

PERFORMANCE OF CONCRETE  
BRIDGE DECK  
SEALERS

by

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A THESIS

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## ABSTRACT

The main objective of this research was to determine an improved method for evaluating bridge deck sealer products for the Alabama Department of Transportation (ALDOT) through a synthesis of two testing methods: NCHRP Report 244 Series II tests and standards developed by the Alberta Ministry of Transportation (BT Series). Five 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  $\mu\text{m}$  into the concrete.

## DEDICATION

I dedicate this thesis to my mother, father, and brother.

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## 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 a concrete bridge sealer, a type of protective surface treatment, to prolong the life of the bridge by reducing the penetration of aggressive substances from the environment (Basheer and Long 1997). ALDOT is currently looking for a potential replacement concrete bridge deck sealer in lieu of the currently-used product, because the manufacturer provides it solely for ALDOT's use, and production could be discontinued. It is crucial to provide an improved testing method to ALDOT to aid their search for an alternative product.

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 by incorporating multiple concrete mix designs and by testing the post-abrasion performance of sealers, yet they lack clarity in some procedural steps and may not be able to effectively evaluate post-abrasion performance of epoxy sealers.

## **1.2 Objectives and Scope**

The objective of this research was to develop an improved method for evaluating the effectiveness of bridge deck sealers using elements of the NCHRP Report 244 Series II and Alberta MOT 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 treated 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; following the 21-day soaking period, the resistance to chloride penetration was evaluated. The chloride content was determined by potentiometric titration by silver nitrate, x-ray fluorescence (XRF) spectroscopy, and the mass gained while immersed in the 15% NaCl solution. Finally, researchers investigated the sealers' penetration depth into the concrete and thickness on the 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 optical microscopy and SEM-EDS.

## **1.3 Organization**

This thesis 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 standard tests. Sealer products and concrete materials used are reported, 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, and the cause and severity of this cracking was investigated because the product 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.

## CHAPTER 2: LITERATURE REVIEW

### **2.1 Synopsis of Corrosion in Reinforced Concrete**

High quality, dense 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 do 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 through 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; 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 chlorides. More chlorides accumulate at the carbonation front and are more likely to initiate 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 contaminants (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 repellent) 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<sup>1</sup>), siloxanes (20 to 75 Å) and other silicate products.

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<sup>1</sup> Å (Angstrom) = 10<sup>-10</sup> m

- **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  $\leq$  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  $\geq$  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.

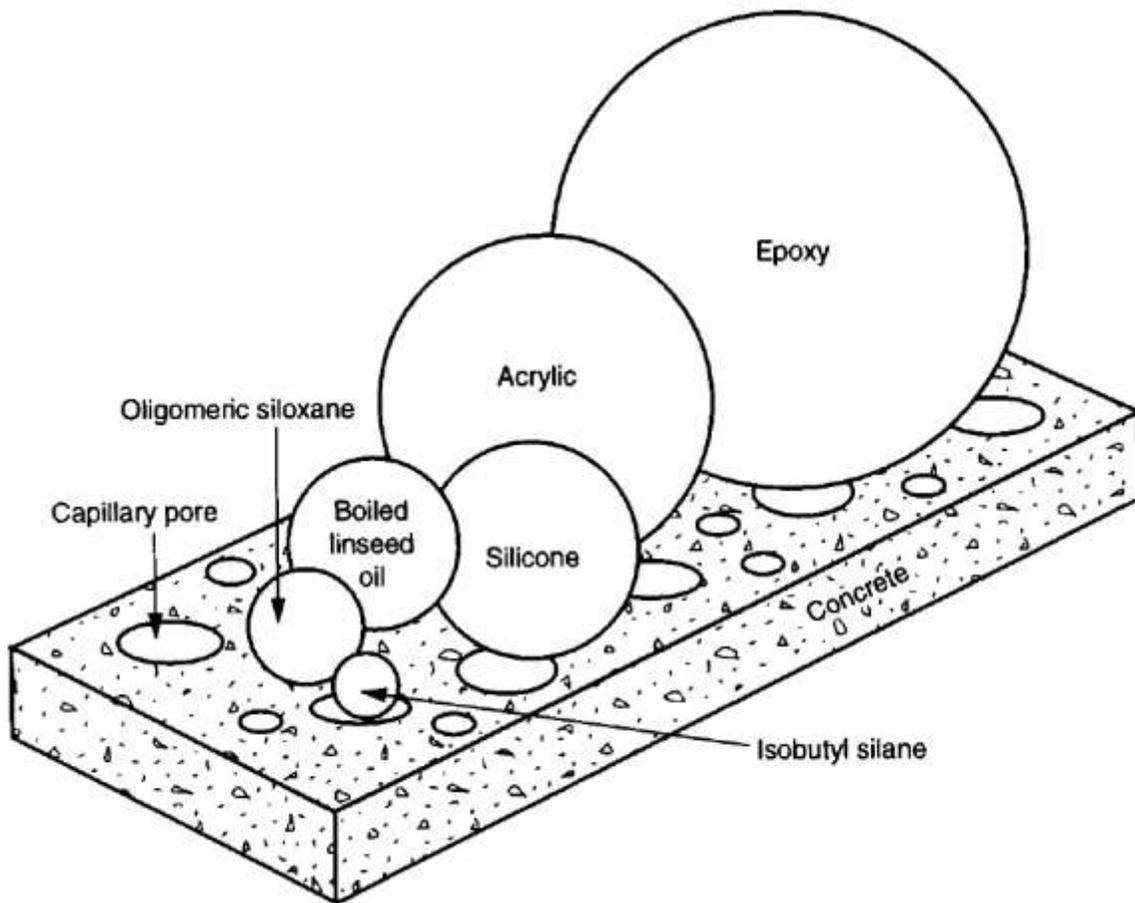


Figure 2.1 Relative molecular sizes of sealer components (Basheer and Long 1997).

**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 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. Bridge deck non-penetrating sealers that have an impermeable surface are ideal, but if water is present in the concrete prior to treatment, water could affect the adhesion of the sealer to the concrete substrate. If water is unable to evaporate, its presence could aid other forms of deterioration; e.g., entrapped water could serve as the water source needed for producing a gel-product from alkali-silica reaction (ASR).

### **2.3 Sealer Evaluation Methods**

National Cooperative Highway Research Program (NCHRP) Report 244, sponsored by 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**

In NCHRP Report 244, four series of tests 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 Report 244 include epoxies, silanes, silicates, silicones, siloxanes and linseed oil. Series I tests provided screening for products for the following tests by evaluating the absorption properties and chloride resistance of concrete sealed with various products. Series II tests studied the effects of concrete moisture content at the time

of sealer application versus chloride intrusion of the concrete. Series III tests 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 Series II and Series IV test were considered useful testing methods for effectively evaluating products (Pfeifer and Scali 1981). Only 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 through sandblasting, 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 untreated 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 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 suggest criteria for acceptable performance criteria for the reduction average mass gain and chloride ingress of sealer-treated concrete compared to unsealed control specimens:

- A minimum of 75% reduction of the mass gained by the treated cubes compared to the untreated control cubes
- A minimum of 75% reduction of the net chloride content retained by the treated cubes compared to the chlorides retained in the untreated control cubes, as measured by titration.
- Any products which do not pass these two limits are not recommended for testing in Series III and Series IV.

The best performance was found in specimens treated with a dual protective system; a silane was used as a secondary defense underneath an epoxy coating. According to NCHRP

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 NCHRP 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 treated at each drying time 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 Alberta MOT B338 Section 1.2. 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).

<p><b>Type 1 Sealers:</b> Penetrating sealers for use on traffic bearing surfaces exposed to abrasion. Categories are based on substrate exposure conditions</p>		
<p><b>Type 1a:</b> Penetrating sealers for application in sheltered conditions such as parking decks where the relative moisture content is a maximum of 55%.</p>	<p><b>Type 1b:</b> Penetrating sealers for application in outdoor conditions such as bridge decks where the deck relative moisture content is a maximum of 70% representing 2 days of drying. Represent mature concrete with a higher w/cm (0.45 - 0.50)</p>	<p><b>Type 1c:</b> 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%. Represent newer, less mature concrete</p>
<p><b>Type 2 Sealers:</b> Clear, film forming sealers for non-traffic bearing elements. Categories are based on the number of components.</p>		
<p><b>Type 2a:</b> One component, clear coatings suitable by less experienced personnel on concrete that has a relative moisture content is a maximum of 70%.</p>	<p><b>Type 2b:</b> 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%.</p>	
<p><b>Type 3 Sealers:</b> 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%.</p>		

### 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 BT010 Sections 2.4-2.8 (Alberta Ministry of Transportation 2000b) and BT001 Section 2.0. BT001 Sections 4.0-4.4 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  $74.0^{\circ}\text{F} \pm 3.0^{\circ}\text{F}$  ( $23.3^{\circ}\text{C} \pm 2.0^{\circ}\text{C}$ ) 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; this is to determine the mass of sealer that adhered to the cube and vapor transmission characteristics of the sealer. The mass measurements during the 15-day drying period are used to make adjustments to equate the relative moisture content of the untreated controls and the treated cubes before testing the waterproofing performance of the sealers. This is done following procedures in BT001 Sections 5.0-6.1. (Alberta Ministry of Transportation 2000a)

The initial waterproofing performance test is described in BT001 Section 6.2. 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 BT001 Sections 6.2 and 6.3. Only the sealed cubes are oven-dried at  $140^{\circ}\text{F} \pm 3^{\circ}\text{F}$  ( $60^{\circ}\text{C} \pm 2^{\circ}\text{C}$ ) 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.  $12.0 \pm 1.0$  g are removed per face for Type 1b sealers, which equates to an approximate depth of 0.5 mm of material removed;  $24.0 \pm 1.0$  g per face are removed for 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 area of the surface, and the mass of material

removed. These cubes are once again immersed in water for 120 hours to determine their waterproofing performance based on the mass of water absorbed in the test. (2000a)

### ***2.3.2.3 Alberta MOT Evaluation Criteria***

Alberta MOT B388 Section 4.2.1 provides criteria that are more stringent than NCHRP Report 244 for evaluating of performance of sealers 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 in the post-abrasion waterproofing performance test.
- Type 1c: an 85% reduction in mass gained relative to the control 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 commonly still used) for determining chlorides in concrete is a wet chemistry technique, titration with silver nitrate. 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 unknown concentration 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. 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 is 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 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 analysis of a concrete 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, placing loose powder and packing it into cups covered by a thin film, grinding powder until the particles are very fine then compressing it into a briquette, 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 Bhatta 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  $\mu\text{m}$ . 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) conducted a study for the Florida Department of Transportation (FDOT) by using 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.

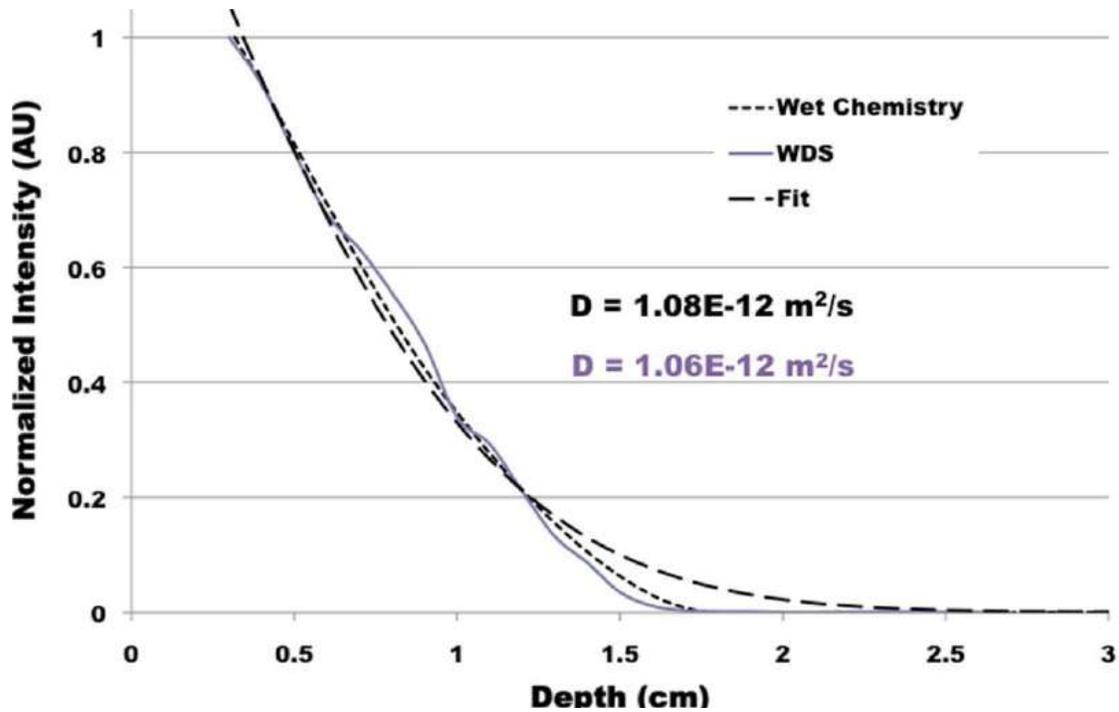


Figure 2.2 Results of one standard sample set gathered from EMPA-WDS and wet chemistry (titration) in the FDOT study. Their data show a good relationship between the two methods of evaluation for chloride content analysis. From (Dempere et. al. 2013).

In this FDOT study, SEM-EDS determined the results faster compared to 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. The authors found SEM microanalysis to be an effective, non-destructive method of measuring chloride content in concrete while minimizing caustic waste material generated by potentiometric titration. (Dempere, et. al. 2013)

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 determining 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 much 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 needed to obtain 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 deep as some water-based penetrating sealers had a heavier molecular weight. 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. (Pincheira and Dorsorst 2005)

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 NCHRP Report 244 Series II tests, the researchers evaluated both *penetrating* and *non-penetrating* bridge deck sealers. The Alberta MOT BT Series protocol can be used 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 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. NCHRP 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 NCHRP 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 NCHRP Report 244 Series II and Alberta MOT BT Series test protocols along with the elements used in the research described in this thesis (ALDOT Project 930-861). Elements of the Series II tests and the BT Series were selected for the research described in this thesis, 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.

<b>Element of Test Protocol</b>	<b>NCHRP R244 Series II Tests</b>	<b>Alberta MOT BT Series</b>	<b>ALDOT Project 930 - 861</b>
Specimen Dimensions	4 in. (100mm)	4 in. (100mm)	4 in. (100mm)
Curing Method	plastic bags in moist-cure room	(unspecified) in moist-cure room	plastic bags in moist-cure 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 cure	sandblasted after cure	sandblasted after cure
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	theoretical analysis and potentiometric titration	N/A	theoretical 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 NCHRP 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 NCHRP Report 244 Series II tests and the Alberta MOT BT Series test protocols for this research:

- The NCHRP Report 244 Series II tests and Alberta MOT standards both use cube specimens with the same dimensions. Similar-sized specimens for each test allowed for synergistic formwork, specimen preparation, and handling during the test procedures.
- The curing method was taken from NCHRP 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.
- The Alberta MOT BT Series procedure for surface preparation was selected because it specified a specific amount of material to remove by sandblasting. NCHRP Report 244 Series II 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 target relative moisture content (RMC) prior to sealer application, instead of drying specimens for a fixed amount of time to unknown moisture contents.
- 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 the NCHRP Report 244 Series II tests.
- The second conditioning phase specified by the Alberta MOT BT Series was selected to adjust the RMC of the treated 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.
- The 21-day waterproofing performance test in 15% NaCl was used from NCHRP Report 244 Series II tests.
- 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 NCHRP 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 suggested for use by the concrete petrographer who was engaged in this research.

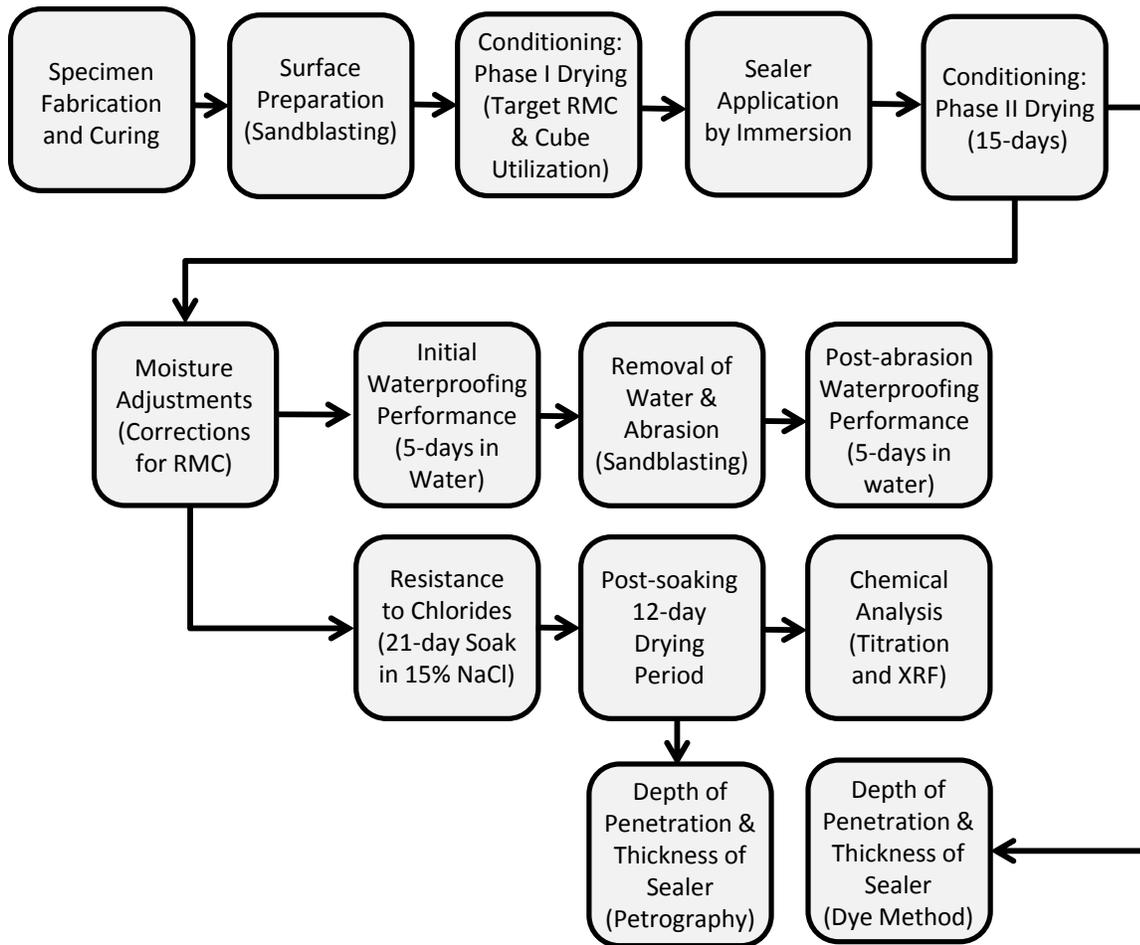


Figure 3.1 Chronological steps of the testing protocol for the selected elements to evaluate sealer products.

### 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 <sup>2</sup> /gal (m <sup>2</sup> /L)	Recommended Method of Application
Sealer A	One Component 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 cement used are reported from the mill certificate and are given in Table 3.3. Properties taken

from the mill certificate for 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.

<b>IDENTIFICATION OF MATERIALS</b>	
<b>MATERIAL</b>	<b>TYPE / PRODUCER / LOCATION / S.G.</b>
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 type I/II cement.

<b>Oxides (%)</b>						Na <sub>2</sub> O <sub>eq</sub> (%)	Limestone (%)	CaCO <sub>3</sub> in Limestone (%)	Insoluble Residue (%)
SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>				
20.5	4.54	3.60	63.04	2.27	2.90	0.49	1.0	97.02	0.35
<b>Bogue Phase (%)</b>				C <sub>3</sub> S + 4.75 * C <sub>3</sub> A (%)	LOI (%)	Heat of Hydration (kJ/kg)	Blaine Fineness (m <sup>2</sup> /kg)	% Passing 45 µm (No. 325) Sieve	
C <sub>3</sub> S	C <sub>2</sub> S	C <sub>3</sub> A	C <sub>4</sub> AF						
55.20	17.20	5.90	10.90	83	1.66	406	402	97.1	

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

<b>Oxides (%)</b>								LOI (%)	Moisture (%)	% Passing 45 µm (No. 325) Sieve
SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	K <sub>2</sub> O	Na <sub>2</sub> O			
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 coarse and fine aggregate.

<b>Property</b>	<b>Coarse</b>	<b>Fine</b>
Specific Gravity (SSD)	2.75	2.63
Absorption Capacity (%)	0.7	0.5
Fineness Modulus	-	2.41

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

Specimen molds were made from cabinet-grade plywood to ensure smooth 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 providing cube 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

Concrete with a higher water to cementitious materials ratio ( $w/cm$ ) has higher permeability and porosity than concrete with a lower  $w/cm$ , when properly consolidated. Concrete made with supplementary cementitious materials (SCM) tends to have a more dense and impermeable microstructure than concrete made using solely portland cement. Three different types of concrete were used in the evaluation of the sealer products. This was to encompass the range of bridge deck concrete that is found in service in Alabama.

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, 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 prequalified requirements stated in Section 501.02 (c) (ALDOT 2012). A 0.4 w/cm 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 ONE CUBIC YARD {METER}

COMPONENT	Class B		
	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.<sup>3</sup> (0.13 m<sup>3</sup>) 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.

<b>Fresh Concrete Properties</b>	<b>MD1</b>	<b>MD2</b>	<b>MD3</b>
Slump (in) {cm}	8.7 {22.1}	3.5 {8.9}	6.0 {15.2}
Unit Weight (pcf) {kg/m <sup>3</sup> }	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}

<b>Hardened Concrete Properties</b>			
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 check 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 for the second layer. 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 can be 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.

<b>Nominal Opening</b>		<b>Requirements</b>	<b>Sil 7</b>
<b>Mesh No.</b>	<b>(microns)</b>	<b>% Retained</b>	<b>% Retained</b>
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. These did not pass the limits specified by Alberta MOT. In Alberta MOT BT010, Section 2.7 *Surface Preparation*, more emphasis is placed 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 the first and last sample sandblasted.

### **3.5 Specimen Conditioning and Sealer Application**

A series of steps was used to condition the specimens, and to optimize 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 used for testing were dried to a target relative moisture content (RMC) before sealer application. Prior to removing specimens from the moist curing room, the three specimens from each mix design were designated as the sample set for total moisture content (TMC) determination. They were removed from the moist room 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 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. Conditions for the environmental chamber were  $74^{\circ}\text{F} \pm 3^{\circ}\text{F}$  ( $23.3^{\circ}\text{C} \pm 2.0^{\circ}\text{C}$ ) and  $50 \pm 4\%$  RH. Figure 3.6 shows the orientation of the cubes on the wire shelves, which allowed for circulating air exposure on all six surfaces.



Figure 3.6 Specimens drying in the environmental chamber.

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 Eqn 3-1:

$$RMC (\%) = \frac{[TMC - (X_0 - X_i)]}{TMC} \times 100 \quad (\text{Eqn 3-1})$$

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. MD1's target RMC was  $70\% \pm 2.0\%$ . The target RMC for MD2 and MD3 was  $80.0\% \pm 2.0\%$ . The drying time required to reach the target RMC was 19 days for MD1, 23 days for MD2, and 9 days for MD3. 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.

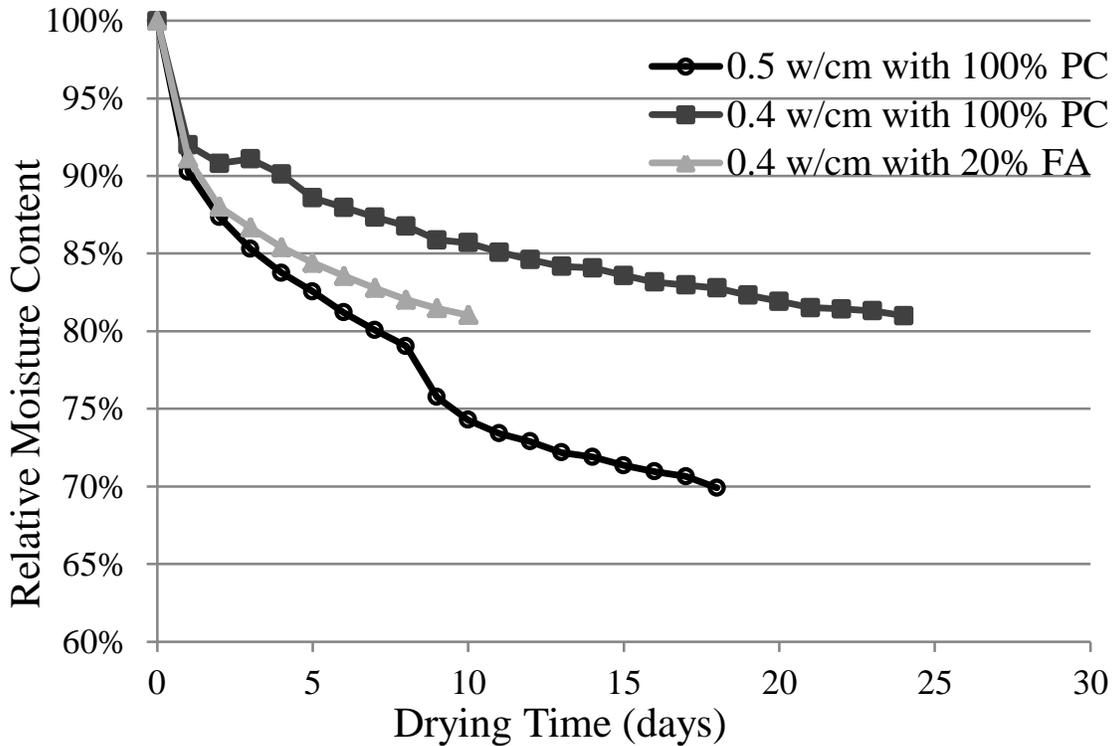


Figure 3.7 Average RMC of specimens during the controlled drying period.

### 3.5.2 Cube Utilization

Per Alberta MOT BT001 (2000a), the sealer application procedures specified the selection of sets of three cubes of similar RMC for 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, 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.

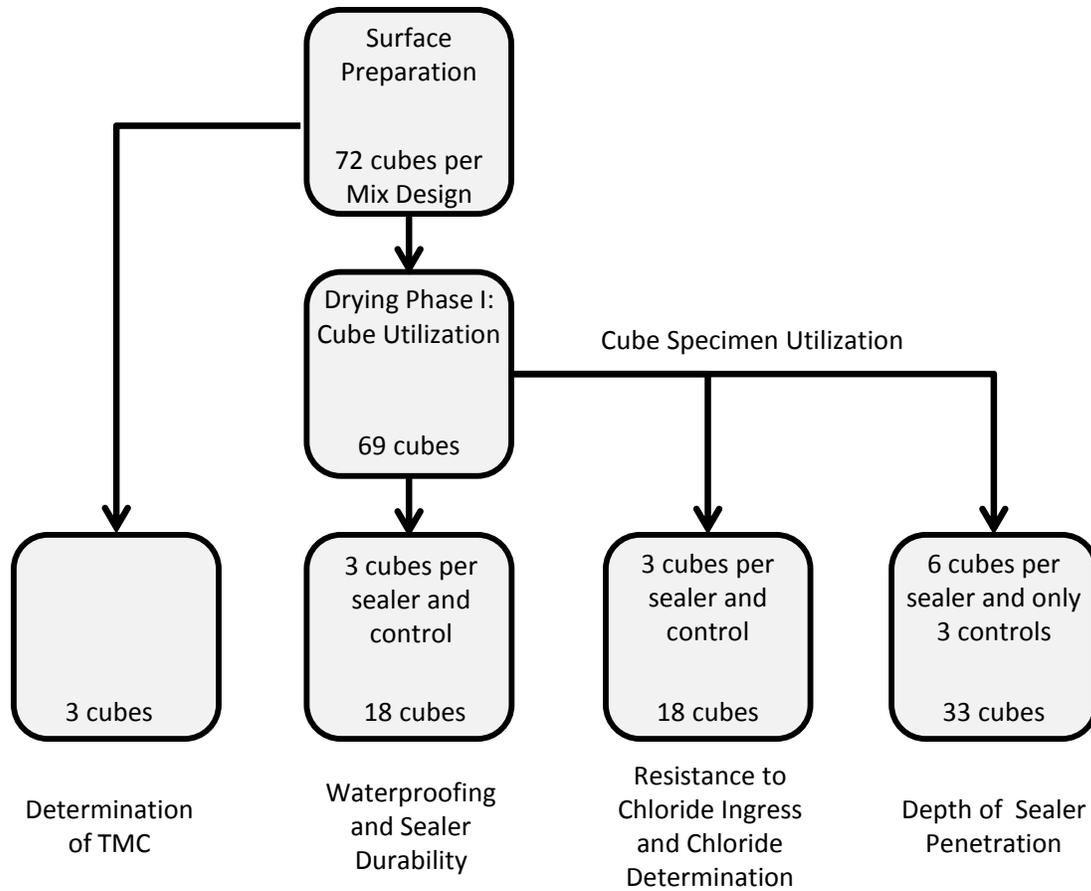


Figure 3.8 Detailed diagram for test specimen utilization by task. Specimen quantities are per mix design.

### 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. The epoxy sealers (Sealers B, C, D and E) were supplied as two-part systems and these were mixed following the manufacturers' recommendations. The specified ratio of components were measured in separate containers, combined in one container and mixed thoroughly for three minutes with a paddle and drill.

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.

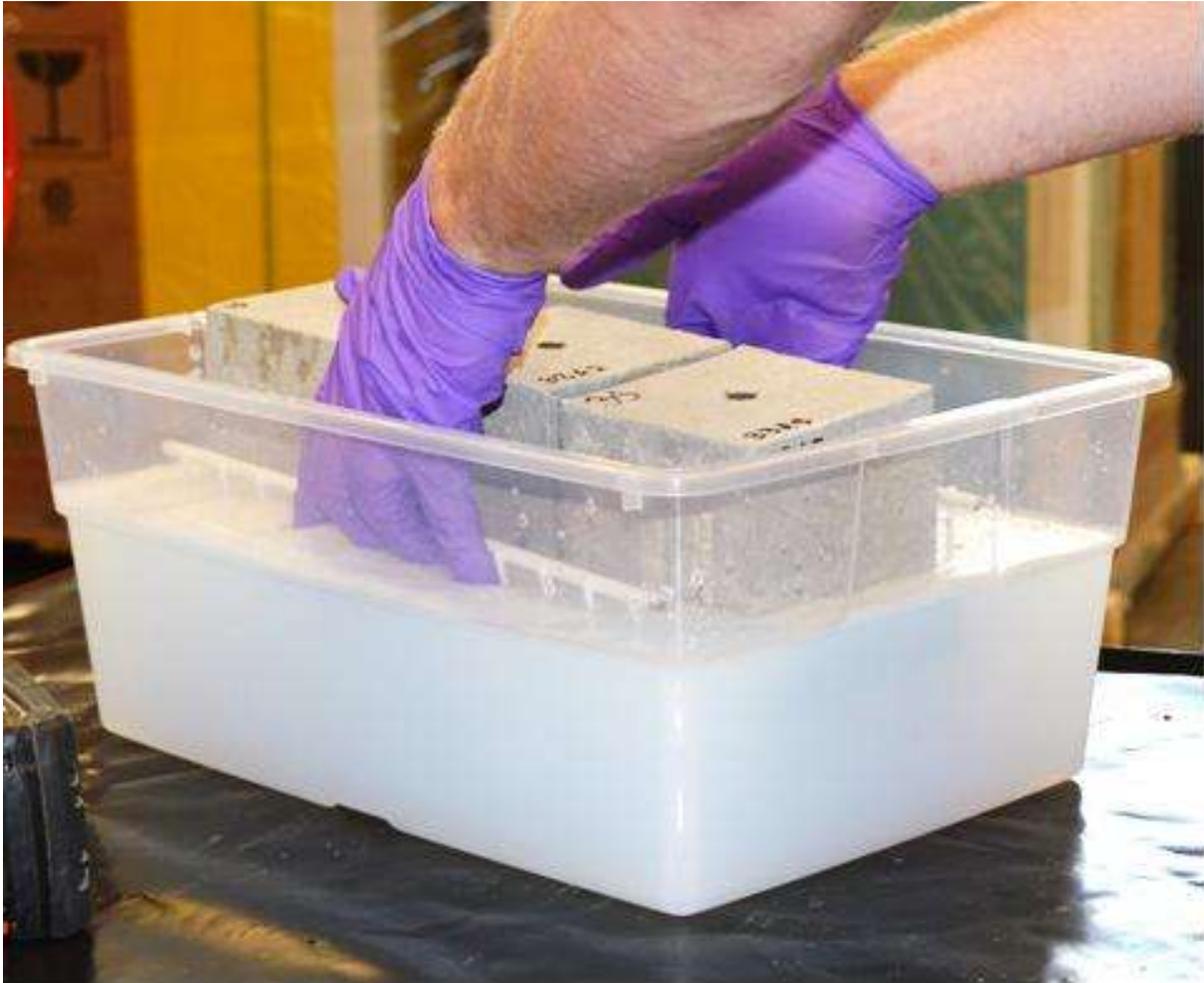


Figure 3.9 A set of specimens being immersed into the sealer.

Measurements were recorded as follows 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 ( $T_2 = \text{mass before second immersion}$ ).
- 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 by Eqn 3-2:

$$SF = (T_1 - T_0) + (T_3 - T_2) \quad (\text{Eqn 3-2})$$

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 wasn't tacky to the touch, but was handled with the same care as the epoxy-based sealers.

### 3.5.3 Conditioning: Phase II Drying

After the sealer was applied, the specimens (treated and 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  $50 \pm 4\%$  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 by 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 after five days ( $C_5$  – for *untreated controls* and  $T_5$  – for *corresponding treated specimens*). The final measurement for the control and treated cubes was recorded after an additional ten days of drying ( $C_{15}$  and  $T_{15}$ ). These measurements were used in intermediate calculations to determine the water loss, sealer loss, and vapor transmission value for the amount of dry sealer mass adhering to individual specimens. Initial mass loss (water) in control cubes ( $WL_0$ ) are calculated by Eqn 3-3:

$$WL_0 = C_0 - C_1 \quad (\text{Eqn 3-3})$$

The initial mass loss (water and sealer) of treated cubes ( $ML_0$ ) was calculated by and Eqn 3-4:

$$ML_0 = T_0 - T_1 \quad (\text{Eqn 3-4})$$

The water loss due to vapor transmission for the control set ( $VLC$ ) was calculated by Eqn 3-5:

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

The water loss due to vapor transmission for the sealed set ( $VLT$ ) was calculated by Eqn 3-6:

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

The vapor transmission value ( $VT$ ) is a ratio comparing the drying performance of the treated 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 the average of water loss in controls due to vapor transmission ( $VLC$ ) and average water loss of the sealed specimens ( $VLT$ ), shown in Eqn 3-7:

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

The average water loss in the treated-test cubes ( $WLT$ ) was calculated as a function of the average vapor transmission value,  $VT$ , and the average of the water lost in the untreated-control cube set,  $WL_0$ , given by Eqn 3-8:

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

Sealer loss ( $SL$ ) was not taken as an average of the three cube set. It was determined by the water loss of the individual treated cubes,  $WLT$ , and the corresponding initial mass loss of individual treated cubes in the set  $ML_0$  showed in Eqn 3-9:

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

Finally, the mass of dry sealer ( $SD$ ) adhered to the treated-specimens is determined. It was not taken as an average of the three cube set, but was calculated from the individual treated cubes' loss of sealer  $SL$ , and the individual mass of fresh sealer ( $SF$ ) gained, given in Eqn 3-10:

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

### 3.5.5 Moisture Adjustments

During the Phase II drying, the untreated control set lost more moisture than the treated sets over the 15-day drying period; this meant the RMC of the treated cubes was greater than the untreated cubes. The treated cubes' RMC needed to be adjusted to the RMC of the untreated controls. After  $SD$  was calculated for each specimen at the end of the 15-day drying period, each treated cube was returned to the environmental chamber and dried to match the average RMC of the untreated control set. This was to ensure each specimen (treated and untreated) had an equal RMC when subjected to tests involving the absorption of moisture. 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.

The untreated control set was then enclosed in heavy-duty plastic freezer bags to prevent further moisture loss, while treated cubes were placed in an oven set to 140°F (60°C). The set of treated cubes designated for the waterproofing performance test was adjusted to the average RMC of their corresponding untreated control set. The same was done for sealed cubes designated for chloride resistance testing.

Adjustments were made by placing the cubes into in an oven at a temperature of 140°F (60°C) to remove moisture until a desired mass ( $DM$ ) was reached. To solve for the  $DM$ , two RMC equations are set equal to each other and solved. Eqn 3-11a shows the relation of the two sets: the average of the three controls' RMC set equal to a treated cube's RMC. Equation 3-11b expands this relationship. The equations for the  $DM$  are as follows:

$$\overline{RMC}_{C15} (\%) = RMC_{T15} (\%) \quad (\text{Eqn 3-11a})$$

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

Where:

*TMC* is the calculated total moisture content of the mix specimens,

*X<sub>0</sub>* is the initial mass recorded for the sealed cube being adjusted when it was at 100% RMC,

$\overline{RMC}_{C15}\%$  is the calculated average of the three cube controls RMC after the 15 days of drying corresponding to the sealed cube set being adjusted for moisture content,

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

*DM* is the unknown desired mass.

*DM* is calculated using Eqn 3-12:

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

The amount of moisture evaporated from the treated cubes varied depending on the sealer type and mass of sealer adhering to the cubes. Cubes that matched the RMC of the untreated controls before their treated counterparts were secured in freezer bags and transferred back to the environmental chamber. They remained sealed in the bags until each treated 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 treated 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. Thirty-six 0.5 in. (12 mm) diameter polyurethane tubes were

paired, symmetrically spaced, and glued to the bottom of each container, as shown Figure 3.10, to ensure the whole surface area of the test specimen was exposed to the water. 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 ( $CGI$ ) subtracted from the average mass of water gained from the treated set ( $TGI$ ), divided by the average mass gained by the untreated control set ( $CGI$ ) and was reported as a percentage shown by Eqn 3-13.

$$WPP_i = \frac{CG_1 - TG_1}{CG_1} \times 100\% \quad (\text{Eqn 3-13})$$

### 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, and then submersing the treated cubes for an additional 120 hours in water. The mass gained by the treated cubes during the post-abrasion submersion was compared to the mass gained by the untreated controls in the initial waterproofing performance test.

After the initial waterproofing performance tests, the treated test cubes were transferred to an oven and dried in the same conditions as described section 3.5.6; 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. Measurements were taken 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 to prevent any moisture loss until they were sandblasted and tested.

Sets of three treated cubes with the same sealer type 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. Samples from MD1 had  $12.0 \pm 1.0$  g per face ( $72.0 \pm 4.0$  g total) removed, and samples from MD2 and MD3 had  $24.0 \pm 1.0$  g removed per face ( $144.0 \pm 4.0$  g total). Figure 3.11 illustrates the contrast between two surfaces of the same specimen before and after this step. 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.



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.

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. Without using the grinder, sandblasting one side of an epoxy-sealed cube took over an hour to complete. 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.

After grinding, it took approximately 45 minutes to sandblast all six sides of each specimen. Due to the extensive time required to sandblast, 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 ( $TG2$ ) to calculate  $WPP_f$  using Eqn 3-14:

$$WPP_f = \frac{CG1 - TG2}{CG1} \quad (\text{Eqn 3-14})$$

### 3.6.3 Resistance to Chloride Penetration

The procedure described in NCHRP Report 244 Series II tests was followed for the soaking and drying periods of the specimens designated 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. The soaking period lasted 21 days. Specimens were removed from the solution for mass gain measurements every three days. Each specimen was lightly toweled dry, measured for mass gain, 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 NCHRP 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. Theoretically, 9.1% chlorides by mass are present in a 15% NaCl solution. In this “mass gain method,” the 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.

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, 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 a detailed 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 a third method, 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 set of samples were tested by XRF. The 18 specimens selected for XRF analysis were:

- 1 untreated, unexposed (baseline) control (2 total)
  - MD2, MD3
- 1 treated, unexposed control per sealer product (5 total)
  - MD2 – All sealer products
- 1 untreated, exposed control (2 total)
  - MD2, MD3
- 1 treated, 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.

### **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 petrographer. Samples subjected to the dye method were observed under an optical microscope, and samples analyzed by 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.

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 treated 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 treated 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.

The coverage rate is based on the specific gravity, fresh sealer mass, and the surface area of the 4 in. (100 mm) cube specimens (sealed surface area per sealer volume). Coverage rates at the lower range indicate that more sealer was applied to the specimen (that is, less area was sealed with a given unit volume of sealer), and conversely for coverage rates at the upper end. 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).

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.

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 (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 <sup>2</sup> /L)	Recommended Coverage (m <sup>2</sup> /L)	Applied Rate (ft <sup>2</sup> /gal)	Recommended Coverage (ft <sup>2</sup> /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 <sup>2</sup> /L)	Recommended Coverage (m <sup>2</sup> /L)	Applied Rate (ft <sup>2</sup> /gal)	Recommended Coverage (ft <sup>2</sup> /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 <sup>2</sup> /L)	Recommended Coverage (m <sup>2</sup> /L)	Applied Rate (ft <sup>2</sup> /gal)	Recommended Coverage (ft <sup>2</sup> /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

Sealers B, C and E were applied within the range of coverage rates recommended by the manufacturers for all three mix designs. Sealer D was applied within the recommended coverage rate for MD1 and MD3, although for MD2 more sealer than recommended was applied. Sealer A did not meet the recommended coverage rate for any of the 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<sup>2</sup>/gal (1.0 m<sup>2</sup>/L), or approximately four times the recommended amount sealer recommended by the manufacturer.

Sealer A had the largest VT value, which was consistently over 100%, which signifies that the specimens were drying faster than if they weren't treated. 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%).

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

All specimens soaked in the dye did not appear to have any penetration depth 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  $\mu\text{m}$  on the top surface of the specimens examined. Sealer C and Sealer B had thickness which ranged from 120 to 900  $\mu\text{m}$ . 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 MD1 specimen treated with Sealer E. The thickness varied depending on the topography of the specimen.

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  $\mu\text{m}$ . 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  $\mu\text{m}$  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 over three times the required amount of sealer needed to satisfy the minimum coverage rate specified by the manufacturer, did not show any increase in penetration depth when evaluated with SEM-EDS (Rothstein 2015).

### **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 method of evaluating the initial waterproofing performance of the sealers in water is speedy and effective. 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. Sealer E had the best overall average performance, followed closely by Sealer D. 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.

Sealer B’s initial waterproofing performance for MD1 was significantly deficient compared to Sealer B’s initial waterproofing performance on the other two mixes as shown in Table 4.2. 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.

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 WATERPROOFING PERFORMANCE AT 5 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
Control	-	-	-	-
Sealer A	13%	10%	19%	14%
Sealer B	67%	80%	71%	73%
Sealer C	89%	82%	73%	81%
Sealer D	86%	86%	69%	80%
Sealer E	80%	85%	77%	81%

#### 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 treated specimens gaining more mass than the untreated controls. None of the products had an

acceptable post-abrasion waterproofing performance. It is easy to see the protection the sealer provided before sandblasting from observing all negative post-abrasion waterproofing performance results with the exception of Sealer A for MD3.

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
Control	-	-	-	-
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 presents the average mass gained for the initial and post-abrasion waterproofing performance tests. Specimens treated with Sealer A had a better post-abrasion waterproofing performance than the other products; however, the mass gained suggests the product did not provide effective protection relative to untreated controls mass gained.

The amount of material removed from each surface in preparation for the post-abrasion waterproofing test completely removes the sealer, re-exposing the substrate concrete. Approximately 0.02 in. (0.5 mm) of material was removed from each sealed MD1 specimen and 0.04 in. (1 mm) removed from MD2 and MD3 specimens. This amount of material removed was greater than the thickness of epoxy sealer on the surface for specimens and greater than the depth to which Sealer A penetrated.

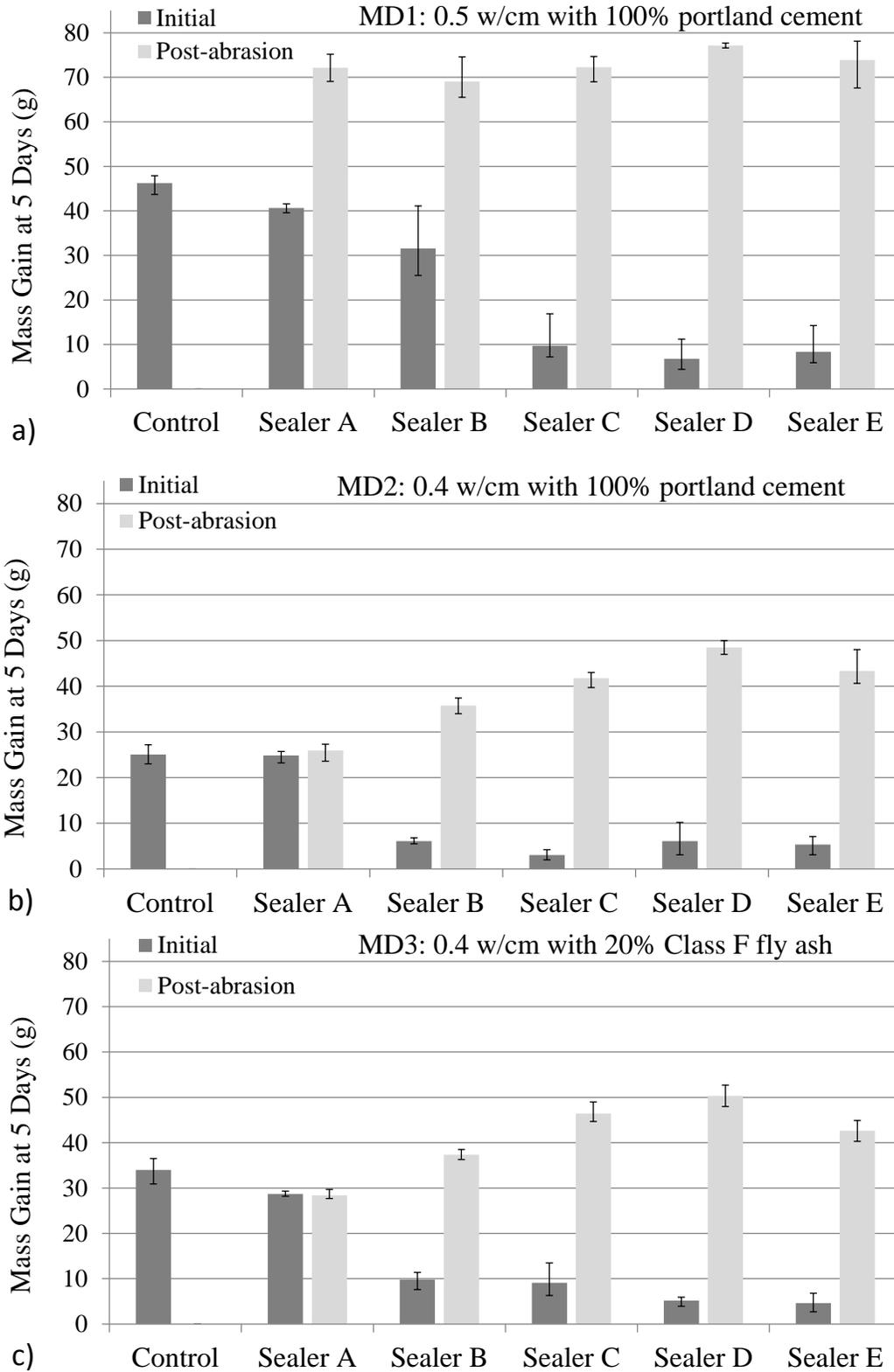


Figure 4.2 Average initial and post-abrasion mass gain at 5 days for (a) MD1 (b) MD2 (c) MD3. Error bars represent the range of values measured between three cube sets.

Results showed 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 parameters stated 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.

#### **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. A discussion of results from mass gain, waterproofing performance, and chloride analyses are discussed in this section.

##### **4.4.1 Mass Gain and Waterproofing Performance**

The waterproofing performance represents the average reduction of mass gained by the treated specimen relative to the untreated controls after soaking in the NaCl solution. Table 4.4 contains the average 21-day waterproofing performance of each sealer for each mix design. These data represent the average reduction of mass gained relative to the unsealed controls. The allowable limit recommended by the NCHRP Report 244 Series II protocol for waterproofing performance at 21 days is a minimum 75% reduction in average mass gained by the sealed specimens relative to the unsealed controls. For the 21-day overall average, only Sealer C met the minimum requirement of 75% reduction in mass absorbed relative to the untreated controls. The overall average for Sealer D and Sealer E were just below 21-day minimum requirement at a 74% average reduction in mass gained. 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 each mix design.

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

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
Control	-	-	-	-
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 3 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.

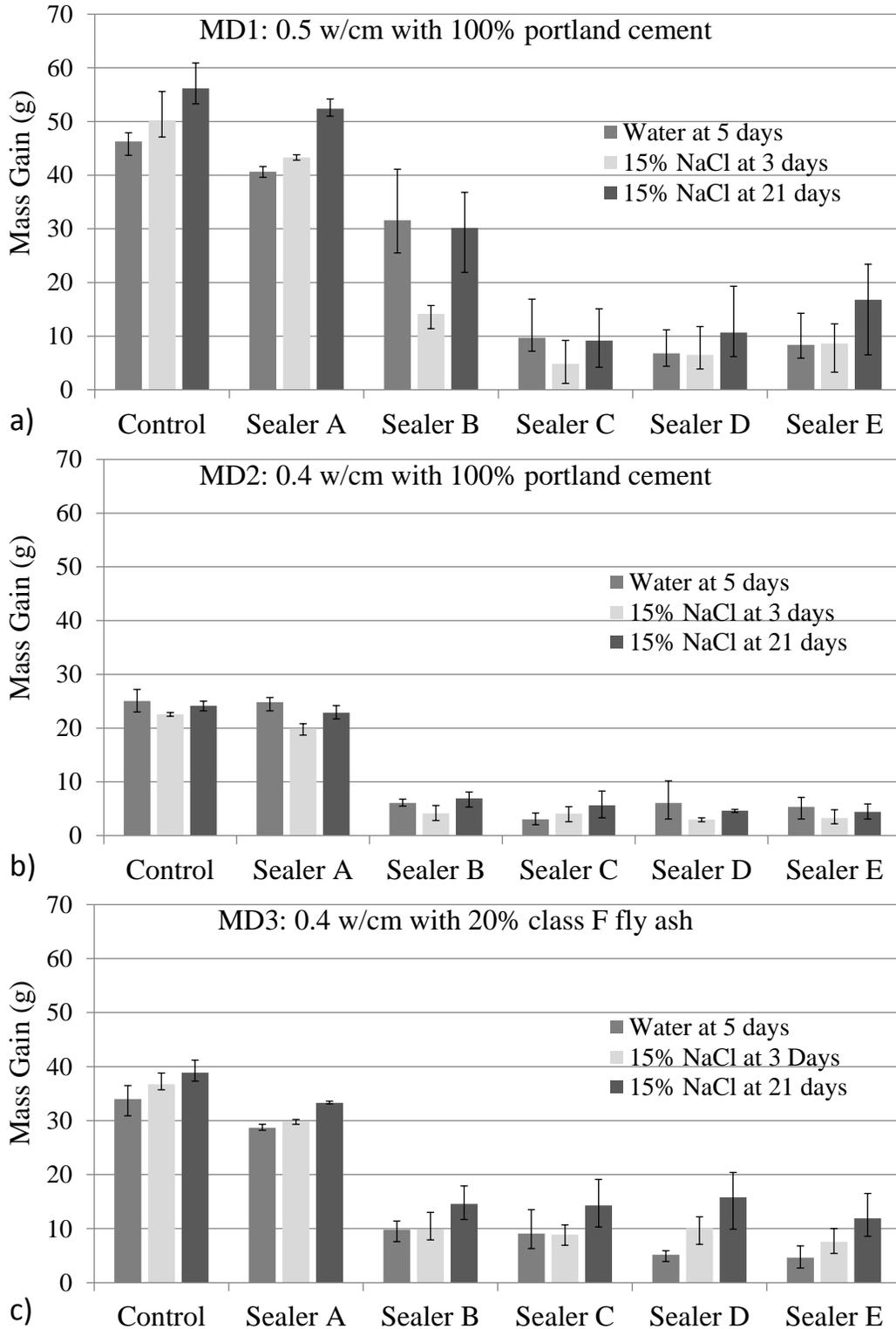


Figure 4.3 Comparison of the average mass gained between specimens which soaked in water and 15% NaCl solution for (a) MD1 (b) MD2 (c) MD3. Error bars represent the range of values measured between three cube sets.

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 amount of mass gained by specimens after 5 days in water and 3 days in 15% NaCl solution for each mix design varied, but they were overall similar with the same exception of Sealer B for MD1. MD1 specimens treated with Sealer B appeared to gain mass at a 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.

#### **4.4.2 Chloride Analysis**

The chloride content of the specimens was also measured by potentiometric titration with silver nitrate and XRF spectroscopy; these chloride contents were also compared with the theoretical 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.

The resistance to chloride penetration of the sealers was evaluated as a percentage of chlorides retained in the treated specimens relative to the chlorides retained in the untreated sample after a 21-day immersion in 15% NaCl solution. Table 4.5 contains 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 treated specimens than in the untreated controls. The NCHRP 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 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 exceeded 80% for all three mix designs.

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
Control	-	-	-	-
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 theoretical chloride content 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 than MD2 and MD3 specimens treated with Sealer B. MD1 and MD3 specimens treated with Sealer A had chloride contents *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	MD	% mass gain after 21days in 15% NaCl solution	Measured Chlorides by Mass Gained (% Mass)	Measured Chlorides by Titration (% Mass)	Measured Chlorides by XRF (% Mass)
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
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

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. Titrations took four to five hours per sample, whereas a sample evaluation by XRF spectroscopy can be completed in minutes.

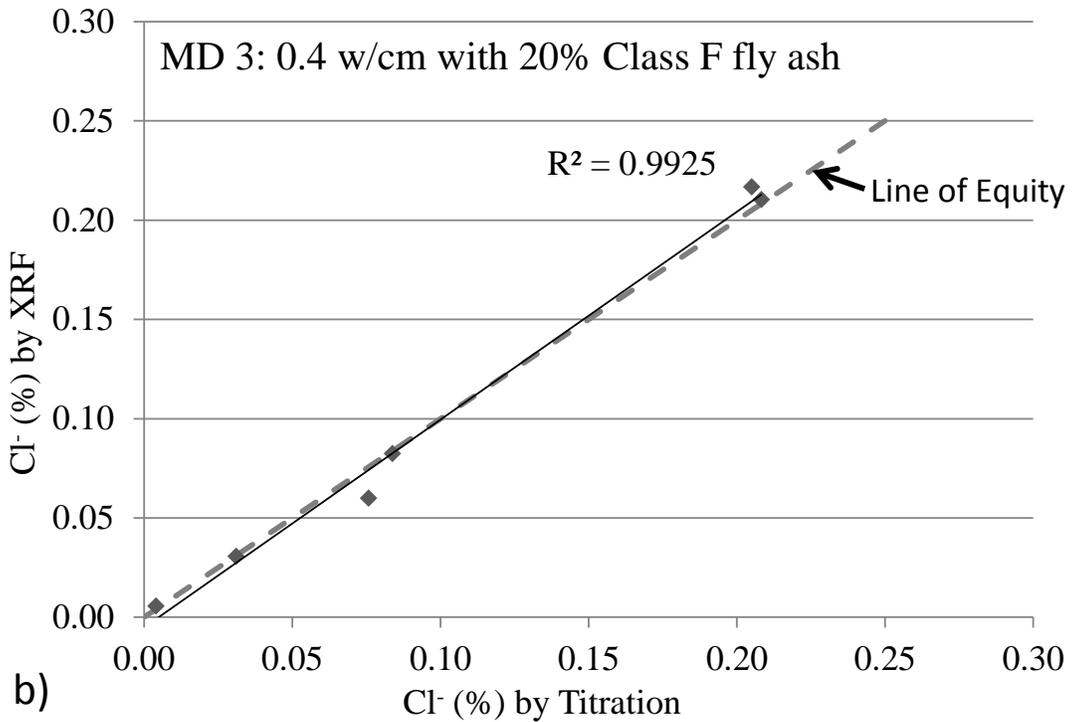
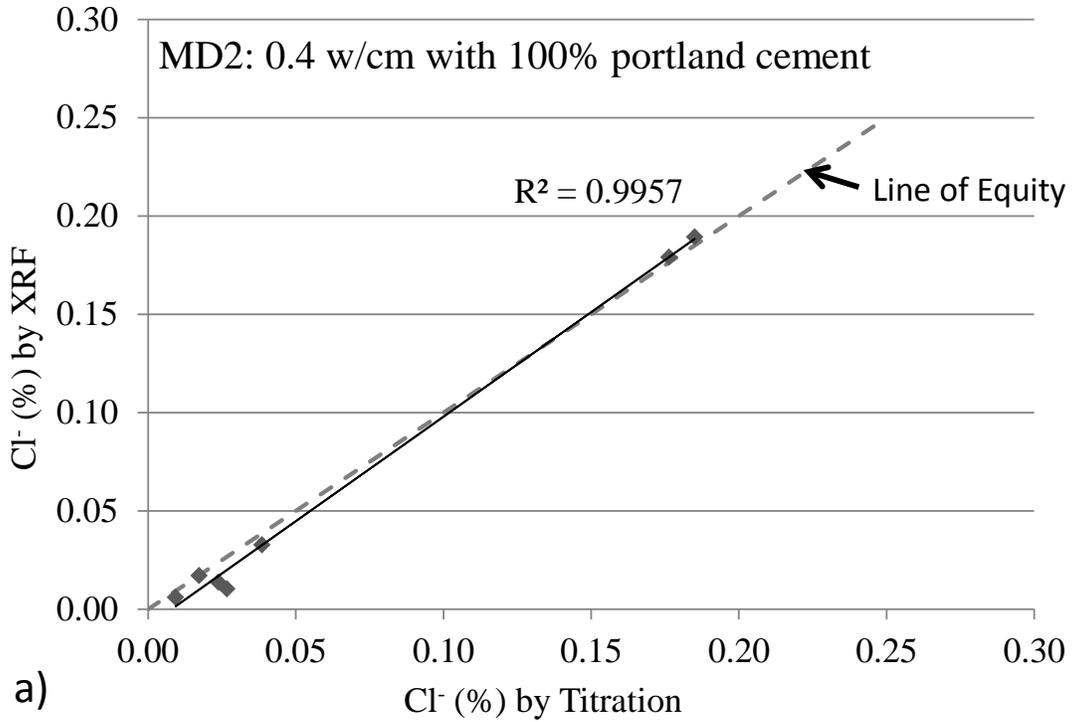
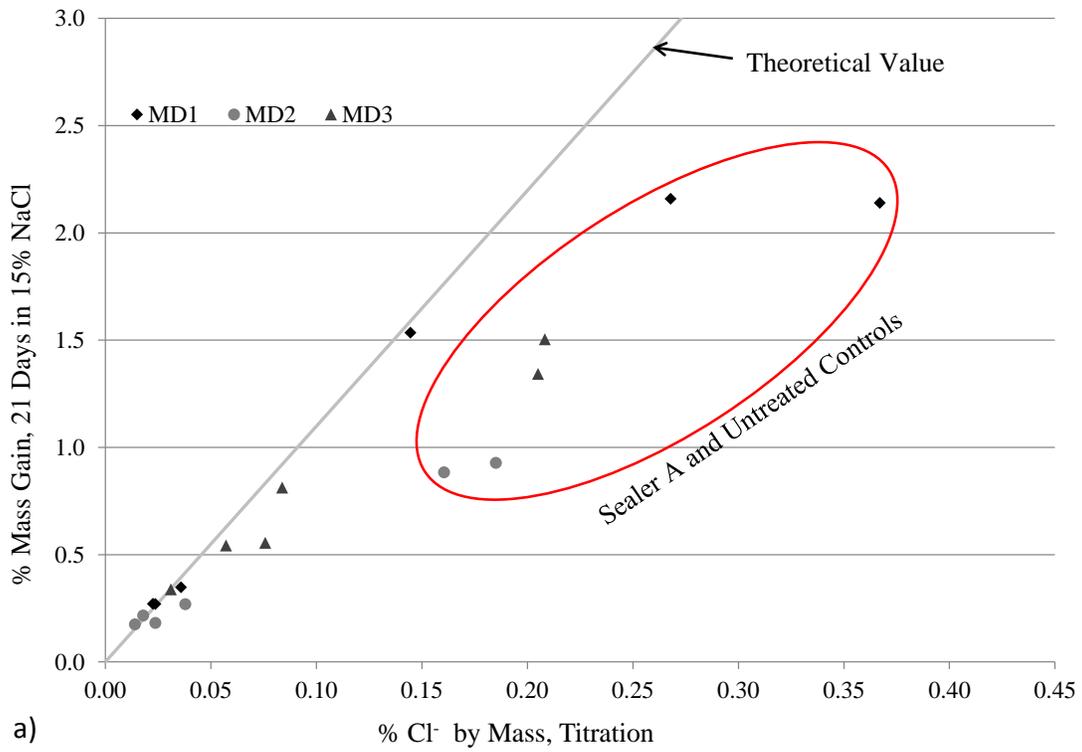


Figure 4.4 Comparison of chloride contents determined by titration and XRF for all combinations of controls and sealers on (a) MD2 (b) MD3. MD3 specimens treated with Sealer C were not included because of cracking of the sealer.

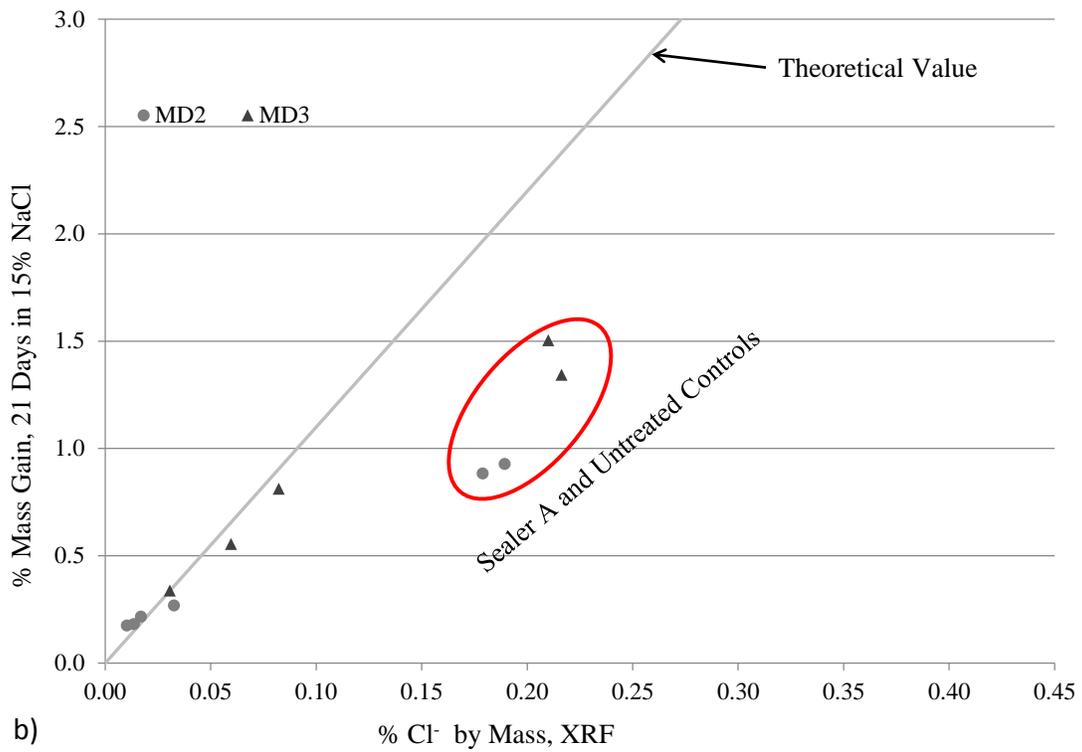
Figure 4.5 presents the chloride content measured by titration and XRF compared with the theoretical chloride content based on the mass gain measurements (9.1% of the mass of the absorbed solution). The theoretical value is shown as a line of equity in the figure and can be used to compare chloride contents 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.

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 need to be conducted to confirm diffusion effects.

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 NCHRP Report 244 Series II tests of 4.6% - 11.1% (Pfeifer and Scali 1981) and varies considerably from the expected 9.1% chlorides in 15% NaCl solution.



a)



b)

Figure 4.5 Comparison between percent mass gain and chloride content determined by (a) Titration (b) XRF.

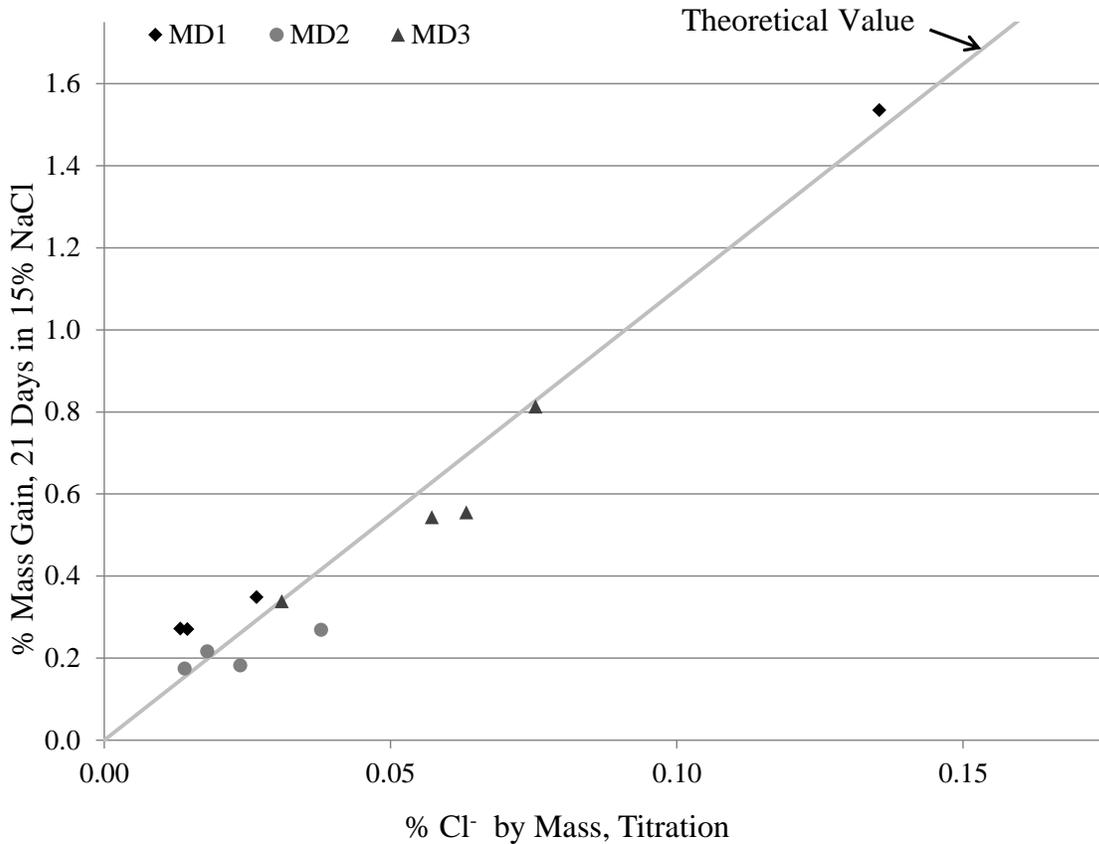


Figure 4.6 Theoretical chloride content based on mass gain measurements versus the chloride content determined from titration for the four epoxy-based sealers.

Based on this research, mass gain measurements are insufficient for evaluating the resistance to chloride ingress because the chloride contents that would be expected based on a 15% NaCl soak solution did not match the chloride contents measured by titration and XRF. Calculating 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 is a more accurate method of measuring the chloride content in concrete; however, it is a time-consuming process. XRF provided nearly identical results as 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 on the top surface of the cube where the sealer coating had the greatest thickness. Cracking of the

sealer was observed on 13 out of 27 specimens treated with Sealer C (the remaining 9 treated specimens were not counted because the sandblasting for the post-abrasion waterproofing tests removed the sealer). Cracks were first noticed approximately 3 weeks after sealer was applied. Figure 4.7 illustrates how the specimens were conditioned and handled between the time that the sealer was applied and when cracking of the sealer was observed.

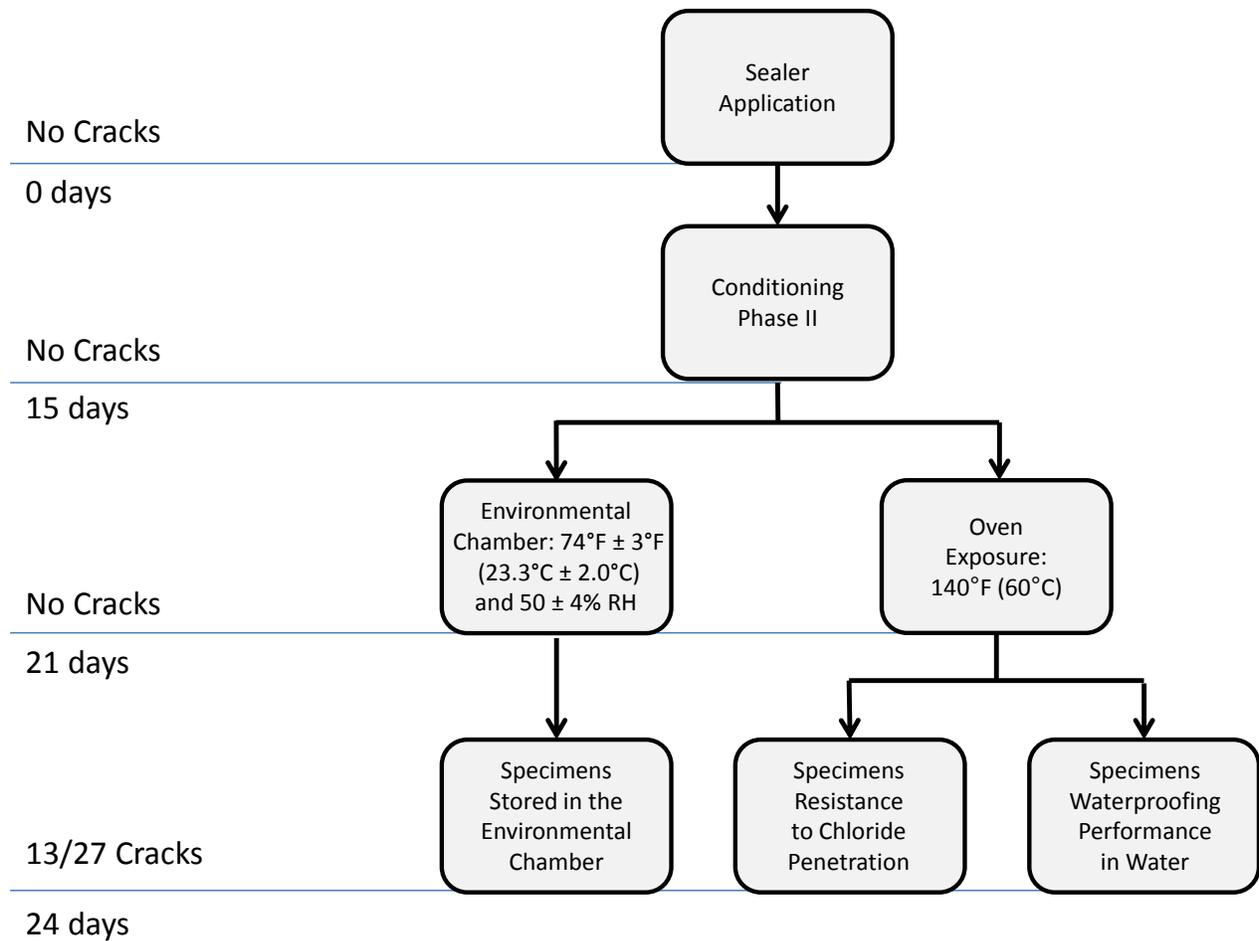


Figure 4.7 Timeline from procedure when cracks were noticed.

Initially, this 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, other specimens treated with Sealer C, which 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  $74^{\circ}\text{F} \pm 3^{\circ}\text{F}$  ( $23.3^{\circ}\text{C} \pm 2.0^{\circ}\text{C}$ ) and  $50 \pm 4\%$  RH. The other three epoxy-based sealer products did not crack before or after they were exposed to oven drying at  $140^{\circ}\text{F}$  ( $60^{\circ}\text{C}$ ) for moisture adjustments. It is worth noting that the excess Sealer C from the sealer application process was placed into a container, where it solidified and 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 4.8 and 4.9 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  $500\ \mu\text{m}$ .

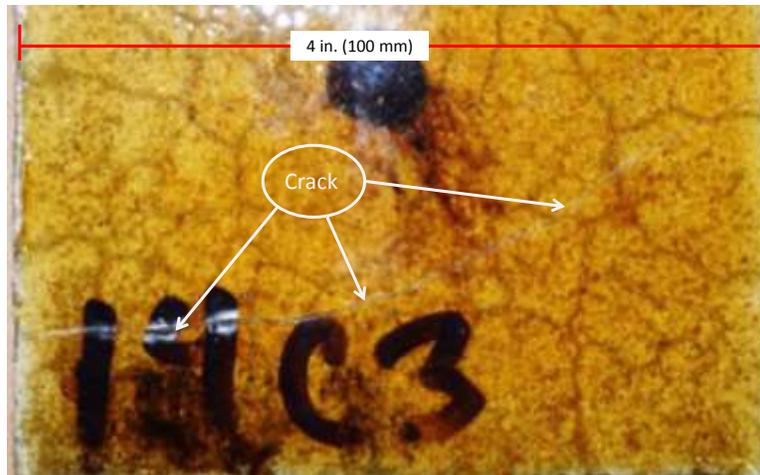


Figure 4.8 Cracking on the top surface of a MD1 specimen treated with Sealer C. This sample was subjected to moisture adjustments in an oven set at 140°F (60°C).

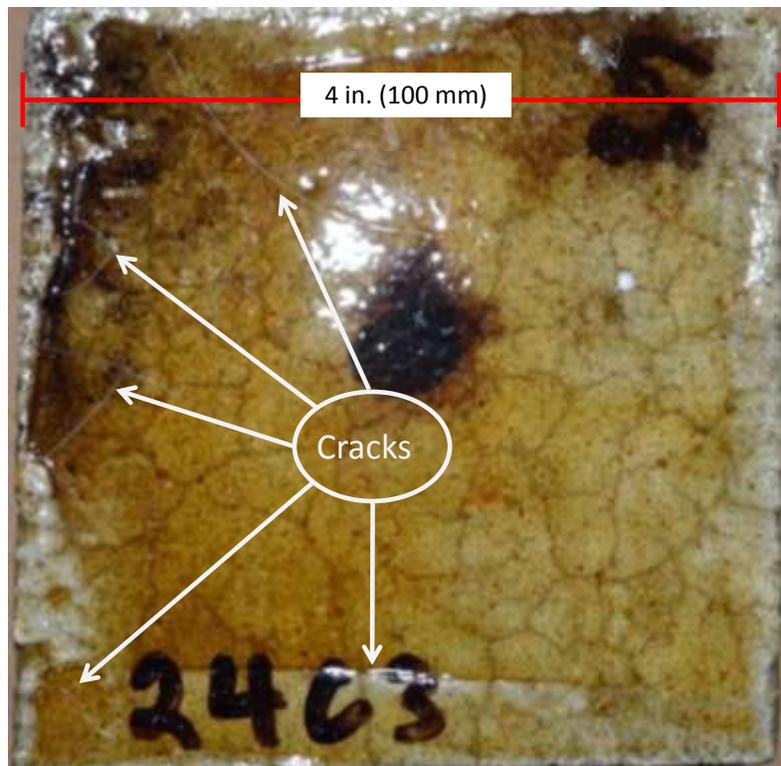


Figure 4.9 Multiple cracks on the top surface of a MD2 specimen treated with Sealer C. This sample was subjected to moisture adjustments in an oven set at 140°F (60°C).

Figure 4.10 provides a 3D cross-section of a crack. 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.

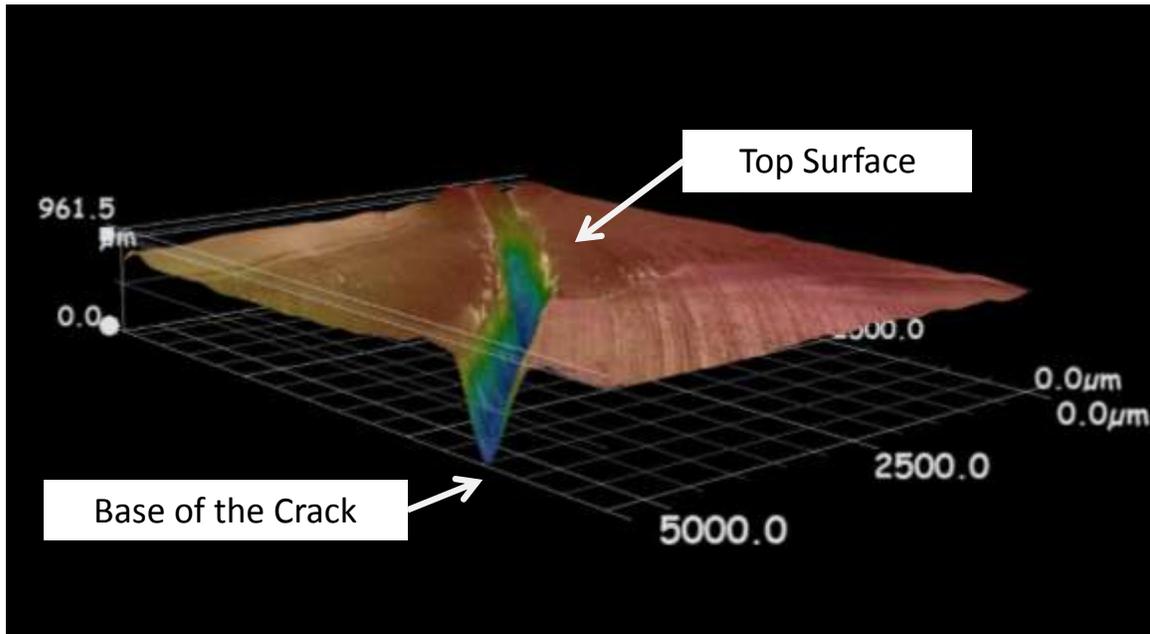


Figure 4.10 Cross-section of a crack profile in a MD1 specimen treated with Sealer C.

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 4.11 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.

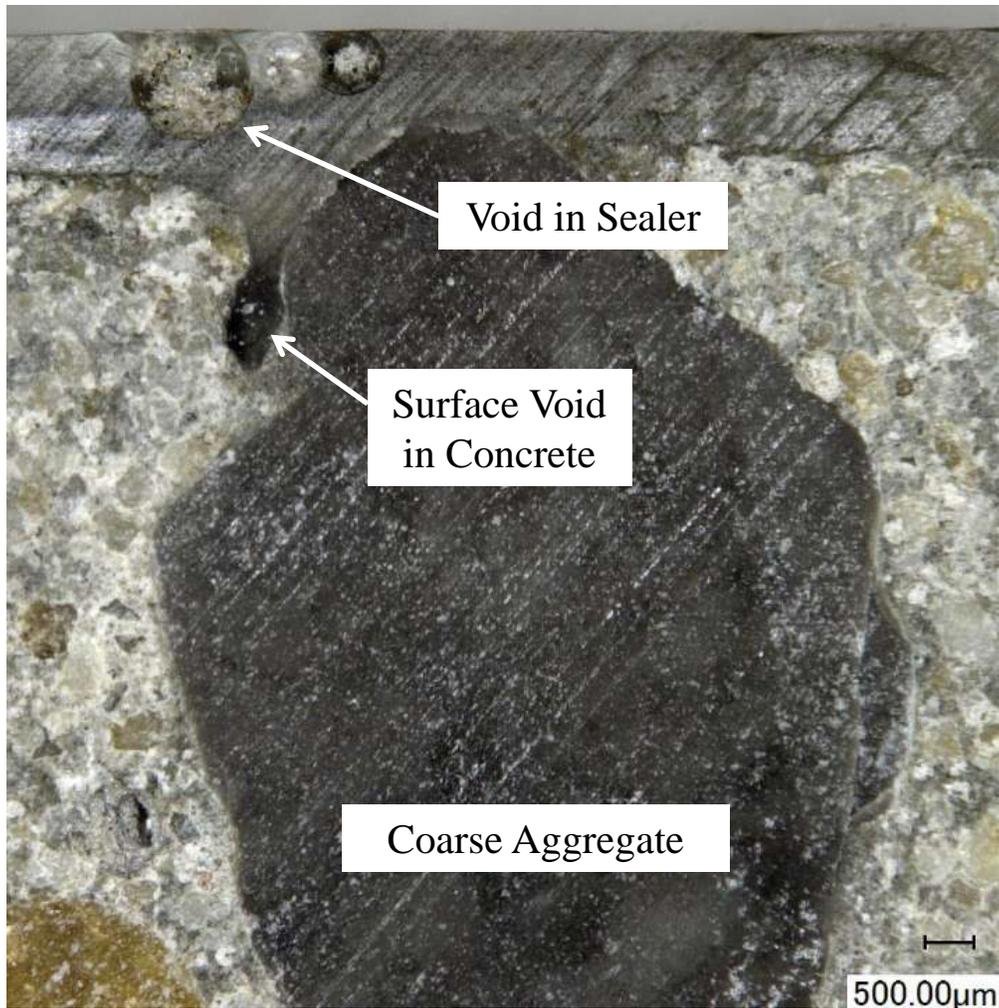


Figure 4.11 A MD2 specimen treated with Sealer D with an air void in the sealer.

Another example of the cracking that developed in Sealer C is shown in Figure 4.12. This figure shows two cracks that developed in the sealer on a specimen while it was stored in the environmental chamber. One crack was traced back to air voids that developed in the sealer, shown in Figure 4.12a and magnified in Figure 4.12b. 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 4.12c 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  $\mu\text{m}$ . 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  $\mu\text{m}$ ; this was also noticed during observations of cracks on other specimens treated with Sealer C.

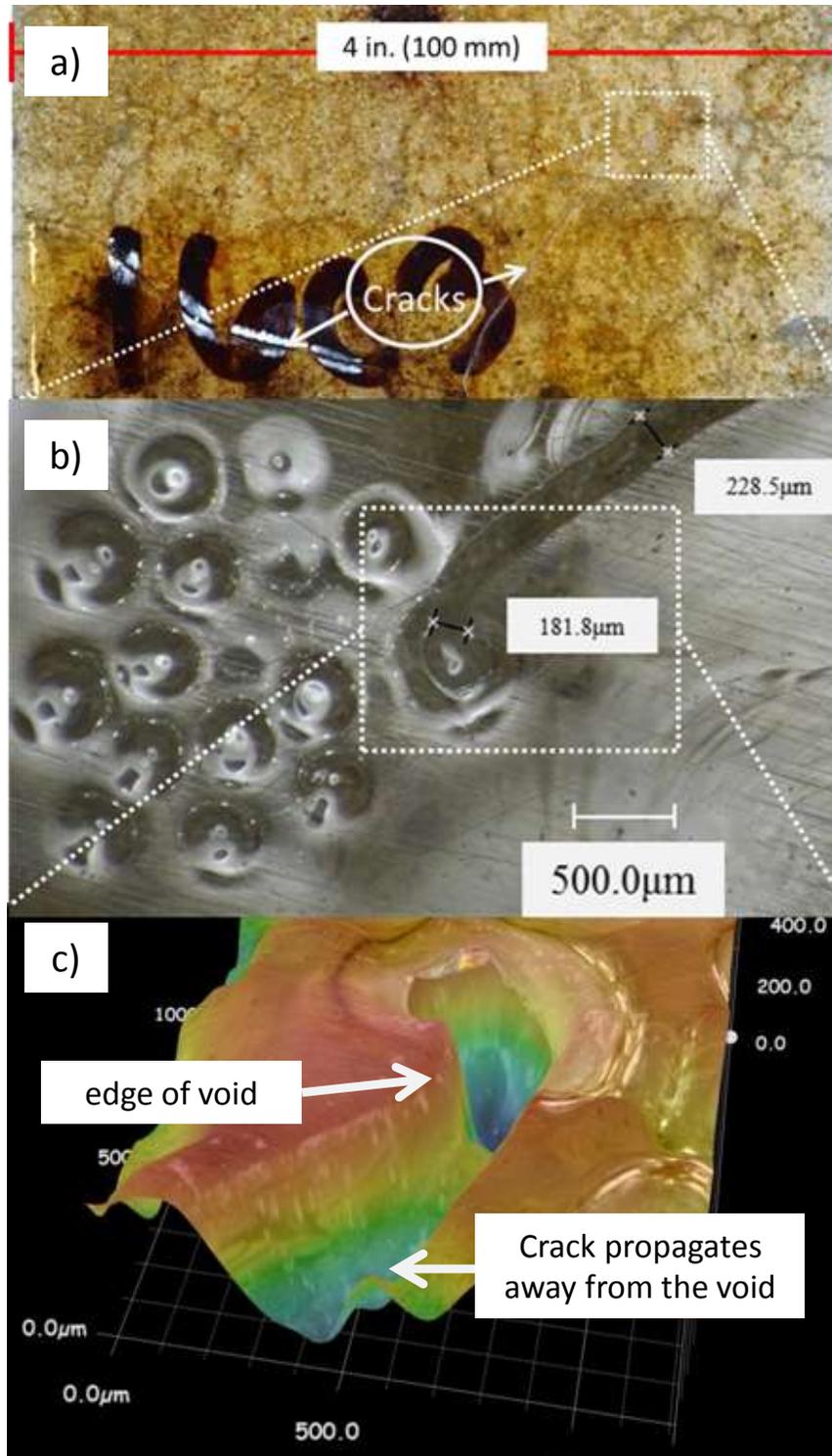


Figure 4.12 (a) Cracks on the top surface of a MD1 specimen. This sample remained in the environmental chamber, and was not placed in an oven. (b) Area of interest of the intersection an air void and a crack propagating through the sealer (c) 3D profile of the air void and crack at 50x magnification.

Figure 4.13 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 4.13a 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 4.13b shows a “U”-shaped crack which propagated 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 4.13c, crazing can be seen at the tip of the crack along with a few microvoids directly in front of the crack tip. This could be a source of where the crack locally initiated at the microvoids and easily 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  $\mu\text{m}$ . The cracks were unrelated to concrete mixture design and exposure conditions following sealer application. Further investigation is needed to confirm the cause and timing of cracking.

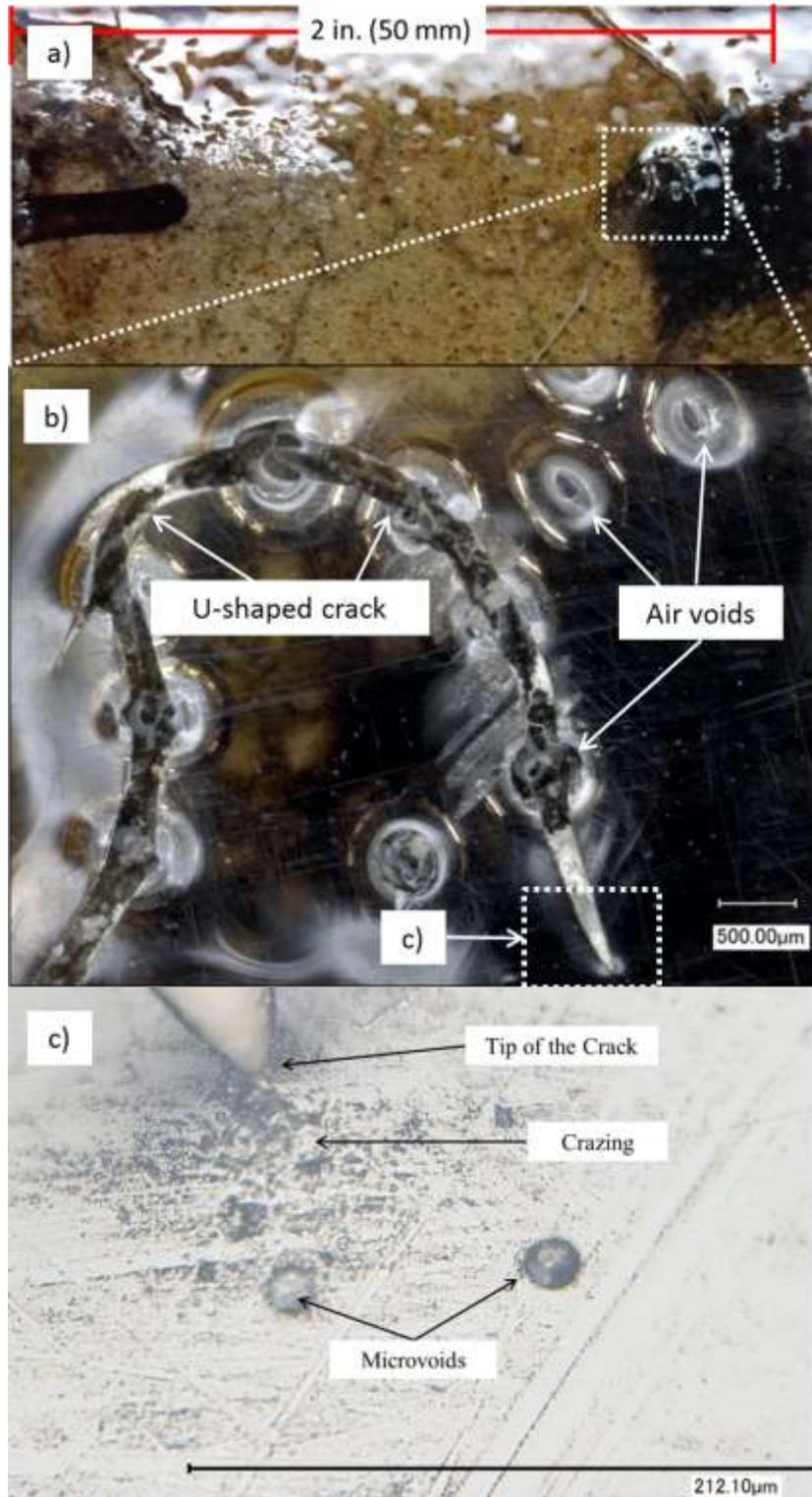


Figure 4.13 MD1 specimen treated with Sealer C that was not subjected to oven drying. (a) Cracks are seen at the center of the cube at the thickest layer of sealer. (b) “U” shaped coalescence of air voids that that developed in the sealer. (c) A crack tip and crazing, examined at 1000x magnification.

#### 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 Eqn 4-1:

$$AWS = [0.5(AWPP) + 0.5(RCP)] \quad \text{Eqn (4-1)}$$

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.

Sealer Product	Mix Design	Alberta MOT Water	NCHRP Report 244 15% NaCl		Average Weighted Score
		Waterproofing Performance (%)		Resistance to Chlorides (%)	
		5 Day	21 Day		
		Weight of Test			
25%	25%	50%			
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; however, since 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. Sealer E, Sealer C, and Sealer D consistently performed better than Sealer B and Sealer A 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  $\mu\text{m}$  thick. None of the epoxy-based products penetrated into the concrete, and Sealer A only penetrated approximately 100  $\mu\text{m}$  into the concrete.

## CHAPTER 5: CONCLUSIONS AND RECOMMENDATIONS

A synthesis of test protocols from NCHRP Report 244 Series II tests and Alberta MOT BT Series tests were 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 mixes 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 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 investigated more 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 wasn't 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  $\mu\text{m}$  into the concrete. This limited penetration depth was also observed even for a specimen immersed in Sealer A for a period of one week.

## **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 can be stored efficiently.
3. Specimen preparation and conditioning procedures detailed in sections 3.4.2 through 3.5.2 of this thesis were an effective way for measuring individual test specimen moisture levels and ensuring similar moisture contents for all specimens prior to applying the sealer 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 thesis 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 accurate results in determining the chloride content when compared with the titration results, yet XRF required less sample preparation and proved to be a faster method of measuring chlorides. XRF is recommended for use 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. 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.
3. 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.
4. 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).
5. 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 of material be removed by sandblasting, they should be prepared for post-abrasion testing by sandblasting (no grinding of the sealer) sealed specimens for an equivalent duration per side that it takes to remove a specified amount of material from an unsealed control specimen.
  - b. Similar to the NCHRP 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 ultraviolet 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 thesis.
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 which are susceptible to higher chloride exposure.

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## APPENDIX A: TITRATION PROCEDURE

The unknown chloride content in concrete can be determined through potentiometric titration by silver nitrate ( $\text{AgNO}_3$ ) 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* was 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™ 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. 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. The 0.05N AgNO<sub>3</sub> standard solution was prepared by diluting 8.4938 g of AgNO<sub>3</sub> with exactly 1 L of water in a volumetric flask. The exact normality of AgNO<sub>3</sub> was determined by titrating<sup>2</sup> 5.00 mL of 0.05N NaCl diluted in 150 mL of water against the AgNO<sub>3</sub> solution, and calculated by Eqn A-1:

$$N = \frac{0.25}{V} \quad (\text{Eqn A-1})$$

Where:

N = exact normality of silver nitrate

0.25 = milliequivalents NaCl (5.0 mL x 0.05N)

V = volume of silver nitrate used

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<sup>2</sup> This titration procedure is detailed following the sample preparation procedure.

### 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<sup>3</sup> 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 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.

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<sup>3</sup> Reasons for the blank are explained in the calculation procedure.

- 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.

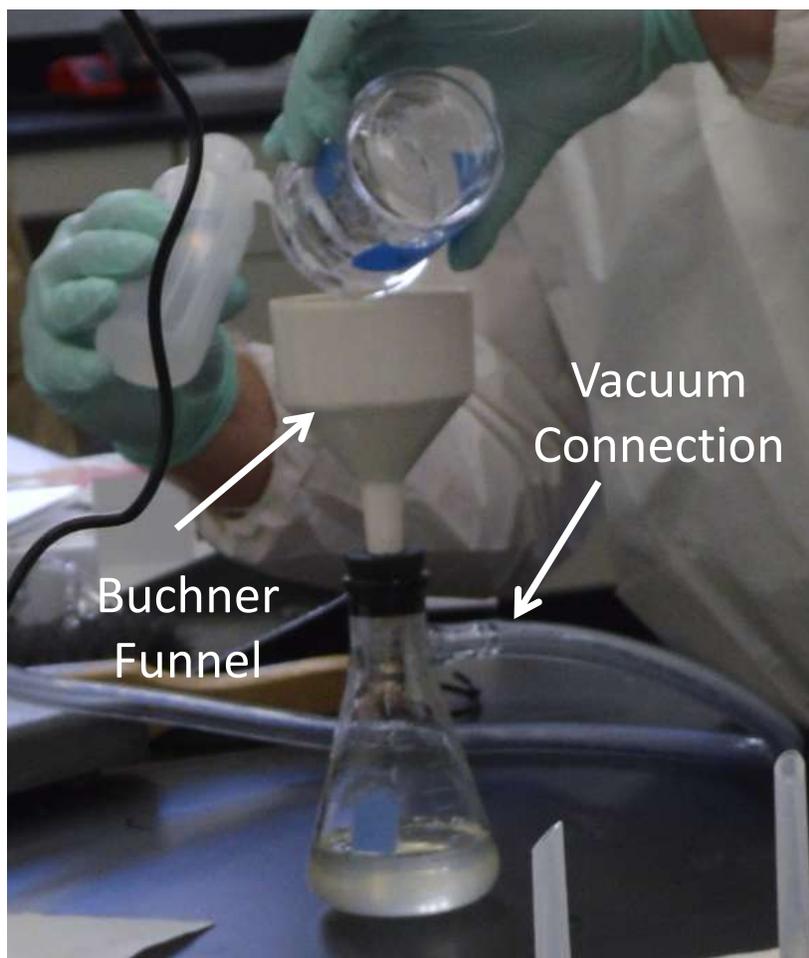


Figure A.1 Sample solution filtration setup.

### 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  $\text{AgNO}_3$ . 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.

$\text{AgNO}_3$  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  $\text{AgNO}_3$  was added. The mV readings increased as the equivalence point was approached with each successive 0.20 mL addition of  $\text{AgNO}_3$ , 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  $\text{AgCl}$  can be seen in the solution.

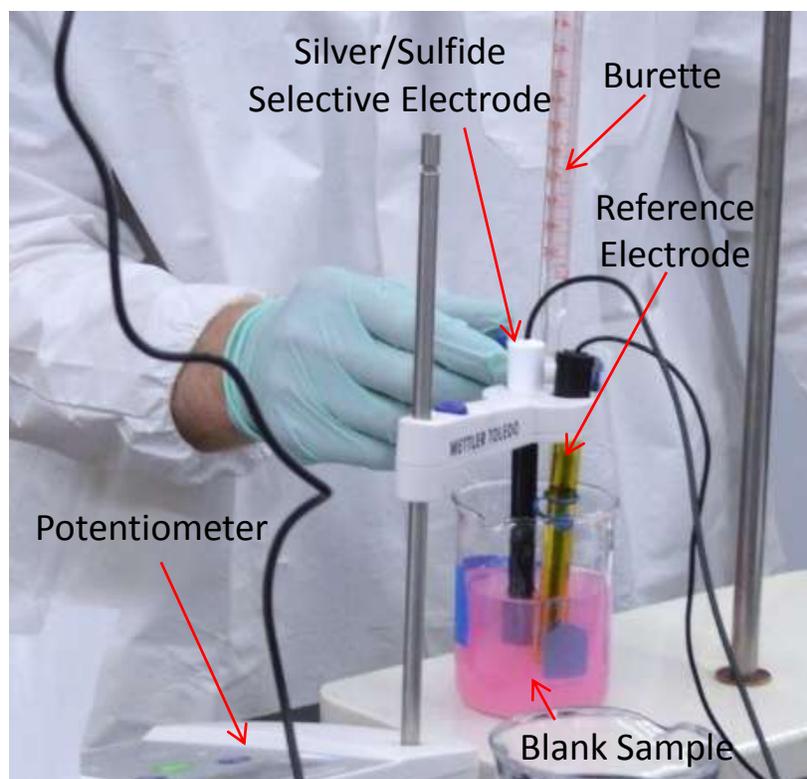


Figure A.2 The configuration for the silver/sulfide selective electrode, reference electrode, and the burette for potentiometric titration of a blank sample.

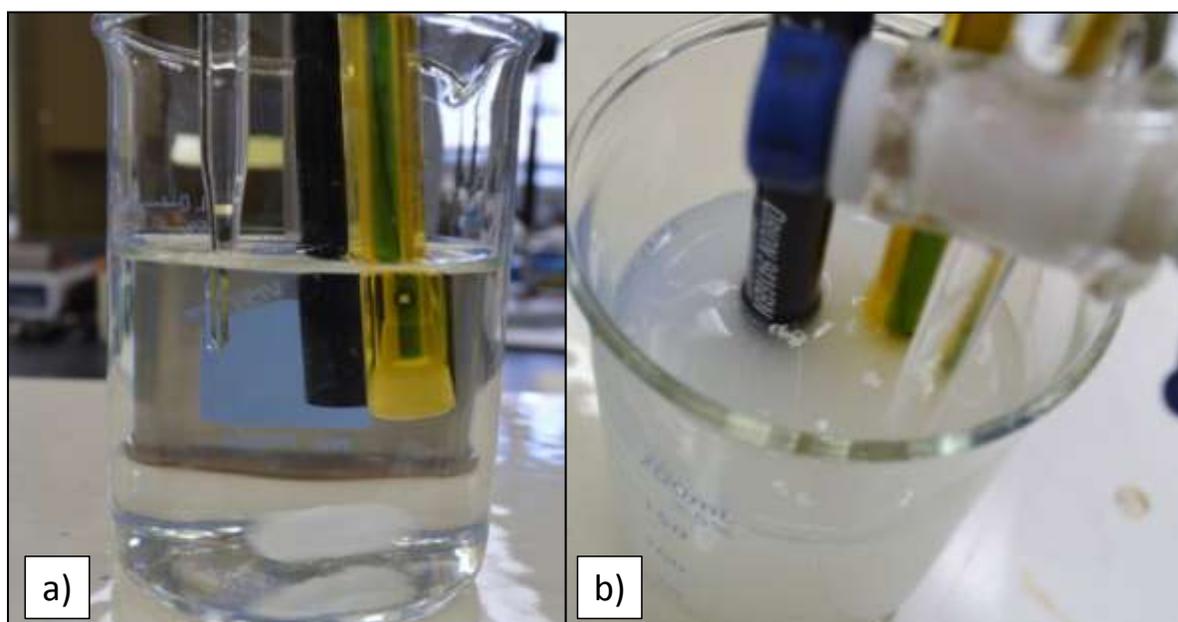


Figure A.3 The burette and electrodes orientation in a sample (a) prior to  $0.05\text{N AgNO}_3$  additions and (b) after reaching the equivalence point signified by  $\text{AgCl}$  precipitate formation.

#### 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<sub>3</sub> 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<sub>3</sub> 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<sub>3</sub> increments and corresponding mV readings. A third column, the 1<sup>st</sup> differential, was calculated showing the difference of subsequent mV readings. In the 4<sup>th</sup> column, the calculated 2<sup>nd</sup> differential is the absolute value of the difference of successive 1<sup>st</sup> differentials. The 1<sup>st</sup> differential will increase as it approaches the equivalence point, and will decrease once it has passed the equivalence point. The successive 2<sup>nd</sup> differentials, corresponding to the maximum 1<sup>st</sup> differential, will be used to interpolate the volume of AgNO<sub>3</sub> 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 plotted against the amount of AgNO<sub>3</sub> used. Using Table A.1 and referring to ASTM C114-11b (2013), the equivalence point (*E*) is calculated using Eqn A-2:

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

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

AgNO <sub>3</sub> Added (mL)	Millivolt reading (mV)	Change in Potential ( $\Delta V$ )	2 <sup>nd</sup> Change in Potential ( $\Delta(\Delta V)$ )
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		

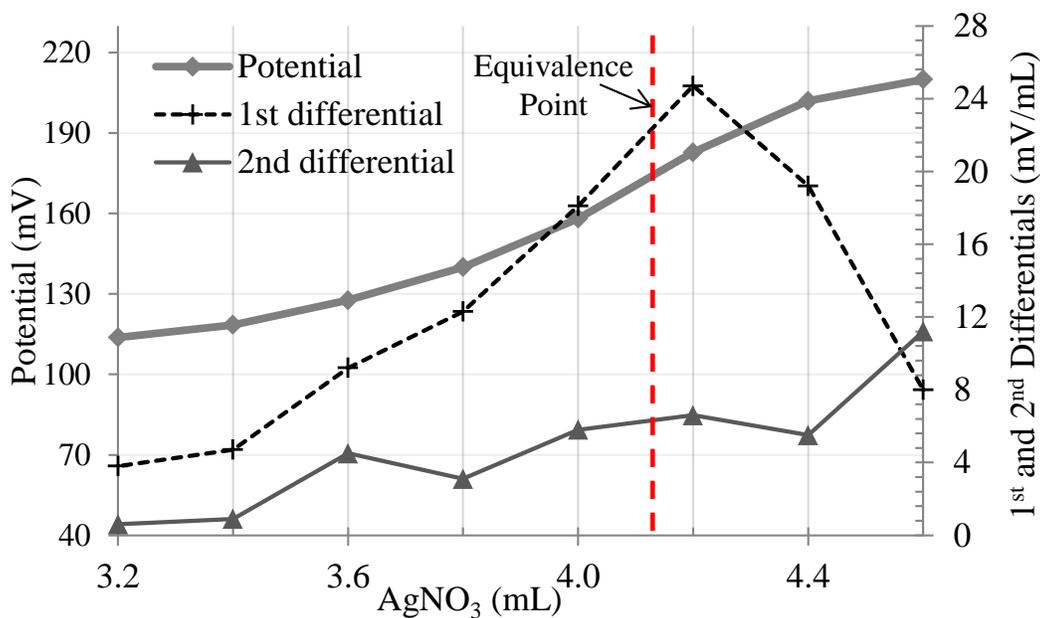


Figure A.4 Graphical representation of the determination of the equivalence point located between 4.0 and 4.2 mL of AgNO<sub>3</sub> using the values of the 2<sup>nd</sup> differential corresponding to the greatest change in slope of the potential (the largest 1<sup>st</sup> differential).

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

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

Where:

$V_1$  = mL of 0.05N  $\text{AgNO}_3$  titrated to reach the equivalence point for the sample

$V_2$  = mL of 0.05N  $\text{AgNO}_3$  titrated to reach the equivalence point for the blank sample

$N$  = calculated exact normality of  $\text{AgNO}_3$

$W$  = mass of sample (g)