can leave the concrete and decrease the degree of concrete saturation, which is a vital parameter in concrete deterioration in cold regions.

Water Absorption/Degree of Saturation

Figure 6 shows the amount of water absorbed by the specimens (*i*) normalized by the cross-section area in contact with water according to ASTM C1585.

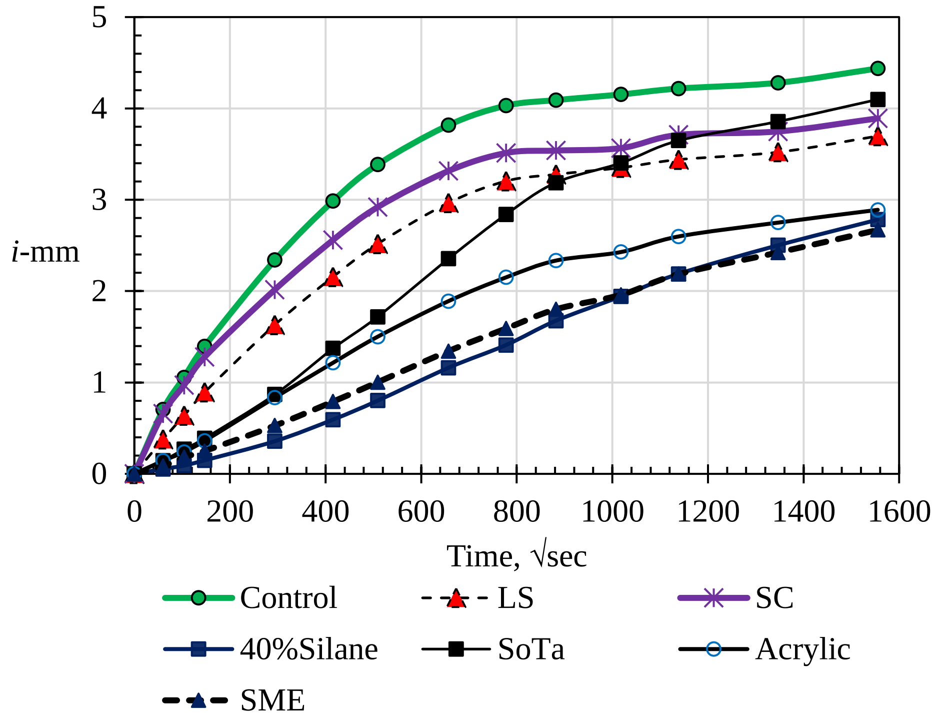


Figure 6. Moisture uptake results with time

LS and SC did not significantly reduce water ingress, and performance was similar to that of the untreated control. Application of these types of sealers cannot guarantee improved durability enhancement.

SoTa and acrylic were in the intermediate absorption group that significantly reduced absorption initially but not in the later ages. The secondary absorption of the concrete treated with SoTa was ultimately identical to that of the untreated control.

The third group included 40% silane and SME, which significantly reduced water absorption in both initial and secondary absorption. The durability performance of these sealers is expected to be good given that water absorption is strongly correlated with concrete durability (Alexander et al. 2009, Hall 1989).

The nick point separates initial absorption and secondary absorption where rapid absorption (capillary suction) stops and water transport dominated by the diffusion process begins. Little difference was observed in concrete treated with 40% silane and SME. This might be associated with minimizing the absorption rate that impedes the capillary process or the capillary and diffusion processes happening simultaneously.

According to the Young-Laplace equation, which defines the capillary suction in an unsaturated medium, the transport process is governed by a capillary force that is a function of the surface tension and contact angle of the wetting liquid, along with the capillary pore radius. Thus, by increasing the contact angle (previous Figure 4), the molecular attraction between water and the concrete can be reduced, and less water infiltrates into the concrete. This strong inverse correlation between the water contact angle and absorption capacity was observed in an unpublished paper by the authors of this report and elsewhere (Dang et al. 2014) as the following equation:

$$P = \frac{2\gamma \cos\theta}{r_c} \quad \textbf{(2)}$$

where P is a pressure difference, γ is the surface tension, θ is the contact angle, and r_c is the capillary pore radius.

Figure 7 presents the evolution of the concrete degree of saturation with time after converting the water absorption data according to equation (2).

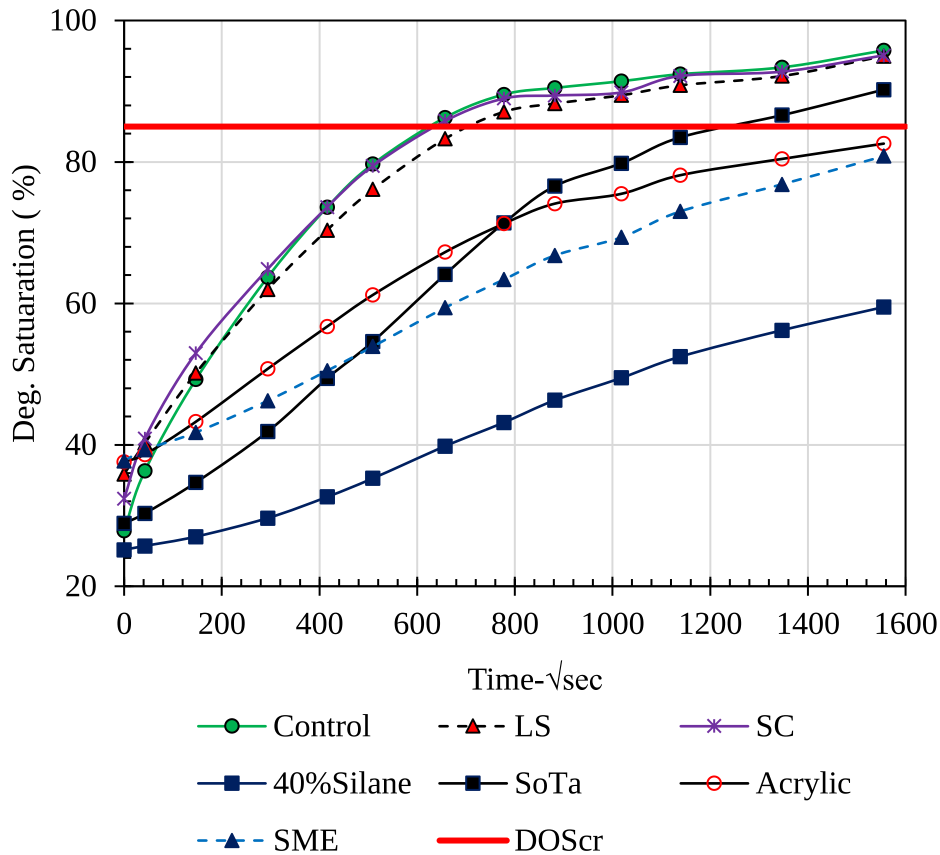


Figure 7. Evolution of the degrees of saturation with time

The 85% line shows the critical degree of saturation, below which deterioration of concrete subjected to F-T cycles is unlikely to occur. The untreated control, LS, and SC had the same time to critical saturation of ≈ 4 days, while concrete treated with SoTa saturated at ≈ 17 days. For the samples that did not reach critical saturation in 28 days, a best fit linear relationship was extrapolated from secondary absorption (from day 7 to 28) to project the time to critical saturation. The time required to saturate acrylic, SME, and 40% silane was ≈ 79, 50, and 113 days, respectively.

The absorption performance after UV testing is shown in Figure 8, where the chart on the left represents the samples before exposure and the one on the right is after 8,000 hrs of UV exposure at 313 nm.

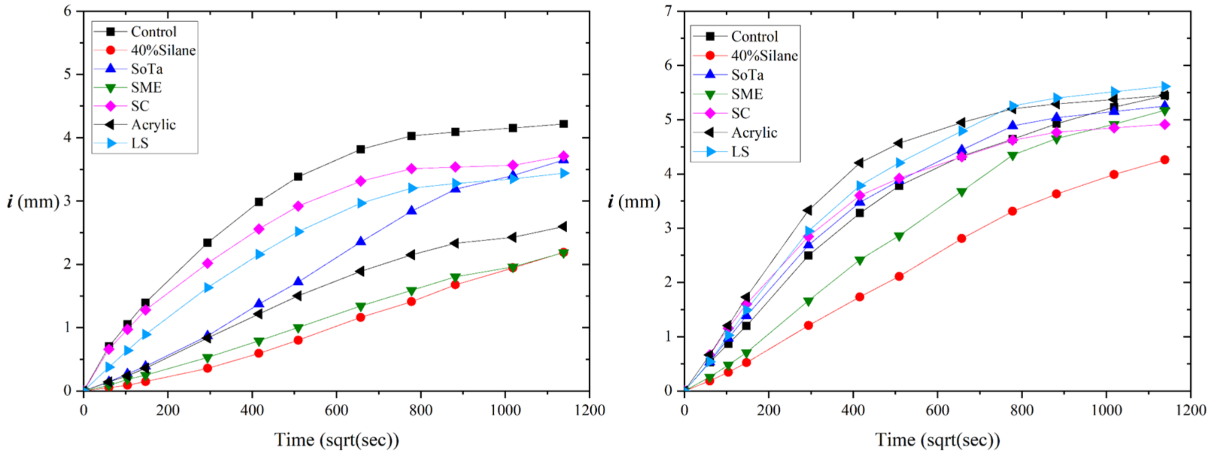


Figure 8. Absorption change from the initial condition (left) and after UV testing (right)

All samples exhibited an increase in absorption after testing with the 40% silane sample being the only that had significantly lower absorption than the control sample. Under initial testing, all of the samples coated with a hydrophobic material exhibited the early age rejection of water from the capillary pores. However, after UV exposure, only the SME-PS and 40% silane samples still delayed capillary absorption. For surface applications in direct exposure to UV radiation, while only tested for a single, long-term condition, breakdown is a concern and should be a consideration.

Freezing-Thawing

Figure 9 shows performance of the concrete treated with sealers for 210 F-T cycles over 15 weeks.

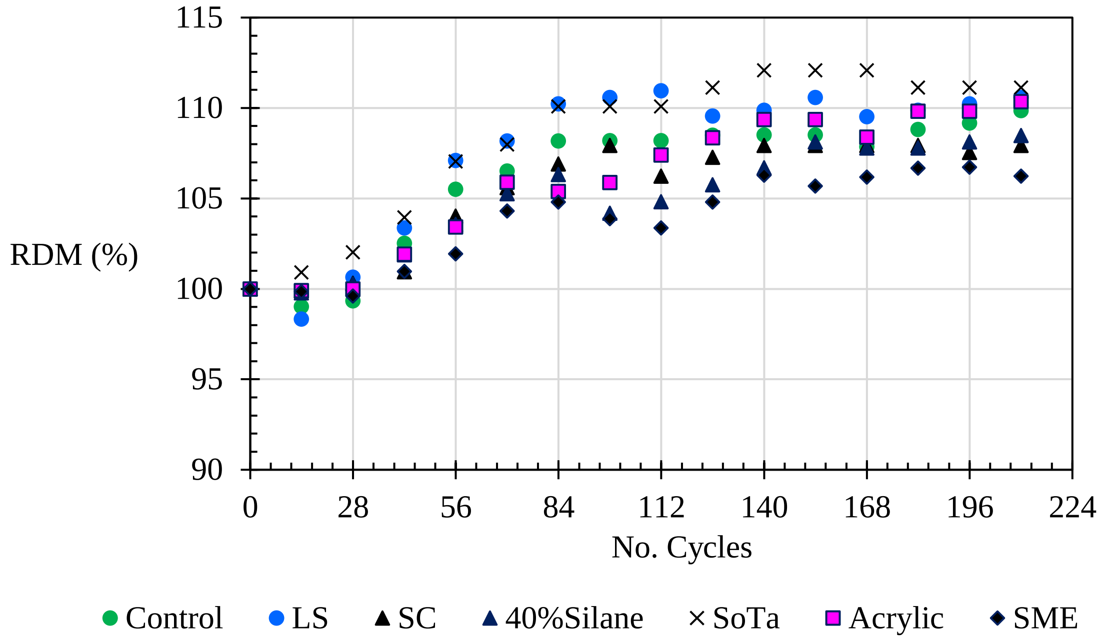


Figure 9. RDM changes with F-T cycles

None of the samples failed the test, which is not surprising considering the air-void system in the concrete. Specimens treated with acrylic visibly shed the coating after a few cycles. The RDM for all the specimens increased with time, which is not typical for conventional tests performed in pure water.

Figure 10 shows the mass changes for the concrete specimens with 210 F-T cycles over 15 weeks.

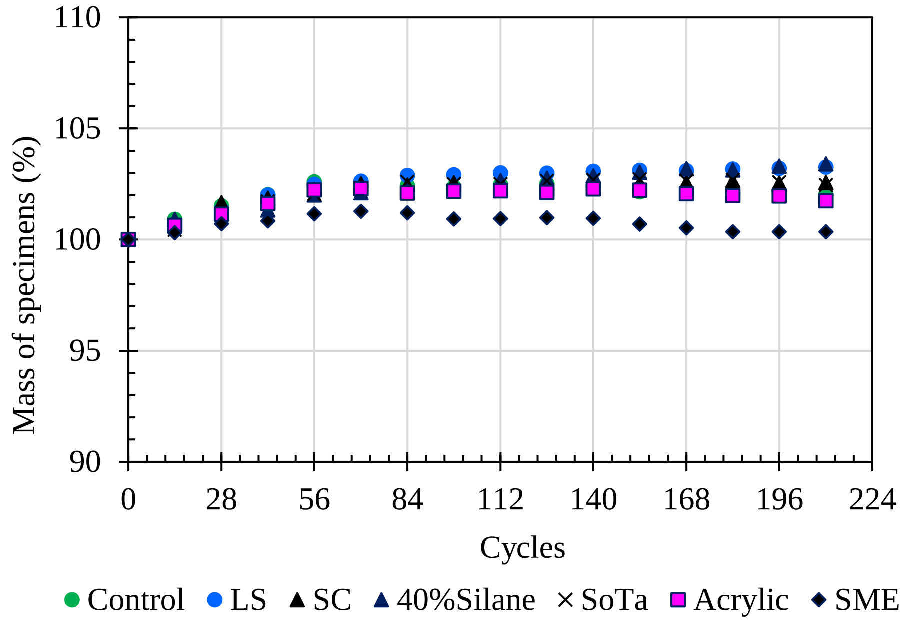


Figure 10. Specimen mass changes with F-T cycles

Sutter et al. (2008) stated that, when concrete is subjected to deicer salt solution and F-T cycles, both physical and chemical interactions occur. The physical interaction stems from internal stress, which is amplified by the presence of deicing salt solutions and surface salt scaling; whereas, the chemical interaction is the interaction between the salt ions and the concrete chemical composition. The increasing RDM of the specimens might be associated with the interaction of unhydrated cement with fluid solution, thus refining the microstructure of the concrete and ultimately increased RDM.

In the same study by Sutter et al. (2008), a 5% increase in the weight of the specimens subjected to an $MgCl_2$ solution was reported. Another study reported precipitating brucite ($Mg(OH)_2$) in the pores and densified mortar specimen in a way that reduced chloride ingress (Poursaee et al. 2010). Formation of ettringite and also brucite in pores was observed elsewhere (Wang et al. 2019). Setzer (2001) described a pumping effect due to pushing supercooled water (during thawing) to empty smaller pores. Thus, increasing specimen weight during F-T testing might be associated with this phenomenon. Also, Liu and Hansen (2016) reported an increase in the weight of specimens that experienced F-T despite contiguous scaling.

The concrete specimens were visually evaluated after 210 cycles (previous Figure 10) following the procedure in ASTM C672 *Standard Test Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals*. Surprisingly, concrete that was treated with penetrating sealers had a higher visual rating (worse performance) than the untreated concrete. SME showed significant reduction in water ingress, and the visual rating was very high compared to the untreated control. Besides the 40% silane and LS treatments, other treatments showed severe scaling compared to the untreated control. The outstanding performance of LS against F-T and salt scaling was not expected given that its performance regarding water contact angle, absorption, and degree of saturation.

Scaling of the treated concrete might not follow the classical theories on internal distress and surface salt scaling. Hazrati et al. (1997) reported that sealers have a negative effect on the durability of proper air-entrained concrete because the sealed layer separates from the bulk. Samaya et al. associated distress with internal pressure of statured water in concrete specimens. Other authors believe that surface scaling might be associated with variations in the coefficient of expansion for unsaturated layer and saturated layers (Valenza II and Scherer 2007a. 2007b).

Chloride Diffusion

Diffusion is a dominant mechanism in the transport of chloride ions. Figure 11 shows the chloride content percentages by mass of the concrete after 91 days of ponding at depths of 0.0625–0.5 in. (top layer) and 0.5–1.0 in. (bottom layer).

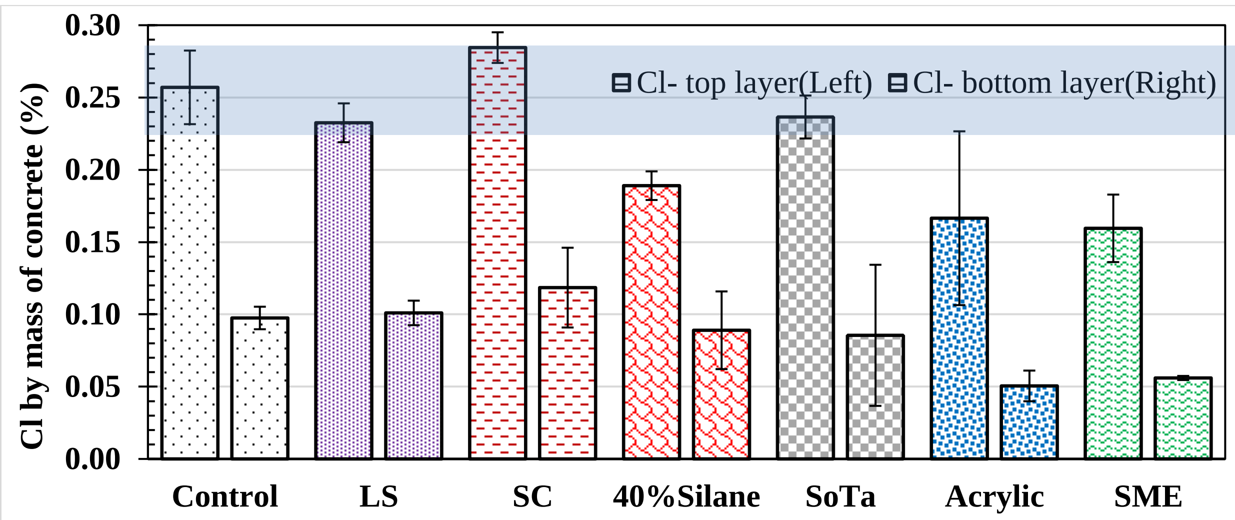


Figure 11. Chloride content percentage results

Concrete treated with LS, SC, and SoTa did not show any improvement in reducing chloride ingress, given the chloride content in their top and bottom layers are close to those for the untreated control. The 40% silane and SME treatments reduced chloride ingress in the top layer by 26% and 36%, respectively. Similarly, SME significantly reduced the chloride in the bottom layer by 43% with a small standard deviation while the reduction for silane was 10%. The correlation between chloride content and water progression in treated concrete can be observed. Treatments that significantly reduced water absorption, likewise, effectively reduced the progression of chloride.

Oxychloride Formation

Figure 12 presents the amount of potential CaOXY formation in the treated concrete discs immersed in the salt solutions as determined by LT-DSC in accordance with AASHTO T 365.

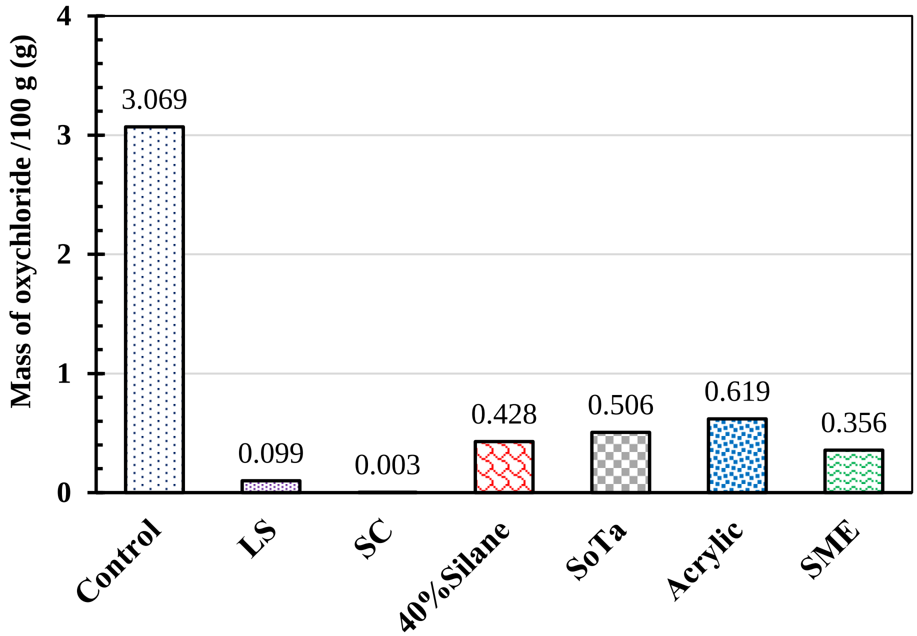


Figure 12. Potential CaOXY formation results

In general, all sealers significantly reduced the potential CaOXY formation. Surprisingly, LS and SC, which did not contribute to improving the concrete against physical interaction, did reduce the potential CaOXY formation. This is likely because the chemical reactions between salt solutions and CH crystals may occur prior to the densification of the microstructure by the pozzolanic reaction. Compared to other sealers, acrylic was less effective in reduction of CaOXY potential, while 40% silane, SoTa, and SME provided a moderate effect.

Discussion

Table 4 summarizes the data obtained.

Table 4. Summarized research data

Characterization	Control	LS	SC	40% Silane	SoTa	Acrylic	SME
Contact angle(θº)	30	76 ↑	74 ↑	105 ↑	120 ↑	83 ↑	100 ↑
Initial absorption (mm)	1.44	0.89 ↓	1.28 ↓	0.15 ↓	0.39 ↓	0.36 ↓	0.25 ↓
Secondary absorption (mm)	4.44	3.7 ↓	3.89 ↓	2.78 ↓	4.1 ●	2.89 ↓	2.67 ↓
Air permeability(m/s)	9.81	9.75 ●	9.75 ●	9.71 ●	9.89 ●	10.1 ↓	9.81 ●
Degree saturation (%)	96%	95% ●	95% ●	59% ↓	90% ↓	83% ↓	81% ↓
Surface scaling	3-4	3 ↓	4.5 ↑	3 ↓	4 ↑	5 ↑	5 ↑
Chloride penetration (%)	0.26%	0.233% ●	0.285% ●	0.189% ↓	0.237% ●	0.167% ↓	0.161% ↓
CaOXY (CaOXY)/100 g (g))	3.069	0.099 ↓	0.003 ↓	0.428 ↓	0.506 ↓	0.619 ↓	0.356 ↓

↑ Increasing, ↓ Decreasing, ● Not significant, Green: Improved performance, Red: Decreased performance

The performance of treated concrete specimens were compared to untreated control specimens. Sealers contributed in converting hydrophilic surfaces of untreated control samples to the exhibition of hydrophobic performance. Similarly, sealer treatments substantially reduced initial and secondary absorption. In other words, sealers reduced concrete sorptivity and appeared to improve potential durability. Concrete treated with SoTa exhibited a notable reduction in initial absorption; whereas, after 28 days in contact with water, the secondary absorption was almost identical to that for the untreated control.

Except for acrylic, which is a pore blocker sealer, the other sealers exhibited similar air permeability to the untreated control. This shows the breathability of treated concrete, and once the RH outside was lower than the internal RH, vapor was allowed to leave the concrete and desaturate the concrete. Surprisingly, decreasing absorption and delaying the time to saturation was not reflected in the performance achievement of concrete against frost action. Sealer applications such as SME aggravated concrete performance against F-T cycles which was not expected. However, all sealers significantly reduced CaOXY formation potential.

Summary

This phase investigated the effects of various penetrating sealers on the performance of concrete when applied to vertically oriented, saw-cut specimens representing concrete joints.

Despite the vertical orientation limiting the application rates and residence time, results indicated improved potential durability. The following specific conclusions can be drawn:

- Sealer treatments effectively reduced the water absorption capacity of the concrete, and a substantial reduction was observed in initial absorption. Reducing the water absorption rate was well reflected in time to saturation; concrete treated with silane, SME, and acrylic did not reach saturation after 28 days in contact with water.

- The acrylic barrier surface coating had the greatest effect on air permeability, followed by the pore blocking sealers, SME and SoTa. The pore lining and refining sealers had no impact on air permeability. When degree of saturation is a concern for durability, air permeability is an important measure to quantify and ensure vapor is allowed to leave the system.

- Aside from 40% silane and LS, it appeared that sealer-treated concrete tends to exhibit an increase in deterioration or not provide appreciable protection once subjected to frost action cycles. The results showed that the sealers did not provide any extra protection against F-T cycles. However, it should be noted that the conclusion herein is based on the F-T test carried out in the laboratory that included continuous contact with freezing fluid and a high cooling/heating rate (10°C/h, which actually rarely exceeds 2°C/h in reality).

- All sealers, regardless of type and mode of action, substantially reduced the potential of CaOXY formation, and the specimens treated with surface densifier sealers were more effective than the others.

- 40% silane was generally found to have the best performance against all of the parameters investigated in this research.

The data indicated the effectiveness of the tests was as follows:

- Water absorption was a good indicator of the ability of a product to prevent fluid penetration
- Wettability was useful in indicating the ability of a product to resist solutions entering the concrete surface
- LT-DSC clearly indicated the benefits of all of the sealants on reducing the risk of oxychloride formation
- F-T performance was influenced by the products but is fundamentally controlled by the air-void system
- Chloride penetration did not seem to indicate the benefits of some products
- Depth of penetration did not provide any useful guidance
- Air permeability did not seem to indicate the benefits of some products

PHASE 2: EFFECT OF SEALERS ON CONCRETES WITH DIFFERENT FAILURE MODES

The objective of this phase was to investigate the effects of sealers based on the durability of concrete that is susceptible to these durability distresses: D-cracking, marginal air system, and oxychloride.

Mixtures and Sampling

To evaluate the effect of sealers on the performance of non-durable concrete, a laboratory investigation was conducted on specimens made using three different concrete mixtures. Each concrete was designed to include susceptibility to durability issues, as follows:

- **Marginal air-void system (mAir):** An otherwise durable mixture with a SAM number higher than 0.45
- **Oxychloride susceptible (mCaOXY):** An otherwise durable mixture that contains a paste susceptible to CaOXY formation
- **D-cracking susceptible (mDcrack)**: An otherwise durable mixture made with known poor-quality aggregate

Ordinary portland cement (OPC) and Class C fly ash that met ASTM C150 *Standard Specification for Portland Cement* and C618 *Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete* were used as cementitious materials. Table 5 presents the chemical compositions of the cement and fly ash used in this work.

Table 5. Chemical composition of cementitious material

Components	OPC	Fly Ash
SiO_2	19.82	40.72
Al_2O_3	5.43	20.45
Fe_2O_3	2.07	6.08
SO_2	3.80	1.02
CaO	63.36	21.61
MgO	3,00	4.35
K_2O	1.05	0.63
Na_2O	0.36	1.36
Others	0.85	2.92

River sand was used for all mixes with 1.49% water absorption and a specific gravity of 2.65 in an SSD condition. Two coarse aggregate samples were selected; the first was crushed limestone with a proven record of excellent performance, and the other was known to be susceptible to D-cracking. The gradation of both aggregates met the requirements of ASTM C33 *Standard Specification for Concrete Aggregates*. Similarly, two binder systems were selected: one with

25% by wt. fly ash to mitigate the chemical attack caused by de-icing salts and the other with 100% portland cement. All mixes were prepared with similar proportions to those in Phase 1.

A commercial air-entraining admixture (AEA) was added in sufficient amounts to obtain concrete mixes with a good (>5%) air content or a marginal (>3% and < 5%) air content. A polycarboxylate-based superplasticizer was used in all three concrete mixtures to maintain workability at the controlled w/cm ratio.

Three penetrating sealers, representing pore lining, pore blocking, and combination pore blocker and pore liner were used. Table 6 presents the labels and chemical compositions of the selected sealers.

Table 6. Identification and chemical composition of sealers

Sealer ID	Chemical Composition	Type
SME	Fatty acids, soy methyl ester, polystyrene	Pore blocker, hydrophobic
SoTa	Water based, contains sodium tartrate, potassium methyl siliconate	Pore blocker, pore liner hydrophobic
40% Silane	Water based, contains chemically reactive alkyltri-alkoxysilane	Pore liner, hydrophobic

Cylindrical specimens measuring 4 in. × 8 in. (100 mm × 200 mm) and beams measuring 16 in. × 4 in. × 3 in. (400 mm × 100 mm × 75 mm) were cast and covered with a plastic sheet and wet burlap. After 24 hours, the specimens were stripped, and cylindrical samples were cut to obtain 2 in. (50 mm) thick discs. This was intended to simulate the crack development caused by saw cutting the joints in a newly constructed concrete pavement. The samples were transferred to the curing room after cutting and cured in a moist condition at 23°C and 100% RH for 7 days. The specimens were then transferred to a drying room set at 23°C and 50% RH for up to 28 days.

The three penetrating sealers were applied to the cut disc surfaces and the beam surfaces in two coats, allowing enough time between the coats to ensure drainage of the excess sealer from the concrete surfaces. The specimens were then returned to the drying room and stored for an additional four weeks with the sealed surfaces in a vertical position.

Test Program

The air-void system of concretes was characterized in both the fresh and hardened states. The fresh air content and SAM number were measured following procedures described in ASTM C231 *Standard Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method* and AASHTO TP 118 *Standard Method of Test for Characterization of the Air-Void System of Freshly Mixed Concrete by the Sequential Pressure Method*, respectively. The hardened air-void parameters, including air content and specific surface area, were measured according to the linear traverse method following the procedure outlined in ASTM C457 *Standard Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete*.

The same tests discussed in Phase 1 were conducted on the samples:

- Air-void system characterization (ASTM C231 and AASHTO TP 118).
- Wettability.
- Air permeability.
- Water absorption, desorption, and saturation.
- F-T durability in accordance with ASTM C666 modified following the recommendations in NCHRP Report 961 (Taylor et al. 2021). Prior to testing, specimens were wrapped with butyl rubber tape, and the bottom face of each specimen was introduced to saturated calcium hydroxide for 7 days in a similar manner as that described in ASTM C1585 prior to testing. During testing, a 3.3% $MgCl_2$ salt solution was introduced to the samples one-dimensionally as a freezing fluid, and the samples underwent two cycles of F-T per day between 20°C and -20°C. Two replicate beams were tested and RDM was measured according to ASTM C215 *Standard Test Method for Fundamental Transverse, Longitudinal, and Torsional Resonant Frequencies of Concrete Specimens*. RDM, weight, and weight loss were measured after each set of 14 cycles (corresponding to a week of F-T exposure). Samples were tested for 140 cycles and the percent RDM was calculated.
- LT-DSC.

Results

Table 7 presents the fresh and hardened properties of the concrete mixtures.

Table 7. Air-void measurement of fresh and hardened of concrete mixtures

ID	SAM		Hardened air		Compressive strength
	Air (%)	SAM #	Air (%)	Spacing Factor (mm)	56-day (psi)
mAir	3.1	0.45	4.47	0.17	4,548
mCaOXY	7.1	0.08	7.06	0.10	4,752
mDcrack	6.8	0.15	7.04	0.11	3,393

Based on the air-void system, specimens from the mAir concrete mixture were expected to exhibit poor performance under F-T conditions. However, mCaOXY and mDcrack mixtures were expected to demonstrate good performance against F-T conditions (Suryavanshi and Swamy 1996, de Vries and Polder 1997, Pan et al. 2017).

Wettability

Table 8 presents the average contact angle of each treatment on the concrete substrate.

Table 8. Water contact angle of specimens

Concrete Mix	Surface Treatment	Mean Contact Angle (Θ)	CV	Classification
mAir	Control	–	–	Hydrophilic
	SME	93	4%	Hydrophobic
	SoTa	111	7%	Hydrophobic
	40% Silane	114	4%	Hydrophobic
mCaOXY	Control	–	–	Hydrophilic
	SME	84	9%	Lower-hydrophobic
	SoTa	94	4%	Hydrophobic
	40% Silane	98	4%	Hydrophobic
mDcrack	Control	–	–	Hydrophilic
	SME	76	30%	Hydrophobic
	SoTa	110	10%	Hydrophobic
	40% Silane	128	3%	Over-hydrophobic

The data verified that all sealer treatments effectively increased the water contact angle of the substrate. The water contact angle of specimens treated with 40% silane demonstrated the highest degree of hydrophobicity, while specimens treated with SoTa featured a slightly lower water contact angle. Both treatments can be classified in as hydrophobic. Specimens treated with SME exhibited a slightly lower water contact angle, and the results were on the threshold between lower-hydrophobic and hydrophobic classification.

The lower contact angle of mCaOXY concrete might be attributed to its cementitious content, which was plain OPC, while the mAir and mDcrack mixtures had 25% of their OPC replaced with Class C fly ash. The pozzolanic nature of fly ash means that pores were refined leading to higher contact angles in those mixtures.

Air Permeability

API results are presented in Figure 13, and based on the API rating, an increase in the API indicates a reduction in air permeability (Alexander et al. 2009).

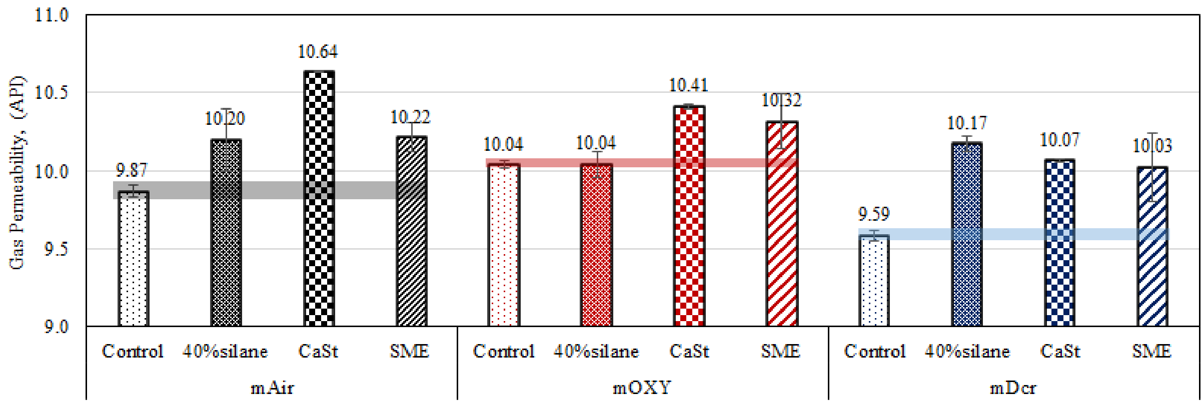


Figure 13. Air (gas) permeability index for concrete

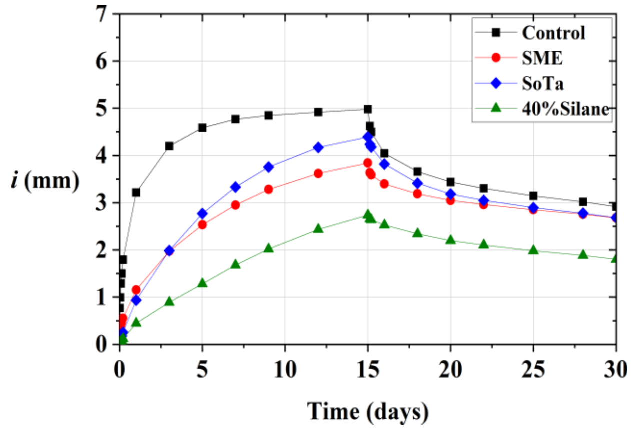
As shown in Figure 13, the 40% silane and SME treatments demonstrated at least a slight reduction in air permeability for mAir and mCaOXY concrete. All of the sealer treatments on the mDcrack specimens significantly reduced the air permeability. Substantial reduction of air permeability was observed (as anticipated) in specimens treated with SoTa (CaSt in Figure 13) given it acts as a partial pore blocker/liner. A similar trend, but with a smaller reduction, was observed for specimens treated with SME.

The higher air permeability of specimens from the mDcrack mixture is likely associated with more porous aggregate compared to the aggregate in the mAir and mCaOXY concrete. In general, the air permeability of the concrete specimens from highest to lowest was as follows: control < 40% silane < SME < SoTa.

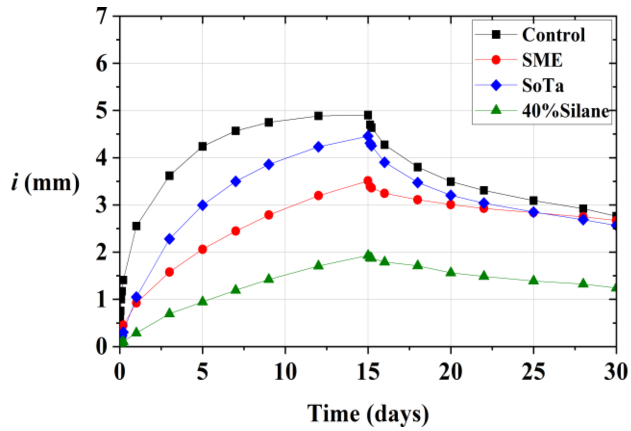
The results show that, depending on the sealer's mode of action, each of them does reduce air permeability. Dai et al. (2010) also reported that, for gas exchange with the exterior environment, concrete beyond hydrophobic are carbonated, which is problematic in reinforced concrete structures. However, this study investigated concrete joints where carbonation is not a matter of concern, unlike studies of reinforced concrete structures. Thus, the breathability of concrete after treatment is recommended to allow vapor exchange and reduce the degree of saturation once the concrete element is not in contact with water.

Absorption Capacity

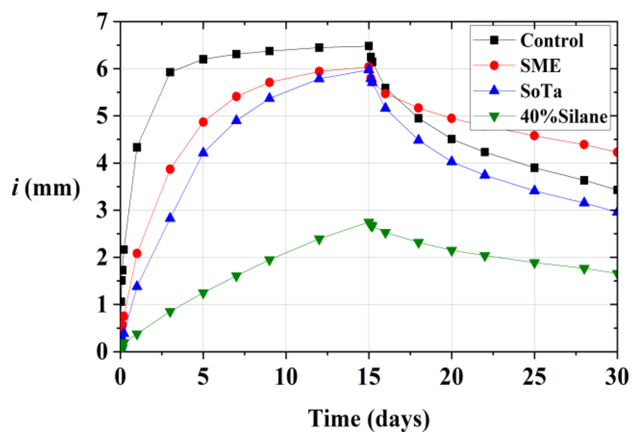
Figure 14 shows the measured amount of water absorbed by the specimens (*i*) normalized by the cross-section area in contact with water according to ASTM C1585 as a function of time.



mAir



mCaOXY



mDcrack

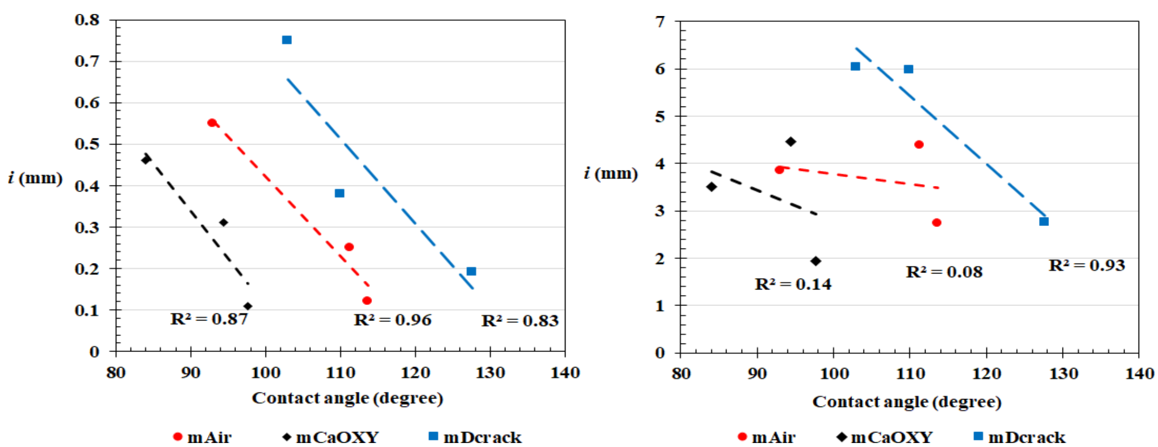
Figure 14. Water absorption and desorption development of concrete specimens

The D-cracking susceptible mixture contained limestone coarse aggregate with a much higher absorption than the good quality limestone used in the other two mixtures. Consequently, the absorption rate and total absorption of the mDcrack specimens was higher. The mAir sample transitioned quickly from initial to secondary absorption, indicating a lack of capillary draw into the entrained air system, as anticipated based on the air system parameters. The more gradual transition from initial to secondary absorption for the mCaOXY specimens is consistent with the good quality air-void system parameters observed from the fresh and hardened specimens. The initial and secondary sorptivity values were higher for all untreated concrete specimens, and a substantial reduction was observed for all sealer treatments. From a critical saturation standpoint, the benefits of penetrating sealers were easily observed.

Concrete treated with 40% silane had the lowest absorption for all mixtures. SoTa and SME provided less reduction in absorption at 15 days. Based on the shape of the absorption curves, the sealers are very effective at preventing moisture ingress at early ages, but the benefit became less pronounced with time, and the secondary rate of absorption was also greater for all of the treated specimens.

Dang et al. (2014) associated the increase in the secondary absorption rate of treated concrete with the formation of a water bridge between the relatively thin treated layer and the absorption potential of the untreated concrete.

Figure 15 presents the water contact angle results as a function of water absorption.



θ vs 5 hrs. absorption θ vs 15 days absorption

Figure 15. Relationship between contact angles and water absorption of coated specimens at 5 hrs and at 15 days

The results suggest that initial water absorption was strongly correlated with the water contact angle (Figure 15 left). Contact angle was not particularly well correlated with absorption after 15 days of exposure. Based on this observation, the effect of sealer treatments on the reduction of initial water absorption can be predicted via a simple laboratory contact angle measurement, which is consistent with the Young-Laplace equation, because, as the water contact angle increases, capillary suction decreases.

Ultimately, for good performance, a penetrating sealer should reduce moisture uptake but also allow drying to reduce critical saturation. For the mAir and mCaOXY specimens, the internal moisture after drying was similar for SoTa and SME demonstrating their pore blocking mechanism. However, and interestingly, the 40% silane specimens underwent comparatively less drying, although starting from a drier condition. Of most importance is the sealer behavior for the mDcrack specimens where the SME effectively sealed in moisture, resulting in higher saturation after drying than the untreated control. These results suggest that sealer function types are not interchangeable for all concrete distresses and that pure pore blocking chemicals may be detrimental to F-T performance.

Degree of Saturation

The saturation levels of the specimens as a function of time are presented in Figure 16.

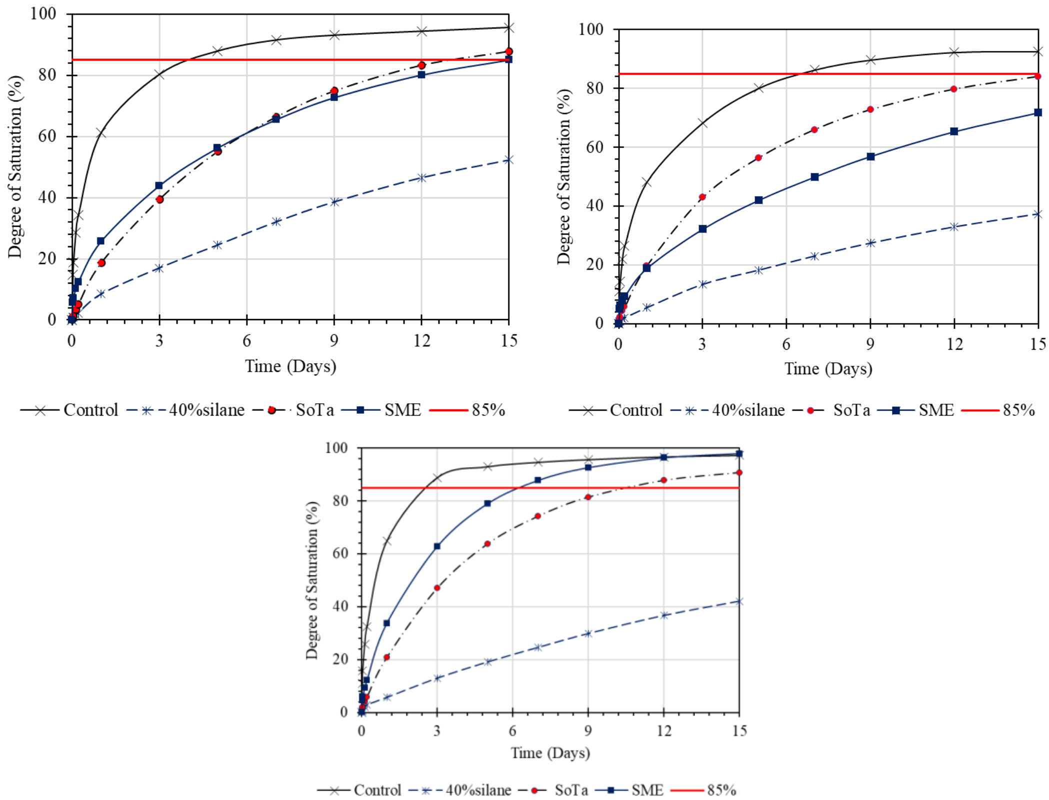


Figure 16. Degree of saturation for concrete specimens: mAir (top left), mOXY (top right), and mDcrack (bottom)

The red line represents the critical degree of saturation (DOS_{crit}) level above which concrete will deteriorate when subjected to F-T cycles (Fagerlund 1977, 2004). The results in Figure 16 show that the control specimens exceeded the DOS_{crit} in less than a week, while all sealer treatments delayed the time to the DOScrit level. Specimens treated with SoTa reached the DOScrit level in 16 or fewer days, while for SME, the mDcrack specimens passed the DOS_{crit} level in 6 days. All specimens treated with SME showed a slight increase in time to saturation. However, regardless of concrete type, the saturation level for concrete treated with 40% silane was less than 60% after 15 days in contact with water.

The degree of saturation governs the secondary absorption rate. For specimens that did not reach the DOS_{crit} level during the testing period, a best fit line from the secondary absorption rate data was extrapolated to determine the time to reach it. Table 9 shows the time to reach the critical saturation level.

Table 9. Required days to reach the critical degree of saturation (DOS_{crit})

Treatment	mAir	mCaOXY	mDcrack
Untreated	4	6.5	2.5
40% Silane	25	38	29
SoTa	13	16	10.5
SME	15	18	6

As indicated, concrete treated with 40% silane was still expected to perform well under F-T conditions.

Desorption

Figure 17 shows the water loss at the exposed surfaces as a function of time after the specimens had been immersed in saturated solution of calcium hydroxide for 30 days.

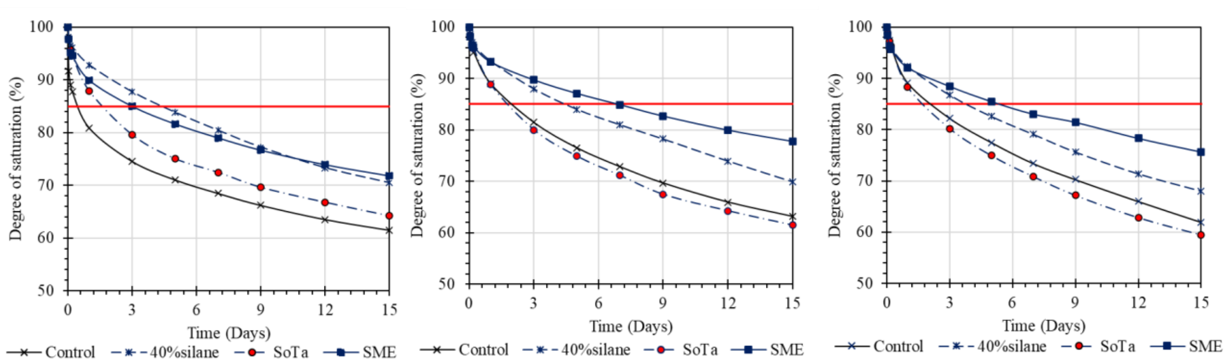


Figure 17. Evolution of absorption and desorption for concrete specimens: mAir (left), mOXY (center), and mDcrack (right)

In this condition, it was assumed that all specimens had reached saturation.

Except for the mAir control sample, none of the specimens had desaturation less than DOS_{crit} in 24 hrs. The mode of action for 40% silane is as a pore liner; thus, it was expected that the water release would be higher than that of the other treatments. The results obtained from concretes treated with SME suggested that specimens dry slower than 40% silane treatments, which was anticipated for SME sealers because their mode of action is as a pore blocker.

On the other hand, substantial water evaporation was seen in samples treated with SoTa sealers, which was not expected. Considerable water loss of concrete treated with SoTa might be attributed to the sodium tartrate in the sealer composition. Sodium is a base material for most super absorbent polymers (SAPs), and once they come in contact with water, they absorb water, swell, and thus act as a partial pore blocker. However, regarding desaturation, the researchers attributed substantial water release for the specimens treated with SoTa to the sodium tartrate layer, which likely absorbs internal moisture and facilitates water evaporation at the surface of the concrete. Hence, further research is needed to investigate and clarify this phenomenon.

An increased API (or decreased air permeability) was not shown to be correlated with desaturation and drying (previous Figure 5). The air permeability results were not correlated well with absorption either. This suggested that using air permeability tests, despite substantial correlation with durability in the published literature, is not useful for characterizing the performance of concrete treated with sealers. The results of this study suggest that the desaturation test can be considered in place of air permeability. The benefits of using the desaturation test are twofold: First, it shows the water exchange mechanism with the exterior environment. Second, it can be utilized simply in most laboratories without extra cost.

Freeze-Thaw Durability

Figure 18 shows RDM results and the accumulation of dried mass scaled on the exposed face of each specimen as a function of the number of F-T cycles using a modified test method.

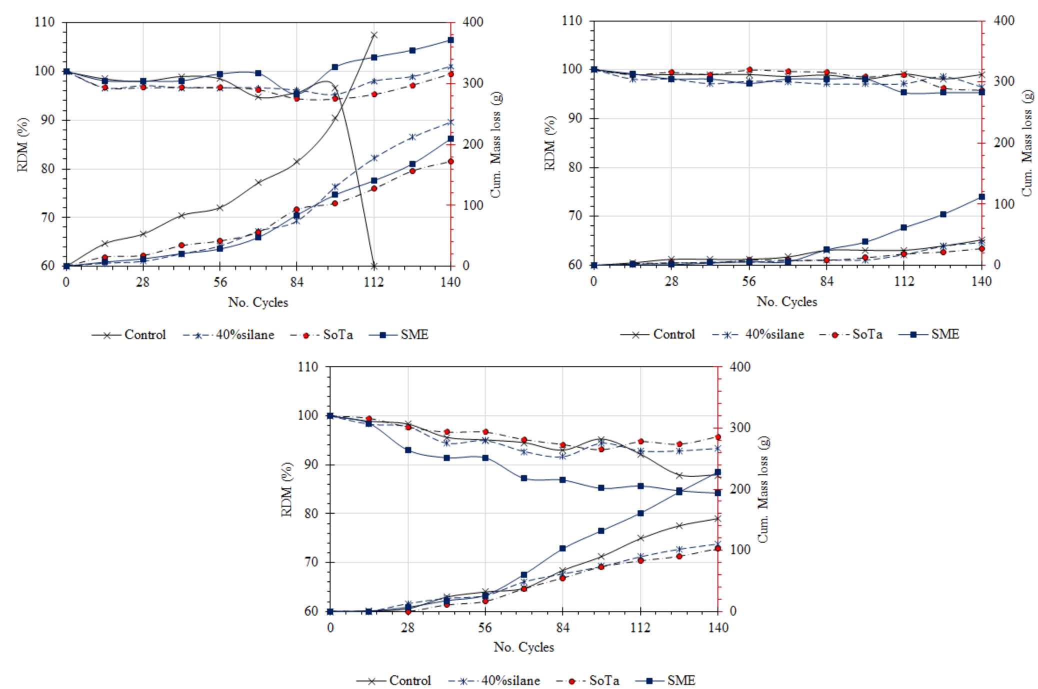


Figure 18. RDM change with F-T cycles and mass scaling of specimens: mAir (top left), mOXY (top right), and mDcrack (bottom)

Following F-T resistance testing, the specimens were evaluated visually according to a procedure outlined in ASTM C672.

Regarding mAir concrete specimens, the RDM results were linear with a slight reduction for all specimens until 84 F-T cycles. Following 84 cycles, the untreated control specimens failed with an RDM less than 60%, and specimens demonstrated scaling over 360 g. Interestingly, after 84 cycles, the RDM results for treated specimens gradually increased resulting in localized damage (Figure 18).

Similar results have been observed by others (Poursaee et al. 2010). The increasing RDM of specimens was likely attributed to scaling of the treated layer, after which the specimens resembled untreated concrete. The concrete likely absorbs more brine, which may eventually react with hydrated cement compositions to form Friedel’s salt or ettringite, which are expansive products that may accelerate damage (Sutter et al. 2006).

No appreciable differences were observed between treated and untreated specimens from the mCaOXY concrete. Even though the cumulative mass loss for the specimens treated with SME ultimately yielded significantly higher mass loss than the untreated specimens, the RDM results for the treated specimens coincided well with those for the untreated control specimens. Resistance of the mCaOXY concrete might be attributed to the concrete’s adequate air-void system. This implies that concrete with a proper air-void system can resist harsh conditions for 140 F-T cycles, noting that the test conditions did not encourage formation of oxychloride.

The behavior of the mDcrack concrete was slightly different than that of the other concretes. The RDM results for the mDcrack specimens gradually decreased. In the meantime, mass scaling significantly increased after 56 F-T cycles (Figure 19).

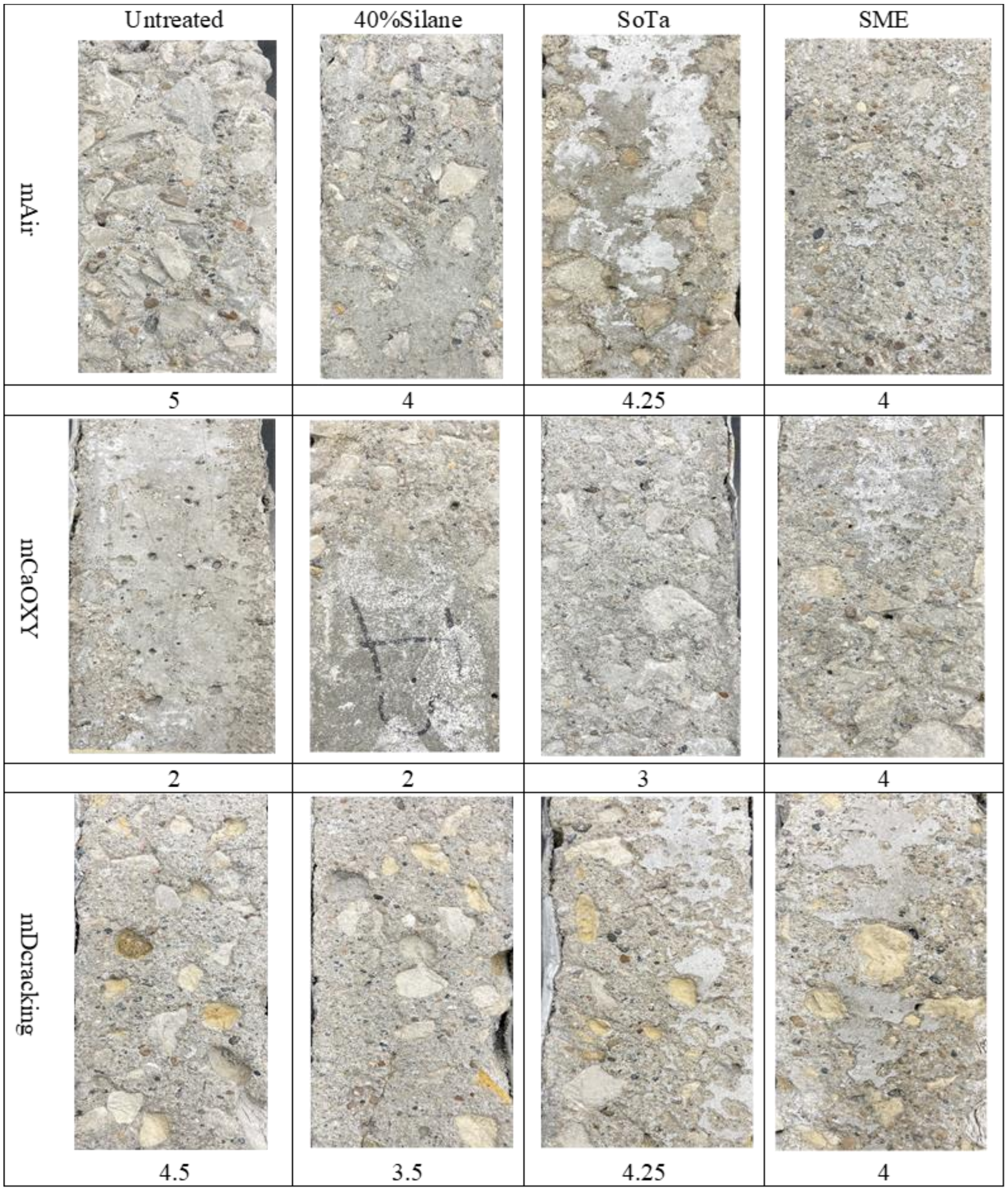


Figure 19. Scaling evaluation of specimens

Localized damage was not featured. Only paste matrix scaling and exposed aggregate particles were observed in most of the concrete specimens. Thus, increased mass scaling stemmed from progressive deterioration of aggregate particles in the form of pop-outs, while the surrounding paste matrix was relatively intact.

The RDM results for SME treatments gradually decreased with cycles, and at the end of the testing period was 84%, while the RDM of the control sample dropped after 112 cycles and was 87%at the end of the testing period. The RDM results for SoTa and 40% silane treatments yielded RDMs higher than 90% after 140 cycles, and the RDM was not affected by F-T cycles.

Based on these results, one can conclude that the SME treatment adversely affected concrete resistance against frost action, while 40% silane and SoTa treatments provided extra protection.

Oxychloride Formation

Figure 20 presents the amount of potential CaOXY formation in the concrete discs immersed in salt solutions in accordance with AASHTO T 365.

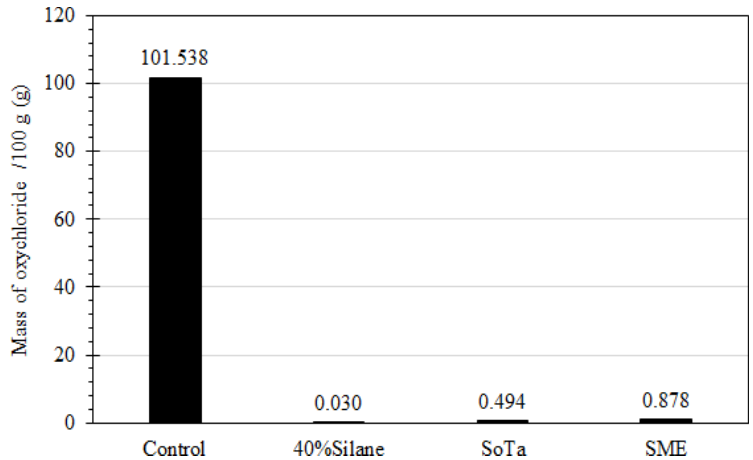


Figure 20. Scaling and visual rating of specimens after 140 F-T cycles

Despite the high potential of cement to form oxychloride, the sealer treatments reduced the potential for oxychloride formation. This is likely because of the chemical reactions between a hydrophobic sealer and hydrated cement. Thus, the formation of a hydrophobic layer reduces the reaction potential between hydrated cement and chloride ions at the surface of sawn joints and pore walls.

Figure 21 shows scanning electron microscope (SEM) images for the untreated control and SoTa treated specimens.

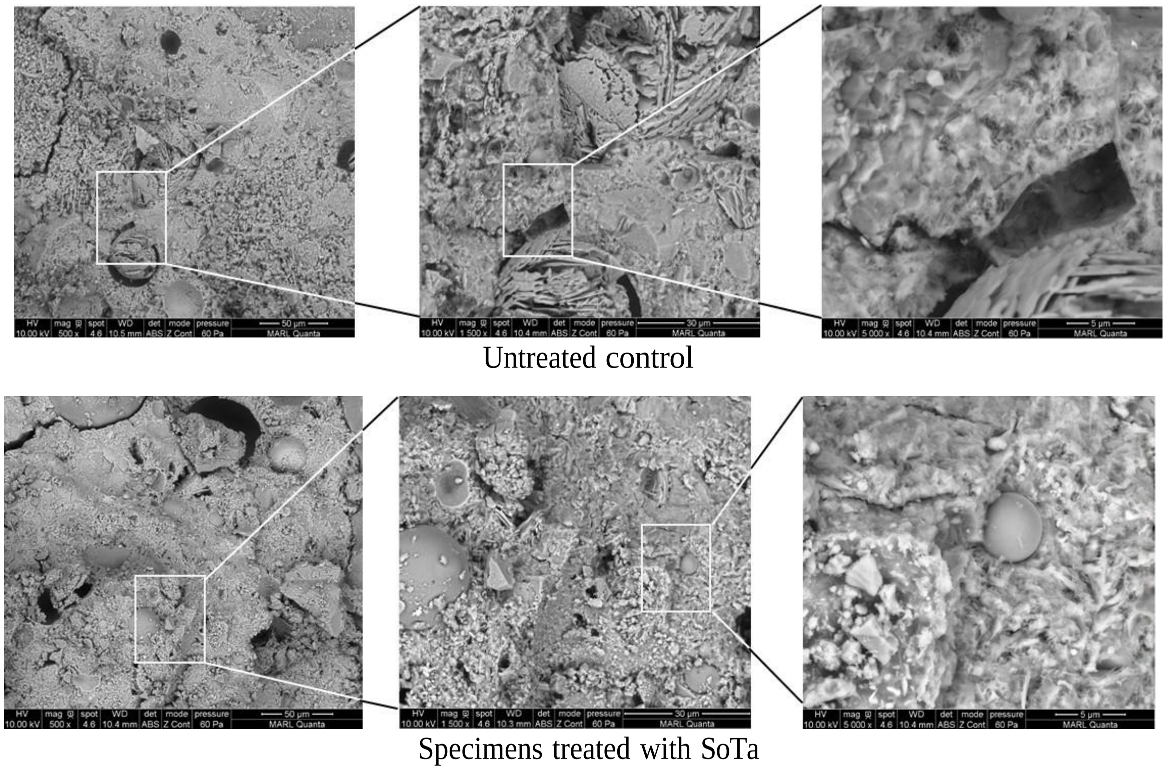


Figure 21. Concrete surfaces with or without SoTa sealer treatment

Clearly, the untreated control specimen showed more large platy crystals in the voids, likely resulting from the oxychloride reaction (Sutter 2006), and un-hydrated fly ash particles were rare, probably due to the accelerated hydration by intruded calcium chloride. On the other hand, the SoTa treated specimen showed much less large platy crystals in the cement paste and many un-hydrated fly ash particles with clean surfaces because of limited calcium chloride intrusion.

Discussion

As can be seen from the previous Figure 5, an increased API (or decreased air permeability) was not consistent with moisture exchange results for the concretes treated with sealers. This suggests that using the air permeability test, despite substantial correlation with durability in the published literature, is not useful for characterizing the performance of concretes treated with sealers. This is not surprising, because the test evaluates gas movement rather than liquid movement. The researchers recommend that the desaturation test be considered instead.

Depending on the mode of action, the sealers generally increased the contact angle of the substrates. Based on the Young-Laplace equation and assuming other parameters constant, increasing the water contact angle significantly reduces the capillary suction potential. The results presented herein and by others (Dang et al. 2014, Xiao et al. 2021), show a significant correlation between the water contact angle and the early water absorption rate. The term early age can be considered from zero to 4 days, and this reflects well with Young-Laplace's equation for flow in an unsaturated medium. However, this correlation minimizes with more water contact, so a consistent correlation cannot be concluded. This shows that, for infrastructure elements that are not in continuous contact with water, the effect of sealers on water absorption can be predicted thorough simple laboratory contact angle measurement as described in this report.

The results from this study show that hydrophobic sealers provide protection to concrete elements that are not in continuous contact with water for long times. This is because hydrophobic sealers can transmit vapor to decrease the saturation level for concrete. However, sealers might adversely affect the durability of concrete elements that are in continuous contact with water and subjected to F-T cycles. Thus, the role of sealers is less effective, and, due to the pumping effect, concrete reaches the DOS_{crit} level much faster. On the other hand, research has shown that initial water content effectively affects the water absorption behavior of the specimen (Spragg et al. 2012). Thus, particularly for concrete elements exposed to cycles of wetting and drying, besides the effect of sealers in reducing water absorption potential, the retained water resists water absorption; thus, slower salt progression is expected. Hence, Spragg et al. (2012) reported the effect of salts on retaining water for longer compared to specimens not exposed to a salt solution.

Note that the different sealers resulted in different effects on concrete resistance to frost cycles. For instance, use of the SME treatment on mAir concrete effectively reduced F-T damage, as indicated by the reduced mass loss. Hence, in the case of concrete with an adequate air-void system, the results were, surprisingly, inverse. However, 40% silane and SoTo sealers did effectively provided extra protection. Thus, it can be concluded that sealer mode of action must be considered for concrete in cold regions and for concrete elements in continuous contact with water. Research has attributed the negative effect of certain sealers to freezing as a saturated layer behind the treated layer causing scaling (Hazrati et al. 1997, Sayams et al. 20220). The negative effect of sealers on concrete containing adequate air-void systems needs further investigation to clarify the deterioration mechanism.

Summary

In this study, three commercially available sealers, with different modes of action, were applied on the vertically sawn face of three different types of concrete. The effects of the sealer applications on concrete F-T durability were investigated. Each concrete contained a variant that made it susceptible to deterioration in cold regions: concrete with marginal air voids, cement that is susceptible to form CaOXY, and aggregate susceptible to D-cracking once subjected to F-T cycles. Based on the experimental results, the following conclusions can be drawn:

- Sealers substantially reduced the capillary suction potential of concrete, leading to decreased water absorption, particularly in the early days of continuous contact with water.

- Increasing the water contact angle by applying sealers on the concrete surfaces correlated well with the reduction of the primary water absorption in the early days (0–5 days). However, this correlation was not consistent with the secondary absorption rate.

- Although sealer treatments decreased concrete air permeability, there was no correlation between API and the water absorption rate. This suggests that the use of the air permeability test is not an effective way to characterize the durability of concrete that is treated with sealers.

- Sealers delayed the time to reach the critical degree of saturation. The degree of saturation for specimens treated with silane was less than 60% after 15 days in continuous contact with water. However, prolonging the time to saturation was not reflected in resistance to frost action.

- Vapor exchange for treated concrete plays a significant role in desaturation of concrete once the concrete is not in contact with water. The SME treated specimens retained more water, while SoTa treated specimens featured substantial reduction in the degree of saturation, which needs further investigation to clarify the phenomenon.

- The effect of sealers on concrete durability varied with the types of concrete. In the concrete that had a marginal air-void system, sealers provided extra protection and increased resistance against frost action cycles, while in the concrete that had a proper air-void system, sealers did not provide an appreciable effect. However, the SME sealer aggravated the resistance against F-T cycles. This demonstrates that the effect of sealers on concrete resistance against frost action depends on the sealer's mode of action.

- Further long-term field research is needed to study the effect of sealers as a means to provide extra protection to concrete joints.

The data indicated the effectiveness of the tests was as follows:

- Water absorption is a good indicator of the ability of a product to prevent distress for all of the mechanisms assessed
- Desorption is important for systems that need to dry out to prevent distress
- Wettability was useful in indicating the ability of a product to resist solutions entering the concrete surface
- LT-DSC clearly indicated the benefits of all of the sealants on reducing the risk of oxychloride formation
- F-T performance was influenced by the products, but is fundamentally controlled by the air-void system
- Air permeability tests did indicate whether or not a sealer prevented gas transmission; however, if breathability is critical for drying, a product that is impenetrable to gases may not be desirable

PHASE 3: FIELD EVALUATION

To complement the laboratory investigations carried out in Phases 1 and 2, sealers were applied to concrete pavement joints at three sites in late summer and fall 2021 for the purpose of long-term field evaluation. The SoTa and 40% silane sealers were selected for use in the field investigation. Sites included newly-constructed pavements in rural and urban areas, as well as an existing urban pavement that was undergoing rehabilitation due to joint deterioration. Site information for these test sections is summarized in Table 10.

Table 10. Field evaluation site information

Project Name	**CR W-61**	**SW State Street**	**SE 8th Street**
Location	Washington County, IA	Ankeny, IA	Ankeny, IA
Project Type	New Construction	Existing Pavement	New Construction
Project Area	Rural	Urban	Urban
Sealer Type	SoTa	40% Silane	SoTa and 40% Silane
Application Date	9/10/21	10/15/21	11/17/21
Conditions	Sunny, 80°F	Overcast, 60°F	Overcast, 49°F

Given that these field applications were completed only shortly before the end of this study, no testing has been performed on these sections to date. However, each site will be monitored to evaluate the long-term performance of the joints in the sealed areas. Cores will also be extracted over both sealed and unsealed joints from these sites to provide samples for follow-up laboratory testing using the same methods as Phases 1 and 2.

Ultimately, the field evaluation should provide further insights into the performance of these sealers by characterizing the performance of the sealed joints over time compared to each other, compared to the unsealed control joints, and compared to the results of the laboratory testing. Further details about the field sites are included in the following sections.

Washington County Highway W-61

A section of a new concrete overlay on Washington County Road (CR) W-61 (Riverside Road) just south of Riverside, Iowa was selected for applications of the SoTa sealer. The overlay was 6 in. thick with integral full-depth shoulders. Transverse joint spacing was 15 ft, with three longitudinal joints (centerline joint plus two joints between the mainline and the integral shoulders) running throughout the pavement. The approximate location of the sealed section is shown in Figure 22.

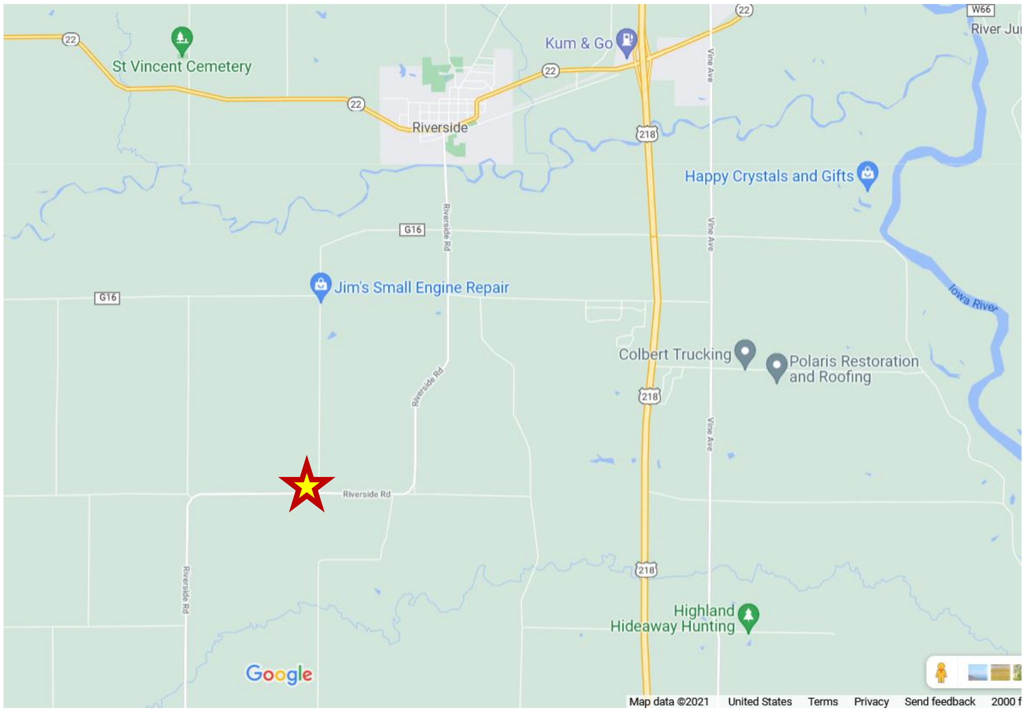


Figure 22. Location of sealer test section on CR W-61 in Washington County

The sealer was applied on September 10, 2021 on a sunny day with a high of 80°F. All transverse and longitudinal joints were sealed for an approximately 515 ft section between station 510+00 (just north of the Miller farm driveway) and two transverse joints north of station 515+00 (across from the Riverside Road sign) near the north end of the project. The sealer was applied using a one gal tank sprayer within the saw cut and out about 6 in. alongside the joint at an application rate of 200 ft^2/gal. The section had been paved nine days prior on September 1, 2021, and all joints were cleaned out prior to sealing on September 10, 2021. All joints were subsequently filled with hot pour joint sealant on September 13, 2021.

A typical section of CR W-61 just after application of the sealer is shown in Figure 23.

Figure 23. SoTa application on CR W-61 in Washington County

It is difficult to distinguish the untreated and treated pavement surfaces alongside the joint in the image due to the sunny conditions.

SW State Street, Ankeny

A section of existing pavement on SW State Street in Ankeny was selected for application with the 40% silane sealer. This existing 10 in. full-depth concrete pavement with integral curb and gutter was constructed in 1996. In late summer and early fall 2021, a rehabilitation project was performed on the southbound lanes to address joint deterioration and joint shadowing that had occurred on this pavement. Activities included partial-depth repairs over joints that were already significantly deteriorated and cleaning and re-filling of all remaining longitudinal and transverse joints.

After joint cleaning but before re-filling with a hot pour sealant, the transverse and longitudinal (centerline and gutterline) joints were treated with 40% silane in the driving lane of an approximately 150 ft long section of pavement. The location of this section, just south of the intersection with SW Vintage Parkway, is shown in Figure 24.

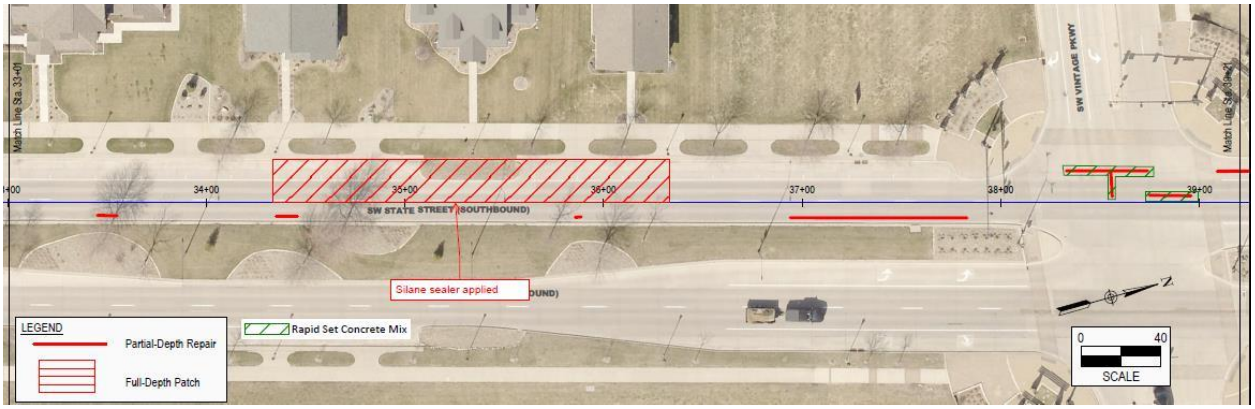


Figure 24. Location of sealer test section on SW State Street, Ankeny

The sealer was applied on October 15, 2021 on an overcast day with a high of 60°F. Given no partial-depth repairs within this section, the sealer was only applied to the existing concrete. The sealer was applied using a one gal tank sprayer within the saw cut and out about 6 in. alongside the joint at an application rate of 330 ft^2/gallon. All joints were subsequently filled with hot pour joint sealant after 24 hours.

SE 8th Street, Ankeny

A section of a new 8 in. full-depth concrete pavement on SE 8th Street in Ankeny was selected for applications using the SoTa and 40% silane sealers. This pavement was constructed with early-entry saw cuts that were not widened, filled, or sealed. Transverse joint spacing was 15 ft with three longitudinal (centerline and gutterline) joints. The approximate location of the sealed treatments is shown in Figure 25.

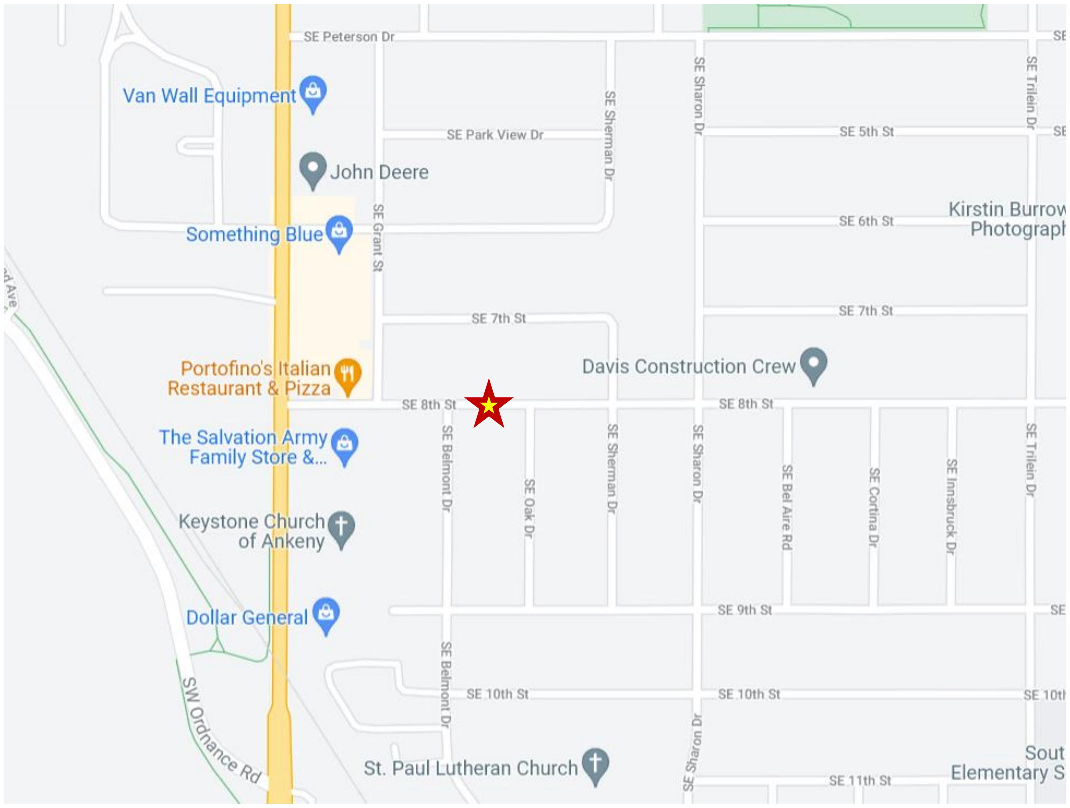


Figure 25. Location of sealer test sections on SE 8th Street, Ankeny

The sealers were applied on November 17, 2021 on an overcast day with a high of 49°F. All transverse and longitudinal joints were sealed for an approximately 300 ft section of pavement between SE Oak Drive and SE Belmont Drive. The west end of this section was sealed with SoTa at an application rate of 200 ft^2/gal, while the east end was sealed with 40% silane at a rate of 330 ft^2/gal. (Application rates differed based on manufacturer recommendations for each product.) The sealers were applied using a one gal tank sprayer within the saw cut and out about 6 in. alongside the joint. Like the rest of the project, treated joints were not subsequently filled or sealed with any other type of joint filler or sealant.

Figure 26 shows SE 8th Street just after application of the sealers.

Figure 26. Sealer application on SE 8th Street

Figure 27 includes images to help identify the boundaries of the treatments with the two different sealers.

Figure 27. Landmarks showing western edge of SoTA application (top left), mid-point where sealers were switched (top right), and eastern edge of 40% silane application (bottom)

CONCLUSIONS

The evidence is clear that sealers can help improve the longevity of concrete surfaces and that they can, to an extent, compensate for mixtures at risk for premature deterioration.

A limited family of chemical families are available, and they act in different ways on the mixtures. The information in Table 11 ties the action of the product to the performance needed.

Table 11. Sealer family/application for different distress mechanisms

	Marginal air-void system	Oxychloride susceptible	D-cracking susceptible	Permeable
Densifier	✓	✓		✓
Pore liner, Hydrophobic	✓	✓	✓	✓
Pore blocker, Hydrophobic	✓	✓		✓
Barrier coating	✓	✓		✓

The test methods listed in Table 12 are recommended for assessing products to mitigate likely distress mechanisms, as indicated.

Table 12. Recommended test methods

	Marginal air-void system	Oxychloride susceptible	D-cracking susceptible	Permeable
Absorption capacity	✓	✓	✓	✓
Wettability	✓	✓	✓	✓
Desorption			✓	
LT-DSC		✓		

IMPLEMENTATION

The research team reviewed a number of test methods for assessing penetrating sealants using a wide range of the available products under several failure modes.

Guidance was developed on how the action of the sealant should be tied to the potential failure mechanism and on which tests should be conducted on new products to ensure that they meet the needs. This guidance can be used by agencies when reviewing the products available to them for a range of applications.

REFERENCES

Adil, G., J. T. Kevern, and D. X. Xiao. 2021. Influence of Penetrating Sealer on Performance of Concrete Joints. *Materials Journal*, Vol. 118, No. 5, pp. 65–74.

Alexander, M., Y. Ballim, and J. Mackechnie. 2009. *Durability Index Testing Procedure Manual*. University of Cape Town, Cape Town, South Africa.

Arribas-Colon, M. d. M., M. Radlinski, J. Olek, and N. Whiting. 2012. *Investigation of Premature Distress Around Joints in PCC Pavements: Parts I and II*. Joint Transportation Research Program, Indiana Department of Transportation and Purdue University, West Lafayette, IN.

Basheer, L. and D. J. Cleland. 2006. Freeze-Thaw Resistance of Concretes Treated with Pore Liners. *Construction and Building Materials*, Vol. 20, No. 10, pp. 990–998.

Cady, P. D. 1994. *NCHRP Synthesis of Highway Practice 209: Sealers for Portland Cement Concrete Highway Facilities*. National Cooperative Highway Research Program, Washington, DC. http://onlinepubs.trb.org/Onlinepubs/nchrp/nchrp_syn_209.pdf.

Christodoulou, C., C. I. Goodier, S. A. Austin, J. Webb, and G. K. Glass. 2013. Long-Term Performance of Surface Impregnation of Reinforced Concrete Structures with Silane. *Construction and Building Materials*, Vol. 48, pp. 708–716.

The Concrete Sealer Guy. 2016. Overview of Different Types of Penetrating Concrete Sealers. *Concrete Sealers USA Blog*. https://www.concretesealersusa.com/blog/overview-of-different-types-of-penetrating-concrete-sealers/.

Cwirzen, A. and V. Penttala. 2005. Aggregate-Cement Paste Transition Zone Properties Affecting the Salt-Frost Damage of High-Performance Concretes. *Cement and Concrete Research*, Vol. 35, No. 4, pp. 671–679.

Dai, J. G., Y. Akira, F. H. Wittmann, H, Yokota, and P. Zhang. 2010. Water Repellent Surface Impregnation for Extension of Service Life of Reinforced Concrete Structures in Marine Environments: The Role of Cracks. *Cement and Concrete Composites*, Vol. 32, No. 2, pp. 101–109.

Dang, Y., N. Xie, A. Kessel, E. McVey, A. Pace, and X. Shi. 2014. Accelerated Laboratory Evaluation of Surface Treatments for Protecting Concrete Bridge Decks from Salt Scaling. *Construction and Building Materials*, Vol. 55, pp. 128–135.

de Vries, I. J. and R. B. Polder. 1997. Hydrophobic Treatment of Concrete. *Construction and Building Materials*, Vol. 11, No. 4, pp. 259–265.

Fagerlund, G. 1973. Critical Degrees of Saturation at Freezing of Porous and Brittle Materials. PhD thesis, Lund University, Lund, Sweden.

Fagerlund, G. 1977. The Critical Degree of Saturation Method of Assessing the Freeze/Thaw Resistance of Concrete. *Materiaux´ et Construction*, Vol. 10, No. 4, pp. 217–229.

Fagerlund. 2004. *A Service Life Model for Internal Frost Damage in Concrete*. Lund Institute of Technology, Division of Building Materials, Lund University, Lund, Sweden.

Freitag, S. A. and S. M. Bruce. 2010. *The Influence of Surface Treatments on the Service Lives of Concrete Bridges*. New Zealand Transport Agency, Wellington, New Zealand.

Fu, C., Y. Ling, and K. Wang. 2020. An Innovation Study on Chloride and Oxygen Diffusion in Simulated Interfacial Transition Zone of Cementitious Material. *Cement and Concrete Composites*, Vol. 110, No. 103585, pp. 1–11.

Hall, C. 1989. Water Sorptivity of Mortars and Concretes: A Review. *Magazine of Concrete Research*, Vol. 41, No. 147, pp. 51–61.

Hazrati, K., C. Abesque, M. Pigeon, and T. Sedran.1997. Efficiency of Sealers on the Scaling Resistance of Concrete in Presence of Deicing Salts. *RILEM Proceedings 30: Freeze-Thaw Durability of Concrete*, pp. 165–196.

Jones, W., Y. Farnam, P. Imbrock, J. Spiro, C. Villani, M. Golias, J. Olek, and W. J. Weiss. 2013. *An Overview of Joint Deterioration in Concrete Pavement: Mechanisms, Solution Properties, and Sealers.* Joint Transportation Research Program, Purdue University, West Lafayette, IN.

Kevern, J. T. 2010. Using Soybean Oil to Improve the Durability of Concrete Pavements. *International Journal of Pavement Research and Technology*, Vol. 3, No. 5, pp. 280–285.

Kosmatka, S. H. and M. L. Wilson. 2016. *Design and Control of Concrete Mixtures*. 16th edition. Portland Cement Association, Skokie, IL.

Liu, Z. and W. Hansen. 2016. Effect of Hydrophobic Surface Treatment on Freeze-Thaw Durability of Concrete. *Cement and Concrete Composites*, Vol. 69, pp. 49–60.

Maso, J. C., editor. 1996. *Interfacial Transition Zone in Concrete*. RILEM Report 11, State-of-the-Art report prepared by RILEM Technical Committee 108-ICC, Interfaces in Cementitious Composites. Taylor & Francis, Abingdon, Oxson.

Moradllo, M. K., B. Sudbrink, and M. T. Ley. 2016. Determining the Effective Service Life of Silane Treatments in Concrete Bridge Decks. *Construction and Building Materials,* Vol. 116, pp. 121–127.

Pan, X., Z. Shi, C. Shi, T.-C. Ling, and N. Li. 2017. A Review on Concrete Surface Treatment Part I: Types and Mechanisms. *Construction and Building Materials*, Vol. 132, pp. 578–590.

Panchmatia, P., J. Olek, and N. Whiting. 2014. Joint Deterioration in Concrete Pavements. 4th International Conference on the Durability of Concrete Structures, July 24–26, Purdue University, West Lafayette, IN.

Pfeifer, D. W. and M. J. Scali. 1981. *NCHRP Report 244: Concrete Sealers for Protection of Bridge Structures*. National Cooperative Highway Research Program, Washington, DC.

Poursaee, A., A. Laurent, and C. Hansson, C. 2010. Corrosion of Steel Bars in OPC Mortar Exposed to NaCl, $MgCl_2$ and $CaCl_2$: Macro-and Micro-Cell Corrosion Perspective. *Cement and Concrete Research*, Vol. 40, No. 3, pp. 426–430.

Setzer, M. J. 2001. Mechanical Stability Criterion, Triple-Phase Condition, and Pressure Differences of Matter Condensed in a Porous Matrix. *Journal of Colloid and Interface Science*, Vol. 235, No. 1, pp. 170–182,

Spragg, R. P., J. Castro, W. Li, M. Pour-Ghaz, P.-T. Huang, and J. Weiss. 2011. Wetting and Drying of Concrete Using Aqueous Solutions Containing Deicing Salts. *Cement and Concrete Composites*, Vol. 33, No. 5, pp. 535–542.

Spragg, R. P., J. Castro, T. Nantung, M. Paredes, and J. Weiss. 2012. Variability Analysis of the Bulk Resistivity Measured Using Concrete Cylinders. *Advances in Civil Engineering Materials*, Vol. 1, No. 1, pp. 1–17.

Suryavanshi, A. K. and R. N. Swamy. 1996. Stability of Friedel's Salt in Carbonated Concrete Structural Elements. *Cement and Concrete Research*, Vol. 26, No. 5, pp. 729–741.

Sutter, L., T. Van Dam, K. Peterson, and D. P. Johnston. 2006. Long-Term Effects of Magnesium Chloride and Other Concentrated Salt Solutions on Pavement and Structural Portland Cement Concrete. *Transportation Research Record: Journal of the Transportation Research Board*, No. 1979, pp. 60–68.

Sutter, L., K. Peterson, G. Julio-Betancourt, D. Hooton, T. Van Dam, and K. Smith. 2008. *The Deleterious Chemical Effects of Concentrated Deicing Solutions on Portland Cement Concrete*. Michigan Tech Transportation Institute, Houghton, MI.

Sutter, L. L. and G. C. Anzalone. 2016. *Investigation of Deterioration of Joints in Concrete Pavements: Field Study of Penetrating Sealers*. National Concrete Pavement Technology Center, Iowa State University, Ames, IA. https://intrans.iastate.edu/app/uploads/2018/03/joint_deterioration_penetrating_sealers_field_study_w_cvr.pdf.

Taylor, P., L. Sutter, and J. Weiss. 2012. *Investigation of Deterioration of Joints in Concrete Pavements*. National Concrete Pavement Technology Center, Iowa State University, Ames, IA. https://intrans.iastate.edu/app/uploads/2018/03/joint_deterioration_report_w_cvr.pdf.

Taylor, P., J. Zhang, and X. Wang. 2016. *Conclusions from the Investigation of Deterioration of Joints in Concrete Pavements*. National Concrete Pavement Technology Center, Iowa State University, Ames, IA. https://intrans.iastate.edu/app/uploads/2018/03/joint_deterioration_investigation_w_cvr.pdf.

Taylor, P., S. Sadati, K. Wang, Y. Ling, X. Wang, W. Sun, J. T. Kevern, and S. Ryazi. 2021. *NCHRP Report 961: Entrained Air-Void Systems for Durable Highway Concrete*. National Cooperative Highway Research Program, Washington, DC. https://nap.nationalacademies.org/download/26071.

Valenza II, J. J. and G. W. Scherer. 2007a. A Review of Salt Scaling: I. Phenomenology. *Cement and Concrete Research*, Vol. 37, No. 7, pp. 1007–1021.

Valenza II, J. J. and G. W. Scherer. 2007b. A Review of Salt Scaling: II. Mechanisms. *Cement and Concrete Research*, Vol. 37, No. 7, pp. 1022–1034.

Vipulanandan, C., A. Parihar, A., and M. Issac. 2011. Testing and Modeling Composite Coatings with Silanes for Protecting Reinforced Concrete in Saltwater Environment. *Journal of Materials in Civil Engineering*, Vol. 23, No. 12, pp. 1602–1608.

Wang, X., S. Sadati, P. Taylor, C. Li, X. Wang, and A. Sha. 2019. Material Characterization to Assess Effectiveness of Surface Treatment to Prevent Joint Deterioration from Oxychloride Formation Mechanism. *Cement and Concrete Composites*, Vol. 104, No. 103394, pp. 1–15.

Weiss, J., M. T. Ley, L. Sutter, D. Harrington, J. Gross, and S. Tritsch. 2016. *Guide to the Prevention and Restoration of Early Joint Deterioration in Concrete Pavements*. National Concrete Pavement Technology Center, Iowa State University, Ames, IA. https://intrans.iastate.edu/app/uploads/2018/03/2016_joint_deterioration_in_pvmts_guide.pdf.

Wiese, A., Y. Farnam, W. A. Jones, P. Imbrock, B. Tao, and W. J. Weiss. 2015. *Evaluation of Sealers and Waterproofers for Extending the Life Cycle of Concrete*. Joint Transportation Research Program, Purdue University, West Lafayette, IN.

Xiao D. X., S. Owusu-Ababio, R. Schmitt, and J. T. Kevern. 2020. *Evaluation of Penetrating Sealers Applied to Saw Cut Faces in Concrete Pavement Joints*. Wisconsin Highway Research Program Madison, WI.

Xiao, D. X., G. Adil, J. Kevern, and S. Owusu-Ababio, S. 2021. Field Performance of Concrete Pavement Joints Protected by Silane Sealer: Case Study. *Journal of Performance of Constructed Facilities*, Vol. 35, No. 6, No. 04021089, pp. 1–8.

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