Comparative Evaluation of Concrete Bridge Deck Sealers

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16. Abstract

The main objective of this research was to compare the performance of five bridge deck sealer products using a synthesis of two testing methods: NCHRP Report 244 Series II tests and standards developed by the Alberta Ministry of Transportation (BT Series). This research also sought to develop an improved method for future evaluation of bridge deck sealer products for the Alabama Department of Transportation (ALDOT). The sealer products were evaluated for their resistance to moisture and chloride ingress, performance after simulated traffic wear, and depth of sealer penetration into the concrete. The products tested included four epoxy-based sealer products and one sodium silicate sealer product.

The sealers were evaluated on three different concrete mix designs representing the range of bridge deck concrete in service with ALDOT. Concrete cube specimens were fabricated, sealed, conditioned, and subjected to different tests. One test evaluated the waterproofing performance of sealers before and after abrasive conditioning (representing traffic wear). Another test determined the products' ability to resist chloride penetration and their waterproofing performance in a saltwater solution. The chloride content was measured by potentiometric titration with silver nitrate and X-ray fluorescence spectroscopy (XRF). Sealer penetration depth as determined by a dye method and scanning electron microscopy (SEM).

Initial (pre-abrasion) waterproofing performance results generally showed a good correlation to chloride resistance. XRF and titration analyses provided nearly identical results for measuring chlorides in concrete; however, XRF is a preferred method for determination of chloride content because it is much faster than titration.

Two epoxy sealers outperformed all products for all evaluation criteria. A third epoxy sealer had good overall performance, but many specimens sealed with this product developed cracks that warranted further investigation. The sodium silicate sealer performed poorly compared to all four epoxy-based sealers. The epoxy-based sealers did not exhibit any measurable sealer penetration into the concrete and the sodium silicate sealer penetrated less than 100 μm into the concrete.

17. Key Words **concrete, bridge decks, sealers, chlorides, SEM, XRD, XRF**	**18. Distribution Statement**

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Executive Summary

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The sealers were evaluated on three different concrete mix designs that represent the range of bridge deck concrete in service with ALDOT. Concrete cube specimens were fabricated, sealed, conditioned, and subjected to different tests. One test evaluated the waterproofing performance of sealers before and after abrasive conditioning (representing traffic wear). Another test evaluated the waterproofing performance of sealers in a saltwater solution, and the specimens were further tested to measure their chloride content. The chloride content was determined using three techniques: (1) potentiometric titration by silver nitrate, (2) x-ray fluorescence (XRF) spectroscopy, and (3) the mass gained while immersed in the 15% NaCl solution. Finally, a dye method, optical microscopy, and scanning electron microscopy (SEM) were used to measure sealer thickness and the depth of sealer penetration into the concrete.

The initial (pre-abrasion) waterproofing performance results generally showed a good correlation to the waterproofing performance in saltwater. Because both tests provided similar results, it would only be necessary to use one of those two tests in future evaluations of sealer products. It is recommended that only the saltwater test be used because it also allows for measurement of chloride penetration.

The XRF and titration analyses provided nearly identical results for measuring chlorides in concrete. The chloride contents calculated based on mass gained during saltwater exposure did not correlate well to the XRF and titration results. Therefore, mass gain is not recommended as a measure of chloride ingress, but may still serve as a valid measure of waterproofing performance. For future sealer evaluations, XRF is the recommended method for determination of chloride content because it is much faster than titration.

The sodium silicate sealer penetrated less than 100 μm into the concrete, while all four epoxy-based sealers did not exhibit any measurable sealer penetration into the concrete. Because the epoxy-based sealers formed a relatively impermeable and abrasion-resistant surface film, the absence of penetration into the concrete pore network did not impair their performance. The dye method was not an effective means of measuring sealer penetration depth or thickness, and the use of optical and scanning electron microscopy is recommended for future sealer evaluations.

Two epoxy sealers outperformed all other products for all evaluation criteria. They also scored above minimum performance standards set by NCHRP Report 244 Series II protocols, so those products are candidates to replace ALDOT’s current product, should that be necessary. A third epoxy sealer had good overall performance, but many specimens sealed with this product developed cracks that warranted further investigation. The fourth epoxy sealer exhibited significantly poorer performance compared to the other three epoxy sealers. The sodium silicate sealer performed poorly compared to all four epoxy-based sealers.

A revised test protocol for evaluating bridge deck sealers is proposed in Appendix B of this report. This test protocol retains the most useful elements of the testing followed in this research, and eliminates aspects that were redundant or ineffective for evaluating sealer performance.

Chapter 1: Introduction

1.1 Motivation

Alabama has a total of 16,078 bridges (ASCE 2013); the Maintenance Bureau of ALDOT maintains over 5,000 of them, and cities and counties maintain the others. The Maintenance Bureau uses an epoxy-based concrete bridge sealer to prolong the life of bridges. The sealer works by reducing the penetration of moisture and aggressive substances from the environment (Basheer and Long 1997). ALDOT is currently looking for a potential replacement concrete bridge deck sealer because the agency is the only user of that product, and production could be discontinued at any time. Thus, ALDOT needs to identify suitable replacement products and wishes to identify a viable method of distinguishing effective sealers from ineffective sealers.

In 1981, the National Cooperative Highway Research Program (NCHRP) evaluated four series of tests to evaluate bridge sealers in NCHRP Report 244 (Pfeifer and Scali 1981). The report recommended only the Series II and IV tests to be used for evaluating sealer products. Though these tests were innovative at the time, NCHRP Report 244 noted some shortcomings in its methodologies for evaluation (e.g., limited criteria for properties evaluated and inconsistent testing results for products between the series of tests) (Pfeifer and Scali 1981). The Alberta Ministry of Transportation (MOT) Bridge Tests (BT Series) provide a potentially modernized approach for the evaluation of concrete sealers. The BT Series tests incorporate multiple concrete mix designs and test the post-abrasion performance of sealers. Unfortunately, they lack clarity in some procedural steps and may not be able to effectively evaluate the post-abrasion performance of epoxy sealers.

1.2 Objectives and Scope

The objectives of this research were to compare the performance of five sealer products, including the currently-used product, and to develop an improved method for evaluating the effectiveness of bridge deck sealers using elements of the NCHRP Series II and BT Series test protocols. Researchers fabricated and conditioned test specimens with three different concrete mix designs, and then applied five concrete bridge deck sealer products to the specimens. Multiple test methods from the two protocols were used to evaluate several sealer properties, and recommendations were made for improving the sealer evaluation process based on the results of the testing.

One test determined the waterproofing performance of the sealers after test specimens were immersed in water for 5 days by measuring the mass of water absorbed in sealed specimens. The same specimens were then sandblasted, representing traffic wear, and subjected again to the same 5-day waterproofing performance test. Another test determined the waterproofing performance and resistance to chloride penetration of sealers by immersing specimens in a 15% NaCl solution for 21 days. After the 21-day soaking period, the resistance to chloride penetration was evaluated by measuring the chloride content of the specimens. The chloride content was determined using three techniques: (1) potentiometric titration by silver nitrate, (2) x-ray fluorescence (XRF) spectroscopy, and (3) the mass gained while immersed in the 15% NaCl solution. Finally, researchers investigated the sealers' penetration depth into the concrete and its thickness on the concrete surface. This was done by observing dye-stained samples under an optical microscope. A concrete petrographer aided in the investigation of the sealers' depth of penetration by employing optical microscopy and SEM-EDS.

1.3 Organization

This report is organized as follows:

Chapter 2 presents a literature review of corrosion of reinforced concrete, classifications of bridge deck surface treatments, methods for evaluating the performance of bridge deck sealers, and analytical methods of measuring chlorides in concrete.

Chapter 3 details the overall plan of synthesizing the NCHRP Report 244 Series II tests and the Alberta MOT BT Series tests. Sealer products and concrete materials used in this research are described, and the step-by-step experimental methodology is described.

Chapter 4 presents and discusses the experimental observations and test results. This includes sealer application, waterproofing performance tests, resistance to chloride penetration, depth of sealer penetration, and sealer thickness. One of the sealer products experienced cracking during testing. The cause and severity of this cracking were investigated because the product otherwise performed favorably in the waterproofing performance and chloride resistance tests.

Chapter 5 presents conclusions regarding the test methods, identifies potential areas of improvement for the test methodology, and recommends areas for future research.

Appendix A provides a detailed test procedure for potentiometric titration using silver nitrate for determining chloride content in concrete. This clarifies certain steps of the procedure provided in ASTM C114 that are not adequately explained in the standard. It should serve as a useful reference for researchers attempting to make similar measurements in the future.

Appendix B identifies the three sealers that exceed the minimum performance scores suggested by NHRP Report 244. It also provides a recommended protocol for evaluating concrete bridge deck sealers. This protocol incorporates the findings of this research, including the recommendations given in Chapter 5 for improving the test protocol used for this project.

Chapter 2: Literature Review

2.1 Synopsis of Corrosion in Reinforced Concrete

Dense, high-quality concrete serves as the front line of defense against waterborne contaminants (ACI Committee 201 2008). Many of these waterborne agents are not harmful to the concrete itself, but they may cause problems for the reinforcing steel embedded in concrete (Basheer and Long 1997). Chloride exposure is the primary cause of corrosion in reinforced concrete (Bottenberg 2008). Corrosion is a natural process of material degradation though means of an electrochemical process called oxidation. External chloride ion exposure, such as dissolved deicing salts or seawater mist, remains the most prominent source of chlorides in concrete; the chlorides ingress the concrete as an aqueous solution, diffuse through the pore network, and eventually reach the reinforcing steel (Bottenberg 2008).

Corrosion depends on many factors, such as the permeability of the concrete, the clear cover depth, the relative humidity, and the ambient temperature. Carbonation of the concrete has a synergistic effect when combined with chloride ingress. Carbonation reduces the pH of concrete and this lowers the threshold chloride concentration needed for corrosion when the carbonation front reaches the reinforcing steel. (ACI Committee 222 2010)

2.2 Surface Treatments

Concrete must be able to perform adequately throughout its service life, and concrete surface characteristics strongly influence concrete longevity, but sometimes surface treatments are needed. Surface treatments can be the most cost-effective solution for delaying time to corrosion initiation (Broomfield 2007). Protective surface treatments for concrete are commonly referred to as “sealers” and all are used for the same functions: preventing the ingress of carbon dioxide, water, and waterborne containments (e.g. chlorides) into concrete in order to prevent corrosion initiation.

2.2.1 Five Surface Treatment Classifications

According to the Construction Industry Research and Information Association (CIRIA) Technical Note 130 and ACI Committee 515 (there are five main classifications for surface treatments: penetrant pore liners, penetrant pore blockers, sealers, coatings, and renderings. Basheer and Long (1997) described these classifications based off their protective action, chemical composition and molecular size as follows:

- **Penetrant pore liners** are the least viscous type of sealers (viscosity < 100 cP), which primarily consist of silicon-based compounds. These sealers have hydrophobic (water repellant) properties, and usually penetrate several millimeters into the capillary pores of the concrete substrate. Most silicon-based sealers are made from a water-based or volatile solvent-based (e.g. ethanol) organo-functional group and a silicon functional group. Once the water or volatile organic constituents (VOCs) have evaporated, the inorganic (silicon functional group) compound that remains either reacts with the substrate or acts as an inert filler to repel aqueous solutions. Some examples of pore liners (along with the size of the molecules) include silanes (10 to 20 Å[1]), siloxanes (20 to 75 Å) and other silicate products.
- **Penetrant pore blockers** have similar viscosity and hydrophobic properties as penetrant pore liners, but pore blockers have a relative heavier molecular weight which reduces the capillary pore sizes they can penetrate. Some of these will penetrate 0.04 to 0.12 in. (1 to 3 mm) into the concrete substrate. Some examples include some silicates, silicofluorides, some epoxy resins and acrylics.
- **Sealers** are more viscous (viscosity between 100-1,000 cP) than penetrant pore liners and penetrant pore blockers. These allow little to no penetration of sealer into the surface pores due to a higher molecular weight, and also form a protective thin film (thickness ≤ 1 mm) on the surface. These include some epoxy resins, acrylics, and linseed oil.
- **Coatings** are more viscous than sealers (viscosity > 1,000 cP), and form a protective film (thickness ≥ 1 mm) on the surface. Coatings function as an impermeable barrier against contaminants. Epoxy resins, acrylics, vinyls, chlorinated rubber, and bitumen are examples of coating sealers.
- **Renderings** are the thickest form of a surface treatment. Usually, these are constructed from mortar and a polymer matrix and protect the concrete as a physical barrier.

2.2.2 Bridge Deck Sealer Classifications

Many low-viscosity protective surface treatments for concrete are commonly referred to as “penetrating sealers” which is misleading, because they do not penetrate the concrete substrate, but will penetrate into concrete cracks. (Pfeifer and Scali 1981). Renderings and coatings do not fit in this low-viscosity category; however, the lower-viscosity surface treatment classifications (penetrating pore liners, penetrating pore blockers and sealers) can be further categorized as two types of sealers, *penetrating* sealers and *non-penetrating* sealers. The characteristics that determine whether they penetrate or not is their molecular size and surface tension; smaller molecules can penetrate while larger molecules cannot. Figure 2.1 presents a schematic of the relative molecular sizes of sealer components and concrete pore sizes. Penetrating sealers and non-penetrating sealers offer protection by different mechanisms as described in the following paragraphs.

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[1] Å (Angstrom) = 10^{-10} m

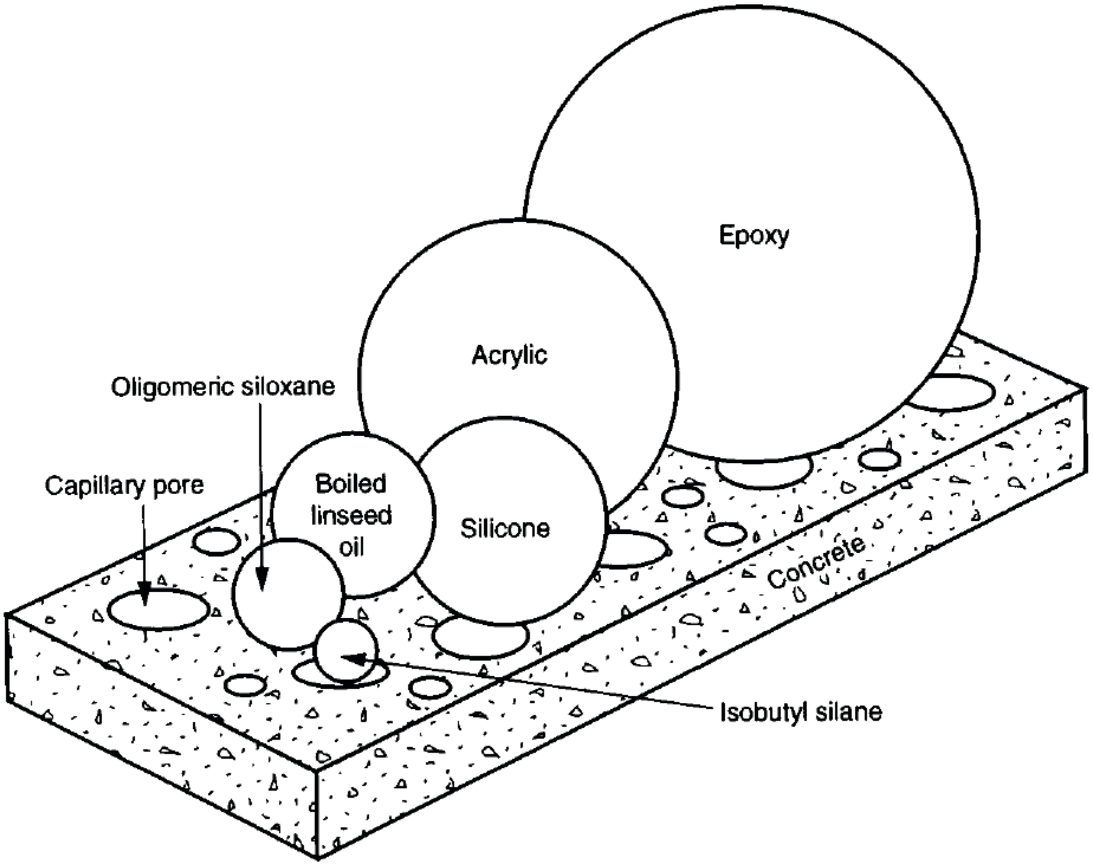


Figure 2.1 Relative molecular sizes of sealer components. From (Basheer and Long 1997)

Penetrating sealers are used more proactively to protect a larger area of concrete before cracks occur. These are usually silicon-based products and are sprayed or flooded onto the surface and soak into the substrate (Pincheira and Dorsorst 2005). Most silicon-based products are combined with a volatile solvent-based or a water-based organo-functional constituent to transport the silicon functional component into the concrete pores. Once the penetrating sealer infiltrates the substrate, the silicon functional component forms barriers in the pore structure that repel liquid water along with any deleterious substance carried by the water. Penetrating sealers (e.g. silanes) are also used to repel liquid water from the capillaries, but are breathable, allowing for water vapor transmission. (Basheer and Long 1997)

Non-penetrating sealers usually consist of epoxy or acrylic sealers. Epoxies usually do not penetrate concrete pores but will form an impermeable surface film (Basheer and Long 1997). These are often a pragmatic and efficient repair system to inhibit moisture ingress by forming a barrier when concrete becomes vulnerable to moisture ingress from cracks (Pincheira and Dorsorst 2005). A protective film forms after the non-penetrating sealer is flooded onto the concrete and dispersed evenly with a roller or brush. Non-penetrating sealers that produce an impermeable surface are ideal, but if excess moisture is present in the concrete prior to treatment, the adhesion of the sealer to the concrete substrate could be negatively impacted. If water is unable to evaporate, its presence could aid other forms of deterioration (e.g. alkali-silica reaction).

2.3 Sealer Evaluation Methods

National Cooperative Highway Research Program (NCHRP) Report 244 (Pfeifer and Scali 1981), sponsored by the Federal Highway Administration, is one of the most referenced guides for testing the performance of bridge deck sealer products. In July 2000, the Alberta Ministry of Transportation published standards for certifying a sealer product for concrete protection. A review of these two test procedures, along with several analytical techniques for measuring chlorides and measuring the depth of penetrating sealers in concrete, are presented in the remainder of this chapter.

2.3.1 NCHRP Report 244 Tests

The research that led to NCHRP Report 244 involved four series of tests. These were conducted on concrete treated with a variety of sealers in order to make recommendations of materials, applications, and test procedures. The types of sealers selected for testing in NCHRP Report 244 include epoxies, silanes, silicates, silicones, siloxanes and linseed oil. The Series I tests were used to screen products for the following three series of tests by evaluating the absorption properties and chloride resistance of concrete sealed with various products. The Series II tests studied the effects of concrete moisture content at the time of sealer application versus chloride intrusion of the concrete. The Series III tests evaluated the effect of different coverage rates on sealer performance. Series IV was an accelerated weathering test to evaluate the durability of sealers and were only used to evaluate products which passed the other series of tests. Series IV used concrete slabs as test specimens, rather than 4 in. (100 mm) concrete cubes which were used in Series I, II and III. The authors of the report concluded that only the Series II and Series IV tests were considered useful testing methods for effectively evaluating products (Pfeifer and Scali 1981). Only the Series II tests were investigated further for this research due to time and cost constraints.

2.3.1.1 NCHRP Report 244 Series II Methodology

The NCHRP Report 244 authors found the Series II tests to have the most realistic curing conditions compared to bridge decks in the field. Concrete test specimens were placed in plastic bags and cured for seven days in a moist curing room at 73 °F (23 °C), as opposed to the curing method used in Series I (6 days submerged in water). After seven days, samples were "lightly abraded" by sandblasting, then weighed and returned to the moist curing room for 14 additional days, for a total of 21 days of moist curing. After the 21 days of moist curing, the specimens were subjected to a conditioning period of slow drying for 33 days. During the 33 days of drying, pairs of cubes were each taken out after 1, 5, and 21 days and were treated with sealer products. The sealer was applied to the cubes' surface with a brush to achieve the manufacturers' recommended coverage rates. Then they were returned to the controlled climate area for the remainder of the 33-day drying period. This procedure allows sealer effectiveness to be tested at different moisture contents of the concrete. (Pfeifer and Scali 1981)

Test specimens were 4 in. (100 mm) cubes. Two cubes treated with same product were paired and used during testing. At an age of 54 days after casting, specimens were placed into an aqueous solution of 15% NaCl for 21 days. Two sets of control cubes were used for the test: one unsealed pair that remained in the controlled climate room, and a second unsealed pair that was exposed to the NaCl solution with the sealed cubes. Each cube was weighed every 3 days to measure the mass of solution absorbed during the 21-day soaking period. The mass measurements were used to determine whether cubes that had been sealed after a longer drying period (e.g. 21 days) absorbed more NaCl solution than cubes that had been sealed after a shorter drying period (e.g. 1 day). Then all specimens returned to the climate-controlled room for an additional 21-day drying period; the purpose of the drying period was to observe the vapor transmission characteristics of the sealers as water from the absorbed NaCl solution evaporated. (Pfeifer and Scali 1981)

After the drying period, the acid-soluble chloride content was determined by potentiometric titration with silver nitrate. A theoretical mass-gain model was also used for evaluating chlorides based on an assumed relationship between total mass gain and chloride ingress. Theoretically, a 15% NaCl solution is 9.1% chlorides by mass. The chloride content was calculated as 9.1% of the total mass gained after 21 days of soaking. (Pfeifer and Scali 1981)

2.3.1.2 NCHRP Report 244 Series II Evaluation Criteria and Main Findings

In their concluding remarks, the authors of NCHRP Report 244 (hereafter referred to as "Report 244" for brevity) suggest criteria for acceptable performance criteria for sealers. Performance is measured as the reduction in average mass gain and chloride ingress of sealer-treated concrete compared to unsealed control specimens. To be considered acceptable, a sealer should demonstrate the following performance in the Series II tests:

- A minimum of 75% reduction of the mass gained by the sealed cubes compared to the untreated control cubes
- A minimum of 75% reduction of the net chloride content retained by the sealed cubes compared to the chlorides retained in the untreated control cubes, as measured by titration.

Products that exceed these two criteria are recommended for further evaluation using the Series IV tests.

The best performance was observed for specimens treated with a dual protective system; a silane was used as a secondary defense underneath an epoxy coating. According to Report 244, the chloride contents measured by titration showed a good correlation to the mass gained in the 21-day soaking period. (Pfeifer and Scali 1981)

Other findings in Report 244 include that epoxy sealers should not be applied to moist concrete, and that the concrete needed to dry for at least 5 days prior to applying the epoxy sealer. Epoxies achieved no measureable penetration depth in the substrate, with an exception of filling cracks. Some of the silanes performed well for specimens regardless of the time of application during the 33-day drying period and provided a measureable penetration depth of up to 0.1 in (2.5mm). (Pfeifer and Scali 1981)

2.3.2 Alberta MOT Specifications and Tests

Unlike the NCHRP Report 244 Series II tests, Alberta MOT tests are conducted solely in water and not a NaCl solution. The Alberta MOT uses two primary test standards from their BT Series (BT001 and BT010) and a specification (B388) for protective concrete bridge sealers to select, test and certify a sealer based on the evaluation of its performance.

Alberta MOT B388, *Specification for Concrete Sealers,* provides the classifications of sealers based on their use in the field, along with approval and performance requirements based on sealer classification. BT010, *Test Procedure for Casting and Storing Concrete Test Specimens for Use in Approval Testing of Sealers*, specifies the mix designs and materials, casting and fabrication requirements, curing times and specimen storage details according to sealer classification in B388. BT001, *Test Procedure for Measuring the Vapour Transmission, Waterproofing and Hiding Power of Concrete Sealers,* provides the procedures for evaluating sealers according to the classification in B388.

2.3.2.1 Alberta MOT Sealer Classification

Table 2.1 defines the classifications of sealers for testing according to Section 1.2 of Alberta MOT B338. Type 1b and Type 1c classifications represent the type of sealer used on traffic bearing surfaces exposed to abrasion such as bridge decks. Type 1b sealers are intended for use on older bridge decks with aged concrete and a higher w/cm, and Type 1c sealers are intended for use on newer bridge decks made with high-performance concrete with a low w/cm. Alberta MOT B388 Section 1.2 only describes penetrating sealers as Type 1 (1a, 1b, or 1c) but does not clearly identify the use of non-penetrating sealers (e.g. two-component epoxies sealers) as bridge deck treatments in their classifications. (Alberta Ministry of Transportation 2010)

Table 2.1 Sealer classifications defined in Alberta MOT B388 (2010)

<table>
<tr><td colspan="6">Type 1 Sealers: Penetrating sealers for use on traffic-bearing surfaces exposed to abrasion. Subtypes are based on substrate exposure conditions.</td></tr>
<tr><td colspan="2">Type 1a: Penetrating sealers for application in sheltered conditions such as parking decks where the relative moisture content is a maximum of 55%.</td><td colspan="2">Type 1b: Penetrating sealers for application in outdoor conditions such as bridge decks where the deck relative moisture content is a maximum of 70% representing two days of drying. Represents mature concrete with a higher w/cm (0.45 - 0.50).</td><td colspan="2">Type 1c: High-performance penetrating sealers for application in outdoor conditions to new bridges and overlays. Cast with a low w/cm (0.35 – 0.45) with a relative moisture content at a maximum of 80%. Represents newer, less mature concrete.</td></tr>
<tr><td colspan="6">Type 2 Sealers: Clear, film-forming sealers for non-traffic-bearing elements. Subtype is based on the number of components.</td></tr>
<tr><td colspan="3">Type 2a: One-component, clear coatings suitable by less experienced personnel on concrete that has a relative moisture content is a maximum of 70%.</td><td colspan="3">Type 2b: Two or more component coatings for use by approved contractors where higher degrees of waterproofing performance are required and where the relative moisture content is a maximum of 70%.</td></tr>
<tr><td colspan="6">Type 3 Sealers: Colored film-forming sealers for use on elements highly exposed to public view where aesthetics are a primary consideration. These products are for use on concrete surfaces where the relative moisture content is a maximum of 70%.</td></tr>
</table>

2.3.2.2 Alberta MOT BT Series Methodology

Once a sealer has been classified, procedural details and criteria for evaluation are provided by Alberta MOT BT010 and BT001. 4-in. (100 mm) concrete cube specimens are fabricated, cured, and conditioned in accordance with Sections 2.4 through 2.8 of BT010 (Alberta Ministry of Transportation 2000b) and Section 2.0 of BT001.

Sections 4.0 through 4.4 of BT001 describe the sealer application method for Type 1b and Type 1c sealers. Three cubes are immersed in the sealer for two minutes at a time, with a maximum of three immersions. The maximum recommended time between immersions is four hours, or when the sample has ceased dripping sealer. For specimens that do not meet the manufacturer's recommended coverage rate, the test is to continue with application rate obtained with no more than three immersions (Alberta Ministry of Transportation 2000a).

BT001 Section 4.4 specifies a 15-day drying period after sealer application to condition the specimens further in a 74.0 °F ± 3.0 °F (23.3 °C ± 2.0 °C) and 50% relative humidity environment. Specimens are weighed before and after immersions in the sealer product and at intermediate times in the 15-day drying period to determine the mass of sealer that adhered to the cube and the vapor transmission characteristics of the sealer. The mass measurements during the 15-day drying period are also used to make adjustments to equate the relative moisture content (RMC) of the untreated control cubes and the sealed cubes before testing the waterproofing performance of the sealers. This is done following procedures in Sections 5.0 through 6.1 of BT001. (Alberta Ministry of Transportation 2000a)

The initial waterproofing performance test is described in Section 6.2 of BT001**.** Test specimens are submersed in a container of water for 120 hours and weighed before and after submersion. After the initial waterproofing testing, a post-abrasion waterproofing performance test is conducted to determine the durability of the sealer; this test uses the same specimens that were used for the initial waterproofing test and is performed following procedures in Sections 6.2 and 6.3 of BT001.

The sealed cubes are then oven-dried at 140 °F ± 3 °F (60 °C ± 2 °C) to remove the moisture absorbed during the initial waterproofing performance test. Specimens are then sandblasted to remove a specified amount of material from each face. 12.0 ± 1.0 g per face are removed from specimens treated with Type 1b sealers, which equates to an approximate depth of 0.5 mm of material removed; 24.0 ± 1.0 g per face are removed from specimens treated with Type 1c sealers, corresponding to an approximate depth of 1.0 mm of material removed. This estimate of the depth of material removed is based on the density of concrete, the surface area of the specimens, and the mass of material removed. These cubes are once again immersed in water for 120 hours to determine their post-abrasion waterproofing performance based on the mass of water absorbed in the test. (2000a)

2.3.2.3 Alberta MOT Evaluation Criteria

Section 4.2.1 of Alberta MOT B388 provides criteria that are more stringent than those found in Report 244 (Series II) for evaluating of performance of sealers. Sealer performance is based on the reduction in mass gained by sealed specimens relative to the untreated controls. No parameters are specified for initial waterproofing performance. Sealers are solely evaluated on post-abrasion (final) waterproofing performance. The minimum acceptable performance criteria for Type 1b and Type 1c sealers are as follows:

- Type 1b: an 86% reduction in mass gained relative to the control specimens in the post-abrasion waterproofing performance test.
- Type 1c: an 85% reduction in mass gained relative to the control specimens in the post-abrasion waterproofing performance test.

2.3.3 Measurement of Chlorides in Concrete

Chlorides in concrete can be measured in several ways. One of the initial methods developed (and still commonly used) for determining chlorides in concrete is titration with silver nitrate, a wet chemistry technique. Other methods of evaluating chlorides include scanning electron microscope-energy dispersive spectroscopy (SEM-EDS), electron probe microanalysis (EPMA), and x-ray fluorescence spectroscopy (XRF); these methods of analysis use different types of detectors to collect results. These techniques are presented and discussed in the following sections.

2.3.3.1 Titration with Silver Nitrate

One technique for measuring the chloride content of concrete is titration with silver nitrate. Titration is a process used to determine an unknown concentration (chlorides) in a solution by adding a measured amount of standard solution (silver nitrate) of a known concentration. The silver ions from the silver nitrate solution will react with chloride ions to form silver chloride and precipitate out of solution. The unknown concentration of chlorides is calculated by the stoichiometry of the reaction of silver nitrate and chloride ions, and the number of moles of the standard solution used to reach an equivalence point in the reaction (when all chlorides in solution have reacted with silver ions).

The equivalence point can be accurately interpolated by measuring the change in voltage potential of the chloride-bearing solution after sequential additions of silver nitrate. Voltage potential is measured with an electrode connected to a potentiometer; this is known as potentiometric titration. Good practice suggests providing constant agitation of the solution during the addition of silver nitrate to prevent a silver chloride precipitate from forming locally in the solution; without agitation, the localized precipitate formation will consequently present a false equivalence point, resulting in an inaccurate calculation of the chloride concentration. (Korkmaz n.d.)

ASTM C1152/C1152M, *Standard Test Method for Acid-Soluble Chloride in Mortar and Concrete*, can be used to determine the acid-soluble (total) chloride content by potentiometric titration with silver nitrate. The method described in ASTM C1152/C1152M is taken from ASTM C114-11b, *Standard Test Methods for Chemical Analysis of Hydraulic Cement* Section

21, *Chloride*. This method was once commonly used by portland cement manufacturers for elemental analysis of clinker and cement.

Potentiometric titration with silver nitrate is not without drawbacks and safety concerns. Caustic chemicals such as nitric acid, potassium chloride and silver nitrate are required for titration with silver nitrate. Silver nitrate must also be stored in an opaque protective container to prevent the decomposition of silver nitrate in light (Korkmaz n.d.). Titration is also a time-consuming test method, and would take a great deal of effort to carry out if large numbers of measurements are needed (Proverbio and Carassiti 1997).

2.3.3.2 Microanalysis by SEM and EMPA

Scanning electron microscopy (SEM) is primarily used for imaging but can perform qualitative chemical microanalysis when equipped with an energy-dispersive spectroscopy (EDS) detector. An electron probe microanalyzer (EMPA) is similar to an SEM in that an electron beam is used to probe the sample, but it is usually equipped with multiple x-ray detectors and used primarily for a quantitative microanalysis, not for high resolution imaging. The basic principle of an SEM and EMPA is the use of a focused beam of electrons to probe a sample; multiple instruments then detect a variety of electrons and x-rays to form an image and/or conduct elemental analysis. (Sarkar, et. al. 2001)

The two types of spectrometers used for microanalysis are EDS and wavelength-dispersive spectroscopy (WDS). For EDS, a single detector detects all characteristic x-rays emitted from the sample and identifies the elements present by their characteristic x-ray energies, which provides a more qualitative analysis (Sarkar, et. al. 2001). WDS is similar to EDS, but identifies elements by the wavelength of characteristic x-rays emitted by elements present in the sample rather than the x-ray energies; WDS is more commonly used in EMPA, which contain multiple spectrometers. However, the spectrometer for WDS is relatively more expensive and slower than EDS (Dempere, et. al. 2013).

Microanalysis of a concrete sample may be conducted by four different modes: analyzing the composition of a single spot (spot analysis), analyzing in bulk for the average distribution of elements of a region (area analysis), conducting a series of spot analyses to determine the distribution density of elements in a region (dot mapping), or analyzing the variation of concentrations of one or more elements along a line (linear traverse or line scan). The resolution of an elemental analysis is limited by size of the interaction volume producing the x-rays (Sarkar, et. al. 2001). A larger interaction volume results from a more powerful probe beam, increasing the amount of x-rays generated, but decreasing the maximum resolution of the image constructed (Dempere, et. al. 2013). Concrete specimen preparation for qualitative analysis requires minimal effort compared to a quantitative analysis which requires a flat-polished surface (Sarkar, et. al. 2001).

2.3.3.3 XRF and micro-XRF

XRF and micro-XRF are two more alternatives to titration for conducting an elemental analysis on a concrete sample. XRF and micro-XRF use high-energy, electromagnetic radiation (x-ray photons) to trigger the emission, or fluorescence, of characteristic x-rays from elements present in a sample material, and these emitted x-rays are identified using either an EDS or WDS detector(s). This is similar to SEM-EDS and EMPA, but an x-ray beam is used to probe the sample instead of an electron beam. The element is then identified based on the characteristic x-ray energy (EDS) or characteristic x-ray wavelength (WDS).

XRF has a spot size of approximately 10 mm and determines the bulk elemental composition of a sample (Sudbrink, et. al. 2012). This method of analysis, frequently used by portland cement manufacturers, requires less sample preparation and relatively inexpensive than titration with silver nitrate. Preparing samples for XRF analysis can be done by a few methods: leaving the specimen undisturbed, packing loose powder into cups covered by a thin film, grinding powder until the particles are very fine then compressing it into a briquette (pressed pellet), or combining powder and flux into a fused glass bead. The fused bead technique has been an accurate method of determining elemental composition and very precise. However, fused beads could be problematic if the elements of interest are volatile; alkali metals and halides (including chlorine) can be released during the fusion process under high temperatures. For this reason, fused bead preparation is not appropriate for chloride analysis. Appropriate methods of preparation are either packing loose powder into cups or leaving the sample undisturbed in order to prevent the loss of the volatile chloride ions. (Broton and Bhatty 2011)

Unlike XRF, micro-XRF is used for mapping the elemental distribution of a sample rather than the bulk concentrations of elements in a sample (Sudbrink, et. al. 2012). Micro-XRF operates similarly to a low-resolution SEM, and has a spot size of approximately 20 to 50 μm. An advantage of micro-XRF analysis compared to SEM-EDS is that micro-XRF allows for microanalysis of a fractured-surface sample without any polishing or other preparation (Garboczi, et al. 2012).

Although SEM-EDS and titration with silver nitrate may be very accurate and precise methods of determining the chloride concentrations, these processes require meticulous sample preparation and may take a lot of time to complete. XRF analysis is a more efficient method of determining chloride content than titrating, and micro-XRF provides similar analysis of SEM-EDS or EMPA with minimal processing of the sample prior to analysis.

2.3.3.4 Case Studies

Dempere, et al. (2013) used SEM-EDS and EMPA-WDS to determine the chloride content in concrete and relate it to chloride diffusion. These techniques were selected for the capability of analyzing the paste in concrete separately from the aggregates, and results were compared with those obtained using potentiometric titration. Two instruments were used for comparing chloride content with potentiometric titration: an SEM equipped with one EDS spectrometer, and an EPMA equipped with four WDS spectrometers.

Dempere, et al. (2013) found that the results from microanalysis tended to indicate higher chloride contents than the results obtained from the titration analyses. The data from microanalysis had to be normalized with data from wet chemistry analysis by regression equations derived from the calibrated models. After normalization, the results from wet chemistry analysis and microanalysis were in better agreement. Figure 2.2 shows a comparison of the normalized microanalysis results of WDS and results from wet chemistry methods for measuring chlorides.

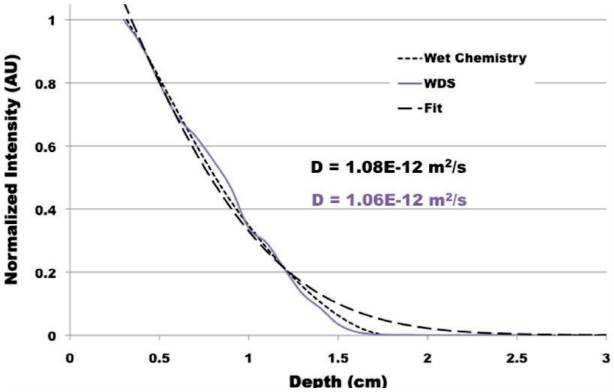


Figure 2.2 Comparison of EMPA-WDS and wet chemistry (titration) chloride measurements on a common sample. From (Dempere et. al. 2013)

SEM-EDS provided results more quickly than EPMA-WDS, while EPMA-WDS provided more accurate results compared with results from the potentiometric titration by silver nitrate. The time taken to evaluate one set of samples by dot mapping was approximately 8 hours for SEM-EDS and 30 hours for EPMA-WDS. Dempere, et al. (2013) concluded that SEM microanalysis is an effective, non-destructive method of measuring chloride content in concrete while minimizing caustic waste material generated by potentiometric titration.

In a study by Proverbio and Carassiti (1997), XRF-WDS was used to determine chloride content in concrete. The objective was to determine the efficacy of XRF analysis for measuring the bulk content of dissolved chlorides in the concrete pore solution, and the influence of the morphology and composition of NaCl grains added in concrete on the accuracy of analysis. Potentiometric titration with silver nitrate was conducted to compare with the results of XRF analysis.

The chloride contents measured by XRF analysis were higher than the chloride concentrations determined by titration (Proverbio and Carassiti 1997). This is similar to the results observed by Dempere, et al. (2013). Proverbio and Carassiti (1997) determined that this difference was determined to be an effect from the techniques used to prepare calibration standards with additions of NaCl. The characteristic x-ray intensities of chlorine are directly proportional with NaCl grains size present; as the NaCl grain size increases, chlorine's characteristic x-ray intensity decreases, reducing the apparent chloride concentration. The elemental characteristic x-rays emitted from the sample are not only depended on the element's concentration, but also on the powder grain size, degree of compaction, and any chemical bindings in the matrix. The use of correctly-prepared calibration standards are essential to obtaining SEM, EMPA, and XRF results of similar accuracy to those obtained by titration.

2.3.4 Measurement of Depth of Sealer Penetration

A simple method implemented to determine the depth of penetration of sealers involves ponding sealed concrete in a commercially available colored dye (e.g. food coloring). The dye stains the unsealed concrete and has no effect on the sealer-penetrated concrete, leaving a distinct boundary of stained and unstained concrete. This is because the sealed concrete prevents penetration from the solution transporting the dye.

Pincheira and Dorsorst (2005) confirmed that a depth of penetration of silanes and siloxanes can be measured by using dye to stain the concrete. Most solvent-based penetrating sealers were found to have deeper penetration than water-based penetrating sealers with similar molecular weights. The solvent-based penetrating sealers that did not penetrate as deeply as some water-based penetrating sealers had a higher molecular weight than the water-based sealers. The maximum penetration depth recorded for a silane was 0.15 in. (3.8 mm). The authors from this study recommended measurements should be taken at least three months from when the sealer is applied to allow sufficient time for the sealer to penetrate and seal the pores of the concrete.

Sudbrink, et al. (2012) used micro-XRF to determine the depth penetration of a silane applied to a bridge deck. Cores with dimensions of 0.5 x 1.0 in. (12.5 x 25.0 mm) were sampled from a bridge deck sealed with a silane and analyzed using micro-XRF (EDS). Samples were only polished enough to remove the saw marks left on the cylinder to improve the quality of optical imaging; ethanol was applied after polishing to remove any debris before microanalysis. A change in the concentrations of sulfur and potassium were observed in sealed samples from microanalysis, indicating the depth of silane penetration. Unsealed control samples did not display any change in the concentration of these elements. The microanalysis data was used to validate the use of a commercial dye to stain the concrete which left a distinct border from the sealer penetration. (Sudbrink, et. al. 2012)

2.4 Summary

Surface treatments for concrete can be classified in five categories: penetrating pore liners, penetrating pore blockers, sealers, coatings and renderings. The lower-viscosity (viscosity < 1000cP) classifications of penetrating pore liners, penetrating pore blockers, and sealers can be further categorized as concrete bridge deck *sealers*. Sealers are either *penetrating* or *non-penetrating* products, and each type offers different mechanisms of protection.

In the Report 244 Series II tests, researchers evaluated both *penetrating* and *non-penetrating* bridge deck sealers. The Alberta MOT BT Series protocol is designed to evaluate *penetrating sealers* for use on traffic-bearing surfaces such as bridge decks, but doesn't contain methodologies for evaluating *non-penetrating* sealers used on traffic-bearing surfaces.

Both the Report 244 Series II and Alberta MOT BT Series test protocols evaluate waterproofing performance, but under different criteria. The Alberta MOT BT Series evaluates a sealer's post-abrasion waterproofing performance whereas the NCHRP Series II tests only use pre-abrasion waterproofing performance criteria. The Report 244 Series II tests evaluate a sealer for resistance to chloride ingress by immersing samples in a saltwater solution, whereas the Alberta MOT BT Series tests samples in water. In Report 244, chlorides were evaluated by mass gain analysis and titration. In addition to titration, alternative methods for analyzing chlorine in concrete are available. These alternative methods are SEM-EDS, EPMA, bulk XRF and micro-XRF, which are all capable of precisely and accurately measuring the chloride content of concrete. The use of dye can also be used to determine the depth of penetration of a penetrating sealer by staining the non-penetrated concrete leaving the penetrated concrete unstained.

Table 2.2 presents a comparison of the main tasks and elements of the Report 244 Series II and Alberta MOT BT Series test protocols along with the elements used in the research described in this report (ALDOT Project 930-861). Elements of the Series II tests and the BT Series were selected for the research described in this report, along with potentiometric titration by silver nitrate and bulk analysis by XRF for evaluating chlorides. A dye method was selected for investigating sealer penetration and thickness. The methodology for selecting the elements used in this research is detailed in Chapter 3.

Table 2.2 Comparison of tasks in NCHRP Report 244 Series II, Alberta MOT BT Series, and ALDOT Project 930-861 testing protocols

Element of Test Protocol	NCHRP R244 Series II Tests	Alberta MOT BT Series	ALDOT Project 930 - 861
Specimen Dimensions	4 in. (100mm)	4 in. (100mm)	4 in. (100mm)
Curing Method	In plastic bags in moist-curing room	(Unspecified) in moist-cure room	In plastic bags in moist-curing room
Curing Duration	21 days – all	5 days – 0.35 w/cm 42 days – 0.50 w/cm	42 days – all
Surface Preparation	Sandblasted during curing period	Sandblasted after curing period	Sandblasted after curing period
Conditioning: Phase I Drying	Dried for 1, 5, and 21 days	Dried to a target moisture content for each mix design	Dried to a target moisture content for each mix design
Sealer Application Specimens per Sealer	2-cube set per sealer	3-cube set per sealer	3-cube set per sealer
Sealer Application Method	1-2 applications with a brush	2-3 two-minute immersions	2 two-minute immersions
Conditioning: Phase II Drying	Dried for 32, 28, and 12 days	Dried for 15 days, adjusted to RMC of unsealed control	Dried for 15 days, adjusted to RMC of unsealed control
Waterproofing Performance	21 days in 15% NaCl	5 days in water	21 days in 15% NaCl, 5 days in water
Post-Abrasion Waterproofing Performance	N/A	5 days after sandblasting in water	5 days after sandblasting in water
Resistance to Chloride Penetration	Mass gain analysis and potentiometric titration	N/A	Mass gain analysis, potentiometric titration, and XRF analysis
Sealer Penetration Sealer Thickness	Yes (unspecified)	N/A	Dye method, optical microscopy, SEM-EDS

Chapter 3: Methodology

3.1 Overall Plan

Selected elements of the Report 244 Series II and Alberta MOT BT Series protocols were used in combination to evaluate bridge deck sealers on specimens representing the range of bridge deck concrete in service with ALDOT (See Table 2.2). The primary criteria used to evaluate the overall performance of the sealer products were the initial waterproofing performance, waterproofing performance after abrasion, and resistance to chloride ingress. Measuring the depth of sealer penetration and sealer thickness was another objective of this research.

Figure 3.1 presents a flowchart of the test protocol followed in this research. The following describes the reasoning for selecting the specific elements (shown in Table 2.2) of the Report 244 Series II tests and the Alberta MOT BT Series test protocols for this research:

- The Report 244 Series II tests and Alberta MOT standards both use cube specimens with the same 4.0 in. (100 mm) dimensions, so the same specimen dimensions were selected for this research. This allowed for synergistic formwork, specimen preparation, and handling during the test procedures.
- The curing method was taken from Report 244 Series II. Specimens were cured in sealed plastic bags because the authors of Report 244 determined that this method produced curing conditions most similar to those experienced by newly-constructed bridge decks in the field (Pfeifer and Scali 1981).
- A curing duration of 42 days was selected from the Alberta MOT BT protocols. This was to give all specimens sufficient time to develop a mature pore structure prior to surface preparation. This research made use of three mixture designs, including one containing 20% fly ash replacement of portland cement that required additional curing compared a 100% portland cement mixture.
- The Alberta MOT BT Series procedure for surface preparation was selected because it specified a specific amount of material to remove by sandblasting. The Report 244 Series II procedure only states that specimens should be "lightly abraded."
- The first conditioning phase specified by the Alberta MOT BT Series tests was selected to dry all specimens to a single relative moisture content (RMC) prior to sealer application, instead of drying specimens for a fixed amount of time to moisture contents that may vary from specimen to specimen.
- The Alberta MOT BT Series method of sealer application by fully-immersing specimens in the sealer was selected to minimize the variation of coverage between the specimens that might occur from applying the sealer by a brush, as was specified in Report 244 for the Series II tests.

- The second conditioning phase specified by the Alberta MOT BT Series was selected to adjust the RMC of the sealed specimens back to the RMC of the untreated controls prior to testing for resistance to moisture and chloride penetration.
- The 5-day waterproofing performance test and the 5-day post-abrasion waterproofing performance test were selected from Alberta MOT test standards. This shorter-duration test would primarily represent resistance to water absorption of sealer-treated specimens.
- The 21-day waterproofing performance test in 15% NaCl was also selected from the Report 244 Series II tests. This test allowed measurement of resistance to moisture ingress and resistance to chloride ingress. Because of the longer duration of this test, it is possible that some chloride ingress would be due to ionic diffusion as well as absorption of the solution.
- Three techniques were selected to measure chlorides in concrete: mass gain analysis, potentiometric titration by silver nitrate, and X-ray fluorescence spectroscopy (XRF). The mass gain and titration methods were both used in the Report 244 Series II tests. XRF was selected as a third test to verify the accuracy of results obtained by the mass gain and titration methods.
- Two methods were selected for both determining the depth of penetration and sealer thickness: the dye method and petrographic analysis by SEM-EDS. The dye method was selected for use based on the successful application of this technique reported by Pincheira and Dorsorst (2005) and Sudbrink, et al. (2012) when used with other penetrating sealers. SEM-EDS was selected by the concrete petrographer who was engaged in this research.

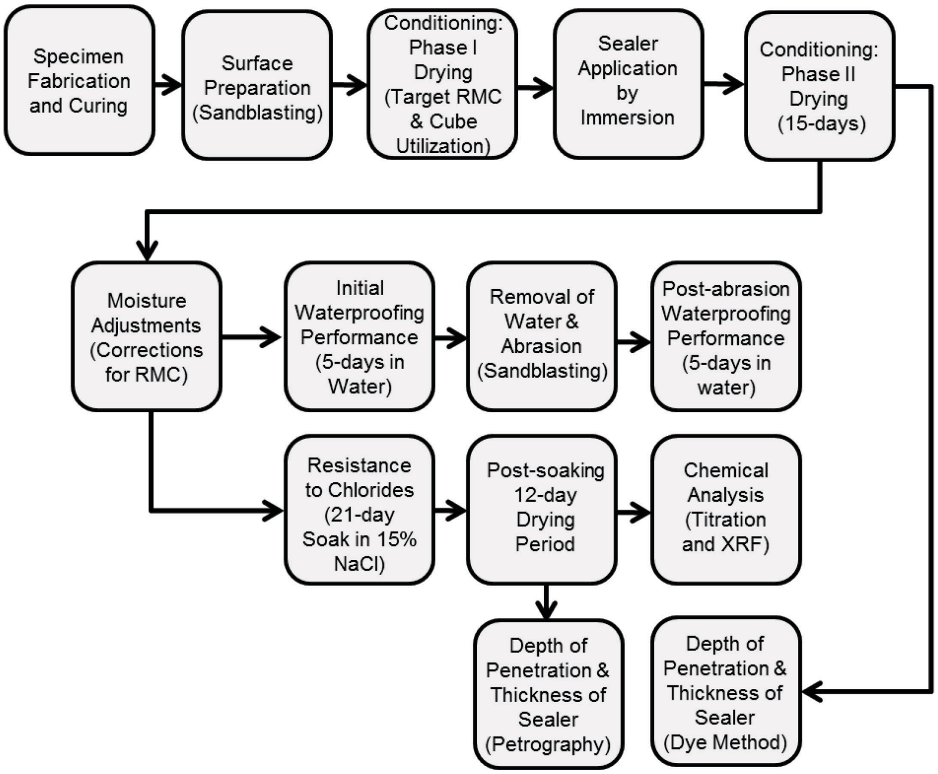


Figure 3.1 **Chronological steps of the testing protocol to evaluate sealer products.**

3.2 Materials

This section describes the five sealer products evaluated, the materials used for the concrete molds, and materials used for fabricating concrete specimens. Sealer B is the currently-used sealer by ALDOT. This product was evaluated against four other products of interest to ALDOT; details regarding all five products are given in Table 3.1.

Table 3.1 The five sealer products evaluated in this study

Product	Composition	Viscosity (cP)	% Solids	Mixing Ratio	Recommended Application Rate ft^2/gal (m^2/L)	Recommended Method of Application
Sealer A	One-Component Sodium Silicate	N/A	~0	N/A	150 – 200 (3.7 – 4.9)	Spray or Flood
Sealer B	Two-Component Epoxy	10 – 30	60	1:1	100 – 300 (3.2 – 7.4)	Flood
Sealer C	Two-Component Epoxy	40	75	1:1	150 – 200 (3.7 – 4.9)	Flood
Sealer D	Two-Component Epoxy	85	100	1:2	150 – 200 (3.7 – 4.9)	Flood
Sealer E	Two-Component Epoxy	105	100	1:2	150 – 175 (3.7 – 4.3)	Flood

The materials specifications that ALDOT uses for structural concrete are outlined in Section 501.02 (a) and Division 800 of the ALDOT *Standard Specifications for Highway Construction*. Approved materials were chosen from ALDOT's lists of qualified sources: I-1 *Sources of Coarse and Fine Aggregates*, I-2 *Portland and Blended Cements*, I-3 *Mineral Admixtures for Portland Cement Concrete*, and II-1 *Chemical Admixtures for Portland Cement Concrete*. Table 3.2 shows the materials used for making concrete in this research. Properties of the ASTM C150 Type I/II cement used are reported from the mill certificate and are given in Table 3.3. Properties taken from the mill certificate for the Class F fly ash used are given in Table 3.4. The aggregate properties were taken from the producers' quality control reports and are given in Table 3.5.

Table 3.2 Identification of materials used from ALDOT qualified sources

MATERIAL	TYPE / PRODUCER / LOCATION / S.G.
CEMENT	TYPE I/II / LEHIGH / LEEDS, AL / 3.15
MINERAL ADMIXTURE	FLY ASH F / GASTON / WILSONVILLE, AL / 2.34
FINE AGGREGATE	#100 SAND / DAVIS SAND AND GRAVEL / MOUNDVILLE, AL / 2.63
COARSE AGGREGATE	#67 LIMESTONE / VULCAN MATERIALS / CALERA, AL / 2.74
WATER	FROM APPROVED SOURCE (UA LAB)
AIR ENTRAINING	MASTERAIR AE 90 / BASF / FLORHAM PARK, NJ / 1.01
HR WATER REDUCER	MASTERPOLYHEED 1025 / BASF / FLORHAM PARK, NJ / 1.07

Table 3.3 Chemical composition and physical properties of the ASTM C150 Type I/II cement

Composition (%)									
SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O_{eq}	Limestone	$CaCO_3$ in Limestone	Insoluble Residue
20.5	4.54	3.60	63.04	2.27	2.9	0.49	1.0	97.02	0.35

Bogue Phase Composition (%)				$C_3S + 4.75^*C_3A$	LOI	Heat of Hydration	Blaine Fineness	% Passing 45 μm
C_3S	C_2S	C_3A	C_4AF	(%)	(%)	(kJ/kg)	(m^2/kg)	(No. 325) Sieve
55.2	17.2	5.9	10.9	83	4.66	406	402	97.1

Table 3.4 Chemical composition and physical properties of the Class F fly ash

Composition (%)								LOI	Moisture	% Passing 45 μm
SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	(%)	(%)	(No. 325) Sieve
49.0	28.07	11.76	3.12	1.26	0.64	2.88	0.83	1.94	0.09	79.11

Table 3.5 Physical properties of the coarse and fine aggregates

Property	Coarse	Fine
Specific Gravity (SSD)	2.75	2.63
Absorption Capacity (%)	0.7	0.5
Fineness Modulus		2.41

Sieve Size		% Passing	% Passing
1 in.	(25 mm)	100.0	-
3/4 in.	(19 mm)	97.0	-
1/2 in.	(12.5 mm)	61.9	100.0
3/8 in.	(9.5 mm)	33.9	100.0
#4	(4.75 mm)	4.7	94.0
#8	(2.36 mm)	1.6	89.4
#16	(1.18 mm)	0.0	86.3
#30	(0.6 mm)	-	73.4
#50	(0.3 mm)	-	15.4
#100	(0.15 mm)	-	1.0
#200	(75 mm)	-	0.0
Pan		0.0	0.0

Specimen molds were made from cabinet-grade plywood to ensure smooth formed surfaces on the specimens. Four molds were constructed to cast a total of 72 cubes for each mix design. Before assembly, as specified in Report 244, epoxy resin paint was applied the surface of each member of the mold. Each mold consisted of a 3 x 6 grid for casting cubes with dimensions of 4 in. (100 mm) as shown in Figure 3.2. Member dimensions for a single mold constructed are: four 4 x 25 in. (100 x 630 mm) side and middle pieces, two 4 x 15 in. (100 x 380 mm) end pieces and a 30 x 20 in. (508 x 762 mm) baseboard. Grooves were notched into longitudinal members for aluminum spacers at intervals providing each cube width of exactly 4 in. (100 mm).

Figure 3.2 Assembled molds for test specimens

3.3 Concrete Mix Design and Properties

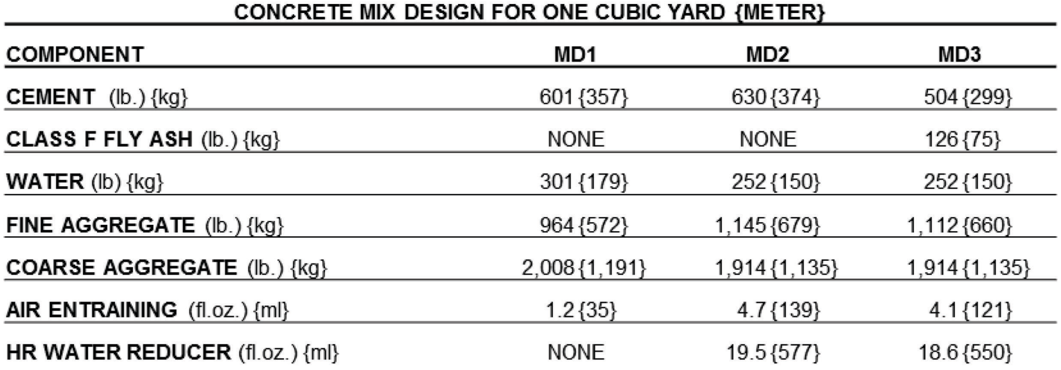
To encompass the range of bridge deck concrete that is found in service in Alabama, three different types of concrete were used in the evaluation of the sealer products. Mix Design 1 (MD1) represents bridge decks constructed in the 1960's. This older concrete could have some carbonation, which would increase the porosity in the carbonated zone. This design used an ALDOT 1964 specification as a guide for proportioning. Mix Design 2 (MD2) and Mix Design 3 (MD3) are more modern mix designs that followed ALDOT's 2012 *Standard Specifications for Highway Construction*, and represent better-quality bridge deck concrete in service. MD2 used 100% portland cement for its cementitious material content, while MD3 used Class F fly ash at a 20% replacement rate.

3.3.1 Mixture Designs and Proportions

Mix Design 1 was categorized as Class A Construction Type 1c concrete in Section 510.03 (b) of the ALDOT *Specification for Proportioning Structural Concrete* (ALDOT 1964). A 0.5 w/cm was used for this mixture. Although fly ash is specified in the 1964 specification, a 100% portland cement mixture is used in its place due to the difficulty of replicating fly ash from the 1960's and the effects of 50 years of aging and service loads. This slump requirement for the 1964 specification was also waived.

Mix Designs 2 and 3 are categorized as Class B – Bridge Superstructure Concrete and were proportioned to meet the requirements stated in Section 501.02 (c) of the 2012 ALDOT construction specifications (ALDOT 2012). A w/cm of 0.40 was used for these two mixtures. Trial mixes were made to determine the amount of admixture needed to meet ALDOT concrete requirements for fresh concrete properties. Table 3.6 contains the proportions used for each mix design.

Table 3.6 Proportions for each concrete mix design. Aggregate quantities are for saturated surface dry (SSD) material



CONCRETE MIX DESIGN FOR ONE CUBIC YARD {METER}

COMPONENT	MD1	MD2	MD3
CEMENT (lb.) {kg}	601 {357}	630 {374}	504 {299}
CLASS F FLY ASH (lb.) {kg}	NONE	NONE	126 {75}
WATER (lb) {kg}	301 {179}	252 {150}	252 {150}
FINE AGGREGATE (lb.) {kg}	964 {572}	1,145 {679}	1,112 {660}
COARSE AGGREGATE (lb.) {kg}	2,008 {1,191}	1,914 {1,135}	1,914 {1,135}
AIR ENTRAINING (fl.oz.) {ml}	1.2 {35}	4.7 {139}	4.1 {121}
HR WATER REDUCER (fl.oz.) {ml}	NONE	19.5 {577}	18.6 {550}

3.3.2 Fresh and Hardened Concrete Properties

Concrete was mixed per ASTM C192. A rotating drum mixer was used to mix a single 4.6 $ft.^3$ (0.13 m^3) batch of concrete for each mix design. All 72 specimens per mix design were cast from this single batch. Table 3.7 reports the fresh and hardened concrete properties of each mix design. The slump, total air content, and unit weight of the fresh concrete were the fresh concrete properties recorded for each mix design per ASTM C413 / C143M, ASTM C231 / C231M and ASTM C138 / C138M, respectively. The mixing temperature of the concrete was also recorded. Three 4 x 8 in. (100 x 200 mm) cylinders were cast for each mix design and tested at 7 days. Three 6 x 12 in. (150 x 300 mm) cylinders were cast for each mix design and tested at 28 days. These cylinders were tested in accordance with ASTM C39 / C39M.

Table 3.7 Fresh and hardened concrete properties for each mix design

Fresh Concrete Properties.	MD1	MD2	MD3
Slump (in) {cm}	8.7 {22.1}	3.5 {8.9}	6.0 {15.2}
Unit Weight (pcf) {kg/m^3}	146.2 {2,342}	148.8 {2,384}	144.4 {2,313}
Air Content (%)	3.6	4.1	5.8
Mixing Temperature at Discharge (°F) {°C}	73.0 {22.8}	72.4 {22.4}	72.8 {22.6}

Hardened Concrete Properties	MD1	MD2	MD3
Average 7 - Day Compressive Strength (psi) {MPa}	3,060 {21.1}	5,450 {37.6}	4,310 {29.7}
Average 28 - Day Compressive Strength (psi) {MPa}	5,240 {36.2}	6,260 {43.2}	5,670 {39.1}

3.4 Specimen Fabrication and Preparation

Specimens for each mix design were cast at one week intervals to allow sufficient time for specimen preparation between mix designs. Specimens were then finished, cured, and prepared for sealer application.

3.4.1 Casting and Finishing

Immediately after recording the fresh concrete properties, concrete was placed into the molds at the same time quality control cylinders were made. The forms were filled halfway, consolidated with a tamping rod, and then placed on a vibrating table for further consolidation. The concrete was vibrated for 15 to 30 seconds. The second layer was overfilled and tamped in the same method as the first layer. The excess concrete was screeded off and then the mold was vibrated again. The top surface was finished with a steel trowel as shown in Figure 3.3.

Figure 3.3 Freshly finished concrete samples

3.4.2 Curing Methods

The specimens were covered with wet burlap for 24 hours, as shown in Figure 3.4, and demolded the day after casting. All specimens were marked in the center of the finished top surface to easily identify the orientation of cubes during testing. The specimens were then sealed in heavy duty one gallon (3.8 L) freezer bags and placed finished surface up in a moist curing chamber for 42 days (Figure 3.5.) The curing chamber was set at 73.0 °F (23.0 °C) and 100% relative humidity.

Figure 3.4 Finished concrete covered with burlap for initial curing for one of the mix designs

Figure 3.5 Bagged specimens curing in the moist curing room

3.4.3 Surface Preparation

Prior to conditioning and sealer application, the surface of the concrete cubes was prepared to expose the internal pore structure of the concrete. Per Section 2.7 of Alberta MOT BT010, specimens were dried in open air in for 24 hours after curing and prior to sandblasting. Specimens were oriented finished surface up and spaced evenly across a table. The table surface allowed equal amounts of air exposure on all sides of the cube. The blasting media used was an abrasive silica sand, Sil 7, which meets the particle size gradation requirements specified in the Alberta MOT procedure. Sil 7 was obtained from Sil Industrial Materials Inc. in Edmonton, Alberta, Canada. Table 3.8 presents the gradation requirements for blasting media in BT010 compared with the gradation of Sil 7.

Table 3.8 Gradation requirements for the blasting media and Sil 7

Nominal Opening		Requirements	Sil 7
Mesh No.	(microns)	% Retained	% Retained
No. 16	1190	4-12	5.5
No. 20	841	8-15	9.0
No. 30	595	15-25	25.0
No. 40	420	35-45	40.0
No. 50	297	15-25	18.4
Pan	-	0-3	2.1

Each cube was weighed before and after sandblasting; intermediate measurements were made after the completion of each surface. The amount of material targeted for removal was 4.0 g per face, per Section 2.7 of Alberta MOT BT010. Each side was lightly abraded to remove a light film of paste and expose the internal pore structure of the concrete. The sides of the cubes were blasted first, and then the bottom and the top surface were abraded last to retain the specimen's orientation. After the mass was recorded, a permanent marker was used to identify the top surface with a single dot and numbered 1 through 72. This process took 5 to 6 hours for the 72 specimens made for each mix design.

The average amount of material removed per cube for MD1 was 20.2 g, MD2 was 16.7 g, and MD3 was 19.4 g. Less material than specified was removed because Section 2.7 of Alberta MOT BT010, *Surface Preparation,* places more emphasis on uniform pore exposure and degree of voids than the exact amount of material removed. Paste was more easily removed from the side and bottom surfaces at faster rate than the finished surface. Whenever 4.0 g of material was removed per face, the finished surface was denser and had the least amount of pore exposure, relative to the others surfaces. The degree of pore exposure from the 4.0 g of material removed from the finished side was used subjectively to prepare the other surfaces of cubes to a uniform condition. Following Alberta MOT BT010 (2000b), the three cubes with largest surface voids, or greatest defects, were preselected for the next phase of determining the total moisture content.

One trivial change was made to compensate the lack of some procedural detail in Section 2.7 of Alberta MOT BT010. Immediately followed sandblasting the final cube, all specimens were returned to the moist curing chamber for no longer than 24 hours; plastic bags were not used to seal the samples in this step. This was to ensure that each sample had a similar moisture content to compensate for the six hours of air exposure between sandblasting of the first and last sample.

3.5 Specimen Conditioning and Sealer Application

A series of steps was used to condition the specimens to equalize their moisture content before and after the sealer was applied to specimens. This was crucial to minimize uncertainties during the testing procedure. The procedures for drying and sealer application were based on Section 2.3 of Alberta MOT BT001.

3.5.1 Conditioning: Phase I Drying

Specimens were dried to a target relative moisture content (RMC) before sealer application. The target RMC for MD1 was 70% ± 2.0%. The target RMC for MD2 and MD3 was 80.0% ± 2.0%.

The average total moisture content (TMC) of three reference specimens from each mix design was used to calculate the RMC of the test specimens during the conditioning process. The three specimens from each mix design designated for total moisture content (TMC) determination were removed from the moist room, weighed, and immediately placed into an oven at 230 °F (110 °C) for seven days. These cubes were weighed to the nearest 0.1 g before and after drying in the oven. The average difference in mass represented the TMC. This set of cubes were not used for any further testing, except for determination of baseline chloride content, which will be discussed later. They were stored in sealed containers, separated by mix design.

Immediately following the removal of the cubes designated for TMC, the remaining cubes were removed from the moist room and weighed. The initial mass, X_0, was recorded for each cube, which represented the cube at 100% RMC. Specimens were towel-dried to saturated-surface-dry conditions before the mass was recorded. After the initial measurement, they were then placed on a cart and covered by a plastic film to prevent moisture loss as they were transferred to the environmental chamber to dry until they reached their target RMC. Conditions in the environmental chamber were 74 °F ± 3 °F (23.3 °C ± 2.0 °C) and 50 ± 4% RH. Figure 3.6 shows the cubes on the wire shelves, which allowed for circulating air exposure on all six surfaces.

Specimens were weighed daily in the environmental chamber to monitor the loss of moisture; the mass recorded each day was used to determine the RMC. These cubes were rotated between top and bottom shelves after each day's measurements to prevent any stagnant air exposure for a prolonged time period. The RMC for each cube is calculated according to Equation 3-1:

$$\boldsymbol{RMC}\ (\%) = \frac{[TMC-(X_0-X_i)]}{TMC} \times \mathbf{100} \qquad \textbf{(Eqn 3-1)}$$

Where:

X_0 = initial weight recorded after curing

X_i = recorded weight at (i = 1 day, 2 day, etc.)

TMC = total moisture content

Figure 3.7 presents the drying rates for each of the mix designs used. The drying rate varied with each mix design due to the different porosities and permeability of each concrete mixture. The drying time required for MD1 specimens to reach the target RMC of 70% ± 2.0% was 19 days. 23 days of drying were required for the MD2 specimens to reach their target RMC of 80.0% ± 2.0%, while only 9 days of drying were required for the MD3 specimens to reach their target RMC of 80.0% ± 2.0%. Out of 69 samples per mix design, coefficients of variation of the RMC after drying were 3.1% for MD1, 1.8% for MD2, and 1.4% for MD3.

Figure 3.6 Specimens drying in the environmental chamber

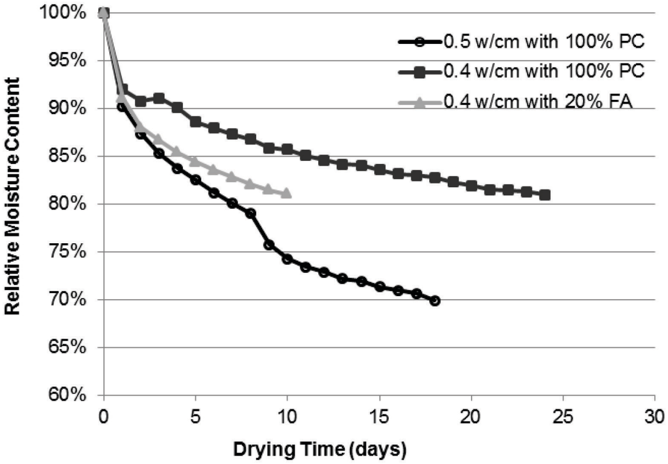


Figure 3.7 Average RMC of specimens during the controlled drying period

3.5.2 *Cube Utilization*

Per Alberta MOT BT001 (2000a), specimens were grouped into sets of three cubes for sealer application and subsequent testing. As the average RMC of the specimens approached the target RMC, they were divided into sets of three for sealer treatment (or controls), and designated for specific tests as shown in Figure 3.8. For each mix design and sealer combination, two sets of specimens (six cubes) with the least surface voids and the least variation from the target RMC were assigned to the waterproofing and chloride resistance tests. This was to minimize variations in sealer absorption that could be potentially caused by variations in the moisture content of the cubes. Six untreated control specimens for each mix design with minimal variation from the target RMC were also selected for the waterproofing and chloride resistance tests. The remaining cubes were used for determination of sealer penetration and sealer thickness by the dye method.

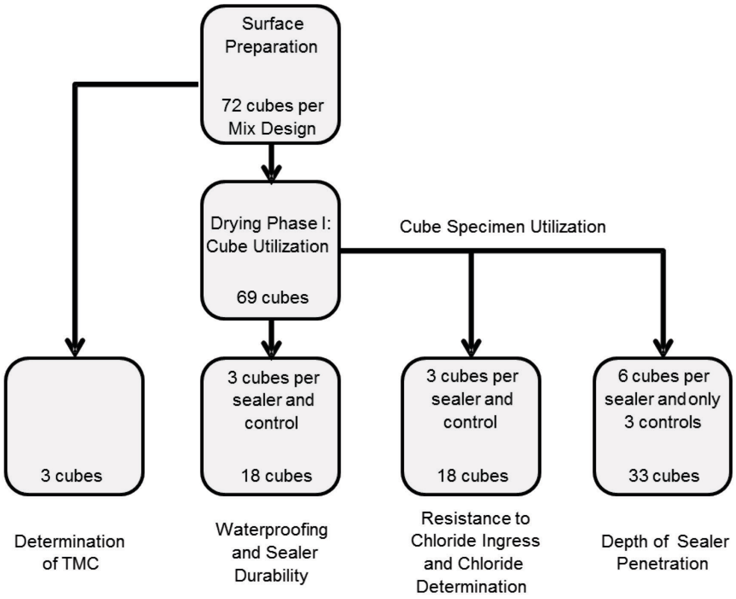


Figure 3.8 Detailed diagram for test specimen utilization by task. Specimen quantities are per mix design

3.5.3 *Sealer Application Procedure*

Immediately before the sealer was applied to the specimens, the sealer products were mixed according to the manufactures recommendations. Sealer A was supplied as a single-component product, ready for application, and required no mixing. The epoxy sealers (Sealers B, C, D and E) were supplied as two-part systems and these were mixed following the manufacturers' recommendations. The two components were measured in separate containers, combined in one container, and then mixed thoroughly for three minutes using a power drill with a mixing paddle.

Three equally-spaced cubes were placed top surface up on a wire tray and lowered into the container of sealer until completely immersed, as shown in Figure 3.9. They were immersed to a depth of at least 0.5 in (12.7mm) above the top surface, as specified by Section 4.1 of Alberta MOT BT001.

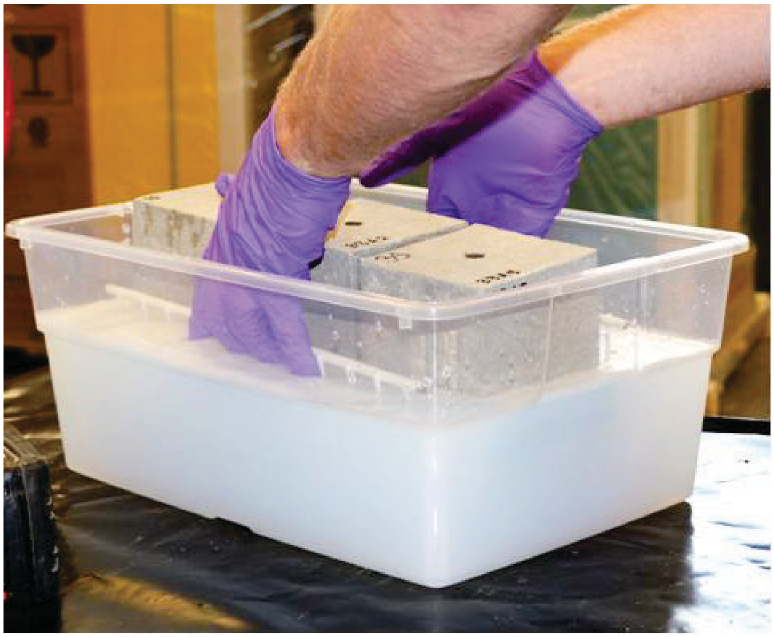


Figure 3.9 A set of specimens being immersed into the sealer

The sealer application process proceeded as follows, and included measurements to determine the mass of fresh sealer (*SF*) gained by the specimens:

1. Before the specimens designated for sealer treatment were removed from the environmental chamber, the mass of the corresponding untreated control specimens (C_0) was recorded. The control specimens remained in the environmental chamber during the sealer application process.

2. All specimens designated for sealer treatment were sprayed with compressed air to remove any dust or particulates and then weighed (T_0) immediately before being immersed in the sealer for two minutes.

3. After 2 minutes of soaking, the specimens were removed from the container of sealer and placed on a wire rack until the sealer ceased to drip. The new mass of the specimens was recorded (T_1) after dripping ceased.

4. The process repeated for the second immersion *(T_2 = mass before second immersion).*

5. After the second coating ceased to drip and the final mass was recorded (T_3), the sealed cubes were returned to the environmental chamber. *SF* was calculated using Equation 3-2:

$$\boldsymbol{SF} = (\boldsymbol{T_1} - \boldsymbol{T_0}) + (\boldsymbol{T_3} - \boldsymbol{T_2}) \qquad \textbf{(Eqn 3-2)}$$

Specimens were always oriented finished (top) surface up. All of the epoxy-based sealers ponded on the top surface and excess sealer was removed by carefully tipping the cube at an angle. The epoxy-based sealers with lower viscosities dripped for a longer duration than the epoxy-based sealers with a relatively higher viscosity. Once an epoxy-based sealer stopped dripping, it was tacky to the touch for up to 4 hours; despite that, the specimens were transferred to the scale for the measurement after dripping ceased. The sealed cubes were carefully grasped at two corners to minimize contact with the tacky sealer and placed on a wire tray and then transferred to a scale tared for that specific tray. The silicate-based sealer was not tacky to the touch, but was handled with the same care as the epoxy-based sealers.

3.5.4 Conditioning: Phase II Drying

After the sealer was applied, all specimens, including the untreated controls, were conditioned a second time by drying for 15 days in the environmental chamber before being tested for resistance to moisture and chloride ingress. The environmental chamber remained at conditions of 73 °F (23 °C) and 50 ± 4% relative humidity. Every specimen was rotated between shelves daily to minimize variations caused by airflow in the chamber. During this drying period, all specimens were weighed at 5 and 15 days after receiving the sealer coating. The timing of these measurements was based on four assumptions, specified in Section 6.1 of Alberta MOT BT001:

1. All sealer evaporation takes place in the initial 5-day drying period.
2. The water loss in the sealed specimens is equal to the water loss in the untreated control specimens during the same 5-day drying period while accounting for vapor transmission ratio.
3. Sealer evaporation is the only form of sealer loss. This takes place in the initial 5-day drying period.
4. The dry sealer mass is the difference of fresh sealer mass and mass of sealer that evaporated at the end of the 15-day drying period.

The mass of each specimen was recorded at the start of the drying period (CD_0 – initial mass of untreated controls, and TD_0 – initial mass of corresponding sealed specimens), and after five days (CD_5 and TD_5) and 15 days (C_{15} and T_{15}) of drying. These measurements were used in intermediate calculations to determine the water loss, sealer loss, vapor transmission value, and the amount of dry sealer mass adhering to individual specimens. Initial mass loss (water) of control cubes (WL_0) was calculated using Equation3-3:

$$\boldsymbol{WL_0 = CD_0 - C_5} \qquad \textbf{(Eqn 3-3)}$$

The initial mass loss (water and sealer) of sealed cubes (ML_0) was calculated using Equation 3-4:

$$\boldsymbol{ML_0 = TD_0 - TD_5} \qquad \textbf{(Eqn 3-4)}$$

The water loss due to vapor transmission for the control set *(VLC)* was calculated using Equation 3-5:

$$VLC = T_5 - T_{15} \qquad \text{(Eqn 3-5)}$$

The water loss due to vapor transmission for the sealed set *(VLT)* was calculated using Equation 3-6:

$$VLT = T_5 - T_{15} \qquad \text{(Eqn 3-6)}$$

The vapor transmission value *(VT)* is a ratio comparing the drying performance of the sealed cubes to that of the untreated controls over the drying period of 10 days. For each three-cube set, *VT* for each sealer was calculated by dividing the average water loss of the sealed specimens (*VLT*) by the average of water loss from the controls due to vapor transmission (*VLC*), as shown in Equation 3-7:

$$VT = \frac{VLT}{VLC} \times 100\% \qquad \text{(Eqn 3-7)}$$

The average water loss in the sealed specimens (*WLT)* was calculated as a function of the average vapor transmission value, *VT,* and the average of the water lost in the untreated control specimens, WL_0, using Equation 3-8:

$$WLT = \mathrm{VT} \times WL_0 \qquad \text{(Eqn 3-8)}$$

Sealer loss *(SL)* was not taken as an average of the three cube set. It was determined by subtracting the water loss of the individual sealed cubes, *WLT,* from the corresponding initial mass loss of individual sealed cubes in the set, ML_0, using Equation 3-9:

$$SL = ML_0 - WLT \qquad \text{(Eqn 3-9)}$$

Finally, the mass of dry sealer adhered to the sealed specimens (*SD*) is determined. It was not taken as an average of the three cube set, but was calculated from the loss of sealer (*SL*) and the mass of fresh sealer *(SF)* gained by the individual sealed specimens, using Equation 3-10:

$$SD = SF - SL \qquad \text{(Eqn 3-10)}$$

3.5.5 Moisture Adjustments

During the Phase II drying, the untreated control set lost more moisture than the corresponding sets of sealed specimens over the 15-day drying period; this meant that the sealed cubes had greater RMC values than the untreated cubes. The RMC of the sealed specimens needed to be adjusted to the RMC of the untreated controls before testing for resistance to moisture and chloride ingress could commence.

After *SD* was calculated for each specimen following the 15-day drying period, the sealed specimens designated for testing the waterproofing performance and resistance to chloride ingress of the sealers were subjected to additional drying to match the average RMC of the untreated control set for that mix design. This was to ensure each specimen (sealed and untreated) had an equal RMC when subjected to tests involving the absorption of moisture. These sealed specimens were placed in a drying oven at a temperature of 140 °F (60 °C) and weighed daily until a desired mass (*DM*) was reached.

The untreated control specimens were enclosed in heavy-duty plastic freezer bags to prevent further moisture loss and stored in the environmental chamber until testing. The specimens designated for determining the depth of sealer penetration and sealer thickness were not adjusted to match the average RMC of the untreated controls and remained in the environmental chamber until they could be examined. In both cases, the conditions in the environmental chamber remained at 73 °F (23 °C) and 50 ± 4% relative humidity.

To solve for the *DM*, two RMC equations are set equal to each other and solved. Equation 3-11a shows the relation of the two sets: the average of the three controls' RMC set equal to a sealed cube's RMC. Equation 3-11b expands this relationship. The equations for the *DM* are as follows:

$$\overline{\boldsymbol{RMC}}_{\boldsymbol{C15}}\ (\%) = \boldsymbol{RMC}_{\boldsymbol{T15}}(\%) \qquad \textbf{(Eqn 3-11a)}$$

$$\overline{\boldsymbol{RMC}}_{\boldsymbol{C15}}\ (\%) = \frac{\boldsymbol{TMC}-[\boldsymbol{X_0}-(\boldsymbol{DM}+\boldsymbol{SD})]}{\boldsymbol{TMC}} \times \boldsymbol{100\%} \qquad \textbf{(Eqn 3-11b)}$$

Where:

TMC is the calculated total moisture content of the reference specimens for each mix design,

X_0 is the initial mass recorded for the sealed cube being adjusted when it was at 100% RMC,

$\overline{RMC}_{C15}\%$ is the calculated average RMC after the 15 days of drying of the three control specimens corresponding to the sealed specimens undergoing moisture adjustments,

SD is the calculated mass of sealer adhered to the sealed specimen, and

DM is the unknown desired mass of a particular specimen.

DM is therefore calculated for each sealed specimen using Equation 3-12:

$$\boldsymbol{DM} = \{\boldsymbol{X_0} + [(\boldsymbol{TMC} \times \overline{\boldsymbol{RMC}}_{\boldsymbol{C15}}\%) - \boldsymbol{TMC}]\} - \boldsymbol{SD} \qquad \textbf{(Eqn 3-12)}$$

The amount of moisture evaporated from the sealed cubes varied depending on the sealer type and mass of sealer adhering to the cubes. Cubes that matched the RMC of the untreated controls (indicated by reaching their *DM*) were secured in freezer bags and transferred back to the environmental chamber. They remained sealed in the bags until each sealed specimen with the same corresponding test designation had been adjusted to match the RMC of untreated controls.

3.6 Testing Procedures

After completing all the conditioning steps, the sealer products were evaluated on the following criteria: initial waterproofing performance, post-abrasion waterproofing performance, resistance to chloride ingress, depth of penetration, and sealer thickness.

3.6.1 Evaluation of Initial Waterproofing Performance

The initial waterproofing performance of the sealers was evaluated per Section 6.2 of Alberta MOT BT001 by submersing cubes in water for 120 hours (5 days). The mass gained by sealed specimens was compared to the mass gained by the untreated control specimens.

A rectangular polyethylene container held all eighteen waterproofing performance test specimens for each mix design. To support the specimens and to ensure their entire surface area was exposed to water, 0.5-in. (12-mm) diameter polyurethane tubes were glued to the bottom in pairs, as shown Figure 3.10. Each tube was cut to approximately 2 in. (50 mm) in length. The container was filled with an adequate volume of tap water until to ensure all the test specimens were submerged to at least a depth of 1 in. (25 mm) above the top surface of the specimens.

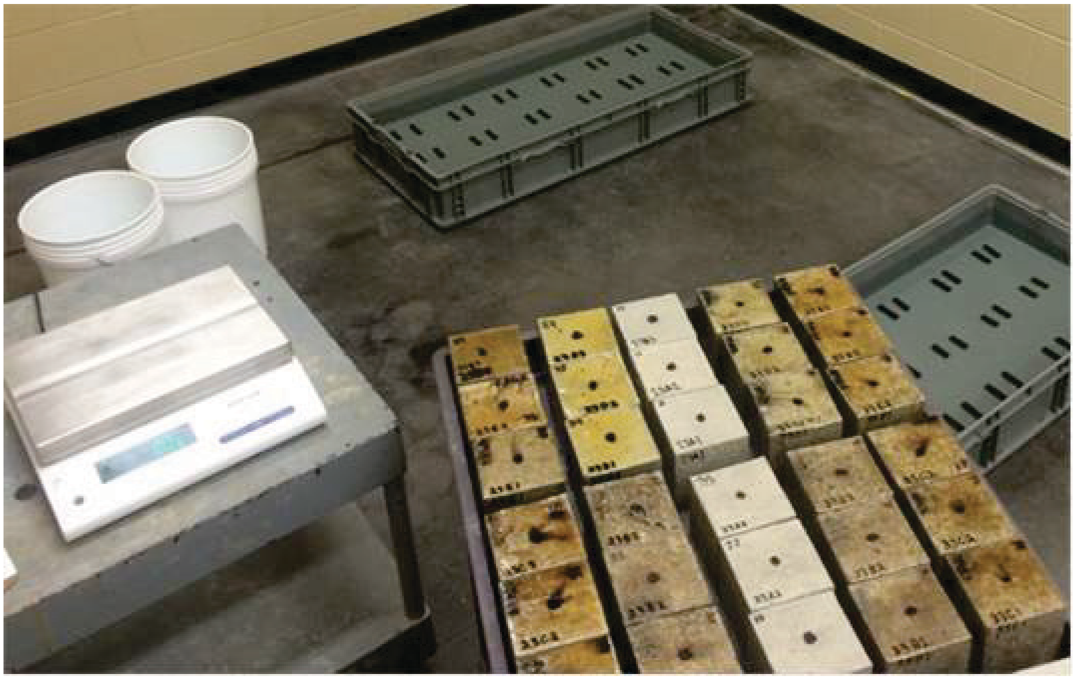


Figure 3.10 Waterproofing performance test configuration

Test specimens were weighed before and after submersion in water for 120 hours. After the soaking period, the cubes were extracted in the same order they were placed. They were lightly wiped free of surface moisture with a clean terry towel and weighed to the nearest 0.1 g. The initial waterproofing performance *(WPP_i)* for each sealer was determined by the average mass of water gained by the untreated control set *(CG1)* subtracted from the average mass of water gained from the sealed set *(SG1)*, divided by the average mass gained by the untreated control set *(CG1)* and was reported as a percentage shown by Equation 3-13.

$$WPP_i = \frac{CG1 - SG1}{CG1} \times 100\% \qquad \textbf{(Eqn 3-13)}$$

3.6.2 Evaluation of Post-Abrasion Waterproofing Performance

Post-abrasion waterproofing performance was evaluated by methods described in Section 6.3 of Alberta MOT BT001. The same containers and specimens used for determining the initial waterproofing performance (section 3.6.1) were reused for evaluating post-abrasion waterproofing performance. This test required removing the water gained in the initial waterproofing performance test, sandblasting the specimens to simulate years of traffic wear, and then submersing the sealed specimens for an additional 120 hours in water. The mass gained by the sealed cubes during the post-abrasion submersion was compared to the mass gained by the untreated controls in the initial waterproofing performance test. This test makes the assumption that additional traffic wear of untreated concrete would not change its absorption properties. Traffic wear of sealed concrete, however, could result in removing some or all of the sealer, reducing the effective performance of the sealer over time.

After the initial waterproofing performance tests, the sealed specimens were transferred to an oven and dried at a temperature of 140 °F (60 °C); the untreated controls were not subjected to post-abrasion testing. Specimens remained in the oven until the amount of water gained during the initial waterproofing performance test was removed. Specimens were weighed daily to prevent any over-drying of the samples. Any samples that finished drying to the target RMC before the others were sealed in plastic bags and placed in the environmental chamber to prevent any moisture loss until they were sandblasted and tested.

Sets of three sealed specimens with same mix design and treated with the same sealer were sandblasted on the same day. The amount of material removed from the sealed cube was dependent on the sealer type specified in Section 6.3 of Alberta MOT BT001 test. MD1 Specimens had 12.0 ± 1.0 g per face (72.0 ± 4.0 g total) removed, and MD2 and MD3 specimens had 24.0 ± 1.0 g removed per face (144.0 ± 4.0 g total). Figure 3.11 illustrates the contrast between two surfaces of the same specimen before and after this step. The exposed aggregate particles are clearly visible on the post-abrasion face. Each side perpendicular to the top surface was sandblasted, followed by the bottom and top surfaces. New identification and orientation marks were placed on the top surface using a permanent marker.

It was not feasible to remove the specified amount of material from the epoxy-sealed cubes solely by sandblasting because the epoxy was very resistant to abrasion. The layer of epoxy was first removed with an angle grinder equipped with a wire brush. Grinding was discontinued before the wire brush made contact with the concrete substrate. After grinding each side, the cubes were weighed and the mass removed was recorded; this was included as part of the total 12.0 or 24.0 g per face to be removed.

Figure 3.11 Surface of a specimen treated with Sealer A, before and after the second round of sandblasting for the post-abrasion waterproofing performance evaluation

After grinding, it took approximately 45 minutes to sandblast all six sides of each specimen, compared to more than one hour per side for sandblasting without first using the angle grinder. Due to the extensive time required to complete this sandblasting step, and the vulnerability of the samples to loss of moisture, only one set of three cubes was sandblasted per day. As a whole, sandblasting all the samples for durability testing took over two weeks to complete for each mix design.

The testing procedure for determining the post-abrasion waterproofing performance *(WPP_f)* is the same described in section 3.6.1. Only the sealed and abraded samples were tested. The average values for the mass gained by the controls *(CG1)* in the first initial submersion were used along with the average mass gained by the sealed and abraded samples *(SG2)* to calculate WPP_f using Equation 3-14:

$$\boldsymbol{WPP_f} = \frac{\boldsymbol{CG1} - \mathbf{SG2}}{\boldsymbol{CG1}} \qquad \textbf{(Eqn 3-14)}$$

3.6.3 Resistance to Chloride Penetration

The procedure described in Report 244 for the Series II tests was followed for determining the resistance to chloride penetration. The chloride penetration tests began at the same time as the waterproofing performance tests.

Separate but identical containers to those used in the waterproofing performance tests were used for evaluating the resistance to chloride ingress of the sealers. A 15% w/v NaCl aqueous solution was prepared with reagent grade NaCl and tap water. This solution was added to the testing containers until the specimens were submerged to a depth of 1 in. (25 mm) over their top surface.

The specimens were immersed in the NaCl solution for a total of 21 days. Specimens were removed from the solution for mass gain measurements every three days. Each specimen was lightly toweled dry, weighed, and returned to the solution. During testing, the solution was periodically stirred to prevent stagnation. After the 21-day soaking period, all specimens were placed into the environmental chamber for 12 days of drying. Specimens were weighed every three days during the drying period per the Report 244 Series II test procedure. After the drying period, three methods were used to measure the amount of chlorides that had penetrated into the specimens.

The first method used for measuring chlorides in the specimens relied on an assumed relationship between total mass gain and chloride ingress. A 15% NaCl solution is 9.1% chlorides by mass. In this "mass gain method," the expected chloride content of the specimens was calculated as 9.1% of the total mass gained after 21 days of soaking in the 15% NaCl solution. This method was applied to all cubes used in the resistance to chloride penetration testing. While simple, this method assumes that the salinity and chloride content of the absorbed solution remains equal to that of soak solution, and ignores the potential for ionic diffusion as a mass transport mechanism (that is, all mass uptake is solely by absorption). Longer exposure periods are typically used to measure the diffusion characteristics of concrete, but some diffusion still could occur in the 21-day exposure period of this test.

Another method determined the total acid-soluble chloride content from the powder by potentiometric titration against silver nitrate per ASTM C1152 / C1152M. Specimens were prepared for chemical analysis after the 12-day drying period. The samples were mechanically crushed and transferred to an oven set to 140 °F (60 °C). After drying overnight, the samples were pulverized until they passed a No. 60 (250 μm) sieve. Careful consideration was taken to thoroughly clean the crushing and pulverizing equipment after each sample to prevent cross contamination of the specimens. The powder was sealed in heavy-duty plastic bags and stored prior to analysis. The specimens used for TMC determination (described in section 3.5.1 of this report) were prepared by the same method to determine the baseline chloride content of specimens prior to exposure to the NaCl solution. This baseline value was deducted from the total chloride content of exposed specimens to provide a net value of chlorides retained from the absorbed solution. Appendix A contains the details of the titration procedure.

All combinations of mix designs and sealers were tested for chlorides by potentiometric titration with silver nitrate; however, not every specimen in the three cube set was tested due to time constraints of the project. From each set of three cubes, the specimen closest to the median mass gained from the 15% NaCl soaking period was selected and prepared for testing by titration. For the first three cubes tested, two powder samples were prepared and titrated per cube. Similar results were obtained for both samples tested from each cube, so only one titration per cube was conducted for the remaining cubes.

Chloride content of the specimens was also determined by XRF analysis. This was conducted per ASTM C114 by Wyoming Analytical Laboratories in Golden, Colorado, and served to validate the results obtained from the mass gain and titration measurements. Samples were selected from the same powder used to for titrations. For cost reasons, only a limited number of samples were tested by XRF. The 18 specimens selected for XRF analysis were:

- 1 untreated, unexposed (baseline) control (2 total)
    - MD2, MD3

- 1 sealed, unexposed control per sealer product (5 total)
    - MD2 – All sealer products

- 1 untreated, exposed control (2 total)
    - MD2, MD3

- 1 sealed, exposed per sealer product (9 total)
    - MD2 – All sealer products
    - MD3 – All sealer products, excluding Sealer C

Specimens from MD2 were selected because lower chloride contents were expected based on the mass gained. Specimens from MD3 were selected over MD1 to compare accuracy of the test between the different mix designs (100% portland cement versus the 20% Class F fly ash). For MD3, Sealer C was omitted from sampling due to sealer cracking.

Selected MD2 specimens were also sent to Dr. Tyler Ley's laboratory at Oklahoma State University to be analyzed for chloride penetration profiles using micro-XRF. This included one untreated control specimen, and one specimen each treated with Sealers A, B, D, and E. The micro-XRF analysis was used to produce compositional maps and to plot chloride concentrations at 0.5-mm intervals to a depth of 20 mm from the specimen surface.

3.6.4 Depth of Sealer Penetration and Sealer Thickness

Depth of sealer penetration and the thickness of the sealer were evaluated by a dye method and assistance of a concrete petrographer. Samples subjected to the dye method were observed under an optical microscope, and samples analyzed by the petrographer were examined under an optical microscope and using SEM-EDS.

3.6.4.1 Dye Method

The dye selected for use was a generic red food coloring product. Cubes of all combinations of sealers and mix designs were cut with an oil-lubricated lapidary saw into three pieces; cuts were perpendicular to the top (finished) and bottom surfaces. Specimens were washed in soapy water to remove any oil droplets deposited in the sample. The middle piece of the three pieces cut was dried in an oven overnight, and soaked in a solution of dye and water the following day. The remaining two pieces were allowed to dry in the open air in the lab at room temperature and were not exposed to dye. After drying, samples were observed under an optical microscope to discern between the dye-stained concrete and the unstained concrete, indicating the sealer penetration depth or sealer thickness.

3.6.4.2 Petrography

Eighteen specimens (six per mix design) were shipped to DRP Consulting for petrographic examination to determine the depth of sealer penetration and the thickness of the sealer. Each set of six specimens includes five specimens that have been exposed to a 15% NaCl solution for 21 days; four were treated with the sealers (Sealers A, B, D, and E) and one was an untreated control. A second control sample in each set was neither treated with sealer nor exposed to the NaCl solution. Specimens treated with Sealer C were not submitted for petrographic analysis because of the cracking observed in the sealer on numerous specimens.

Each specimen was observed for depth sealer penetration and sealer thickness by an optical microscope and SEM-EDS. The samples observed by SEM-EDS were analyzed by use of elemental analysis to determine the depth of penetration, indicated by changes in the chemical composition between sealed and untreated concrete.

After receiving preliminary results, one additional specimen was shipped to DRP Consulting for evaluation with SEM-EDS. The additional specimen was an oven-dried slice of a cube fabricated from MD1 that had been soaked continuously in Sealer A for one week, and was intended to represent a “best-case” scenario for sealer penetration.

3.7 Summary

Five different bridge deck sealers were applied to cube specimens made from three different types of concrete proportioned using ALDOT specifications for bridge decks. Sealer evaluation incorporated elements of the testing protocols of described in Report 244 Series II tests and the Alberta MOT BT Series standards. Specimens were fabricated, cured, and conditioned before the sealer was applied. After the sealer application, a series of calculations were used in a second conditioning phase to determine the vapor transmission ratio, and the dry sealer mass adhered to cubes, and to equate the RMC of the sealed specimens and untreated controls.

After the calculations, specimens were evaluated on their initial waterproofing performance, post-abrasion waterproofing performance, resistance to chloride penetration, and determination of sealer penetration and sealer thickness on the concrete. The resistance to chloride penetration was determined by a mass gain analysis, titration by silver nitrate, and XRF spectroscopy. The depth of sealer penetration and sealer thickness was determined by observing specimens stained with a dye under an optical microscope, and petrographic analysis including elemental analysis by SEM-EDS. The results from these tests are presented and discussed in Chapter 4.

Chapter 4: Results and Discussion

4.1 Sealer Application and Specimen Conditioning

This section provides information about the sealer application process and the specimen conditioning procedures. The fresh sealer mass recorded at time of application was used to determine the coverage rate of the sealer. The vapor transmission value (VT) and the dry sealer mass were determined from the evaporable components of sealer products calculated during the second conditioning phase.

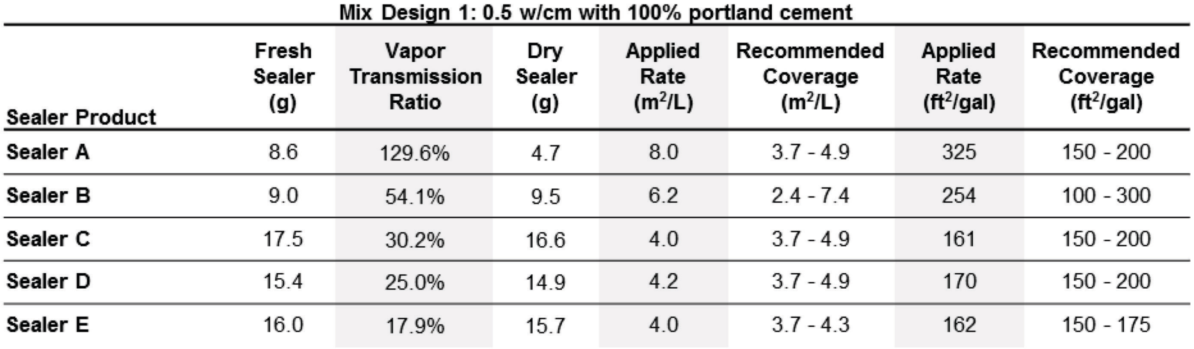
Table 4.1 presents the average fresh and dry sealer mass, average vapor transmission value (VT), the actual coverage rate, and the coverage rate recommended by the manufacturers. This information is presented for each mix design. All values are an average of the six cubes used for waterproofing performance and resistance to chloride penetration tests for each mix design. The coverage rate is based on the specific gravity of the sealer, fresh sealer mass, and the surface area of the 4 in. (100 mm) cube specimens (sealed surface area per sealer volume). Low values for coverage rates indicate that more sealer was applied to the specimen (that is, less area was sealed with a given unit volume of sealer), while high values for coverage rates indicate that less sealer was applied to the same surface area.

Sealers B, C and E were applied within the range of coverage rates recommended by the manufacturers for all three mix designs. The viscosity of epoxy-based products increased as they approached the gel time, causing a larger amount of fresh sealer to collect on the top surface of the cubes. This affected the coverage rate and can be seen in the results for MD2 specimens treated with Sealer D (Table 4.1b). Sealer D was applied within the recommended coverage rate for MD1 and MD3, although for MD2 more sealer than recommended was applied. Sealer A exceeded the recommended coverage rate for all three mix designs. That is, less sealer was applied to the specimens than recommended by the manufacturer. The applied coverage rate for the additional specimen that was soaked in Sealer A for one week was 41 ft^2/gal (1.0 m^2/L), or approximately four times the recommended amount sealer recommended by the manufacturer.

Specimens treated with Sealer A had the largest VT value, which was consistently over 100%. This signifies that the specimens were drying faster during the 15-day period after sealer application than if they had not been sealed. With an exception of MD1 specimens treated with Sealer B, all the epoxy sealers had VT values ranging from 15 to 30%, with Sealer E having the lowest VT values. This means the epoxy-based sealers considerably reduced the evaporation of moisture from the sealed cubes compared to the unsealed control specimens. The increased VT for Sealer B when applied to MD1 specimens could be an effect of the sealer's interaction with drier concrete (RMC = 70% for MD1) than the other two concrete mixtures (RMC = 80%).

Table 4.1 **Comparison of the average fresh sealer mass, vapor transmission value (VT), dry sealer mass, applied coverage rates, and the recommended coverage rates for the sealers applied to specimens made with (a) MD1, (b) MD2, and (c) MD3**

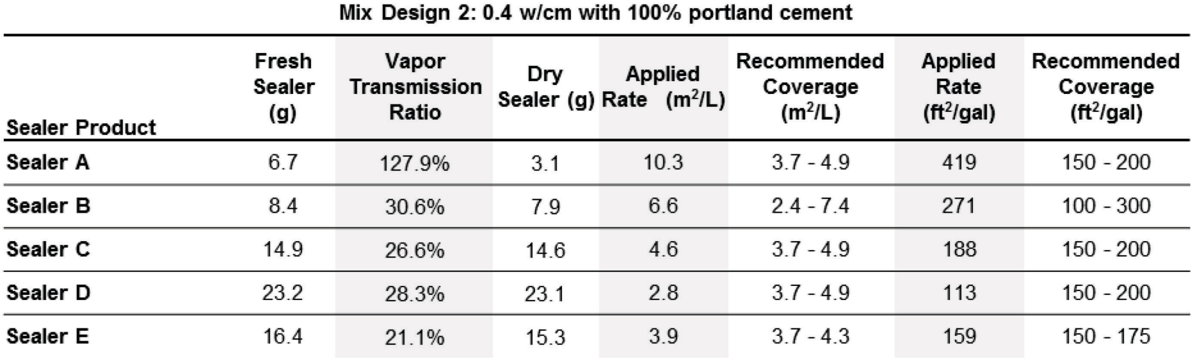
a)



Mix Design 1: 0.5 w/cm with 100% portland cement

Sealer Product	Fresh Sealer (g)	Vapor Transmission Ratio	Dry Sealer (g)	Applied Rate (m^2/L)	Recommended Coverage (m^2/L)	Applied Rate (ft^2/gal)	Recommended Coverage (ft^2/gal)
Sealer A	8.6	129.6%	4.7	8.0	3.7 - 4.9	325	150 - 200
Sealer B	9.0	54.1%	9.5	6.2	2.4 - 7.4	254	100 - 300
Sealer C	17.5	30.2%	16.6	4.0	3.7 - 4.9	161	150 - 200
Sealer D	15.4	25.0%	14.9	4.2	3.7 - 4.9	170	150 - 200
Sealer E	16.0	17.9%	15.7	4.0	3.7 - 4.3	162	150 - 175

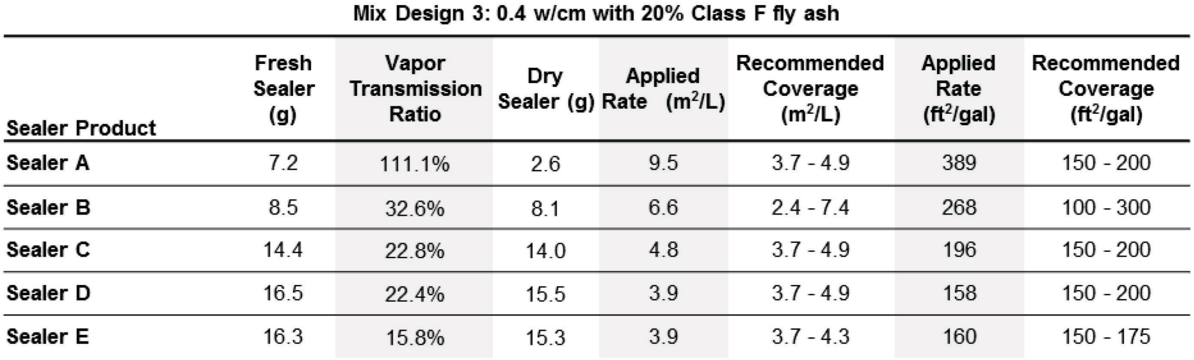
b)



Mix Design 2: 0.4 w/cm with 100% portland cement

Sealer Product	Fresh Sealer (g)	Vapor Transmission Ratio	Dry Sealer (g)	Applied Rate (m^2/L)	Recommended Coverage (m^2/L)	Applied Rate (ft^2/gal)	Recommended Coverage (ft^2/gal)
Sealer A	6.7	127.9%	3.1	10.3	3.7 - 4.9	419	150 - 200
Sealer B	8.4	30.6%	7.9	6.6	2.4 - 7.4	271	100 - 300
Sealer C	14.9	26.6%	14.6	4.6	3.7 - 4.9	188	150 - 200
Sealer D	23.2	28.3%	23.1	2.8	3.7 - 4.9	113	150 - 200
Sealer E	16.4	21.1%	15.3	3.9	3.7 - 4.3	159	150 - 175

c)



Mix Design 3: 0.4 w/cm with 20% Class F fly ash

Sealer Product	Fresh Sealer (g)	Vapor Transmission Ratio	Dry Sealer (g)	Applied Rate (m^2/L)	Recommended Coverage (m^2/L)	Applied Rate (ft^2/gal)	Recommended Coverage (ft^2/gal)
Sealer A	7.2	111.1%	2.6	9.5	3.7 - 4.9	389	150 - 200
Sealer B	8.5	32.6%	8.1	6.6	2.4 - 7.4	268	100 - 300
Sealer C	14.4	22.8%	14.0	4.8	3.7 - 4.9	196	150 - 200
Sealer D	16.5	22.4%	15.5	3.9	3.7 - 4.9	158	150 - 200
Sealer E	16.3	15.8%	15.3	3.9	3.7 - 4.3	160	150 - 175

MD 1 specimens treated with Sealer B also appear to gain mass in the second drying phase. The cause for mass gain is unknown, but likely from a weighing error after the second immersion in Sealer B during the sealer application process. This error resulted in the RMC value of these sealed specimens to increase by only 0.1%, which was not enough to be significant.

The sealer application method used in this research was inadequate to apply Sealer A at the recommended coverage rate, assuming that the recommended coverage rate can be achieved in the field. Improvements to the application method for penetrating sealers, such as Sealer A, should be considered. This could include repeating immersions or extending the duration of the immersions until the recommended coverage rate is met. Another adjustment in the application procedure should be made for non-penetrating epoxy sealers to provide an evenly distributed amount of sealer on all six surfaces of the cubes:

1. Use the method of immersion for only the first application.

2. Remove the excess sealer ponded on the surface with a brush or roller.

3. Determine the coverage rate applied from the first immersion. If needed, apply the second application with a brush or roller.

4.2 Depth of Sealer Penetration and Sealer Thickness

None of the specimens soaked in the dye appeared to have any detectable penetration depth beyond the concrete surface when observed under an optical microscope. The use of dye wasn't necessary for evaluating non-penetrating epoxy sealers, which only penetrated into surface voids and cracks; not into the smaller capillaries of the substrate. Sealer E and Sealer D had the overall greatest sealer thickness. This varied between 120 to 1,300 μm on the top surface of the specimens examined. Sealer C and Sealer B had thickness which ranged from 120 to 900 μm. Figure 4.1 shows a cross section of the top surface of a specimen treated with Sealer E, and illustrates typical variations in sealer thickness on the surface of a single specimen.

Figure 4.1 Sealer thickness variations for MD1 specimen treated with Sealer E.

Unlike Sealer A, epoxy-based sealers would pond on the top surface of the cube; this effect resulted in a greater sealer thickness in localized areas. The side and bottom surfaces of the specimens had uniform film thickness of sealer approximately 100 μm. The sealer thickness on the top surface may be a more accurate representation of the sealer thickness on bridge decks in the field, so more emphasis was placed on examining this surface than the sides of the cubes. Usually, the thickest layer of sealer was located in the center of the cube or above concrete air voids on the top surface.

Dr. David Rothstein aided the investigation by examining specimens using a combination of optical microscopy and SEM-EDS. None of the epoxy sealers penetrated into the concrete substrate. Sealer A penetrated no more than 100 μm into the concrete. No penetration was observed with an optical microscope; only analysis by SEM-EDS was able to determine the penetration by analyzing variations in sodium concentrations. (Rothstein 2015)

The limited penetration of Sealer A was thought to have been influenced by the fact that the sealer was applied at less than the recommended coverage rate. However, the specimen soaked in the sealer for one week, despite having nearly *four times* the amount of sealer needed to satisfy the minimum coverage rate specified by the manufacturer, did not exhibit any increase in penetration depth when evaluated with SEM-EDS (Rothstein 2015). The full petrographer's report has been provided under separate cover because of length and file size.

4.3 Waterproofing Performance

The results of each product's initial and post-abrasion waterproofing performance relative to the untreated control specimens are reported and discussed in the following sections.

4.3.1 Initial Waterproofing Performance

The initial waterproofing performance results are presented in Table 4.2 as a percentage mass gain relative to the control specimens. Higher values are indicative of better waterproofing performance. Sealers D and E had the best overall average performance, with an initial waterproofing performance of 82%. Sealer C had the third overall best results, though it outperformed all sealers for MD2. Sealer B performed similarly to the other epoxy sealers when applied to MD2 and MD3 specimens, but did not perform as well when applied to MD1 specimens. Sealer A consistently performed poorly, relative to the other products, with an average initial waterproofing performance of only 10%.

Table 4.2 Average initial waterproofing performance results for each sealer, reported as a percentage of the sealed cubes reduction in mass gain to that of the untreated control

AVERAGE INITIAL WATERPROOFING PERFORMANCE IN WATER

SEALER PRODUCT	MD1: 0.5 w/cm with 100% portland cement	MD2: 0.4 w/cm with 100% portland cement	MD3: 0.4 w/cm with 20% Class F fly ash	Average of all three mix designs
Sealer A	12%	1%	16%	10%
Sealer B	32%	76%	71%	60%
Sealer C	79%	88%	73%	80%
Sealer D	85%	76%	85%	82%
Sealer E	82%	79%	86%	82%

Sealer B's initial waterproofing performance for MD1 was significantly deficient compared to Sealer B's initial waterproofing performance on the other two mixes. One factor stands out as a possible source for the decreased performance, observed in Table 4.1(a), is that Sealer B has a relatively higher VT of 54% when applied on a drier concrete surface (70% RMC for MD1) compared to a VT of 30% when applied the other two mixes (80% RMC). For Sealer B, a relationship could exist between the concrete RMC at sealer application, VT, and waterproofing performance. That is, a higher VT is observed when the Sealer B is applied on drier concrete, resulting in a decreased resistance to moisture ingress, and decreasing the waterproofing performance.

4.3.2 Post-abrasion Waterproofing Performance

The post-abrasion waterproofing performance results, relative to the initial waterproofing performance of the control set, are presented in Table 4.3. Negative values are indicative of the sealed specimens gaining more mass than the untreated controls. None of the products had an acceptable post-abrasion waterproofing performance by the standards of the Albert MOT BT Series protocol. With the exception of MD3 specimens treated with Sealer A (WPP_f = 16%), all specimens had negative post-abrasion waterproofing performance values. This demonstrates the loss of protection caused by the grinding and sandblasting process.

Table 4.3 Average post-abrasion waterproofing performance results for each sealer, reported as a percentage of the sealed cubes reduction in mass gain after abrasion to the initial waterproofing performance of the untreated control

AVERAGE POST-ABRASION WATERPROOFING PERFORMANCE IN WATER

SEALER PRODUCT	MD1: 0.5 w/cm with 100% portland cement	MD2: 0.4 w/cm with 100% portland cement	MD3: 0.4 w/cm with 20% Class F fly ash	Average of all three mix designs
Sealer A	-56%	-4%	16%	-14%
Sealer B	-49%	-43%	-10%	-34%
Sealer C	-56%	-67%	-37%	-53%
Sealer D	-67%	-94%	-48%	-69%
Sealer E	-60%	-73%	-25%	-53%

Figure 4.2 compares the average mass gained in the initial and post-abrasion waterproofing performance tests, further illustrating the difference in performance before and after simulated traffic wear.

The results suggest that the post-abrasion waterproofing test method was not an effective evaluation for simulating the effects of traffic wear on a sealer product, if the procedures for sandblasting in Section 6.3 of Alberta MOT BT001 were followed. For any penetrating sealer with a lighter molecular weight (e.g. silanes and siloxanes), this test method could potentially have some quantifiable results for evaluating traffic wear on the sealer, but confirmation of an adequate depth of penetration should be determined before spending long hours sandblasting samples. An alternative method of evaluating simulated traffic wear for epoxy sealers is needed.

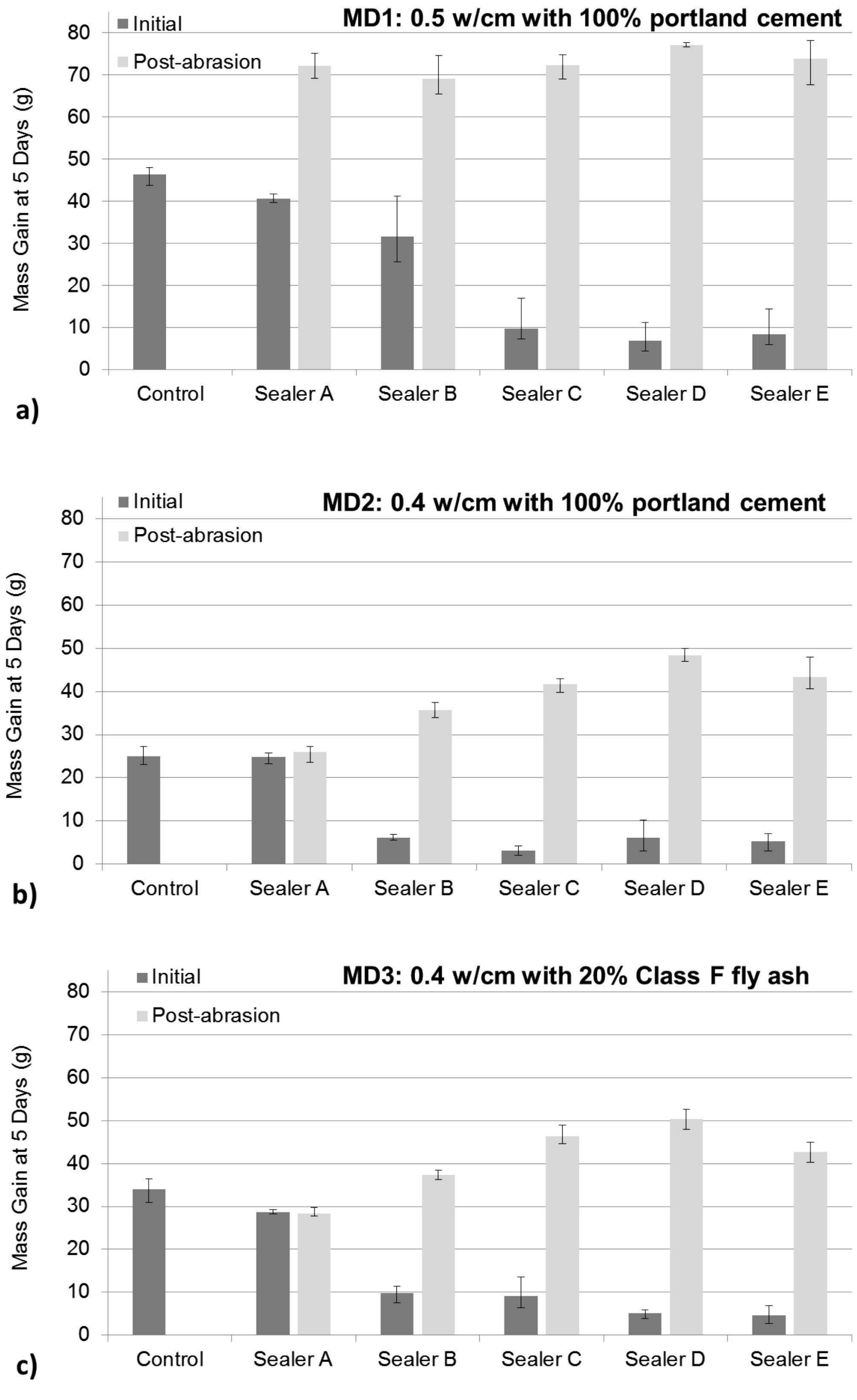


Figure 4.2 Average initial and post-abrasion mass gains at 5 days for (a) MD1, (b) MD2, and (c) MD3. Error bars represent the range of values measured between three cube sets

4.4 Resistance to Chloride Penetration

Test specimens were subjected to a 21-day immersion period in a 15% NaCl solution and then evaluated by their waterproofing performance and resistance to chloride ingress. The resistance to chloride penetration was determined by the mass gained while immersed in the NaCl solution, potentiometric titration, and XRF spectroscopy. Results from the mass gain, waterproofing performance, and chloride analyses are discussed in this section.

4.4.1 Mass Gain and Waterproofing Performance

Table 4.4 contains the average 21-day waterproofing performance of each sealer for each mix design. The waterproofing performance represents the average reduction of mass gained by the sealed specimens relative to the untreated controls after soaking in the 15% NaCl solution. For acceptable performance, the Report 244 Series II protocol recommends a minimum 75% reduction in average mass gained by the sealed specimens relative to the unsealed controls after 21 days in 15% NaCl. Sealers C and D had acceptable performance for MD1 and MD2, but not MD3. Sealer D passed the 21-day performance criteria for MD1 and MD2, but not MD3; Sealer E met the 21-day criteria only for MD2. Sealer A and Sealer B failed to meet the 21-day performance criteria for all three mix designs. The 21-day waterproofing performance of Sealer C across all three mix designs also met the 75% threshold for acceptable performance, while Sealers D and E fell just below the threshold with an average 74% waterproofing performance.

Table 4.4 Average waterproofing performance results for each sealer, reported as a percentage of the reduction in mass gained by the sealed specimens after 21 days in a 15% NaCl solution relative to the untreated controls

AVERAGE WATERPROOFING PERFORMANCE AT 21 DAYS IN 15% NaCl

SEALER PRODUCT	MD1: 0.5 w/cm with 100% portland cement	MD2: 0.4 w/cm with 100% portland cement	MD3: 0.4 w/cm with 20% Class F fly ash	Average of all three mix designs
Sealer A	7%	5%	14%	9%
Sealer B	46%	71%	63%	60%
Sealer C	84%	77%	63%	75%
Sealer D	81%	81%	59%	74%
Sealer E	70%	82%	69%	74%

Figure 4.3 presents a comparison of the average mass gained by all test specimens that were soaked in water and specimens that were soaked in the 15% NaCl solution. The average mass gained at three days for specimens in the 15% NaCl solution was included to compare with the average mass gained at 21 days in the 15% NaCl solution and the average mass gained at 5 days for the specimens soaking in water. Error bars represent the range of values measured for each set of three specimens.

It is clear to see the majority of the mass was typically absorbed in the first three days for untreated control specimens soaked in the 15% NaCl solution. The mass gains by specimens after five days in water and three days in 15% NaCl solution were typically similar, with the notable exception of MD1 specimens treated with Sealer B; these appeared to gain mass at a much slower rate in the 15% NaCl solution than the corresponding specimens tested in water. Most of the MD1 samples had a larger range of values measured for mass gain than specimens made from MD2 and MD3. This may be, in part, a result of the higher w/cm and porosity.

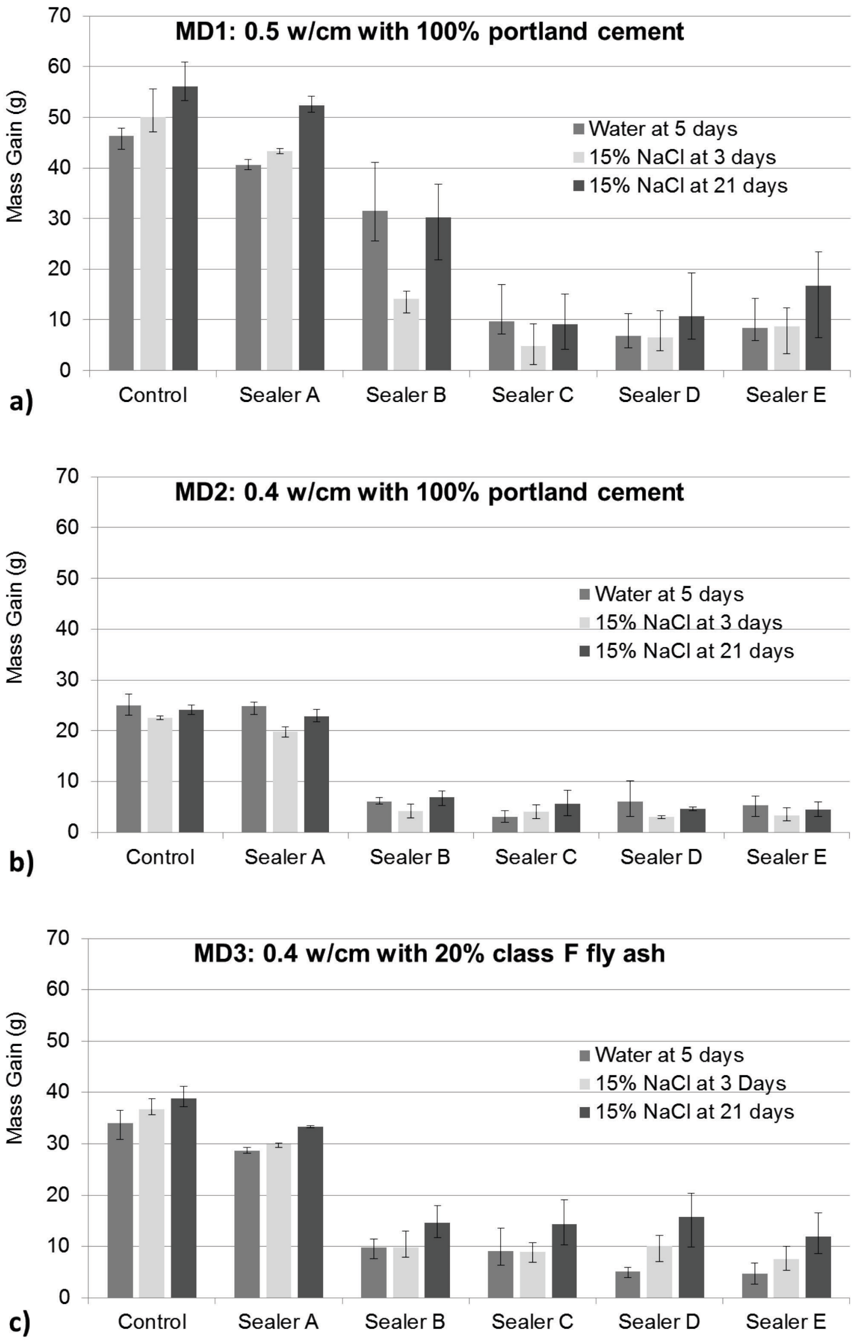


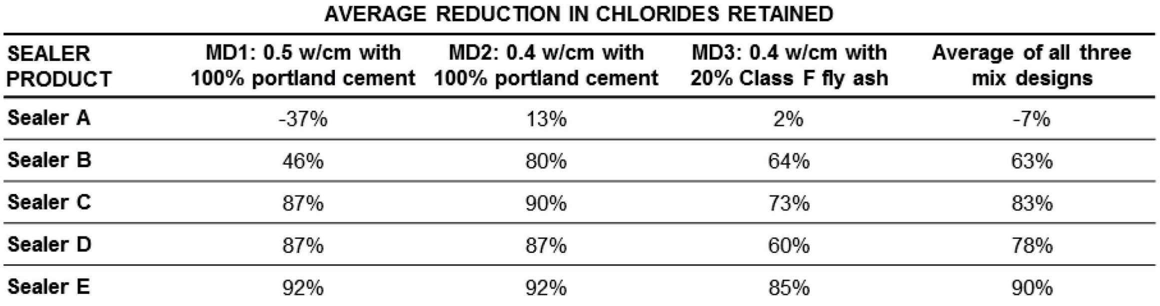
Figure 4.3 Comparison of the average mass gained between specimens which soaked in water and 15% NaCl solution for (a) MD1, (b) MD2, and (c) MD3. Error bars represent the range of values measured between three cube sets.

4.4.2 Chloride Analysis

The chloride content of the specimens was measured by potentiometric titration with silver nitrate and XRF spectroscopy; these chloride contents were also compared with the expected chloride content based on the mass of 15% NaCl solution gained by the specimens. The results obtained from all three techniques used to measure chlorides retained are discussed in this section, along with the chloride penetration profiles measured using micro-XRF.

The resistance to chloride penetration of the sealers was evaluated as a percentage of chlorides retained in the sealed specimens relative to the chlorides retained in the untreated control specimens after a 21-day immersion in 15% NaCl solution. Table 4.5 presents the resistance to chloride penetration for each sealer product relative to the unsealed controls as measured by titration. Negative values indicate that more chlorides were retained in the sealed specimens than in the untreated controls. The Report 244 Series II test protocol recommends a minimum reduction of the average net chloride content retained in sealed cubes of 75% of the average net chloride content in the unsealed controls. Sealer E performed the best for all three mix designs, averaging a 91% net chloride content reduction relative to the control. Sealers C and Sealer D met this criterion for MD1 and MD2, but not MD3; although these two were below 75% for MD3, their average reduction in chloride content for all three mix designs exceeded 80%. It is also worth noting that all four epoxy-based sealers performed better on the basis of chlorides measured by titration than they did on the basis of the mass gain measurements (waterproofing performance in 15% NaCl). Sealer A, however, performed worse on average based on chlorides measured by titration.

Table 4.5 Average reduction of chloride content in sealed specimens, measured by titration. Data are reported as a percentage of the chlorides measured by titration in the unsealed control specimens. All specimens were soaked in a 15% NaCl solution for 21 days



AVERAGE REDUCTION IN CHLORIDES RETAINED

SEALER PRODUCT	MD1: 0.5 w/cm with 100% portland cement	MD2: 0.4 w/cm with 100% portland cement	MD3: 0.4 w/cm with 20% Class F fly ash	Average of all three mix designs
Sealer A	-37%	13%	2%	-7%
Sealer B	46%	80%	64%	63%
Sealer C	87%	90%	73%	83%
Sealer D	87%	87%	60%	78%
Sealer E	92%	92%	85%	90%

Table 4.6 compares the expected chloride content calculated based on the mass gain measurements with the total chloride content measured by titration and XRF. Specimens treated with Sealer E had the lowest chloride content retained for each mix design. Specimens treated with Sealer C had the second overall lowest chloride content. Specimens treated with Sealer D contained the fewest chlorides for MD1 and MD2. The chloride content of MD 1 specimens treated with Sealer B was much higher that MD2 and MD3 specimens treated with Sealer B. MD1 and MD3 specimens treated with Sealer A had chloride contents that were *higher* than the untreated control set, as measured by titration and XRF.

Table 4.6 The total chloride content measured by each of the three techniques, reported as percentage of the total mass of concrete

Sealer Product	Mix Design	Mass gain after 21days in 15% NaCl solution (% Mass)	Calculated Chlorides by Mass Gained (% Mass)	Measured Chlorides by Titration (% Mass)	Measured Chlorides by XRF (% Mass)
Control	1	2.16	0.196	0.259	-
	2	0.93	0.084	0.185	0.189
	3	1.50	0.137	0.167	0.210
Sealer A	1	2.14	0.196	0.358	-
	2	0.88	0.084	0.161	0.179
	3	1.34	0.137	0.200	0.216
Sealer B	1	1.53	0.195	0.135	-
	2	0.27	0.080	0.038	0.033
	3	0.55	0.122	0.058	0.060
Sealer C	1	0.35	0.140	0.027	-
	2	0.22	0.024	0.018	0.017
	3	0.54	0.050	0.052	-
Sealer D	1	0.27	0.025	0.015	-
	2	0.18	0.017	0.024	0.014
	3	0.81	0.074	0.070	0.082
Sealer E	1	0.27	0.025	0.013	-
	2	0.17	0.016	0.014	0.010
	3	0.34	0.031	0.026	0.031

Figure 4.4 presents for a comparison of the chloride contents determined by XRF and titration for the same specimens. A diagonal dashed line serves as a line of equity, or perfect agreement between the two test methods. A very strong correlation exists ($R^2 > 0.99$) with the results from titration and XRF for both MD2 and MD3. Moreover, the slope of the best-fit line is only slightly greater than line of equity, indicating a slightly higher chloride content measured by XRF, but otherwise nearly equivalent results. XRF is a faster method compared to titration for evaluation of chlorides in concrete according to these data, and provides nearly identical data. Titration took four to five hours per sample, whereas a sample evaluation by XRF spectroscopy can be completed in minutes.

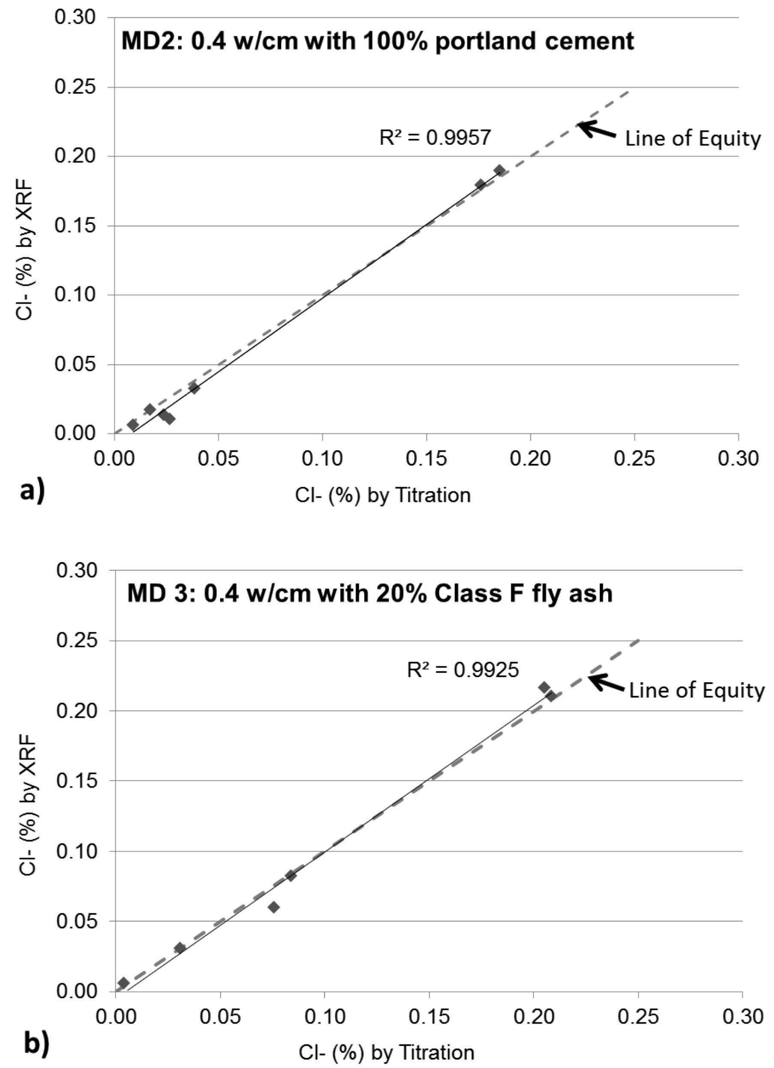


Figure 4.4 Comparison of chloride contents determined by titration and XRF for all combinations of controls and sealers on (a) MD2 and (b) MD3. MD3 specimens treated with Sealer C were not included because of cracking of the sealer

Figure 4.5 presents the chloride content measured by titration and XRF compared with the expected chloride content based on the mass gain measurements (9.1% of the mass of the absorbed solution). The expected value is shown as a line of equity in the figure and can be used to compare chloride contents determined by titration and XRF with the percentage of mass gained after 21 days of soaking in a 15% NaCl solution. Any data plotted above the line of equity indicate that fewer chlorides were measured than would be expected based on the mass gain measurements. Data which fall below the line indicates that more chlorides were measured than would be expected based on the mass gain measurements.

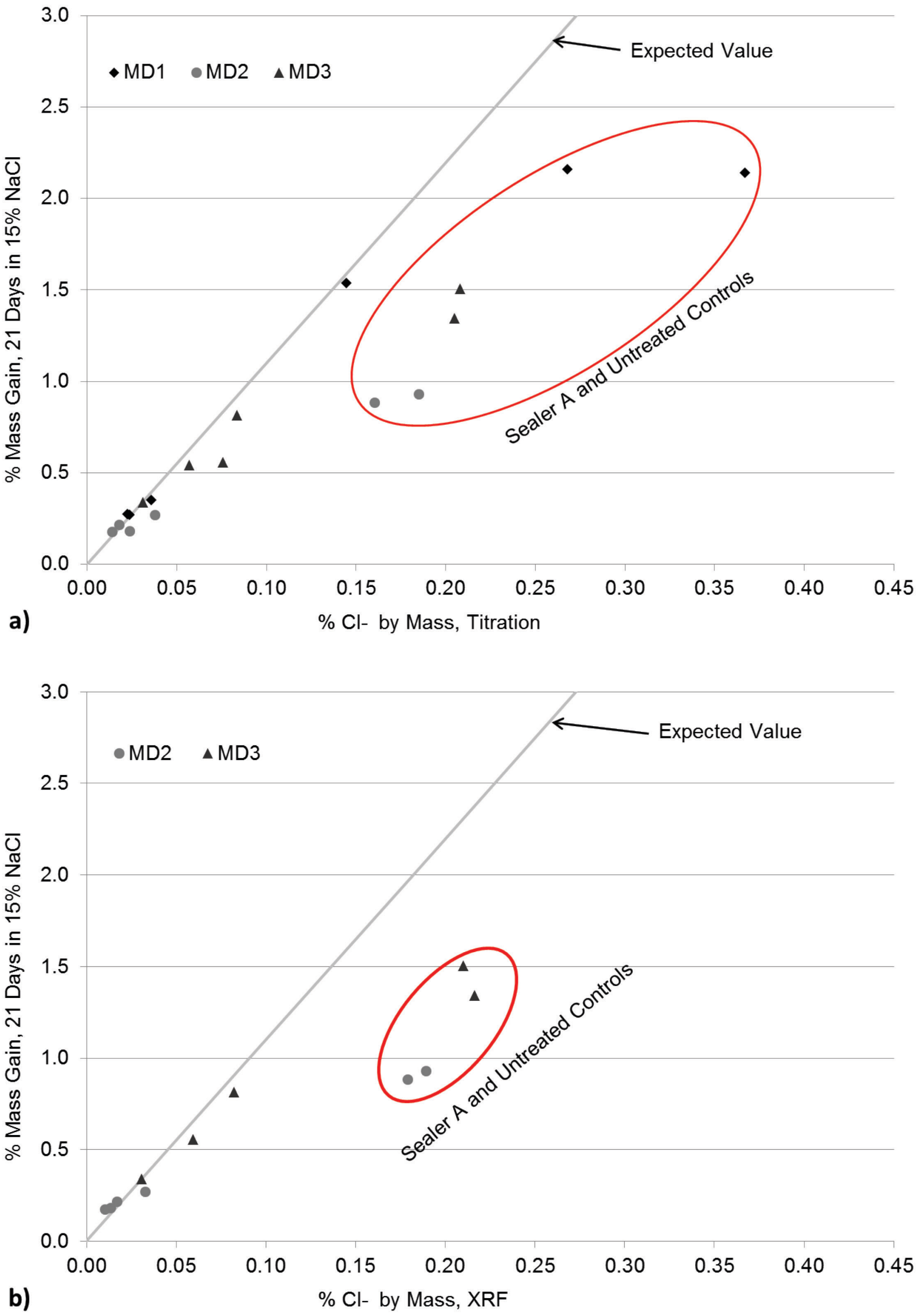


Figure 4.5 Mass gain vs. chlorides measured by (a) titration and (b) XRF

According to Figure 4.5, the untreated controls and specimens treated with Sealer A retained more chloride ions than expected. A possible explanation for the higher chloride contents in the specimens treated with Sealer A and the control specimens could be due to chloride diffusion in the concrete once the specimens were saturated. More work would be needed to confirm the role of diffusion.

Figure 4.6 presents the same data as Figure 4.5(a), but removes the outliers (Sealer A and untreated controls) from this observation to focus on the data for specimens treated with the epoxy-based sealers only. The data for these specimens shows a better fit to the theoretical value as suggested from clustered data along the line. By dividing the chloride contents measured by titration by the mass of NaCl solution absorbed, the chloride content of the absorbed NaCl solution in the concrete can be calculated. For the specimens in Figure 4.6, the chloride concentration of the absorbed solution ranged from 4.9% to 14.1%. This range is consistent with the range of values reported in Report 244 Series II tests of 4.6% - 11.1% (Pfeifer and Scali 1981), but also varies considerably from the expected 9.1% chlorides in 15% NaCl solution.

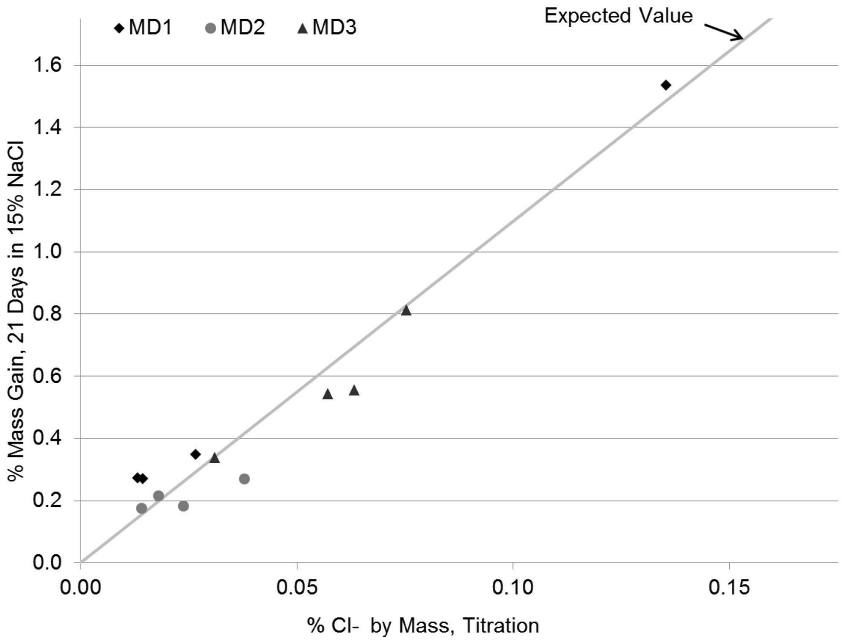
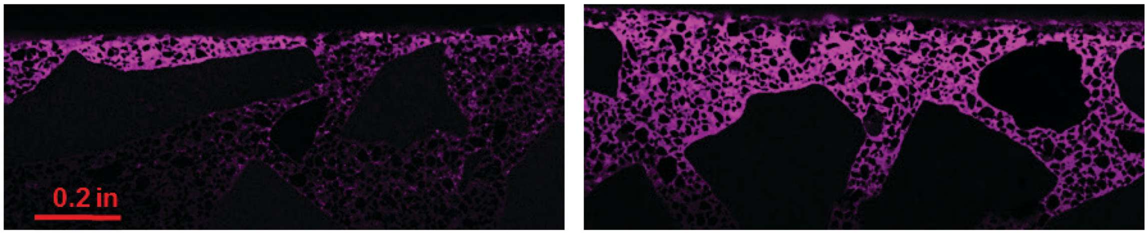


Figure 4.6 Mass gain measurements vs. chloride measured by titration for the four epoxy-based sealers

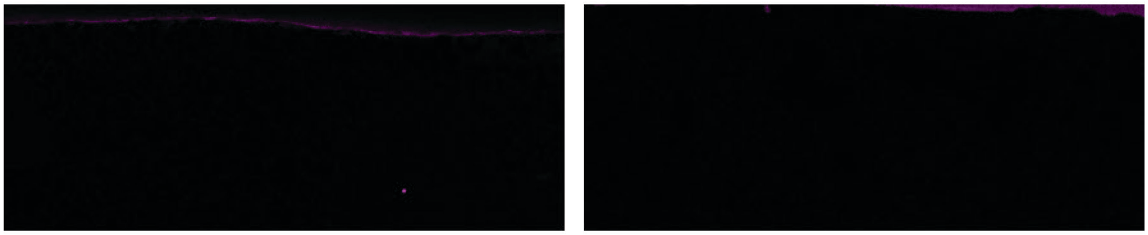
Selected specimens used in the petrographic investigation of sealer thickness and penetration depth were also examined using micro-XRF to measure chloride penetration profiles to depths of 10 to 20 mm. Figure 4.7 shows micro-XRF dot maps (area scans) of cut and polished cross-sections of specimens treated with Sealers A, B, and E, and a control specimen. The presence of chlorides is indicated by purple dots, and the intensity of the color is related to the concentration of chlorides. Black regions contain no detectable chlorides. The aggregate particles can therefore be identified in areas where chlorides have penetrated as black regions surrounded by a matrix of purple (cement paste). Only a thin strip of purple can be seen in the images of specimens treated with Sealers B and E; these epoxy-based sealers appear to have blocked any measurable chloride ingress.

Figure 4.8 presents the concentration vs. depth profiles for these four specimens plus one specimen treated with Sealer D. These concentrations were calculated based on the area scans by workers in Dr. Tyler Ley's research group at Oklahoma State University. Results of a point scan of the specimen treated with Sealer A are also shown; the chloride concentrations at six points of increasing depth were measured using micro-XRF to verify the concentrations calculated based on the area scan. These data confirm that concrete treated with Sealer A experienced significantly greater chloride ingress than the untreated control concrete. Chloride concentrations for the specimen treated with Sealer A were greater at all measured depths than for the untreated control specimen. The data also show that chlorides were essentially unable to penetrate the epoxy-based sealers.



Untreated Control

Sealer A



Sealer B

Sealer E

Figure 4.7 Micro-XRF area scans of polished surfaces of four specimens showing chloride penetration.

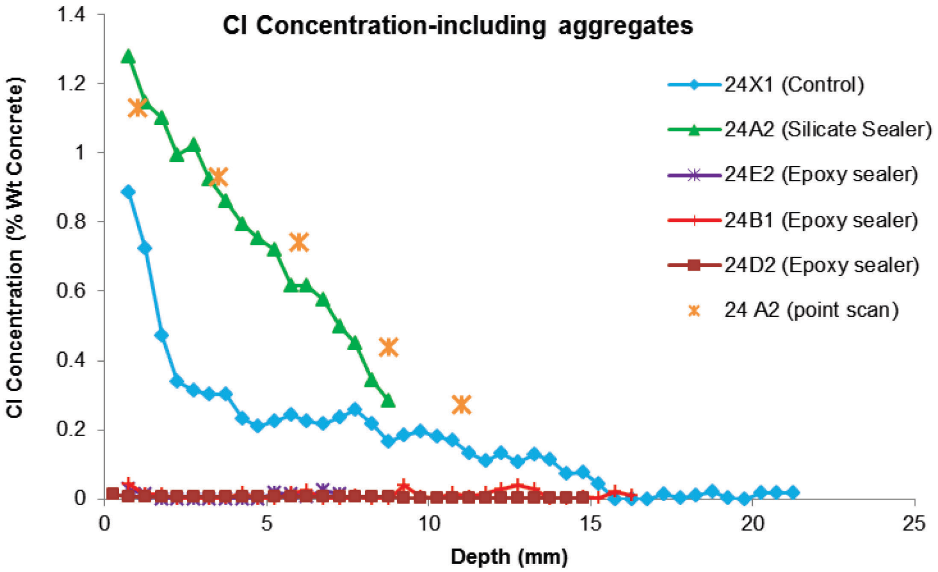


Figure 4.8 Chloride concentration profiles determined from micro-XRF area scans of five specimens. Point scan results for the specimen treated with Sealer A are also shown

Based on this research, mass gain measurements are insufficient for evaluating the resistance to chloride ingress because the expected chloride contents based on a 15% NaCl soak solution did not match the chloride contents measured by titration and XRF. Calculating expected chloride contents based on mass gain measurements assumes that all chloride ingress occurs by absorption, and does not account for the possibility of diffusion. Titration and XRF are more accurate methods of measuring the chloride content in concrete. Titration, however, is a time-consuming process. XRF provided nearly identical results to titration and required less sample preparation, resulting in a faster method of evaluation.

4.5 Early-Age Sealer Cracking

Many specimens from each mix design that were treated with Sealer C developed cracks in the sealer on the top surface of the cube where the sealer coating had the greatest thickness. Air voids in the sealer appear to have also played a role in crack initiation. Cracking of the sealer was observed on 13 out of 27 specimens treated with Sealer C (the remaining 9 sealed specimens were not counted because the sealer was removed in preparation for the post-abrasion waterproofing tests). Cracks were first noticed approximately 3 weeks after sealer was applied. Figure 4.9 illustrates how the specimens were conditioned and handled between the time that the sealer was applied and when cracking of the sealer was observed.

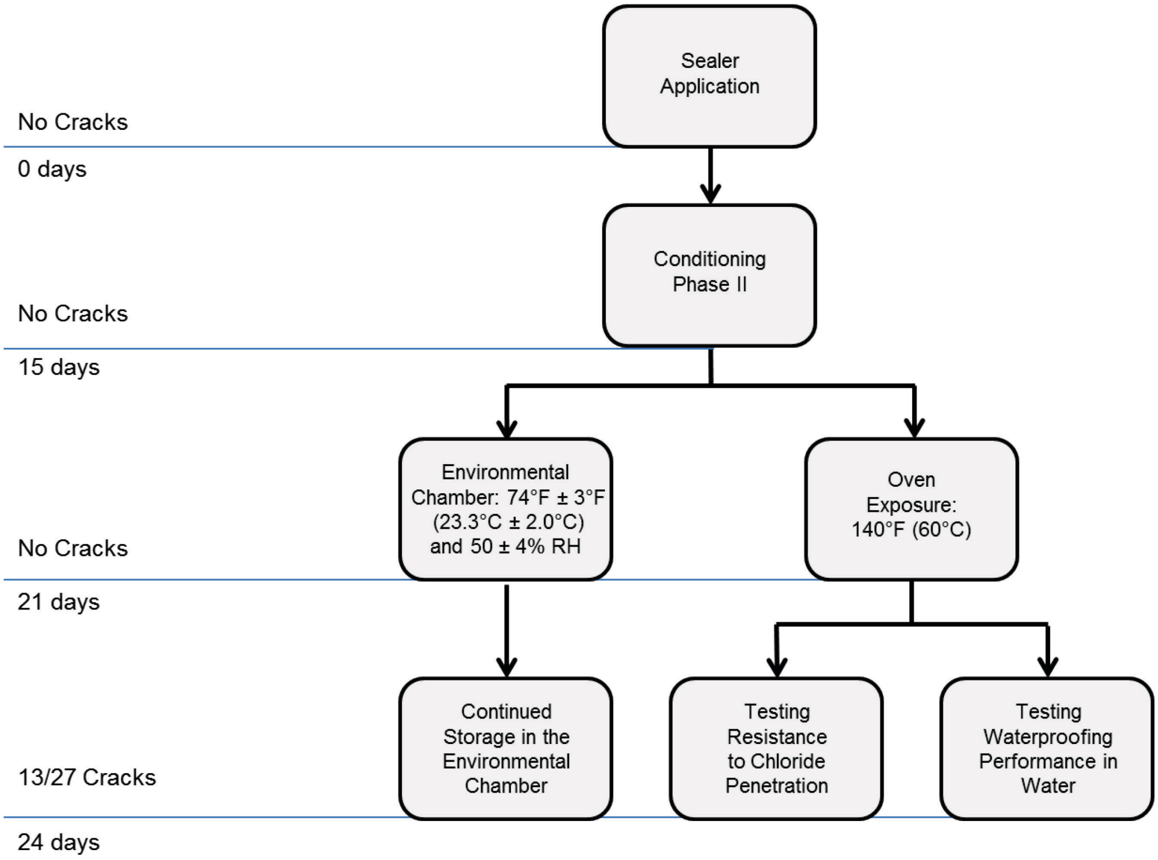


Figure 4.9 Timeline from after sealer application when cracks were noticed

Initially, the sealer cracking was assumed to be a side effect of thermal strains that developed in the sealer as specimens cooled once removed from the oven for moisture adjustments. However, specimens treated with Sealer C that were not subjected to oven drying for moisture corrections also developed cracks in the sealer. These specimens were stored in the environmental chamber set at conditions of 74 °F ± 3 °F (23.3 °C ± 2.0 °C) and 50 ± 4% RH. No cracking was observed in the other three epoxy-based sealer products. It is worth noting that the excess Sealer C from the sealer application process was placed into a container, where it solidified and then completely disintegrated from severe cracking within one week.

To investigate the cracking of Sealer C further, 5 of the 13 samples with cracked sealer were examined using a Keyence VHX-1000 optical microscope equipped with a digital camera. Specimens treated with the other epoxy sealer products were examined briefly for cracks when determining the depth of sealer penetration and sealer thickness; they did not exhibit cracks anywhere on the surface. Figures 4.10 and 4.11 show two specimens with cracking that were exposed to oven drying for moisture adjustments. The darker shades of amber are indicative of the thickest layer of sealer; cracking was only observed to occur in these areas where the sealer thickness was greater than 500 μm.

Figure 4.12 provides a three-dimensional cross-section of a crack generated by the microscope. This was a MD1 specimen treated with Sealer C and the crack cross-section in the figure was similar to those observed on other specimens. Cross-sections of all the cracks were triangular, with the widest space at the top surface of the sealer which indicates the stress field was greater on the surface of the sealer and cracked downward.

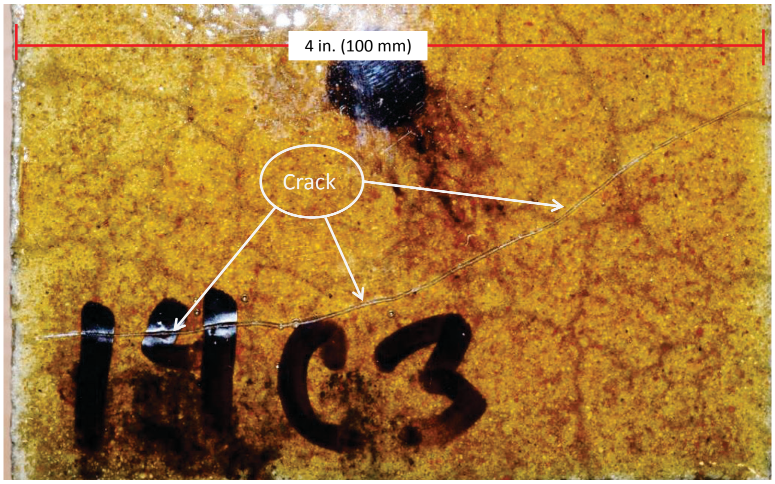


Figure 4.10 Cracking on the top surface of a MD1 specimen treated with Sealer C. This sample was subjected to moisture adjustments in an oven set at 140 °F (60 °C)

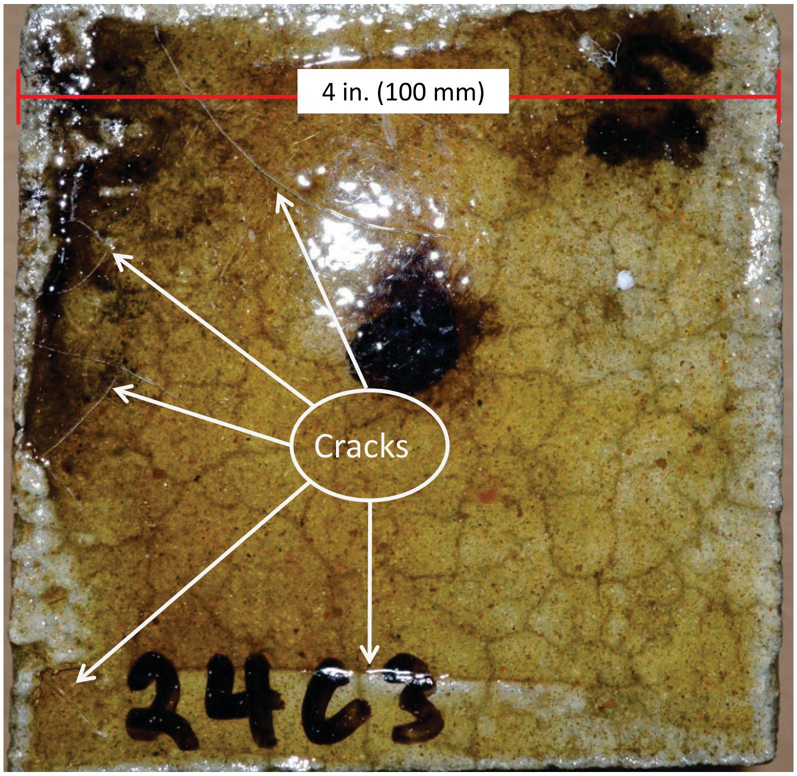


Figure 4.11 Multiple cracks on the top surface of a MD2 specimen treated with Sealer C. This sample was subjected to moisture adjustments in an oven set at 140 °F (60 °C)

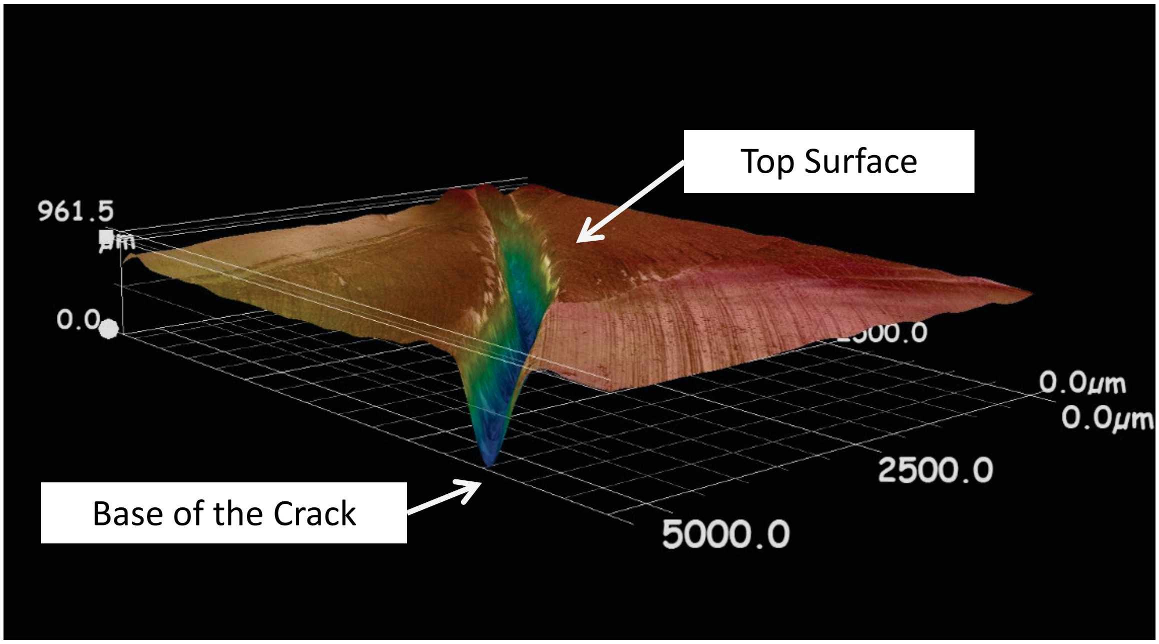


Figure 4.12 Cross-section of a crack profile in a MD1 specimen treated with Sealer C

Air voids in the sealer were observed on all specimens treated with all four of the epoxy-based sealer products. Air voids varied in size from 80 µm to over 1,000 µm and formed in epoxies at any thickness. Most voids larger than 200 µm were clustered together near coarse aggregates. Liquid sealer may have displaced pockets of air when filling concrete surface voids; this air may have become entrapped before reaching the surface as the sealer gelled. Figure 4.13 shows an example of a void that formed in a Sealer D, which did not exhibit any cracking issues. In the case of Sealer C, these air voids in the sealer may have acted as stress concentrations in the sealer, and may be where the cracks originated.

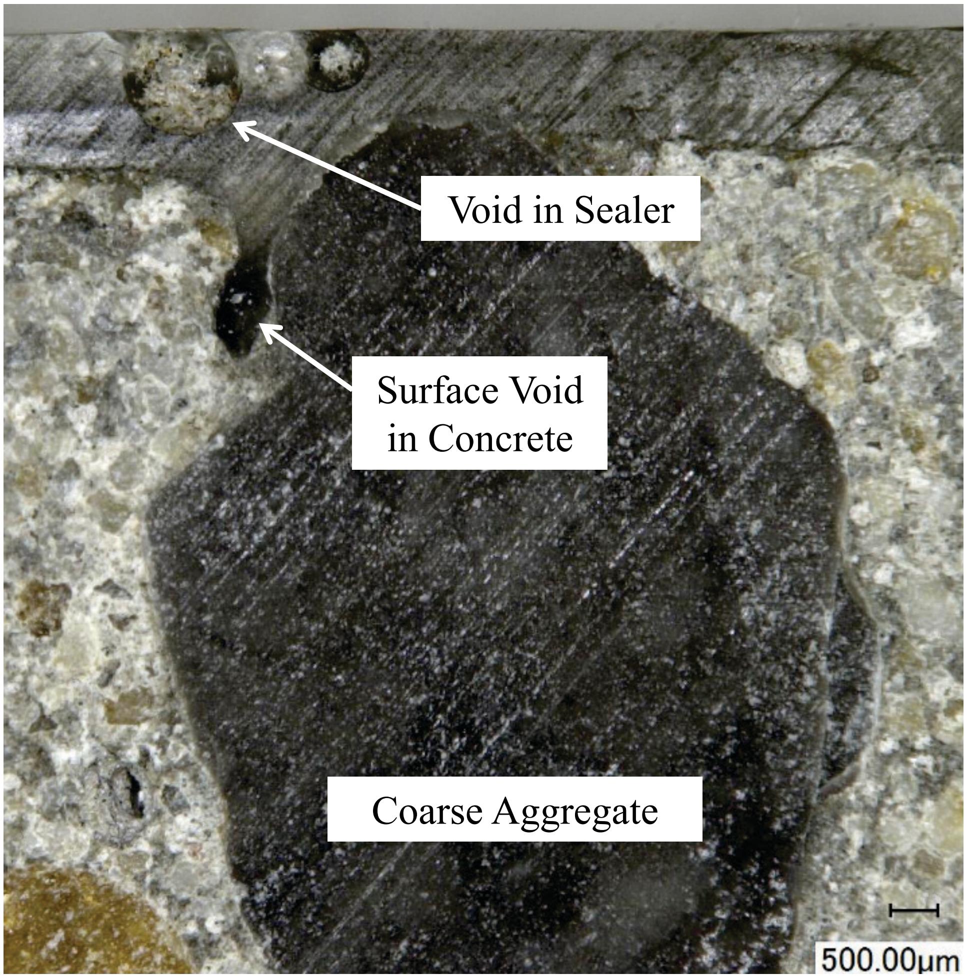


Figure 4.13 A MD2 specimen treated with Sealer D with an air void in the sealer

Another example of the cracking that developed in Sealer C is shown in Figure 4.14. This figure shows two cracks that developed in the sealer on a MD1 specimen while it was stored in the environmental chamber. One crack was traced back to air voids that developed in the sealer; this is shown in Figure 4.14a and magnified in Figure 4.14b. This area of the specimen was investigated because the sealer thickness was relatively less compared to other regions on the surface of this specimen that exhibited sealer cracking. Figure 4.14c is a three-dimensional image of the intersection of the crack and air void. This image was taken to observe the depth profile at the location where the crack and air void interacted under the assumption cracking started on the surface and moved through the sealer to the interface with the concrete substrate. This 3D profile shows a change in topography of the crack where it passes through an air void. The depth of the crack is approximately 550 μm. It is possible this crack could have initiated from the top surface of the sealer at the void and propagated down and through the sealer on the specimen. Cracking of the sealer on this specimen appears to have occurred preferentially in locations where the sealer thickness was greater than 500 μm; this was also noticed during observations of cracks on other specimens treated with Sealer C.

Figure 4.15 shows a MD 1 specimen with cracks in Sealer C; the specimen was stored inside the environmental chamber for the duration of testing and never exposed to oven drying. Figure 4.15a shows that this specimen had a collection of air voids that coalesced, and cracks that appear to have initiated at the voids. A closer look at this region in Figure 4.15b shows a crack that follows a U-shaped path through a tightly-grouped formation of air voids. This may have been the path of least resistance for the crack to follow being it did not propagate through some of the air voids spaced further apart. In Figure 4.15c, crazing can be seen at the tip of the crack along with a few microvoids directly in front of the crack tip. The crack initiation may have been triggered in part by stresses occurring near the microvoids and then propagated through the larger air voids.

Sealer C was the only sealer to crack of the products tested. All of the cracks appeared to originate from the surface and propagate down to the concrete in areas where the sealer thickness was greater than 500 μm, and the presence of air voids in the sealer may have been related to crack initiation. The cracks in the sealer were unrelated to concrete mixture design and exposure conditions following sealer application. The other three epoxy-based sealers were applied in a similar manner as Sealer C, subjected to the same environmental conditions, and also exhibited voids and regions of >500 μm thickness, so it is not clear why cracking was limited to Sealer C.

Further investigation may be able to confirm the cause, timing, and implications of cracking. It would be desirable to apply the sealer at several thicknesses and conduct continuous observations under a microscope to determine when, where, and how the cracks form. Further use of micro-XRF could be used to examine the cross section of specimens exposed to NaCl solution to determine if chloride ingress was locally accelerated at the cracks. This would considerably reduce the corrosion protection provided by the sealer.

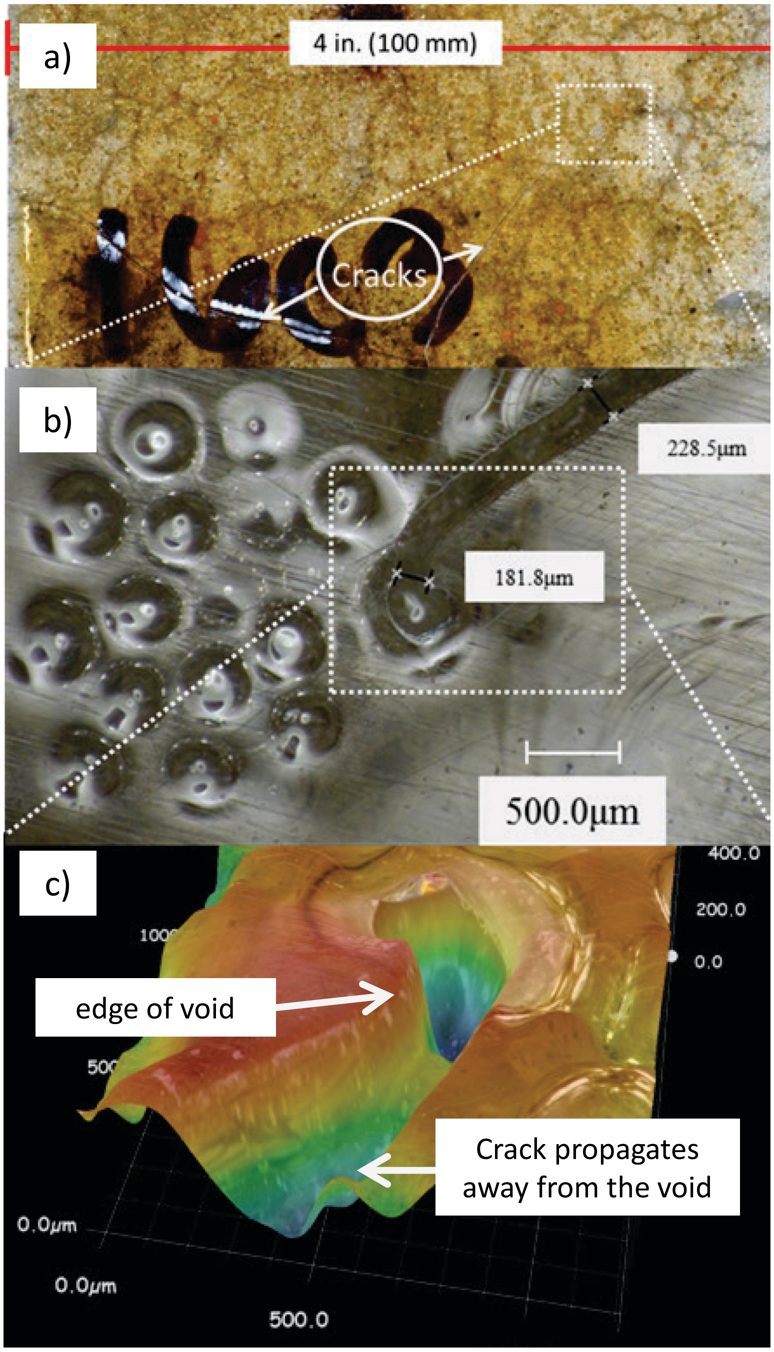


Figure 4.14 **(a) Cracks on the top surface of a MD1 specimen. This sample remained in the environmental chamber, and was not placed in an oven. (b) Area of interest of the intersection an air void and a crack propagating through the sealer, and (c) 3D profile of the air void and crack at 50x magnification**

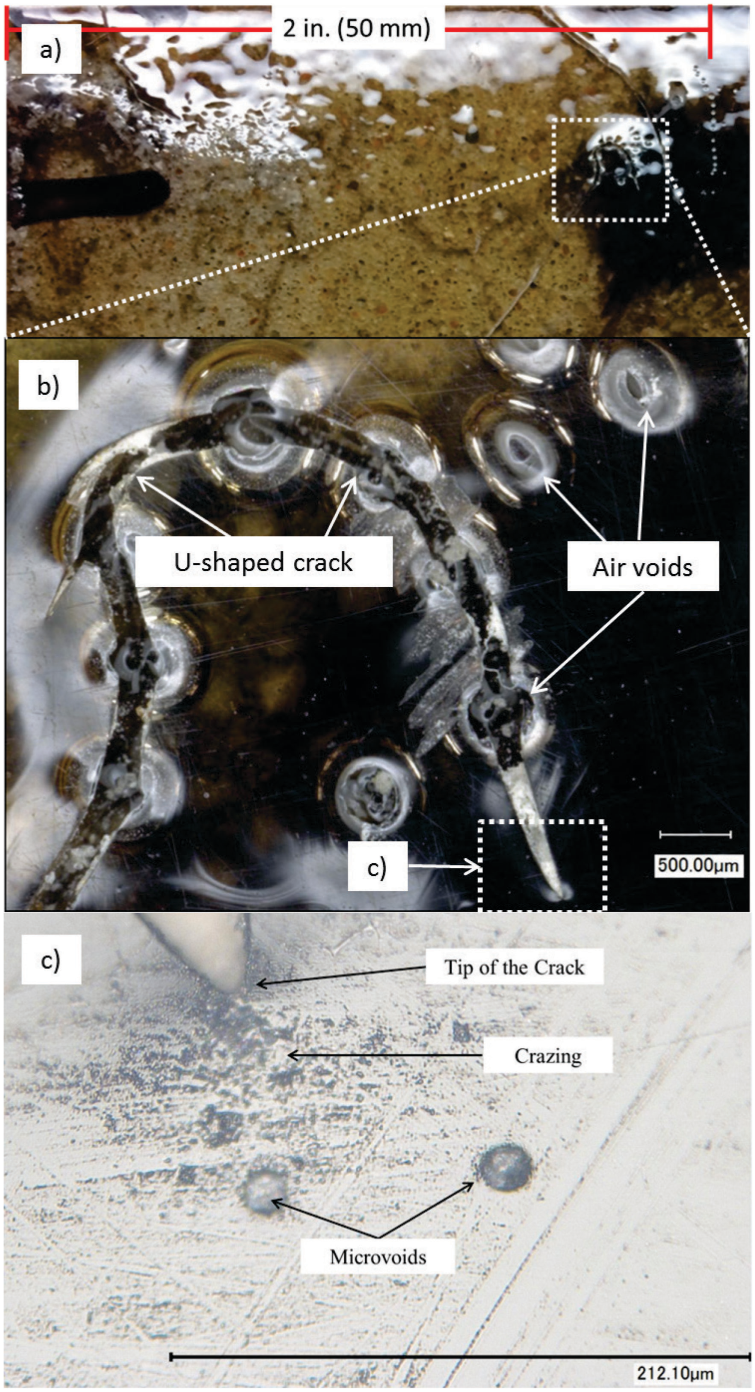


Figure 4.15 MD1 specimen treated with Sealer C that was not subjected to oven drying. (a) Cracks are seen at the center of the cube at the thickest layer of sealer. (b) “U” shaped coalescence of air voids that that developed in the sealer. (c) A crack tip and crazing, examined at 1000x magnification.

4.6 Overall Performance Evaluation

Each sealer received a quantitative overall score that depended on evaluation results from waterproofing performance and resistance to chloride penetration tests. The post-abrasion waterproofing performance was omitted from the scoring because the sealer was completely removed in the test preparation.

The waterproofing performance results at 21 days for specimens tested in the 15% NaCl solution and the waterproofing performance results at 5 days for specimens tested in water were averaged together. In each case, the waterproofing performance of the sealer was calculated as the percentage reduction in mass gained by sealed specimens relative to the unsealed control specimens. The averaged waterproofing performance results were weighted as 50% of the overall score for each sealer.

A weight of 50% was also given to the resistance to chloride penetration test results because chlorides have a significant influence on corrosion in reinforced concrete. The resistance to chloride penetration of a sealer was calculated as the percentage reduction in the mass of chlorides measured in sealed specimens relative to the mass of chlorides measured in the unsealed control specimens; chloride contents used in this calculation were the values measured by titration.

The average weighted score was calculated for each mix design by Equation 4-1:

$$\boldsymbol{AWS} = [\mathbf{0.5}(\boldsymbol{AWPP}) + \mathbf{0.5}\,(\boldsymbol{RCP})] \qquad \textbf{Eqn (4-1)}$$

Where:

AWS is the average weighted score

AWPP is the average of the waterproofing performance results

RCP is the resistance to chloride penetration results

Table 4.7 provides a summary of the results used to calculate the average weighed score *(AWS)* for each product used with each mix design. The results from each of the three mix designs were equally weighted. The *AWS* for the three mix designs were averaged to calculate the overall score. Table 4.8 presents the overall score for each product.

Table 4.7 The overall performance for each mix design used to determine the average for scoring the products.

		Alberta MOT	NCHRP Report 244 Series II		
		Weight of Test			
		25%	25%	50%	
Sealer Product	**Mix Design**	**5 Day Waterproofing Performance in Water (mass gain)**	**21 Day Waterproofing Performance in 15% NaCl (mass gain)**	**Resistance to Chloride Penetration in 15% NaCl (titration)**	**Average Weighted Score**
Sealer A	1	12	7	-37	-14
	2	1	5	13	8
	3	16	14	2	8
Sealer B	1	32	46	46	43
	2	76	71	80	77
	3	71	63	64	65
Sealer C	1	79	84	87	84
	2	88	77	90	86
	3	73	63	73	70
Sealer D	1	85	81	87	85
	2	76	81	87	83
	3	85	59	60	66
Sealer E	1	82	70	92	84
	2	79	82	92	86
	3	86	69	85	81

Table 4.8 Ranks and scores of products based on overall performance

Performance Rankings

Product	Score	Rank
Sealer E	84/100	1
Sealer C	80/100	2
Sealer D	78/100	3
Sealer B	61/100	4
Sealer A	1/100	5

4.7 Summary

The method of sealer application used in this research may need to be modified in future studies to meet recommended coverage rates for certain products, but it was generally effective for the epoxy sealers. The waterproofing performance test method proved to be effective for evaluating each product in both water and the 15% NaCl solution, but conducting these two tests in parallel is redundant. Because the 3-day measurements in 15% NaCl and the 5-day measurements taken in water provided similar results, a 5-day measurement can be taken for specimens soaking in the 15% NaCl solution to replace testing in a second set of specimens in water. The post-abrasion waterproofing performance test is not an effective evaluation for non-penetrating epoxy sealers or sealers that do not penetrate more than 1 mm (0.4 in.) into the concrete. XRF may be used as an alternative to titration for measuring chlorides because it takes less time to conduct and produces nearly identical results compared to titration.

Sealers C, D and E consistently performed better than Sealers A and B for every method of evaluation. Sealer E had the best overall performance and Sealer A performed the worst overall. The cracks in Sealer C appear to occur in the areas where the sealer thickness is over 500 μm thick. Despite the fact that Sealer C performed well in the tests conducted for this study, the cracks could reduce the field performance of this product by allowing localized chloride ingress. However, if a way to avoid cracking in Sealer C can be identified, it has the potential to be a very effective bridge deck sealer product, based on its performance in this study. Sealer A only penetrated approximately 100 μm into the concrete and none of the epoxy-based products penetrated into the concrete.

Chapter 5: Conclusions and Recommendations

A synthesis of the NCHRP Report 244 Series II and Alberta MOT BT Series test protocols was used to evaluate five bridge deck sealer products on 4.0-in. (100 mm) cube specimens fabricated from three different types of concrete. The three concrete mixtures used in this research were: 0.5 w/cm with 100% portland cement (Mix Design 1), 0.4 w/cm with 100% portland cement (Mix Design 2), 0.4 w/cm with Class F fly ash at a 20% replacement rate (Mix Design 3). The products were evaluated for waterproofing performance before and after abrasion by sandblasting and resistance to chloride penetration. The depth of sealer penetration and the sealer thickness were also determined by optical microscopy and SEM-EDS. This chapter provides conclusions for the overall performance of the products, recommendations for the testing protocols used for evaluation, and potential areas for future research.

5.1 Sealer Performance

The four epoxy-based products (Sealers B, C, D and E) performed better than the silicate-based sealer product (Sealer A) in all tests. Concluding remarks regarding each sealer product follow:

- Only Sealer C, Sealer D, and Sealer E had average overall performance scores over 75%; this was the minimum acceptable performance suggested in the NCHRP Report 244 Series II test protocol.
- Sealer E had the best overall performance. It outperformed other sealers in almost every test method on the three concrete mix designs. This product was very effective for preventing moisture and chloride ingress.
- Sealer C had the second-best overall performance. It performed similarly to Sealers D and Sealer E in almost every test, but the cracking observed on specimens treated with this product resulted in lower confidence in the sealer to provide adequate protection. These cracks appeared to be inherent and not influenced by an outside factor. The cause of, and potential for prevention of, cracks in Sealer C should be further investigated because of the positive performance it displayed in testing.
- Sealer D had the third-best overall performance, and provided the best protection for specimens made using Mix Design 1. This product was very similar to Sealer E, but it was not as effective as Sealer E for protecting specimens fabricated from Mix Design 3.
- Sealer B had the fourth-best overall performance. The other non-penetrating sealer products provided significantly better protection against moisture and chloride ingress than Sealer B.
- Sealer A had the lowest overall performance. In the tests conducted in this research, it did not perform as well as the other sealers by a large margin. Sealer A was not applied at the coverage rate recommended by the manufacturer, and therefore may have been at a

disadvantage to the other products. However, the method of application used was taken from the BT Series tests and targeted towards testing penetrating sealers such as Sealer A.

- The results of a petrographic investigation suggest that Sealer A did not penetrate more than 100 μm into the concrete. This limited penetration depth was also observed even for a specimen immersed in Sealer A for a period of one week. Vehicle traffic could easily be expected to wear off the thin layer of sealer-penetrated concrete, further limiting its effectiveness.

5.2 Evaluation of Test Methods for Sealer Performance

The testing procedures were evaluated for simplicity (or complexity), repeatability, and time to completion. As noted in Section 2.2, products categorized a *penetrating* sealer or *non-penetrating* sealer offer protection by different mechanisms. The methodology followed in this research evaluated all products using the same procedures as an attempt to provide an equal foundation for an effective evaluation for both sealer classifications. Modifications to some procedures are suggested and other procedures followed in this research are no longer recommended for use. The following sections highlight key observations from the research conducted and will be incorporated in a draft procedure to be provided to ALDOT in the final report for this project.

5.2.1 Useful Test Methods and Procedures

1. The use of multiple mix designs that represent a range of concretes that a sealer may be applied to should be retained since some variations in results were observed between the three mix designs in testing. This could indicate a certain sealer may be more effective in providing protection for a certain type of concrete and not as well for another mixture type.

2. Cube specimens are not representative of a bridge deck as a whole, but many samples can be fabricated for effective evaluation of sealer product's overall performance. Cubes are easy to handle, they do not require a large area for testing, and they can be stored efficiently.

3. Specimen preparation and conditioning procedures detailed in sections 3.4.2 through 3.5.2 of this report were an effective means of measuring individual test specimen moisture levels, ensuring similar moisture contents for all specimens prior to sealer application, and testing for waterproofing performance and resistance to chloride ingress. This process of preparation and conditioning is recommended for future sealer evaluations.

4. The 15-day drying period detailed in section 3.5.3 of this report for determining the vapor transmission (VT) value is recommended for future use. It is primarily used for testing silanes or other sealers that are intended to provide a vapor-permeable coating, but it can be used for non-penetrating epoxy products as well. This is a necessary conditioning step,

and determines the amount of water vapor transmitted through the sealer. This will indicate if a sealer will allow a concrete bridge deck to dry out.

5. Adjusting the relative moisture content (RMC) of the sealed cubes to that of the controls is recommended to ensure a baseline relative moisture content to evaluate sealer products.

6. The 21-day soaking period in the 15% NaCl solution was an effective exposure period for evaluating resistance to chloride penetration. However, it is not necessary to weigh the specimens every three days. Only two mass measurements should be recorded for mass gain after the start of the exposure period: a measurement at 5 days to replace the waterproofing performance in water, and measurement at 21 days to determine the overall mass gain should be sufficient. A separate waterproofing performance test involving a 5-day immersion in water is not necessary. Using a single waterproofing performance test also allows for a reduction in the number of samples required to evaluate a sealer.

7. XRF of bulk powder samples provided similar results as titration for determining the chloride content of the concrete. However, XRF required less sample preparation and proved to be a faster method of measuring chlorides. XRF is recommended over the much slower titration process.

5.2.2 Suggested Modifications and Ineffective Methods

1. Testing any more than three products simultaneously may become too demanding for labor and time. Especially at the time of sealer application, meticulous handling of the specimens is needed to assure the sealer product is applied consistently and uniformly. It is highly recommended that only two to three products are tested together.

2. Control specimens from MD3 (20% fly ash) generally absorbed more water and chlorides than their counterparts from MD2 (0.40 w/cm, 100% OPC). This suggests that the MD3 specimens may require further curing in order to have permeability characteristics representative of field concrete containing fly ash. To avoid further prolonging the sealer evaluation process, this mixture should be eliminated, and leaving only 0.40 w/cm and 0.50 w/cm concrete with 100% OPC binder.

3. A major improvement in sealer evaluation would start with altering the sealer application method based on the product classification to ensure it is applied at the manufacturers' recommended coverage rate. A proposed solution uses the category of surface treatment to dictate the method of application:

    a. *Penetrating* sealers (silanes, siloxanes, etc.) should be applied by full immersion until the recommended application rate is reached; this may require multiple immersions.

   b. *Non-penetrating* sealers (epoxies) should be limited to one immersion and then finished with a brush or roller. If a second sealer application is needed to reach the recommended rate, only a brush or roller should be permitted to avoid exceeding the recommended coverage rate. This procedure would be more representative of the method by which the sealer is applied on a bridge deck, and it would ensure that all specimens reach the coverage rate recommended by the manufacturer.

4. The 5-day initial waterproofing performance test is a quick and effective procedure for determining the ability of a product to resist moisture ingress, but this same test can be accomplished using the 15% NaCl solution. Using the 5-day waterproofing performance in the 15% NaCl solution will minimize the amount of samples fabricated.

5. Testing post-abrasion waterproofing performance by sandblasting according to Alberta MOT procedures is ineffective for evaluating non-penetrating (epoxy) sealers because the protective layer of sealer is completely removed. These parameters are meant for *penetrating* sealers only. This method should not be considered for use unless it is further tested on sealer products confirmed to penetrate further than 1 mm (0.4 in).

6. The dye method to determine sealer penetration depth was not effective in this research; however, none of the sealer products tested penetrated the concrete enough for an effective evaluation of the technique. It may be more effective for sealers that are able to penetrate more deeply into the concrete substrate.

5.3 Future Areas for Research

During the course of the research, several opportunities for future research were noted. Some pertain to improving techniques for evaluation, and others pertain to investigating sources of potential premature sealer failure.

1. The cracks in Sealer C should be investigated further because of the positive waterproofing performance it displayed in testing. A controlled study involving multiple film thicknesses and time-lapse observation under a microscope is recommended.

2. Evaluate the waterproofing performance and chloride resistance of epoxy sealers after skid-resistant sand is broadcasted on the freshly-applied sealer surface. The discontinuities from the sand in the sealer's surface may affect the moisture resistance properties leading to inadequate field performance.

3. Develop a useful testing method that can accurately quantify the durability of sealers; this is targeted mainly for non-penetrating products. Two proposed methods to improve the durability testing of sealers are:

   a. Epoxy-based non-penetrating sealers are significantly more resistant to sandblasting than unsealed concrete. Rather than requiring that a certain amount material be removed by sandblasting, they should be prepared for post-abrasion testing by sandblasting (no grinding of the sealer) each side of sealed specimens for a fixed amount of time. This would be the amount of time needed to remove a specified amount of material from one side of an unsealed control specimen.

   b. Similar to the NCHPR Report 244 Series IV tests, the research on sealer durability should extend beyond evaluating simulated traffic wear by abrasion. It would be valuable to assess qualities such as resistance to ultraviolent light exposure, extended chloride exposure, and even impact resistance. This may require a change from the 4-in. (100-mm) cube specimens to fabricating small slabs or beams. This testing should only be applied to products that exhibit passing performances in the recommended methods described in this report.

4. Evaluate the use of a dual treatment system of a silane (or other penetrating sealer) used as secondary form of protection under an epoxy-based sealer. This could provide the best solution for high-risk areas (such as bridges near the Gulf coast) that experience higher chloride exposure.

References

ACI Committee 201. *ACI 201.2R-08: Guide to Durable Concrete.* Farmington Hills, MI: American Concrete Institute, 2008.

ACI Committee 222. *ACI 222R-01: Protection of Metals in Concrete Against Corrosion.* Farmington Hills, MI: American Concrete Institute, 2010.

Alberta Ministry of Transportation. *B388 Specifications for Concrete Sealers.* Alberta Transportation Technical Standards Branch, 2010.

Alberta Ministry of Transportation. *BT001 Test Procedure for Measuring the Vapour Transmission, Waterproofing and Hiding Power of Concrete Sealers.* Alberta Transportation Technical Standards Branch, 2000a.

Alberta Ministry of Transportation. *BT010 Test Procedure for Casting and Storing Concrete Test Specimens for Use in Approval Testing of Sealers.* Alberta Transportation Technical Standards Branch, 2000b.

ALDOT. *Standard Specifications for Highway Construction.* Montgomery, AL, 1964.

ALDOT. *Standard Specifications for Highway Construction.* Montgomery AL, 2012.

ASCE. *2013 Report Card for America's Infrastructure.* 2013. http://www.infrastructurereportcard.org/alabama/alabama-overview/ (accessed 2015).

ASTM C114-11b. "Standard Test Methods for Chemical Analysis of Hydraulic Cement." West Conshohocken, PA: ASTM International, 2013.

ASTM C1152 / C1152M-04. *Standard Test Method for Acid-Soluble Chloride in Mortar and Concrete.* West Conshohocken, PA: ASTM International, 2012.

Basheer, P. A. M., and A. E. Long. "Protective Qualities of Surface Treatments for Concrete." *Structures and Buildings* 122, no. 3 (1997): 339-346.

Bottenberg, R. "Cathodic Protection of Historic Bridges." *Concrete International* 30, no. 9 (2008): 37-41.

Broomfield, J. P. *Corrosion of Steel in Concrete: Understanding, Investigating and Repair.* 2. New York, New York: Taylor and Francis, 2007.

Broton, D., and J. I. Bhatty. "Analytical Techniques in Cement Materials Characterization." In *Innovations in Portland Cement Manufacturing*. Portland Cement Assn; 2nd edition, 2011.

Dempere, L. A, B. Willenberg, and R. Deist. *Use of Scanning Electron Microscopy and Microanalysis to Determine Chloride Content of Concrete and Raw Materials.* Gainsville: FDOT, 2013.

Garboczi, E. J., D. P Bentz, J. M. Davis, and Y. Lu. "Modeling Chloride Transport in Cracked Concrete: A 3-D image-based microstructure simulation." *COMSOL Conference Boston 2012.* Boston, MA, 2012. 1-15.

Korkmaz, D.; *Precipitation Titration of Chloride by the Mohr Method.* Retreived from http://academic.brooklyn.cuny.edu. n.d.

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

Pincheira, J. A., and M. A. Dorsorst. "Evaluation of Concrete Deck and Crack Sealers." Wisconsin Highway Research Program #0092-03-09, Wisconsin Department of Transportation, University of Wisconsin-Madison, Madison, WI, 2005.

Proverbio, E., and F. Carassiti. "Evaluation of Chloride Content in Concrete by X-Ray Fluorescence." *Cement and Concrete Research* 27, no. 8 (1997): 1213 - 1223.

Rothstein, D. "Petrographic Examination of Labcrete Samples Treated with Different Sealers." Report No. DRP14.1251, Boulder, CO: DRP Consulting, Inc., 2015. Unpublished.

Sarkar, S. L., Xu Aimin, and D. Jana. "Scanning Electron Microscopy, X-Ray Microanalysis of Concretes." In *Handbook of Analytical Techniques in Concrete Science and Technology*, by V. S. Ramachandran, 231-241. Park Ridge, New Jersey, U.S.A.: NOYES PUBLICATIONS, 2001.

Sudbrink, B., T. Ley, and J. M. Davis. "Applications of Micro X-Ray Fluorescence in Concrete." *Microscopy Microanalysis* Vol. 18, no. S2 (2012): 952-953.

Appendix A: Titration Procedure

The unknown chloride content in concrete can be determined through potentiometric titration by silver nitrate ($AgNO_3$) per ASTM C1152/C1152M *Standard Test Method for Acid-Soluble Chloride in Mortar and Concrete*. This standardized test is derived from ASTM C114-11b *Standard Test Methods for Chemical Analysis of Hydraulic Cement* and refers to the better-detailed C114-11b method often throughout the process. ASTM C14-11b is referred below because of elaboration on some steps, and both standards follow the some process (ASTM C1152 / C1152M-04 2012).

A.1 Potentiometer and Electrode Selection

This method requires the use of a *potentiometer* with a millivolt (mV) scale capable of a 1 mV precision or better, and a *chloride, silver/sulfide ion selective electrode.* There are two main categories of these electrodes: silver/sulfide selective electrode and a silver billet electrode. Both may be used as a combination electrode (silver chloride and potassium chloride are included in the electrode chambers), or a half-cell electrode which requires a separate reference electrode (contains the silver chloride and potassium chloride). Ion selective electrodes have an epoxy body with a silver crystalline membrane where the silver billet is coated with potassium chloride membrane. These two differ by the physical body but operate under the same principle; they measure the electrochemical reaction of chloride ions through the outer membrane silver against the potential of known silver chloride within the electrode (combination) or in the reference electrode (half-cell). For this research, *Thermo Scientific's Orion Half-Cell Silver/Sulfide Selective Ion Electrode* with a *Thermo Scientific Orion Double Junction Reference Electrode* were used. The double junction reference electrode required two filling solutions, potassium chloride and silver chloride. Most electrodes are compatible with pH or ion selective electrode meters capable of millivolt (mV) readings. A *Mettler Toledo FE20 FiveEasy^{TM} pH Meter* served as the potentiometer used in this research.

A.2 Procedure

To obtain chloride concentrations, four procedural steps were conducted: preparation of chemical reagents, sample preparation, titration, and calculations.

A.2.1 Chemical Reagents

All of the stock chemicals used were converted into standard solutions specified per ASTM C114-11b. All water used was deionized (ultrapure 18 MΩ resistivity) water. A 0.05 N NaCl standard solution was prepared by diluting 2.9222 g of oven-dried NaCl with exactly 1 L of water in a volumetric flask. The 0.05N $AgNO_3$ standard solution was prepared by diluting 8.4938 g of $AgNO_3$ with exactly 1 L of water in a volumetric flask. The exact normality was determined by titrating[2] 5.00 mL of 0.05N NaCl diluted in 150 mL of water against the $AgNO_3$ solution, and calculated by Equation A-1:

$$N = \frac{0.25}{V} \qquad \textbf{(Eqn A-1)}$$

Where:

N = exact normality of silver nitrate

0.25 = milliequivalents NaCl (5.0 mL x 0.05N)

V = volume of silver nitrate used

Stock 70% nitric acid was diluted with water to a 50% w/w solution. The indicator solution was made by the following proportions: 2.0 grams of methyl red per 1 L of 95% ethanol.

A.2.2 Sample Preparation

Prior to titration, the concrete powder was digested in acid and filtered to extract the chloride ions retained. Also, a blank[3] sample was made for each titration. Each sample (and blank) was prepared for titration as follows:

- 5.0 g of concrete powder was weighed out into a 250 mL beaker and combined with 75 mL of water. This was stirred thoroughly to break up any clusters of powder for 20 to 30 seconds. Blank samples did not contain any concrete powder, only 75 mL of water.
- 25 mL of 50% nitric acid was slowly added while stirring. Approximately one minute later, 3 to 4 drops of methyl red is added, stirred again for 10 to 15 seconds.
- The beaker was then covered with a watch glass and allowed to sit for 2 minutes. An opaque, light-pink color persisted above the solids in the solution which indicated the solution was sufficiently acidic.
- The beaker was placed on a hot plate set at 300 °C (570 °F) and brought to a rapid boil. It took approximately 20 to 40 minutes for a sample to reach a boil. A watch glass was used to cover the beaker to prevent chlorides from evaporating.
- The covered beaker was removed and allowed to cool for 15 to 20 minutes; this time was taken to prepare the filtration station.
- Figure A.1 presents the filtration setup. A Buchner funnel and 250 mL filtration flask were prepared for use. A No. 42 filter paper was placed in the funnel and dampened with water prior engaging the vacuum and filtering the sample solution. The solution was slowly poured

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[2] This titration procedure is detailed following the sample preparation procedure.

[3] Reasons for the blank are explained in the calculation procedure.

in the funnel and allowed to filter before rinsing any remaining residue. The beaker, watch glass, and funnel were rinsed free of residue with no more than a total of 100 mL of water. Blank samples were not filtered, but an additional 100 mL of water was added. This process took 15 to 20 minutes to complete per sample.

- The filtrate was transferred to a 250 mL beaker, covered, and allowed to cool to room temperature before proceeding to titration. The filtrate took 1 to 2 hours to cool before the solution could be tested further.

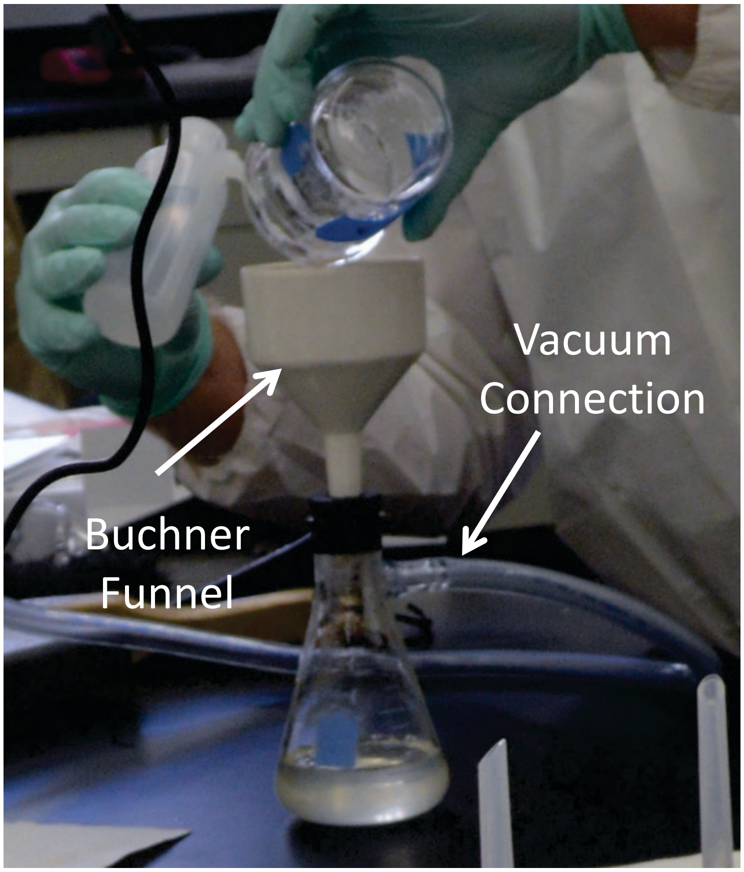


Figure A.1 Sample solution filtration setup.

A.2.3 Titration

Figure A.2 shows the orientation of the burette relative to the two electrodes in the beaker. Electrodes were connected to the potentiometer and were placed in water to obtain a neutral reading for the voltage potential (mV reading) corresponding to an equivalence point; this same mV reading was used to indicate the value of the approaching equivalence point during a titration. The equivalence point represents when $[Ag^+]$ equals the amount of $[Cl^-]$ in the solution, and therefore the silver has fully reacted with the chlorides. A 50 mL burette was filled with 0.05N $AgNO_3$. The beaker containing the filtrate sample was then placed onto a magnetic stirrer and a TFE-fluorocarbon-coated stir bar was added for a consistent agitation of the solution while titrant was added. The tip of the electrodes and burette were lowered no more than 1 in. (25.4 mm) into the solution, and the initial mV reading was recorded.

$AgNO_3$ titrant was added in 0.20 mL increments. The volume of each increment was recorded with the corresponding change in mV reading. Millivolt readings took 5 to 10 seconds to stabilize after an increment of $AgNO_3$ was added. The mV readings increased as the equivalence point was approached with each successive 0.20 mL addition of $AgNO_3$, and then the rate of changes in mV readings then decreased noticeably once the equivalence point was passed. After this change was observed, three additional increments of titrant were added to solution as specified in ASTM C114-11b. Figure A.3 shows the solution before and after the titration was complete; precipitate formation of AgCl can be seen in the solution.

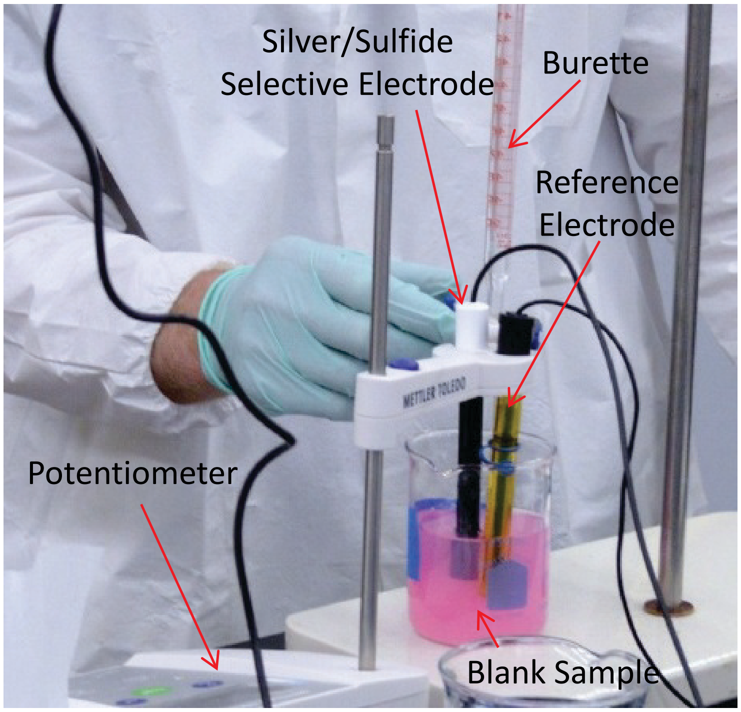


Figure A.2 **The configuration for the silver/sulfide selective electrode, reference electrode, and the burette for potentiometric titration of a blank sample.**

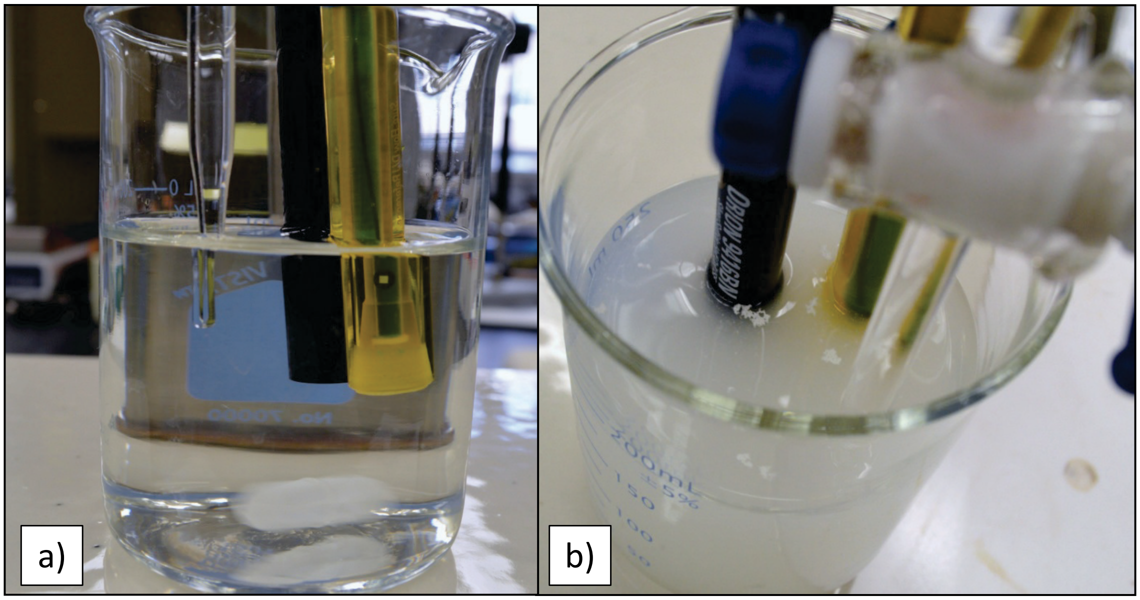


Figure A.3 The burette and electrodes orientation in a sample (a) prior to 0.05N $AgNO_3$ additions and (b) after reaching the equivalence point signified by AgCl precipitate formation.

A.2.4 Calculation

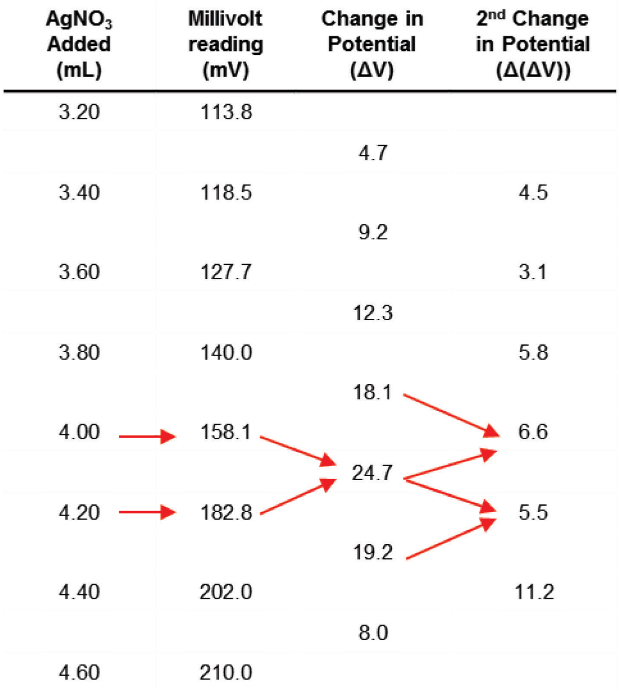
Samples with low chloride concentrations may not be accurately measured due to large changes in mV readings. To compensate, specimens with lower expected chloride contents had 2.0 mL of 0.05N NaCl pipetted into the solution before titration; this addition of NaCl was accounted for by subtracting the volume of $AgNO_3$ used to titrate the blank sample which also had 2.0 mL of 0.05N NaCl included. Specimens that were expected to have higher concentrations of chlorides did not need the additional standardized NaCl solution. The volume of $AgNO_3$ used to reach the equivalence point determines the amount of chlorides in the filtrate, and can be used to calculate the chloride concentration by mass of the original concrete powder sample.

Appendix XI in ASTM C114-11b provides an example of the calculation for the equivalence point by using the differentials in potential (mV readings) to interpolate an approximate value. This was determined by plotting two columns from the recorded volumes of sequential $AgNO_3$ increments and corresponding mV readings. A third column, the 1st differential, was calculated showing the difference of subsequent mV readings. In the 4th column, the calculated 2nd differential is the absolute value of the difference of successive 1st differentials. The 1st differential will increase as it approaches the equivalence point, and will decrease once it has passed the equivalence point. The successive 2nd differentials, corresponding to the maximum 1st differential, will be used to interpolate the volume of $AgNO_3$ used for the equivalence point.

Table A.1 provides example data in the four-column format. Figure A.4 represents this concept graphically where the potential and differentials are a plotted against the amount of $AgNO_3$ used. Using Table A.1 and referring to ASTM C114-11b (2013), the equivalence point *(E)* is calculated using Equation A-2:

$$\boldsymbol{E = 4.0 + \left[\frac{6.6}{(6.6-5.5)}\right] \times 0.2 = 4.11\ mL} \qquad \textbf{(Eqn A-2)}$$

Table A.1: Four-column table used for calculating the equivalence point for a sample.



$AgNO_3$ Added (mL)	Millivolt reading (mV)	Change in Potential (ΔV)	2nd Change in Potential (Δ(ΔV))
3.20	113.8		
		4.7	
3.40	118.5		4.5
		9.2	
3.60	127.7		3.1
		12.3	
3.80	140.0		5.8
		18.1	
4.00	158.1		6.6
		24.7	
4.20	182.8		5.5
		19.2	
4.40	202.0		11.2
		8.0	
4.60	210.0		

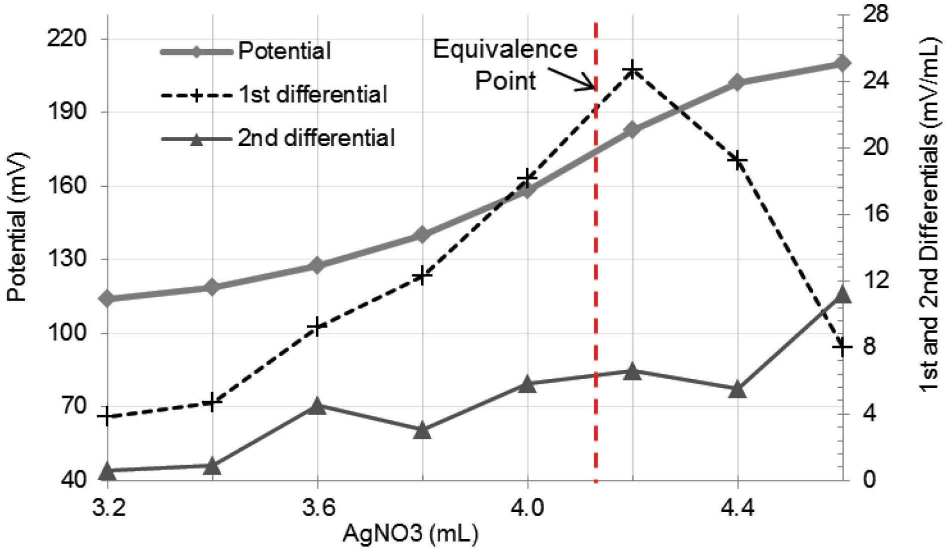


Figure A.4 Graphical representation of the determination of the equivalence point located between 4.0 and 4.2 mL of $AgNO_3$ using the values of the 2^{nd} differential corresponding to the greatest change in slope of the potential (the largest 1^{st} differential).

Once the equivalence point was calculated for a sample, the chloride content by mass of concrete is calculated by Equation A-3:

$$Cl, \% = 3.545 \frac{[(V_1 - V_2) \times N]}{W} \qquad \textbf{(Eqn A-3)}$$

Where:

V_1 = mL of 0.05N $AgNO_3$ titrated to reach the equivalence point for the sample

V_2 = mL of 0.05N $AgNO_3$ titrated to reach the equivalence point for the blank sample

N = calculated exact normality of $AgNO_3$

W = mass of sample (g)

Appendix B: Recommended Sealer Evaluation Protocol

The objective of this test protocol is to evaluate bridge deck sealers for resistance to moisture and chloride ingress. It is strongly recommended that a previously approved product be included as a reference because concrete properties may vary slightly from one test to the next.

Fabrication, Conditioning, and Sealer Application

B.1 Specimen Fabrication

Fabricate cube specimens using two concrete mixture designs (0.40 and 0.50 w/cm) containing only ASTM C150 Type I, II, or I/II portland cement as the binder. For each mixture design, fabricate six control specimens plus an additional six specimens per sealer. (If three sealer products are to be evaluated, this would require 24 test specimens).

The 0.40 w/cm concrete mixture shall meet the requirements of Class B – Bridge Superstructure Concrete in the ALDOT Construction Specifications and contain 630 lb/yd^3 (374 kg/m^3) of cement. The 0.50 w/cm concrete shall contain 600 lb/yd^3 (357 kg/m^3) of cement, and the proportions in Table 3.6 of this report may be used as a guide in proportioning the mixture.

Concrete shall be mixed in accordance with ASTM C192 / AASHTO R39. The mixer capacity shall be sufficient to produce concrete for the full set of test specimens, plus quality control tests. Slump, unit weight, and air content of the mixture shall be measured and reported in accordance with ASTM C138 / AASHTO T121, ASTM C143 / AASHTO T119, and ASTM C231 / AASHTO T152. Several trial batches may be necessary to produce concrete with the required air content.

The cube specimens shall be 4.0 in. (100 mm) on a side and cast in molds in two layers. Each layer shall be rodded and vibrated to aid consolidation of the concrete. No form oil or other lubrication shall be used on the molds because this may interfere with water absorption during the testing process. Instead, mold pieces may be shrink-wrapped to aid in demolding the specimens.

Cube specimens shall be screeded and hand-troweled in accordance with good concrete finishing practice.

Three 6 x 12 in. (150 x 300 mm) cylinders shall be cast for quality control and tested for compressive strength at 28 days per ASTM C39 / AASHTO T22.

B.2 Curing Methods

After initial set of the concrete, cover the specimens in wet burlap or plastic and cure in molds in a room with an ambient temperature of 73 ± 2 °F (23 ± 1 °C). This may take place in a mixing room meeting AASHTO M 201 or ASTM C 511.

Demold specimens at 24 ± 0.5 hrs after casting. Upon demolding, number each specimen for identification and mark all specimens in the center of the finished top surface with permanent marker to identify the orientation of cubes during testing.

Seal the specimens in heavy duty one gallon (3.8 L) freezer bags and place them in a moist curing chamber until an age of 42 days after casting with the finished surface facing up. The curing chamber shall be set at 73 ± 2 °F (23 ± 1 °C) and >98% relative humidity in accordance with AASHTO M 201 / ASTM C 511.

B.3 Surface Preparation

After 42 days of curing, dry all specimens in open air for 24 hours in preparation for sandblasting. All six sides of each specimen shall be exposed to the air (that is, do not place one side on an impermeable surface such as a plastic sheet).

After 24 hours of drying, lightly sandblast each specimen to expose the internal pore structure of the concrete using abrasive silica sand.

Weigh each cube before and after sandblasting each face. Remove 4.0 ± 0.2 g of concrete from the top surface. Use this abraded surface as a reference for sandblasting the other 5 sides. Do not remove more than 4.0 ± 0.2 g from each face.

Reapply the numbers and center markings to identify the top (finished) surface of the test cube. Note the three specimens with the largest surface voids. These specimens will be used for total moisture content (TMC) determination.

Return all specimens to the moist curing room for storage after all specimens have been abraded and identification markings restored.

B.3 Storage

Specimens shall be stored in the moist room for 24 hours to return them to a fully-saturated state (100% relative moisture content). Do not bag specimens for this step; bags will block moisture and prevent re-saturation of the specimens.

B.4 Conditioning: Phase I

Specimens shall be conditioned to a target relative moisture content (RMC) prior to sealer application. The drying requirements to the target moisture content are dependent on the concrete being represented for testing. The target RMC:

- 70% RMC – 0.50 w/cm concrete.
- 80% RMC – 0.40 w/cm concrete.

B.4.1 TMC Determination

For each mixture design, three control specimens shall be used to determine the total moisture content of the specimens in the fully-saturated state.

Remove the specimens from the moist room, dry the specimens with a towel to a saturated surface-dry condition, and immediately weigh each specimen to the nearest 0.1 g to determine the initial mass, X_0.

Transfer the specimens to a 230 °F (110 °C) oven for seven days of drying to remove all moisture. After seven days, weigh each specimen again to determine the final mass, X_7, and calculate the TMC of each cube using Equation B-1.

$$\boldsymbol{TMC}\ (\mathrm{g}) = \boldsymbol{X_0} - \boldsymbol{X_7} \qquad \textbf{(Eqn B-1)}$$

The average TMC of the three control specimens shall be considered representative of the remaining test specimens.

Reserve the three TMC specimens for determining baseline chloride content (B.8.2).

B.4.2 Controlled Dry to Target RMC

Remove the remaining specimens from the moist room, dry the specimens with a towel to a saturated surface-dry condition, and immediately weigh each specimen to the nearest 0.1 g to determine the initial mass, X_0.

Transfer the specimens to an environmental chamber for controlled drying at conditions of 74 °F ± 3 °F (23.3 °C ± 2.0 °C) and 50 ± 4% relative humidity.

Weigh each specimen daily to the nearest 0.1 g, (X_i), and rotate among the shelves to minimize variations caused by air flow within the chamber.

The greatest mass will be lost in the first day (typically about 10 g). Mass lost after three days is usually around 1 to 2 g per day. Mass loss after seven days is typically about 0.5 to 1.0 g per day.

Use Equation B-2 to calculate the RMC of each cube after each daily measurement.

$$\boldsymbol{RMC}\ (\%) = \frac{[\boldsymbol{TMC} - (\boldsymbol{X_0} - \boldsymbol{X_i})]}{\boldsymbol{TMC}} \times \mathbf{100} \qquad \textbf{(Eqn B-2)}$$

Note: The RMC cannot be calculated until the TMC is calculated after seven days of drying, but the specimens are unlikely to reach their target RMC before this time. At seven days, calculate the RMC for the previous six days.

For each mixture design, calculate the average RMC of all specimens. Controlled drying shall end when the average RMC has reached the target RMC of 70% or 80%. Seal specimens in plastic freezer bags to prevent further changes in moisture content prior to sealer application.

B.4.3 Cube Utilization

Select cubes for moisture and chloride resistance testing based on a visual assessment of surface voids and the variance from the average RMC of the full set of specimens. Cubes that have the least variance from the average RMC and a minimum of surface voids shall be used for these tests, while the remaining cubes shall be used for sealer penetration depth and thickness determination.

B.5 Sealer Application

Prepare each sealer for application following the manufacturers' instructions and apply according to the manufacturer's recommended coverage rates, and following the appropriate instructions below. For each mixture design, six specimens shall receive the sealer treatment. Sealer application shall take place as soon as practicable after the specimens reach their target RMC.

Note: The pot life of epoxy-based sealers may be considerably shortened from that described in the product literature. Monitor sealer temperature and discard if it begins to rise rapidly.

B.5.1 Penetrating (Non-Epoxy) Sealers

Remove specimens from the freezer bags and spray all specimens with compressed air to remove any dust or particulates and then weigh them (T_0) immediately before immersing the specimens in the sealer.

Place three equally-spaced cubes top surface up on a wire tray and lower them into a container of sealer until completely immersed, as shown in Figure 3.9. The depth of sealer should be at least 0.5 in (12.7mm) above the submerged top surface of the three concrete cubes.

After two minutes of immersion in the sealer, remove the set from the container of sealer and place them on a wire rack until the sealer ceases to drip. Record the mass of the specimens after the sealer ceases to drip (T_1).

Weigh the specimens again immediately before the second immersion (T_2) and repeat the immersion process.

After two minutes have passed, remove the specimens and allow them to drip dry on a rack.

Once the sealer ceases to drip after the second immersion, weigh the specimens again and record the mass (T_3). Calculate the mass of fresh sealer applied to each specimen using Equation B-3.

$$\boldsymbol{SF} = (\boldsymbol{T_1} - \boldsymbol{T_0}) + (\boldsymbol{T_3} - \boldsymbol{T_2}) \qquad \textbf{(Eqn B-3)}$$

If the mass of fresh sealer indicates that the sealer has been applied within manufacturer's recommended range of coverage rates, return the sealed cubes to the environmental chamber for a second round of controlled drying.

However, if insufficient sealer has been applied, additional immersions may be required. Repeat the steps above as necessary until the sealer application rate falls within the range recommended by the manufacturer.

B.5.2 Epoxy (Non-penetrating) Sealers

Remove specimens from the freezer bags and spray all specimens with compressed air to remove any dust or particulates and then weigh them (T_0) immediately before immersing the specimens in the sealer.

Place three equally spaced cubes top surface up on a wire tray and lower them into the container of sealer until completely immersed, as shown in Figure 3.9. The depth of sealer should be at least 0.5 in (12.7mm) above the submerged top surface of the three concrete cubes.

After two minutes of immersion in the sealer, remove the set from the container of sealer and place them on a wire rack until the sealer ceases to drip. The epoxy-based sealers will pond on the top surface of the cubes. Remove this extra sealer with a brush or roller. Record the mass of the specimens after the sealer ceases to drip (T_1).

Weigh the specimens again immediately before the second application (T_2) and apply the second layer of sealer using a brush or roller.

Once the sealer ceases to drip after the second application, weigh the specimens again and record the mass (T_3). Calculate the mass of fresh sealer applied to each specimen using Equation B-3.

$$\boldsymbol{SF} = (\boldsymbol{T_1} - \boldsymbol{T_0}) + (\boldsymbol{T_3} - \boldsymbol{T_2}) \qquad \textbf{(Eqn B-3)}$$

If the mass of fresh sealer indicates that the sealer has been applied within manufacturer's recommended range of coverage rates, return the sealed cubes to the environmental chamber for a second round of controlled drying.

However, if insufficient sealer has been applied, repeat the steps for the second application as necessary until the sealer application rate falls within the range recommended by the manufacturer.

B.6 Conditioning: Phase II

Cubes are to be conditioned for a second time in preparation for testing resistance to moisture and chloride ingress. During this second conditioning phase, the vapor transmission value, sealer loss, and mass of dry sealer are determined. The sealed cubes will also be adjusted to the same RMC of the unsealed controls before testing.

This step is not required for the sealed specimens that will be used for depth of penetration and thickness determination.

B.6.1 Controlled Dry for 15 Days

The environmental chamber should remain at conditions of 74 °F ± 3 °F (23.3 °C ± 2.0 °C) and 50 ± 4% relative humidity. Rotate every specimen between shelves daily to minimize variations caused by airflow in the chamber. Upon returning the specimens back in the environmental chamber, record the mass of the sealed cubes (T_0), and the untreated controls (C_0).

Record the mass of each specimen after five days *(C_5 – for untreated controls and T_5 – for corresponding treated specimens)* and fifteen days (C_{15} *and* T_{15}) of drying.

Calculate the initial mass loss (water) of the control specimens (WL_0) using Equation B-4:

$$\boldsymbol{WL_0 = C_0 - C_5} \qquad \textbf{(Eqn B-4)}$$

Calculate the initial mass loss (water and sealer) of the sealed specimens (ML_0) using Equation B-5:

$$\boldsymbol{ML_0 = T_0 - T_5} \qquad \textbf{(Eqn B-5)}$$

Calculate the water loss due to vapor transmission for the control specimens (*VLC*) using Equation B-6:

$$\boldsymbol{VLC = C_5 - C_{15}} \qquad \textbf{(Eqn B-6)}$$

Calculate the water loss due to vapor transmission for the sealed specimens (*VLT*) using Equation B-7:

$$\boldsymbol{VLT = T_5 - T_{15}} \qquad \textbf{(Eqn B-7)}$$

The vapor transmission value (*VT*) is a ratio comparing the drying performance of the sealed cubes to that of the untreated controls over the final 10 days of the drying period. Calculate the *VT* of each sealer using the average *VLC* and the average *VLT,* following Equation B-8:

$$\boldsymbol{VT} = \frac{\boldsymbol{VLT}}{\boldsymbol{VLC}} \times \mathbf{100\%} \qquad \textbf{(Eqn B-8)}$$

For each sealer, calculate the average water loss from the sealed cubes (*WLT*) using the average VT and WL_0, following Equation B-9:

$$\boldsymbol{WLT} = \boldsymbol{VT} \times \boldsymbol{WL_0} \qquad \textbf{(Eqn B-9)}$$

For each sealed specimen, sealer loss (*SL*) is calculated using the *WLT* of the sealer*,* and the corresponding initial mass loss of the specimen, $ML_{0,}$ following Equation B-10:

$$\boldsymbol{SL} = \boldsymbol{ML_0} - \boldsymbol{WLT} \qquad \textbf{(Eqn B-10)}$$

Finally, the mass of dry sealer (*SD*) adhered to the sealed specimens is determined. It is calculated from the *SL* and *SF* of each specimen, following Equation B-11:

$$\boldsymbol{SD} = \boldsymbol{SF} - \boldsymbol{SL} \qquad \textbf{(Eqn B-11)}$$

B.6.2 Moisture Corrections

Once the *SD* is calculated for each specimen at the end of the 15-day drying period, each sealed cube should be adjusted to match the average RMC (target RMC of 70% or 80%, depending on the mixture design) of the unsealed control set. This is to ensure each specimen (sealed and unsealed) has an equal RMC when subjected to tests involving the absorption of moisture.

The control specimens shall remain enclosed in heavy-duty plastic freezer bags in the environmental chamber to prevent further loss in moisture.

Place the sealed specimens in an oven set to 140 °F (60 °C) and weigh the specimens daily until a desired mass (*DM*) corresponding to the target RMC is reached.

Calculate the *DM* for each specimen using Equation B-12:

$$\boldsymbol{DM} = \{\boldsymbol{X_0} + [(\boldsymbol{TMC} \times \overline{\boldsymbol{RMC}}_{\boldsymbol{C15}}\%) - \boldsymbol{TMC}]\} - \boldsymbol{SD} \qquad \textbf{(Eqn B-12)}$$

Where:

TMC is the calculated total moisture content of the reference specimens for each mix design,

X_0 is the initial mass recorded for the sealed cube being adjusted when it was at 100% RMC,

$\overline{RMC}_{C15}\%$ is the calculated average RMC after the 15 days of drying of the three control specimens corresponding to the sealed specimens undergoing moisture adjustments,

SD is the calculated mass of sealer adhered to the sealed specimen, and

DM is the unknown desired mass of a particular specimen.

The amount of moisture which evaporates from the sealed cubes varies depending on the sealer type and the mass of sealer adhering to the cube. Cubes in that reach the RMC of their corresponding control specimens before their sealed counterparts shall be enclosed in freezer bags and transferred back to the environmental chamber. They will remain in sealed bags until each sealed specimen has been adjusted to the desired RMC. Once all of the sealed cubes are adjusted, they are ready for testing.

Testing Procedure

B.7 Resistance to Moisture and Chloride Ingress Test

Testing containers shall have a means of supporting the specimens above the bottom surface with minimal contact. The test setup described in Section 3.6.1 of this report and shown in Figure 3.10 is suitable for this purpose.

For each mixture design, prepare sufficient 15% w/v NaCl solution to immerse three specimens per sealer and three untreated control specimens to a depth of 1 in. (25 mm) above the top surface of the specimens. Fill the test containers with the 15% NaCl solution.

Weigh each specimen to the nearest 0.1 g, and then immerse the specimen in the 15% NaCl solution for 21 days.

After five days in the solution, remove each specimen, pat dry with a towel, weigh to the nearest 0.1 g, and return the specimen to the solution.

After 21 days, remove each specimen, pat dry with a towel, and weigh to the nearest 0.1 g. Reserve the specimens for later chloride content determination.

The waterproofing performance (WPP) of a sealer is a measure of its resistance to moisture ingress. It is reported as a percentage reduction of the mass gained by the untreated control specimens (higher percentages indicate better performance).

WPP is calculated separately for each mixture design at both five days (WPP_5) and 21 days (WPP_{21}). To calculate WPP, subtract the average mass of solution gained by the three sealed specimens (*SG*) from the average mass gained by the control specimens (*CG*), and divide by *CG*, as shown in Equation B-13.

$$\boldsymbol{WPP_i = \frac{CG-SG}{CG} \times 100\%} \qquad \textbf{(Eqn B-13)}$$

B.8 Chloride Content Determination

The chloride content of the specimens may be determined either by potentiometric titration with silver nitrate (described in ASTM C1152 and Appendix A) or by X-ray Fluorescence Spectroscopy (XRF) (described in ASTM C114). Both techniques can be expected to provide equivalent results, but XRF can be expected to provide results more quickly. If titration is used, the procedure in Appendix A should be followed.

Microanalysis by SEM-EDS, EMPA, or micro-XRF may be used to provide confirmation of the depth and intensity chloride ingress, but is not required.

B.8.1 Specimen Preparation

Cut each specimen in half using a lapidary or cut-off saw intended for use on masonry and concrete. The cut shall be made perpendicular to the top surface of the specimen. Reserve one half for later microanalysis, if needed. The other will be reduced to a powder for XRF or titration.

Use a jaw crusher and pulverizer to mechanically crush the half specimen to be used for XRF or titration. Place the pieces in a 140 °F (60 °C) oven to dry for 24 h, then pulverize until the entire sample passes a No. 60 (250 μm) sieve. After crushing or pulverizing a specimen, thoroughly clean the equipment to avoid cross-contamination of specimens.

Reduce the resulting powder from each specimen to three sub-samples by quartering. Each sub-sample shall contain ~10 g of powder and be individually bagged prior to analysis.

B.8.2 Baseline Chloride Measurements

The untreated control specimens used for TMC determination in B.4.1 shall be used to determine the baseline chloride content of the concrete for each mixture design. This shall be reported as an average of all three specimens, and in terms of the percent mass of chlorides by mass of concrete. The baseline chloride content is subtracted from the chloride contents measured in the specimens exposed to the 15% NaCl solution to give the net chloride content of a specimen.

B.8.3 Net Chloride Measurements and Reporting

Determine the chloride content of each sub-sample by titration or XRF, and average the three measurements to determine the gross chloride content of each specimen. Then subtract the baseline chloride content to obtain the net chloride content of each specimen. Chloride contents shall be reported in terms of the percent mass of chlorides by mass of concrete.

For each sealer, report the resistance to chloride penetration (*RCP*) for each mixture design as the percentage reduction in net chlorides relative to the untreated control specimens. Calculate RCP in the same manner as WPP in Equation B-13.

B.9 Sealer Performance Evaluation

Acceptable performance of a sealer shall be defined as a minimum WPP and RCP of 75% for both mixture designs.

B.10 Sealer Penetration and Thickness Determination

If desired, sealer thickness and/or penetration depth can be determined using the three sealed specimens not exposed to the 15% NaCl solution. Some sealers contain sodium or chlorine, and NaCl solution exposure could interfere with these measurements if micro-analytical tools are used.

Sealer thickness and penetration measurements may be helpful in confirming even coverage of the specimen or predicting durability under traffic wear. However, they should not be used alone to determine sealer performance.

Epoxy-based sealers are unlikely to penetrate the concrete substrate, except into surface voids and cracks. Artificially-induced cracks may be used to test the crack-filling ability of an epoxy-based sealer. Optical microscopy of a sawn-open specimen at <100X magnification should be sufficient for sealer thickness measurements.

Penetrating sealers, such as silanes, siloxanes, and silicates, are intended to penetrate the concrete substrate. Determination of the depth of penetration should be undertaken by a concrete petrographer skilled in the use of SEM-EDS, EMPA, or micro-XRF. An evaporated sample of the sealer can provide the chemical composition of the non-evaporable solids. This will aid in determining penetration depth by measuring gradients in the concentration of these elements in the near-surface cement paste.

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