Evaluation of Penetrating Sealers for Concrete

Literature Review
May 2019

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EVALUATION OF PENETRATING SEALERS FOR CONCRETE

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INTRODUCTION

Background and Problem Statement

The main factors negatively impacting concrete joint durability in Iowa are governed by moisture saturation and deleterious chloride ion reactions from deicing 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. Specifically, extending time to critical saturation reduces freeze-thaw exposure conditions, and limiting chloride ingress reduces the potential for oxychloride formation. The reduction in intrusion of moisture and chloride ions is also a means of improving resistance against reinforcing steel corrosion and further durability of bridge decks.

Numerous products exist in the sealer marketplace with a range of performance claims. A 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 these products.

Sealers can be categorized based on the chemical structure, functional type, amount of active chemical, carrier solution, and the action mechanism, and vary widely in performance. Indeed, the parameters to define satisfactory performance are not agreed upon.

There is a need to establish a suite of performance criteria to define acceptability of a given product, and a protocol for agencies to evaluate a product submitted for approval. The aim of the proposed work will meet those needs.

Research Objectives

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

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

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

- Examine 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 for long-term performance

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

LITERATURE REVIEW

The incorporation of durable materials is an inseparable part of sustainable construction. Given its widespread use, durability of concrete can significantly affect the maintenance cost and service life of transportation infrastructure. The concrete degradation mechanisms are typically tied to the environmental conditions and exposure situation, including variations in seasonal temperature and relative humidity, and the availability of deleterious materials and ions. Frost action, scaling caused by deicing salts, chemical attack, surface damage caused by abrasion and steel corrosion are some of the mechanisms that can result in deterioration of different segments of transportation infrastructure, including structural elements and pavement panels.

Concrete is a naturally porous medium. The interconnected voids can develop a percolating network that can be penetrated by moisture, gas, and ions that accelerate deterioration. Corrosion of the paste and embedded steel, alkali silica reaction, sulfate attack, bulk freeze and thaw (F/T) damage, and scaling can occur upon the intrusion of these external fluids (Kosmatka and Wilson 2016). The size distribution and connectivity of the pores in concrete depend on mix design parameters including the type and content of the binder, water-to-cementitious materials (w/cm) ratio, quality of consolidation, curing conditions, and degree of hydration (Kosmatka and Wilson 2016).

The ingress of moisture and ions responsible for concrete deterioration occurs through three main mechanisms (Christodoulou et al. 2013):

- *Diffusion:* Transport of ions under a concentration gradient
- *Permeation:* Transport of fluids through saturated concrete under pressure
- *Capillary suction:* Transport of liquids due to surface tension in capillaries of porous and non-saturated solids

While the mechanisms act together under natural conditions, capillary suction tends to be the governing mechanism in many cases (Christodoulou et al. 2013).

Durability of Joints in Concrete Pavement

Pavement joints are among the locations where failures are commonly observed, even in cases where the bulk of the pavement exhibits proper durability against frost action and deicing salt scaling. Joints in a rigid pavement can act as local reservoirs trapping deicing salts and moisture. Such a harsh micro-environment inside the joints impacts the durability of concrete in sawn joint surfaces, resulting in premature distress. 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 primary physical and chemical

mechanisms that contribute to joint deterioration are elaborated on in the following sections (Spragg et al. 2011, Sutter et al. 2008, Taylor et al. 2012, Taylor et al. 2016).

Paste Deterioration Due to Deicer Attack

Pressure development in concrete pores caused by salt crystallization above a critical salt concentration is a well-known mechanism of deterioration in concrete exposed to salt solutions (Basheer and Cleland 2006). However, the damage caused by salts is not limited to this one mode of action. Formation of expansive oxychloride, as a result of chemical reactions between the cementitious paste system and deicing salts, causes cracking at regular intervals parallel to the saw cut and/or the loss of paste, thus leaving clean aggregates in the voids (Farnam et al. 2015, Julio-Betancourt 2009). The mechanism of action and type of products vary depending on the type and concentration of the incorporated deicing agent, as well as the ambient temperature.

Deicing salts, similar to many external solutions, may dissolve Calcium Hydroxide (CH) and result in leaching, which leads to permeability increment and concrete alkalinity reduction (Farnam et al. 2005a). In addition, at high concentrations, a reaction between salts and cementitious constituents can result in secondary deposits in concrete and diminish durability. High concentration of salts is also a common result of anti-icing practices, where deicer solutions are applied to dry pavements. Even at a low concentration, the evaporation that occurs during drying cycles will eventually produce a solution with high salt concentration (Farnam et al. 2015). At some point, these concentrations will reach a level of super saturation that drives the precipitation of secondary mineral deposits in the concrete (Farnam et al. 2015, Julio-Betancourt 2009).

Saturated Frost Damage

The expansion of water in the saturated capillaries of the concrete as it freezes causes cracking. Cycles of freezing and thawing open these cracks, allowing more water to penetrate, and, as a result, the concrete deteriorates incrementally. Concretes that are highly saturated are prone to accelerated damage. Providing a sufficient amount of evenly distributed air bubbles and enhancing the resistance to moisture transport are considered the main factors to improve resistance of the paste to frost damage.

Characteristics of an entrained air-void system are typically quantified in terms of the volume of air in fresh and hardened concrete (% volume), distribution of the bubbles within the hardened paste known as spacing factor, and fineness of the bubbles known as specific surface area. Typically, a spacing factor no larger than 0.008 in. (200 µm) and a specific surface area of over 600 in.^{2}/in.^{3} (25 mm^2/mm^3) is considered to be adequate for protection against freeze and thaw cycles. An automated stereomicroscope can be employed for measuring the properties of the air-void system in hardened state. Measurements in fresh state using the super air meter (SAM) are also considered as a new means for evaluating the quality of the air-void system in concrete. It is reported that a SAM number of 0.20 or lower (Ley et al. 2017) can guarantee a desired hardened air-void system with a spacing factor lower than 0.008 in. (200 µm).

Note that in a concrete with a degree of saturation over the critical limit of about 0.85 (AASHTO PP 84), the availability of a well-distributed air-void system may not necessarily be able to secure frost durability. Degree of saturation is defined as the portion of pore volume filled with a liquid phase. Therefore, limiting the moisture penetration into concrete is an essential step in developing frost-durable concrete mixtures.

As stated earlier, capillary suction is typically considered the dominant transport mechanism for concrete exposed to atmospheric exposures. A non-steady state transport mechanism exists for short-term contact between the liquids and the concrete surfaces in such environments. This transport mechanism can be quantified by monitoring the increase in mass due to capillary water absorption as a function of the square root of the time, usually termed as rate of absorption ($kg/m^2/h^{0.5}$) as introduced in equation (1):

$$Rate\ of\ water\ absorption = \frac{W_w}{(A_c\sqrt{t})} \tag{1}$$

where W_w is the weight gained by the specimen in contact with water (g), A_c is the surface area of the specimen in contact with water (mm^2), and t is the time of exposure (s).

The non-steady state transport will convert into a steady-state diffusion mechanism for elongated periods of contact between the liquid and concrete surface. Rate of water absorption is typically determined using the ASTM C1585 test method. An example of a moisture uptake profile typically obtained using this test is presented in Figure 1 (Liu and Hansen 2016, AASHTO PP 84).
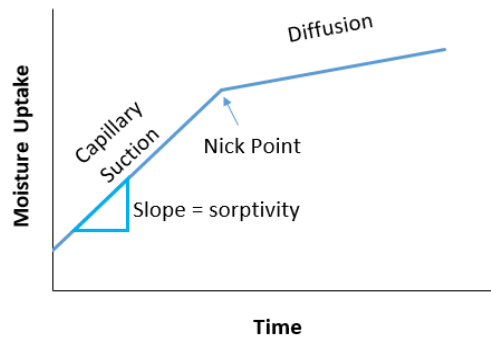
Note the nick point separating the capillary suction and diffusion mechanisms
After Liu and Hansen 2016

Figure 1. Typical moisture uptake profile obtained during sorptivity testing

Durability Cracking of Coarse Aggregate

Expansive freezing of water trapped inside some types of aggregate particles leads to damage that normally starts near joints and forms a characteristic D-shape crack pattern. These cracks typically start forming from the bottom of the pavement where ample moisture is provided by a saturated base. D-cracking typically starts with a darker color at joint locations that suggest

higher internal moisture content in comparison with mid-panel areas. Upon to exposure to F/T cycles (3 to 20 years), these areas will exhibit cracking, as stated earlier, and progressive pavement distress will be the final outcome. (Kosmatka and Wilson 2016). Figure 2 presents a sample of D-cracking damage in concrete pavement.

Figure 2. Severe D-cracking in a pavement section in Pennsylvania inspected by the research team

The F/T resistance of aggregate is believed to be a factor of its porosity, absorption capacity, permeability, pore structure, and tensile strength (Kosmatka and Wilson 2016). If saturated to a critical degree, an aggregate with a high absorption rate may not be able to tolerate the expansive pressures caused by the water freezing inside. This can eventually result in cracking within the aggregate structure as shown in Figure 3.

Figure 3. Cracks propagate within the coarse aggregate structure

The physics are similar to what has been observed for F/T distress in cementitious pastes, where air entrainment is employed for pressure dissipation and proper means are required to avoid critical degrees of saturation.

It is generally reported that the coarse aggregates with high porosity values that contain medium-sized pores (within the range of 3.9 to 197 µin. [0.1 to 5 µm]) are prone to F/T damage. The capillary forces in such pores can cause the aggregates to become saturated easily. At a certain rate of freezing, water in the pores cannot move out and thus develops internal pressure high enough to fracture the aggregate particle (Kosmatka and Wilson 2016).

Highly permeable aggregates mostly contain large pores that do not reach the critical saturation easily. Also, the availability of such large pores permits easy water movement, making it possible for the water to expel from aggregate during freezing without generating stress.

Low-permeability aggregates with porosities limited to 0.3% are shown to be strong enough to absorb the stress resulting from freezing water within their elastic limit. Moreover, the water available in the fine pores does not freeze readily.

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 also may be caused by aggregate particles being dislodged during sawing, typically because the concrete strength is too low when sawing is conducted.

7

Early-Age Drying Damage

High evaporation rates during placement results in large differences in moisture content through the depth of the concrete slab. These differences may lead to stresses high enough to cause fine horizontal cracks and delamination. In areas where these horizontal cracks intersect vertical cracks or joints, concrete material can break free and "flat bottom" or delamination spalling can occur.

Considering the aforementioned mechanisms of damage, the following solutions have been proposed to reduce the risk of distress:

- A low w/cm ratio (i.e., about 0.40 to 0.42) to reduce permeability
- Use of supplementary cementitious materials to maintain high Si/Ca ratio that are more resistant to oxychloride formation
- Optimizing the construction techniques and enhancing the stability of the air-void system to secure an adequate air-void system behind the paver
- Providing an adequate drainage system beneath the concrete
- Limiting the use of aggressive salts, especially magnesium and calcium salts, to times and temperatures (typically $< 15°F$ [$-9°C$]) when necessary for safety
- Use of surface treatments to slow water and salt penetration into the microstructure

A challenge with the last recommendation is that there has been no guidance available on what products to use.

Reinforcement Corrosion in Bridge Decks

Reinforcement corrosion is one of the dominant factors that govern the service life of reinforced concrete (RC) structures in harsh exposures, e.g., marine environments. Corrosion can also result in premature deterioration of bridge decks exposed to salt treatment during the cold seasons. Corrosion is an electrochemical reaction that only can take place if all the elements of a corrosion cell are available, including the anode, cathode, electrolyte, and the electrical connection.

Corrosion can initiate if the chloride ion concentration reaches a threshold on reinforcing steel bar surfaces, or if the pH of the surrounding concrete drops (due to carbonation) and the oxide layer on the reinforcing bar surface de-passivates (Sadati et al. 2016). Progressive reinforcement corrosion can affect the structural integrity in following ways: (1) reduction in cross sectional area of the reinforcing steel, (2) reduction in bond strength between the reinforcing bars and the surrounding concrete, and (3) cracking and spalling of the concrete cover in light of the formation of expansive corrosion products (Sadati et al. 2016).

In addition to the exposure conditions, chloride-induced deterioration is typically affected by the chloride diffusion coefficient, threshold chloride, and subsequent corrosion rate (Glass and

Buenfeld 2000). Lowering the rate of chloride ion ingress is a proven methodology for delayed corrosion and elongated service life of reinforced concrete structures.

When chloride ions enter concrete, they may get trapped in bonds with the matrix or continue to be transported within the pore solution, accumulate on the surface of reinforcing bars, and initiate corrosion (Sadati et al. 2017). Being categorized as physical or chemical, chloride binding in concrete is defined as the interaction between the chloride ions and the matrix. Given the fact that unbound free chloride ions are able to move toward the rebar in concrete, the rate of chloride ingress and service life of RC structures may be affected by the binding capacity of the matrix (Khan et al. 2016).

Adsorption of chloride ions to the hydration products, including calcium silicate hydrate (CSH), calcium aluminate hydrate (CAH), ettringite, and monosulfate accounts for the physical binding in concrete (Luo et al. 2003, Hirao et al. 2005). However, ions can also take part in reactions with alumina-bearing phases of cement (C_3A and C_4AF), which result in chemical binding. Reaction with C_3A produces calcium chloroaluminate hydrate (C_3A $CaCl_2$ $10H_2O$), usually regarded as Friedel's salt. Reaction with C_4AF leads to calcium chloroferrite (C_3F $CaCl_2$ $10H_2O$) production. The former is usually reported to play the major role in the chemical binding process (Cheewaket et al. 2010).

Chloride ion concentration, cement composition and content, supplementary cementitious material (SCM) type and content, w/cm ratio, and curing conditions are considered to be some of the main factors affecting the binding capacity (Andrade et al. 2015). Given the fact that Friedel's salt will not be stable at low pH values, variation in pH of the pore solution due to such effects as carbonation can also affect the chloride binding (Kobayashi et al. 1989, Suryavanshi and Swamy 1996). Similar behaviors were also reported by Anstice et al. (2005) who reported an increase in the concentration of free chloride ions in CO_2-enriched environments. Such a mechanism can increase the depth of chloride ion migration and enhance the corrosion risk as reported by Eto et al. (2014) and Kuosa et al. (2014).

Use of Surface Treatments for Enhanced Concrete Durability

As stated earlier in this report, the review of existing literature, both in joints and in a bridge deck, indicates that all mechanisms of damage are correlated with ingress of fluids into concrete. The use of high-performance concrete mixtures prepared with SCMs, suitable aggregate sources, water reducing agents, and air entrainment is considered the most effective means of securing long-term durability of concrete materials in harsh exposures. With the use of such high-performance mixtures, significant improvements have been made in in enhancing the service life of concrete elements. However, even though beneficial, such approaches may not be sufficient for the most severe applications and exposure situations.

Surface treatment can be considered an additional measure for protection of concrete against ingress of deteriorating substances, e.g., chloride ions and moisture. Concrete sealers can be divided to three main categories based on their mechanism of action as shown in Figure 4.
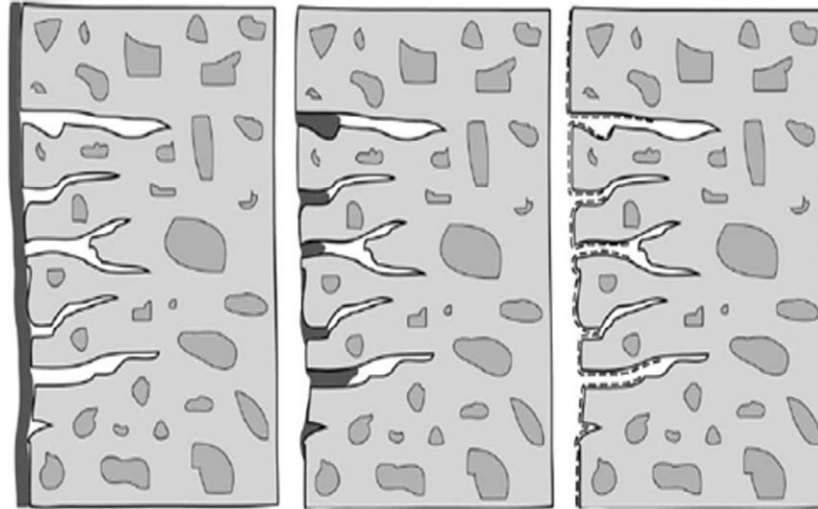
Surface coatings   Pore blockers   Pore liners

Medeiros and Helene 2009, adapted from Bentur et al. 1997

Figure 4. Concrete sealers based on mechanism of action

- *Water repellents:* These sealers 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:* Sealers with a viscosity sufficiently low to allow penetration into the concrete pores will seal the pores while leaving little or no measurable coating on the exterior surface of the concrete.
- *Barrier coatings:* These coatings are too viscous to penetrate inside the concrete pores, but they form a thick barrier layer on the surface that blocks the pores.

Several studies have demonstrated the positive effects of surface coatings on the durability of concrete structures. Moradllo et al. (2012) and Sadati et al. (2015, 2017) reported extended time to corrosion initiation and longer service life of marine reinforced concrete structures protected with a variety of surface barriers, including polyurethane, aliphatic acrylic, bitumen and rubber emulsion, and polymer-modified cementitious coatings. However, given the potential influences on skid resistance, barrier coatings may not be considered the best candidates for pavement surfaces and bridge decks.

Barrier coatings are also vulnerable to premature failure due to abrasion damage under traffic loading. Moreover, it is reported by Wright et al. (1993) that the enhanced internal hydraulic pressures during freeze and thaw cycles in coated concrete specimens can accelerate the damage in concrete elements exposed to subzero environments. Therefore, use of surface treatments that facilitate the breathability seems to be necessary. Pore liners, also known as penetrating sealers, can be considered an effective means for treating concrete surfaces in rigid pavement construction and bridge deck applications.

The capillary suction, earlier introduced as the main mechanism of transport in atmospheric exposures, is considered to be a function of the surface tension of the liquid and its contact angle with a pore as demonstrated in equation (2) (Liu and Hansen 2016):

$$\Delta P = \frac{2\sigma \cos\theta}{r} \qquad (2)$$

where σ is the surface tension of the wetting liquid, θ is the contact angle, and r is the pore radius.

Given a contact angle of lower than 90°, capillary suction can occur spontaneously and in the form of an attraction between the liquid molecules and the porous substrate. This leads to a capillary rise and a concave meniscus as depicted in Figure 5.
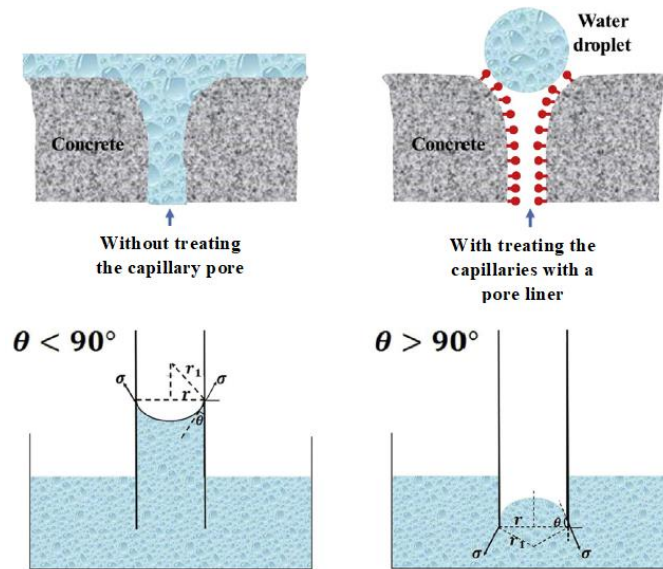
Figure 5. Contact angle, meniscus formation, and capillary suction with (right) or without (left) a pore liner

In the case of a treated substrate where capillary pores are lined with a hydrophobic sealer, such pressure differences will be reversed and extra energy will be required for moisture penetration (Liu and Hansen 2016).

A wide range of sealers are being used for concrete protection. Concrete sealers can be used on both fresh concrete and old concrete that is already in service. The sealer families listed in Table 1 are among those most commonly used.

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 [i]	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 [ii]	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 [iii]	Reactive resins
Polyesters	Pore blocking or barrier coat [iii]	Synthetic resins
Chlorinated rubber	Pore blocking or barrier coat [iv]	Chlorinated polyisoprene
Silicones	Water repellent	Silicon based with two organofunctional group
Vinyls	Pore blocking or barrier coat [iii]	Polymers of acrylic and methacrylic acid

[i] Acting as pore blocker when less than 50% active ingredient
[ii] Pore blocking if solvent based, barrier coating if water based or high molecule weight
[iii] Pore blocking if diluted and barrier coat if not diluted
[iv] Mainly barrier, but can be pore blocker if too diluted

Historical Performance of Penetrating Sealers

Silane, Siloxanes, and Siliconates

Silanes, siloxanes, and siliconates are chemically classified as organosilicon compounds. All three are sourced from chlorosilane (see Figure 6) and have three silicon functional groups and one organofunctional group (Christodoulou et al. 2013).
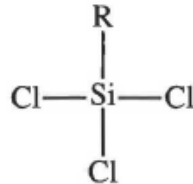
Cady 1994, NCHRP

Figure 6. Chlorosilane is the raw material used for production of silanes, siloxanes, and siliconates

Silanes, siloxanes, and siliconates molecules bond to the silicates structures of the hydrated cement paste (substrate) and also to each other by means of the silicon functional groups. The exposed organofunctional groups, on the other hand, provide a hydrophobic layer on the pore walls (Cady 1994, Christodoulou et al. 2013). The formed water repellent layer avoids penetration of liquid water (and the ions dissolved in it) into concrete, while enabling passage of water vapor and other gases.

Silanes and siloxanes are more commonly used as concrete sealers compared to the siliconates (Basheer and Cleland 2006). However, the efficiency of these treatments tends to degrade over time due to exposure to ultraviolet light, physical abrasion, weathering, and alkaline attack of the hydrophobic molecules, i.e., breakdown of silicon bonding by the alkalinity of the pore solution (Moradllo et al. 2016).

Dang et al. (2014) investigated the effect of two alkyltrialkoxy silane-based sealers on durability of concrete designated for bridge decks. The authors reported improvements in abrasion resistance, which was attributed to the formation of a strong surface layer in light of reactions between the silicates and portlandite. Sorptivity tests according to ASTM C1585 indicated significant reduction in initial absorption rate, highlighting the reduced capillary suction with use of silanes. Significant reduction in mass loss due to deicing salt scaling was observed for the treated samples. Gas permeability was similar to that of the control sample without any surface treatments. The use of silane-based sealers also increased the contact angle of the water in comparison with the control specimen.

Christodoulou et al. (2013) investigated the long-term in situ performance of reinforced concrete structures impregnated with silane. The incorporated sealer was a water-based alkyl alkyloxi

silane with 20% active ingredient, applied at a rate of 0.074–0.123 gal/ft^2 (3–5 l/m^2) using a brush.

The study involved an examination of 32 concrete cores extracted from 8 reinforced concrete bridge supporting cross beams. The beams were treated with silane within 12 to 20 years before taking the core samples. The cross beams were in service for 23–31 years before sealer application. The specimens were tested for water absorption in accordance with BS EN 13057 (2002). Results were compared to the data obtained from testing 16 cores extracted from 4 similar elements, but not treated with sealers.

The authors indicated a residual protective hydrophobic effect by the silane treatment, even 20 years after sealer application. In almost all examined samples, it was observed that the younger sealers outperformed the older ones, exhibiting a time dependent performance for silane (Christodoulou et al. 2013).

Basheer and Cleland (2006) investigated the effect of silane and siloxane sealers on F/T durability. The study involved one alkyl trialkoxy silane and one oligomeric siloxane. The silane was used at two active ingredient levels of 40% and 100% in ethanol and the siloxane was undiluted. Silane was applied in a rate of 0.008 gal/ft^2 (0.33 l/m^2) and siloxane was applied at rate of 0.005 gal/ft^2 (0.22 l/m^2), both 10% higher than the minimum recommended dosage by the producer. The treatments were applied in two coats, with a six-hour interval between the coats. The treated slabs were cured at 68°F (20°C) and a 55±5% relative humidity (RH) for seven days. Results indicated improvement in F/T performance in terms of resistance to scaling while exposed to deicing solution or deionized water. Moreover, an increase in penetration depth of the sealer proved to be effective in increasing the protection level.

Liu and Hansen (2016) investigated the effect of silane-based water repellents on F/T durability of concrete. Two sealers were used, one with 40% active ingredients and the other one with 100% active ingredients. Specimens from a laboratory investigation exhibited an increase in contact angle between water and the treated concrete surface, as presented in Figure 7, followed by significant reduction in sorptivity and moisture uptake.
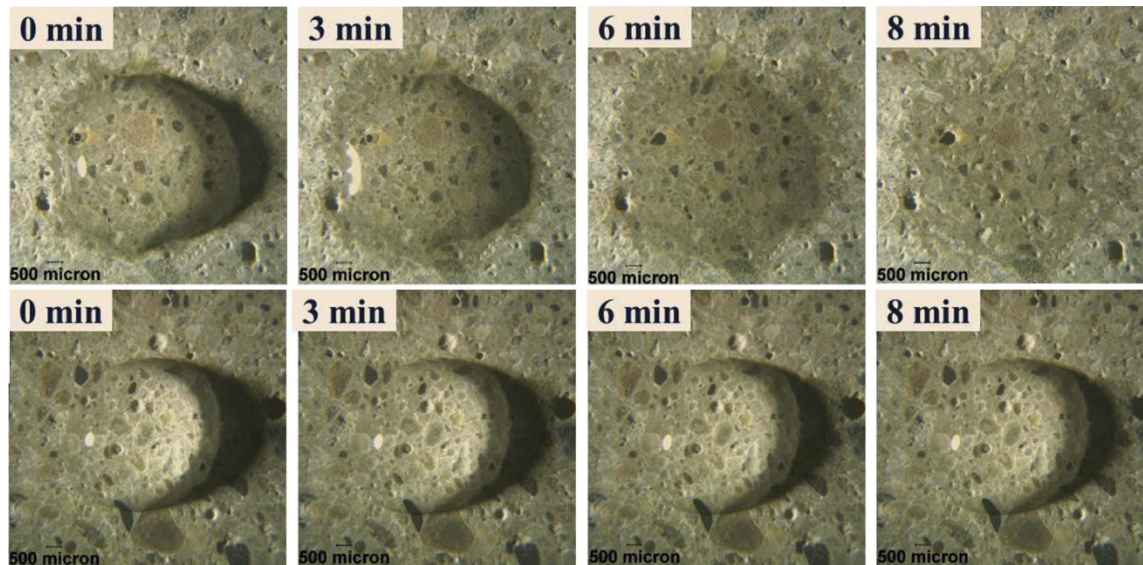
Figure 7. Increase in contact angle due to the hydrophobic effect of silane with 40% active ingredient showing untreated control sample (top row) and silane-treated concrete (bottom row)

Results obtained from testing the laboratory and field specimens indicated the positive effect of silane treatment on scaling resistance of the concrete with a high w/cm ratio exposed to deicers. However, the authors reported that the silane treatment failed to protect the concrete with an inadequate air-void system against bulk moisture uptake and internal distress caused by F/T cycles.

Moradllo et al. (2016) investigated the field performance and effective life of silane-based water repellents applied on bridge decks in Oklahoma. The study summarized data obtained from testing 360 concrete cores, extracted from 60 bridges in service for 6 to 20 years. The authors employed a visual technique, based on staining the split cores to determine the depth of sealer penetration as shown in Figure 8.
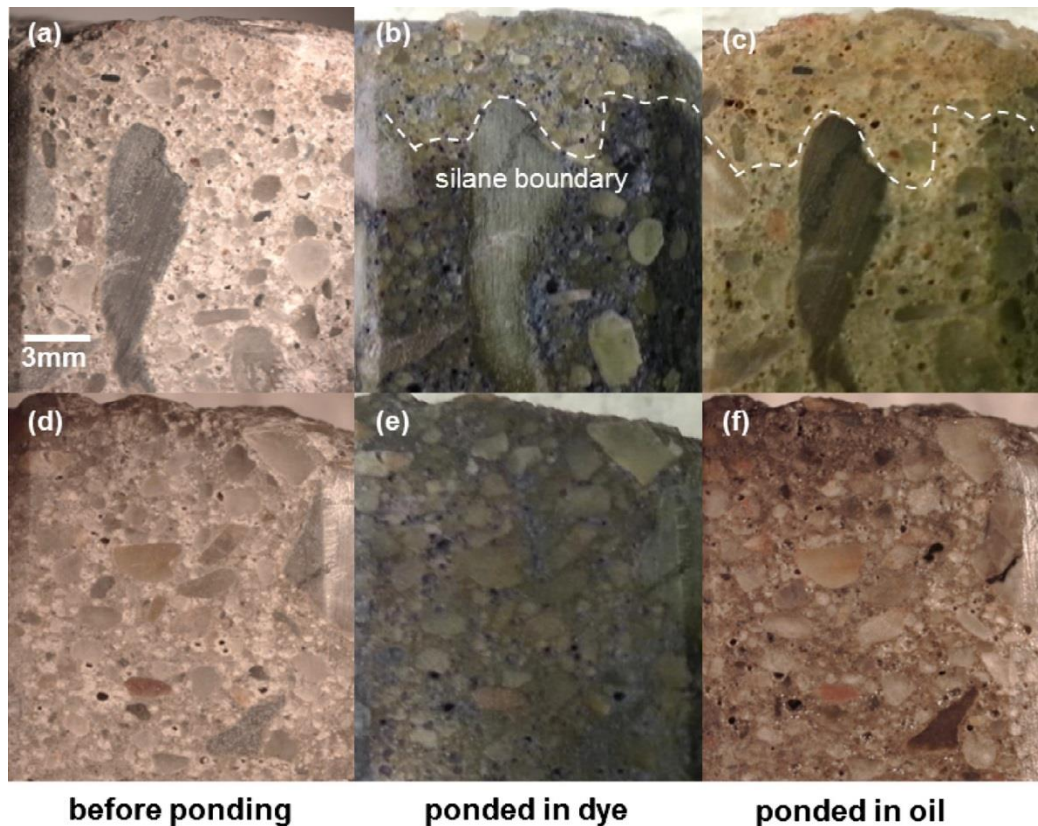
Figure 8. Staining technique incorporated by Moradllo et al. (2016) to investigate the depth of silane-based sealer penetration into bridge deck concrete showing cores treated with silane (top row) and specimens without any treatment (bottom row)

A time-dependent efficiency was reported by the authors, where 100% of the sealers younger than 12 years old exhibited a penetration depth of greater than 1/8 in. (3.2 mm) as the minimum requirement. This was reduced to 68% in the case of sealers applied over 15 years before testing, and further reduced to 16% for sealers between 17 and 20 years old. The authors also reported similar silane thickness for the travel lane and the shoulder areas, indicating minimal degradation due to abrasion. More importantly, fewer than 5% of the investigated samples exhibited deterioration of silane at the concrete surface, indicating minimal damage caused by ultraviolet light and weathering. The reduction in overall thickness of the silane layer over time, however, was reported to be mainly due to the interaction with the alkaline pore solution.

In addition to observed deterioration from the bottom from attack of the high pH, the researchers also observed the lack of sulfate in the surface, silane layer (Ley et al. 2011). The sulfate-bearing phases including ettringite and monosulfate had been dissolved and migrated or forced out completely where silane was present.

A research study led by the South Dakota DOT evaluated two types of sealers for enhancing the joint durability. The investigated sealers were a siloxane-based and a silane-based product. The results obtained from a laboratory investigation indicated the efficiency of the incorporated

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materials in reducing the moisture transport and degree of damage caused by the deicing chemicals (Sutter et al. 2008).

Research at the Minnesota DOT (MnDOT) MnROAD facility investigated the effect of silane- and siloxane-based sealers on chloride ion ingress into joints of existing pavements. Results obtained by the authors indicated no significant difference between the properties of the treated and reference joints during the two-year study window (Sutter and Anzalone 2016).

Mostafa and Sanders (2018) conducted a study on sealers to enhance scaling resistance of Nevada bridge decks. The testing included chloride ion intrusion (AASHTO T 259/260), F/T resistance (ASTM C666), rapid chloride ion permeability (ASTM C1202), and salt water absorption (Pfeifer and Scali 1981 with NCHRP 244 Series II). Overall, the authors indicated that silane sealers performed better than the siloxanes sealers, and water-based sealers performed better than the solvent based sealers. Sealers with chemical family of alkylalkoxy silane performed better than all the other sealers, and it was recommended to use a sealer of alkylalkoxy silane and a water-based sealer.

Soy Methyl Ester (SME)

Soy methyl esters (SMEs) consist of plant oils made from long-chain fatty acids, esterified to a methyl group. SME is a derivation of soybean oil, which has a different chemical composition and structure after esterification from the original oil. SMEs are nontoxic and biodegradable products. The potential for water repellency and reduction in fluid transport in concrete, stemmed from the hydrophobic nature of the SMEs, makes them suitable candidates for use in the construction industry. Contrary to soybean oil, the SMEs can act as desirable solvents for synthetic polymers. Polystyrene (PS) and polyvinyl chloride (PVC) can be dissolved in SMEs to produce SME-polymer combinations with properties tailored for specific applications. Alterations in such fluid properties as viscosity and facilitating the deposition of PS in concrete pores are among these modifications. The addition of PS is shown to increase the viscosity of SME-PS mixtures, exhibiting a gradual increase with up to 10% PS addition and a significant jump afterward (Coates et al. 2009). The increase in viscosity is believed to be due to the entangling of the long chain-shaped molecules of PS.

Coates et al. (2009) investigated the feasibility of using SME with 1%, 5%, and 10% PS additions as penetrating sealers. Mortar samples were used for evaluating the effect of SME-PS combinations on water absorption. Both horizontal and vertical surfaces were investigated. The results indicated up to 85% and 93% reduction in total water absorption when between 0.005 and 0.008 oz/in^2 (0.020 and 0.036 g/cm^2) of sealer was applied on flat surfaces. The authors also ponded the notch prepared on hardened mortar specimens to simulate the impregnation of the vertical surfaces, e.g., pavement joints. A solution of SME with 5% PS was used on an oven-dry surface and allowed to penetrate for 48 hours. X-ray measurements indicated no detectable penetration of water into the treated specimens, while a moisture penetration depth of 0.3 in. (8 mm) was detected for reference specimens after two hours.

Golias et al. (2012) have also investigated the performance of SME-PS as concrete sealer. A SME with 5% PS was compared to two other sealants in this study; (1) a solvent-based alkylalkoxysilane sealer (SBS) with greater than 50% active ingredients, which is actually a solution of silane in isopropanol solvent, and (2) a water-based alkylalkoxysilane penetrating sealer containing 40% silane, which is actually an emulsion of silane in water.

Sample conditioning similar to that prescribed for ASTM C1585 sorptivity test was employed by Golias et al. (2012) to investigate the depth of SME-PS penetration at different levels of relative humidity. Results indicated a sharp decrease in the rate of SME-PS absorption into concrete as a function of the increase in ambient RH from 50% to 80%.

Results also indicated superior performance of SME-PS treated specimens compared to the reference and SBS treated concrete, which failed after exposure to rapid F/T cycles. It was reported by the authors that, when silane-bearing sealants are used, the carrier solution will eventually evaporate, leaving behind a layer of the protective silane membrane on the specimen surface. Thermal deformations imposed during F/T cycles can lead to cracking of the membrane and efficiency loss. However, the SME will not cure or harden, hence not experiencing such thermal stresses. This was further elucidated with testing the treated specimens for water absorption at the end of the F/T cycles. Again, SME treated samples exhibited a desirable performance, with minimal reduction in performance (i.e., minimal increase in sorptivity) due to F/T deterioration (Golias et al. 2012).

Linseed Oil

Being categorized as a vegetable oil, linseed oil is produced from flaxseed and consists of a mixture of organic acid triglycerides. While drying, linseed oil hardens due to oxidative polymerization, a process that is very slow with raw linseed oil. The oil is therefore boiled over mild oxidizers to accelerate the polymerization process while exposed to atmospheric oxygen. Dilution in mineral spirits, kerosene, or Stoddard is considered afterward to reduce the viscosity of the boiled linseed oil. The final product can be used as a concrete sealer (Cady 1994).

Linseed oil sealers were commonly used as anti-scaling compounds and chloride barriers for concrete pavements in the late 1960s. The effect of linseed oil on the compressive strength of concrete has been a concern. It was reported (Cady 1994, Blankenhorn et al. 1978) that linseed oil softens and deteriorates the concrete shortly after impregnation as a result of chemical reaction with free lime. The softening is reported to be temporary and followed by a hardening, which is attributed to the solidification or polymerization of the linseed oil that forms an internal reinforcement system.

Wright et al. (1993) investigated the performance of linseed oil treatment in comparison to a silane and a siloxane system both in laboratory and in field conditions. Depth of sealer penetration was determined for laboratory specimens. Cube specimens measuring 3.0 in. (76 mm) were conditioned at 73°F (23°C) and 50% RH for 14 days before sealer application. This was followed by 45 days of drying with the same conditions as after impregnation with sealers. Cubes were then cut with a dry saw and split samples were immersed in water for 1 min. Depth

of sealer penetration was determined based on the visual inspection and measurements using a digital caliper. Penetration depths of 0.1 in. (2.3 mm), 0.008 to 0.08 in. (0.2 to 2.0 mm), and lower than 0.02 in. (0.5 mm) were reported for the linseed oil, silane, and siloxane, respectively (Wright et al. 1993).

The use of sealers resulted in reduced vapor transmission compared to the reference sample, with the lowest vapor transmission values (rate of moisture evaporation) obtained for the sample treated with linseed oil (Wright et al. 1993). Abrasion of the top 0.02 in. (0.5 mm) of the specimens did not affect the efficiency of the linseed oil sealer against absorption, while significant increase in absorption was observed for silane and siloxane treated samples. Depth of chloride ion penetration, determined according to AASHTO T 259, was also reduced with the use of linseed oil and siloxane. Samples treated with silane exhibited the highest depth of penetration after 90 days of ponding with 4% sodium chloride solution.

The authors reported desirable performance of linseed oil treated concrete against deicing salt scaling regardless of the air entrainment. Moreover, the mass loss due to exposure to rapid F/T cycle was minimized with the use of linseed oil, compared to the maximum values obtained for siloxane treated specimens. This was attributed to better breathability of the linseed oil treated concrete and development of hydraulic pressure in samples treated with siloxane.

The same sealers were also applied to the following pavement surfaces: seven-day-old city street and highway and seven-year-old airfield pavement in Manitoba, Canada. Core samples were extracted from the pavements at different time intervals up to three years after application. The cores were tested for salt-water absorption and chloride ion intrusion. Results indicated a better performance for linseed oil treated samples, compared to those sealed with silane and siloxane. Linseed oil exhibited a uniform quality in terms of reducing the relative salt water absorption for three years. However, a slight increase in chloride ion intrusion at year three was observed for linseed oil treated samples (Wright et al. 1993).

Crystalline Waterproofing Products

Crystalline waterproofing products are argued to be beneficial while used as admixtures or as topical treatments. The silicate-based agents available in crystalline waterproofing systems are reported to react with hydration products to form insoluble crystals that block the concrete pores. Cappellesso et al. (2016) used crystalline waterproofing materials as both admixtures and coatings. The authors reported a reduction in water penetration when crystalline waterproofing was used as a surface treatment for concrete specimens.

Chemical and Particle-Based Silicate Materials

Lithium/sodium silicates and colloidal silica are additional classes of materials that have potential for use as penetrating sealers. Traditionally, these materials are used to densify or harden concrete surfaces for polishing purposes.

Depending on the mass fraction and size of nano-silica, these coatings can fill the spaces between particles and calcium silicate hydrate (CSH) gels to densify the microstructure. The nano-sized or silicate chemicals react with free CH to create additional CSH and reduce permeability (Aggarwal et al. 2015).

Although products exist in the marketplace that suggest benefits as a chemical admixture, fundamentally, CH must be produced before the secondary densification reaction can take place. As a curing method, lithium silicates have not been shown to prevent chloride penetration better than conventional curing techniques (Hajibabaee et al. 2018).

Evaluating Performance of Penetrating Sealers

For a certain type of sealer, the efficiency can depend on several factors, including the pore structure of the substrate, initial moisture content of the substrate, and surface preparation (Dai et al. 2010). The application process and coating intervals between coats can vary for different products and manufacturers. However, the results obtained during this review suggest the time-dependent performance of the sealers and their degradation over time. This highlights the need for proper understanding of different materials and considering re-coating intervals for sealed concrete.

Most laboratory tests seeking to evaluate penetrating sealants are based on measuring their influence on ion penetration, fluid permeability, water sorption, concrete surface abrasion and skid resistance, ability to resist freeze and thaw, and microstructure phase changes.

A review of the existing literature demonstrates the fact that there is no uniform approach for evaluating the performance of concrete sealers. This highlights the need for proper tests for laboratory evaluation of fundamental transport properties that correlate well with the field data. A review of the existing test protocols by neighboring DOTs is underway.

Results of a similar survey on state DOT crack and deck sealing practices conducted by Mamaghani et al. (2007) are summarized as follows:

- **Colorado DOT**: State approved silane sealers are used for bridge decks. However, the DOT is considering avoiding the use of silane or siloxane sealers to reduce the need for re-application of sealers.

- **Michigan DOT**: Penetrating sealers are used for bridge decks and vertical surfaces. The substrate should be at least 28 days old and surface should be prepared with high-pressure washing. Sandblasting is recommended for new surfaces to ensure removal of curing compound.

- **Montana DOT**: High molecular weight methacrylate (HMWM) is used for sealing deck cracks.

- **Nevada DOT**: Linseed oil is used for roadways, with great satisfactory results in reducing the concrete permeability.

- **Ohio DOT**: HMWM is used for treating concrete wearing surfaces.

- **South Carolina DOT**: Monomeric alkyltrialkoxy silane compound with 40% active ingredient mixed with an anhydrous alcohol solvent or mineral spirits is used. A maximum rate of 125 ft^2/gal (3 m^2/l) and a minimum depth of penetration of 0.2 in. (4 mm) in deck concrete is required. Performance is evaluated based on the following tests: 1) ASTM D1653 for vapor transmission, with a criteria of minimum 98% in relation to uncoated specimen; 2) NCHRP 244 Series II cube test for accelerated weathering, where weight gain should be limited to 20% of the weight gain observed for uncoated cubes and absorbed chloride should be limited to a maximum of 20% of uncoated cubes (Pfeifer and Scali 1981); and 3) NCHRP 244 Series IV (Pfeifer and Scali 1981) slab test (southern climate), where absorbed chloride should be limited to 10% of the uncoated slabs at the end of 24 weeks.

- **South Dakota DOT**: Bridge deck crack and surface sealing activities is recommended within three to six months after construction and repeated at five-year intervals.

- **Alberta DOT**: The use of penetrating sealers and coating systems with the Alberta DOT goes back to the 1960s. Epoxy coatings, epoxy and acrylic coatings, and silane sealers are used. The Alberta DOT generally considers a four-year interval for sealing their bridge decks.

Results of a research project by the Missouri DOT (Wenzlick 2007) indicated linseed oil as the bridge deck sealer listed in standard specifications for resistance to scaling but not recommending it as a crack sealer. The same report summarizes the following as specifications used by other DOTs:

- **Iowa** (non-riding surfaces): AASHTO T 259, requiring less than 10% chloride ion compared to control sample at 1/16 in. to 1/2 in. (1.6 mm to 12.7 mm).

- **Illinois**: Linseed oil is used for resistance against scaling.

- **Kentucky** (non-riding surfaces): F/T testing for 50 cycles with no visible defects, ASTM D822 for accelerated weathering with slight color change allowed, ASTM B117 for salt spray resistance with no loss of adhesion after 300 cycles, FS TT-P-29 for fungus growth resistance with no growth at 21 days.

- **Tennessee**: AASHTO T 259 with maximum 1.0 pcy at ½ to 1.0 in. (12.7 mm to 25.4 mm) after 90 days of ponding.

- **Arkansas** (Silane or Siloxane): ASTM C642 for absorption with maximum of 1% by weight

at 48 hours and 2% by weight at 50 days, and AASHTO T 259 with maximum 0.76 pcy at ½ to 1.0 in. (12.7 mm to 25.4 mm).

- **Oklahoma**: Absorption testing according to OHD L39 with maximum of 1% by weight after 48 hours and minimum penetration depth of 0.15 in. (3.81 mm) according to OHD L40 test method.

- **Nebraska**: AASHTO T 250 with maximum 0.76 pcy at ½ to 1.0 in. (12.7 mm to 25.4 mm).

- **Wisconsin**: No sealer specifications at the time, but the following proposals from University of Wisconsin-Madison: AASHTO T 259 with half of the chloride ion penetration compared to control, ASTM C672 with 1 rating lower than the untreated sample.

- **Texas**: Linseed oil or penetrating types of sealers to be applied to concrete surfaces. Following requirements according to DMS81-40: maximum 1% absorption to ensure water repellency and minimum penetration depth of 0.25 in. (6.35 mm). Maximum of 2.25% absorption after 1,000 hours of weathering according to Tex-898-B. Density should not vary by more than 0.05 lb/gal (5.99 g/l) (ASTM D1475) compared to pre-approved sample. Infrared spectrum to match with that of the pre-approved sample according to (Tex-888-B). Gas chromatograph to match with that of the pre-approved sample.

- **Ohio**: Reactive silica is recommended with the following performance criteria: scaling (ASTM C672) to be limited to rating 0 after 100 cycles, ASTM C642 absorption to be limited to 1% after 48 hours and 2% at 50 days. Skid resistance (ASTM E274) not to reduce by more than 10%.

Literature Review Summary and Conclusions

There is a need to establish a suite of performance criteria to define acceptability of a given product, and a protocol for agencies to evaluate a product submitted for approval. The aim of the proposed work will meet those needs, and this Literature Review helped define the basis of this investigation going forward.

- The bulk of the technical literature agrees on the fact that the incorporation of penetrating sealers offers significant improvements in long-term durability and longevity of the transportation infrastructure.

- Test procedures employed by different DOTs mainly focus on water absorption of an immersed sample, chloride ion penetration after ponding, deicing salt scaling, and sealer penetration depth.

- Results obtained through the literature search indicate that the degree of efficiency of the sealers degrades with time, and re-applications are required to ensure durability. The interval, however, may vary depending on the sealer type and extent of weathering.

- A review of the current literature confirms the need for establishing laboratory testing specifications and selection criteria that correlates well with field observations.

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