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**NEXTGEN Concrete - Tests of the Future: Chloride  
Diffusion**

**Deliverable #6: Final Report**



**PRIME CONTRACTOR**

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<p>16. Abstract</p> <p>FDOT is seeking to maximize the durability of newly constructed structural elements. As part of this goal, FDOT needs to do a better job assessing concrete mixes for specific structural applications. This assessment needs to adapt to the ever-changing physical, chemical, and market issues associated with concrete's constituent ingredients. FDOT needs to allow the industry to use innovative new materials as well as existing materials in new combinations to optimize the performance of concrete. We need to apply to our mixes a series of screening tests that robustly assess their durability in specific FDOT applications and environments. Specifically, we need tests that measure a mix's chloride and sulfate durability. These are key, materials-related factors that influence the durability of our new structures.</p> <p>In light of this, a research project, BEE02 "NEXTGEN Concrete - Tests of the Future: Chloride and Sulfate Durability," was developed to establish thresholds for several screening test methods, including AASHTO T358 (SR), AASHTO TP119 (BR), and Modified ASTM C1202 for sulfate permeability. In addition to the test methods included in BEE02, it is also important to investigate chloride diffusion so that the SR and BR test results can be verified and validated with direct chloride diffusion results. Moreover, the chloride diffusion coefficients can be used as inputs for service life models. Thus, this project was developed as a companion project to BEE02 to investigate the chloride diffusion rates using both bulk diffusion (ASTM C1556) and rapid migration (NT Build 492) tests. Based on testing results, the target diffusion coefficients were derived based on a design service life of 75 years for different structural components. The correlations between resistivity and bulk diffusion coefficients were also established. Using this information, the thresholds were recommended for both SR and BR tests for the extremely aggressive environment.</p>			
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## Executive Summary

FDOT is seeking to maximize the durability of newly constructed structural elements. As part of this goal, FDOT needs to do a better job assessing concrete mixes for specific structural applications. This assessment needs to adapt to the ever-changing physical, chemical, and market issues associated with concrete's constituent ingredients. FDOT needs to allow the industry to use innovative new materials as well as existing materials in new combinations to optimize the performance of concrete. We need to apply to our mixes a series of screening tests that robustly assess their durability in specific FDOT applications and environments. Specifically, we need tests that measure a mix's chloride and sulfate durability. These are key materials-related factors that influence the durability of our new structures.

In light of this, a research project, BEE02 "NEXTGEN Concrete - Tests of the Future: Chloride and Sulfate Durability," was developed to establish thresholds for several screening test methods including AASHTO T358 for surface resistivity (SR), AASHTO TP119 for bulk resistivity (BR), and Modified ASTM C1202 for sulfate permeability. In addition to the test methods included in BEE02, it is also important to investigate chloride diffusion so that the SR and BR test results can be verified and validated with direct chloride diffusion results. Moreover, the chloride diffusion coefficients can be used as inputs for service life models. Thus, this project was developed as a companion project to BEE02 to investigate the chloride diffusion rates using both bulk diffusion (ASTM C1556) and rapid migration (NT Build 492) tests.

### **RESEARCH OBJECTIVES**

The two objectives of this project are the following:

1. To investigate and determine chloride diffusion rates using bulk diffusion (ASTM C1556) and rapid migration (NT Build 492) tests for concrete mixes intended for use in an extremely aggressive chloride environment.
2. Establish a correlation between the chloride diffusion test results and the bulk (TP 119) and surface (T 358) resistivity results obtained in contract BEE02.

## **CONCLUSIONS AND RECOMMENDATIONS**

In order to achieve these two objectives, the research team conducted both bulk diffusion (BD) and rapid migration test (RMT) tests for 31 selected mix designs. Using the Fick's Second Law service life model, the target diffusion coefficients were derived based on a design service life of 75 years for different structural components. There are several key parameters in the concrete service life model, including the critical concentration at depth and time, surface chloride concentration, depth, time for diffusion, and apparent diffusion coefficient. For this study, the extremely aggressive environment condition is considered, and the surface chloride concentration is assumed to be 2000 ppm as specified in FDOT SDG. In this study, it is assumed that the propagation period of 20 years for epoxy-coated steel and 6 years for all other types of rebars. Then the required initiation period to reach 75 years of service life will be 55 years for epoxy-coated steel and 69 years for all other types of rebars. Chloride concentration necessary to initiate corrosion is another important parameter. The research team is using the assumptions that Life 365 (2020) uses, which are black steel = 0.05% wt. concrete, epoxy-coated = 0.05%, and stainless steel = 0.5%. The depth  $t$  depends on the type of structure and its respective concrete cover.

The research team has investigated the relationship between the chloride migration coefficients  $D_{nssm}$  derived from the RMT and the SR results after 28 days of curing. The fitted power-law regression curve showed an  $R^2$  value of 0.8787, indicating a strong inverse correlation between SR and  $D_{nssm}$ . Similar correlations were also observed between BR and  $D_{nssm}$ . The correlation between apparent diffusion coefficients from BD tests and resistivity results from SR and BR tests were also investigated. The coefficients of determination ( $R^2$ ) are 0.8576 and 0.87 for SR vs.  $D_a$  and BR vs.  $D_a$ , respectively, implying correlation coefficients of 92.6% and 93.3%, which indicate good correlations between them.

Based on the testing results, the target diffusion coefficients, and the correlations between resistivity (SR and BR) and BD results, the following thresholds are recommended for the structures exposed to an extremely aggressive environment:

- a. For structures with 3" cover, use 98 k $\Omega$ -cm and 26 k $\Omega$ -cm for SR and BR, respectively.
- b. For structures with 4" cover, use 52 k $\Omega$ -cm and 19 k $\Omega$ -cm for SR and BR, respectively.
- c. For structures with 4.5" cover, use 41 k $\Omega$ -cm and 16 k $\Omega$ -cm for SR and BR, respectively.

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# 1 Introduction

## 1.1 Research Problem and Background

FDOT is seeking to maximize the durability of newly constructed structural elements. As part of this goal, FDOT needs to do a better job assessing concrete mixes for specific structural applications. This assessment needs to adapt to the ever-changing physical, chemical, and market issues associated with concrete's constituent ingredients. FDOT needs to allow the industry to use innovative new materials as well as existing materials in new combinations to optimize the performance of concrete. We need to apply to our mixes a series of screening tests that robustly assess their durability in specific FDOT applications and environments. Specifically, we need tests that measure a mix's chloride and sulfate durability. These are key, materials-related factors that influence the durability of our new structures.

In light of this, a research project, BEE02 "NEXTGEN Concrete - Tests of the Future: Chloride and Sulfate Durability," was developed to establish the thresholds for several screening test methods, including AASHTO T358 for surface resistivity (SR), AASHTO TP119 for bulk resistivity (BR), and Modified ASTM C1202 for sulfate permeability. In addition to the test methods included in BEE02, it is also important to investigate chloride diffusion so that the SR and BR test results can be verified and validated with direct chloride diffusion results. Moreover, the chloride diffusion coefficients can be used as inputs for service life models. Thus, this project will serve as a companion project to BEE02 to investigate the chloride diffusion rates using both bulk diffusion (ASTM C1556) and rapid migration (NT Build 492) tests.

## 1.2 Research Objectives

The two objectives of this project are the following:

1. To investigate and determine chloride diffusion rates using bulk diffusion (ASTM C1556) and rapid migration (NT Build 492) tests for concrete mixes intended for use in an extremely aggressive chloride environment.

2. Establish a correlation between the chloride diffusion test results and the bulk (TP 119) and surface (T 358) resistivity results obtained in contract BEE02.

## 2 Literature Review

### 2.1 Correlations between Chloride Diffusion Rate and Surface and Bulk Resistivity Results for Different Mix Designs.

Several studies have investigated correlations between bulk resistivity and chloride diffusion rate in concrete. In theory, the Nernst-Einstein equation can be used to describe the relationship between the diffusivity of ion species  $i$  and their partial conductivity (Lu, 1997):

$$D_i = \frac{RT\sigma_i}{Z_i^2 F^2 C_i} \quad \text{Eq. 1}$$

where  $D_i$  is the diffusivity of ion  $i$  ( $\text{m}^2/\text{s}$ );  $\sigma_i$  is the partial conductivity of ion  $i$  ( $\text{m/s}$ );  $R$  is the gas constant ( $8,314 \text{ J/mol}$ );  $T$  is absolute temperature ( $\text{K}$ );  $Z_i$  is the charge of ion  $i$ ;  $F$  is Faraday's constant ( $96,500 \text{ Coulombs/mole}$ ); and  $C_i$  is the concentration of ion  $i$  ( $\text{mol}/\text{m}^3$ ).

Andrade (2002) presented his findings on concepts of the chloride diffusion coefficient, particularly, he summarized the relation between rapid chloride migration coefficient ( $D_{RCM}$ ) and electrolytic resistivity ( $\rho_{TEM}$ ) as follows:

$$D_{RCM} = A\rho_{TEM}^b \quad \text{Eq. 2}$$

where  $A$  and  $b$  are constants based on testing results.

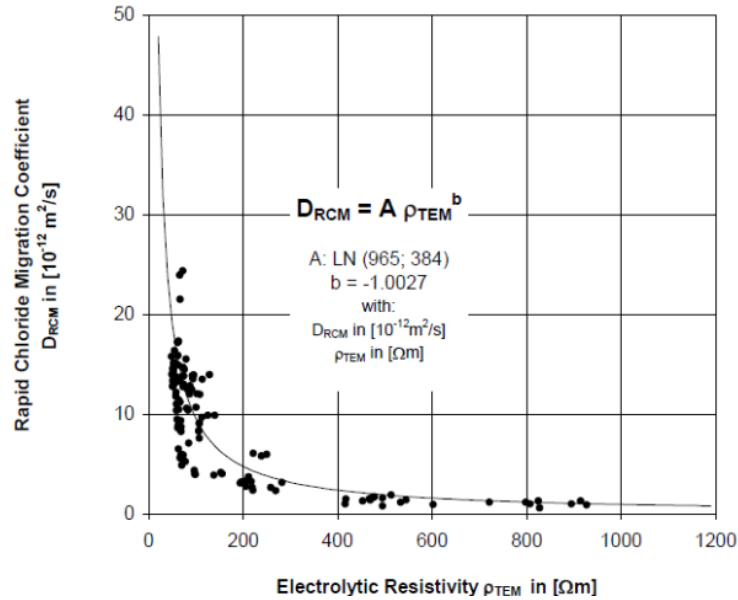


Figure 1. Relation between a  $D_{RCM}$  and bulk resistivity (Andrade, 2002)

Based on the measured data in the laboratory as shown in Figure 2, Han (2004) proposed an exponential equation to correlate between rapid chloride migration (RCM) and the two-point electrode method (TEM) in Eq. (3):

$$RCM = ae^{bTEAM} \quad \text{Eq. 3}$$

where the empirical model parameters  $a$  and  $b$  can be determined by means of fitting the RCM and TEM with Eq. (3). This equation can also converted into a linear function by taking the logarithm on both sides of Eq. (4):

$$y = A + Bx \quad \text{Eq. 4}$$

where  $y = \ln(RCM)$  and  $x = \ln(TEAM)$ . The model parameters  $A$  and  $B$  can be determined based on the testing data.

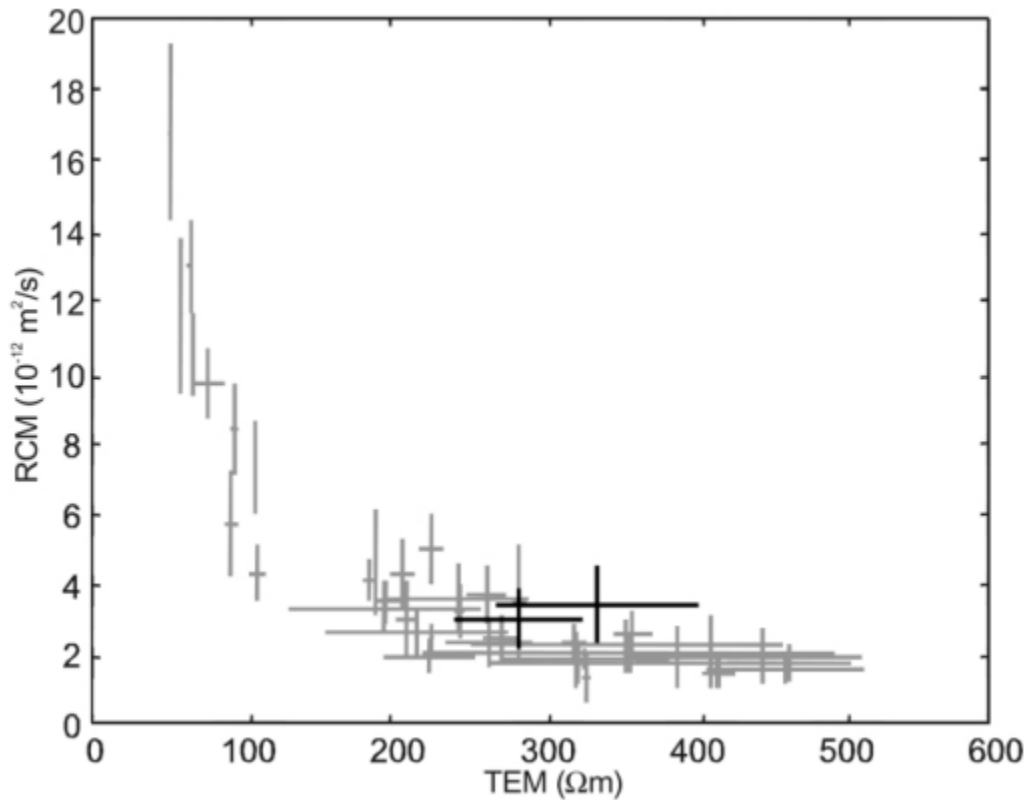


Figure 2. Overview of the measured data in the laboratory with the measuring uncertainty ( $\pm 1$  standard deviation) (Han, 2004).

Bagheri and Zanganeh (2012) conducted a comparative study regarding different rapid tests for the evaluation of chloride resistance of concrete. They have studied the correlation between the electrical conductivity (the reciprocal of the electrical resistance) and chloride diffusivity obtained from the rapid chloride migration test (RCMT) according to NT Build 492. As shown in Figure 3, high correlation coefficients of 0.93 and 0.94 were obtained for the ages of 28 and 90 days, indicating a strong linear correlation between electrical conductivity and chloride diffusivity.

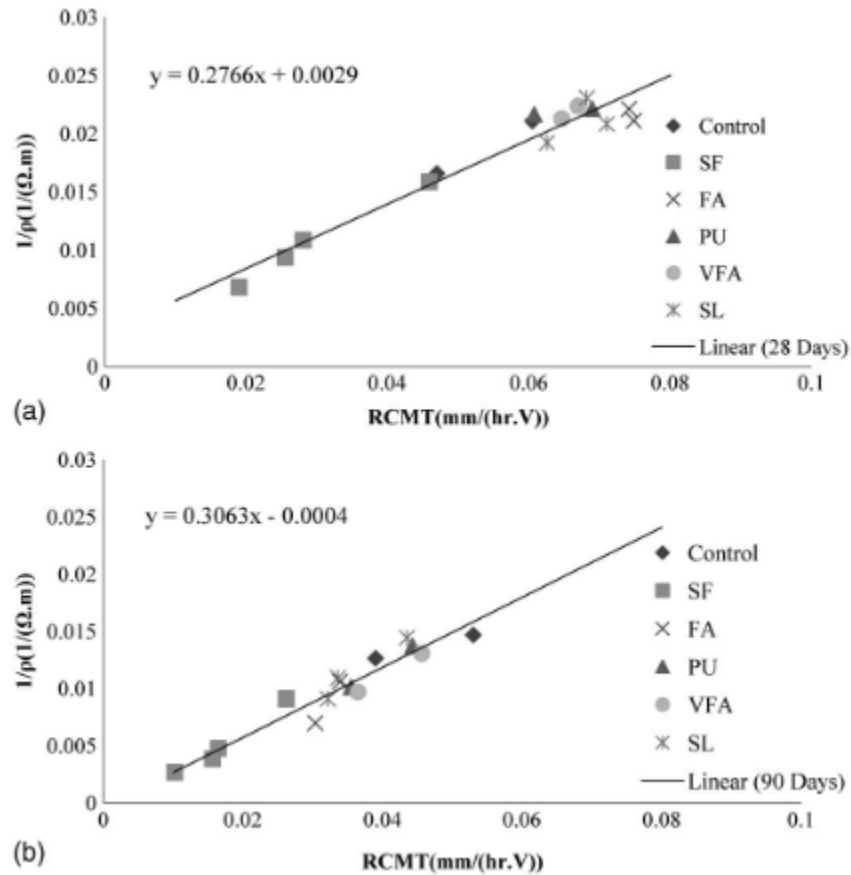


Figure 3. Relationship between the results of the RCMT and electrical conductivity tests at (a) 28 days; (b) 90 days (Bagheri and Zanganeh, 2012).

Sengul (2014) also derived the relationship between the chloride diffusivity (NT Build 492, 1999) and the electrical resistivity (ASTM C 1760). Figure 2 shows this relationship based on the test results of 29 mixes at various ages. A very good relationship ( $R = 0.97$ ) was observed between electrical resistivity and chloride diffusivity with a standard error of 0.016. If using the electrical conductivity, which is the reciprocal of the resistivity, a linear relationship can be established between conductivity and chloride diffusivity as shown in Figure 5. It is worth noting that these relationship equations are empirical equations based on certain data set which can't be used directly for other mixes.

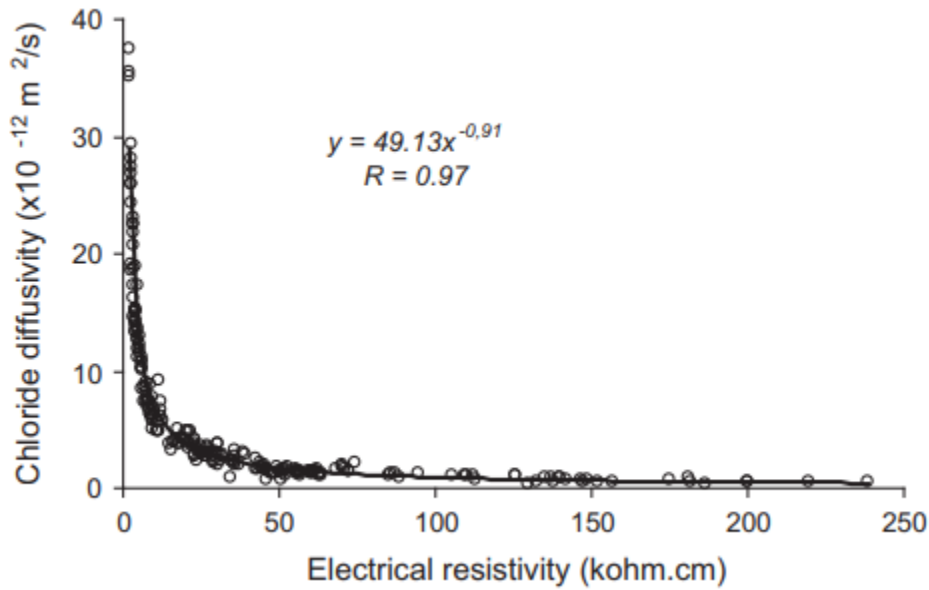


Figure 4. Relationship between chloride diffusivity and electrical resistivity (Sengul, 2014).

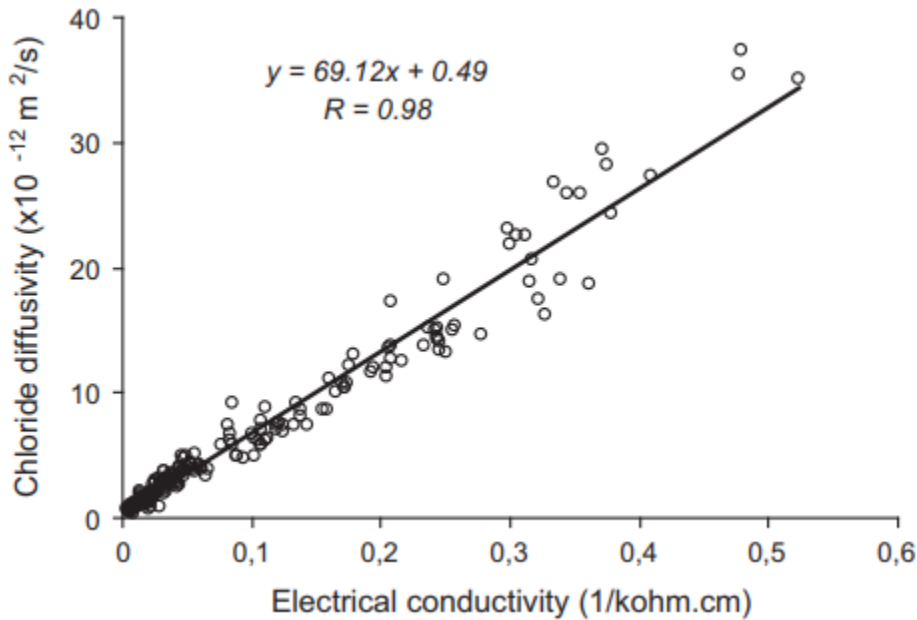


Figure 5. Relationship between chloride diffusivity and electrical conductivity (Sengul, 2014).

Noort et al. (2016) performed investigations of the resistivity and chloride migration coefficient ( $D_{RCM}$ ) for slag cement-based concretes based on the tests of specimens taken from 47 different concrete mixes. As shown in Figure 6b, in a plot of the log scale, the linear correlation is

observed with a direction coefficient of approximately -1. Thus, in Figure 6c, a linear correlation is observed between conductivity and  $D_{RCM}$ .

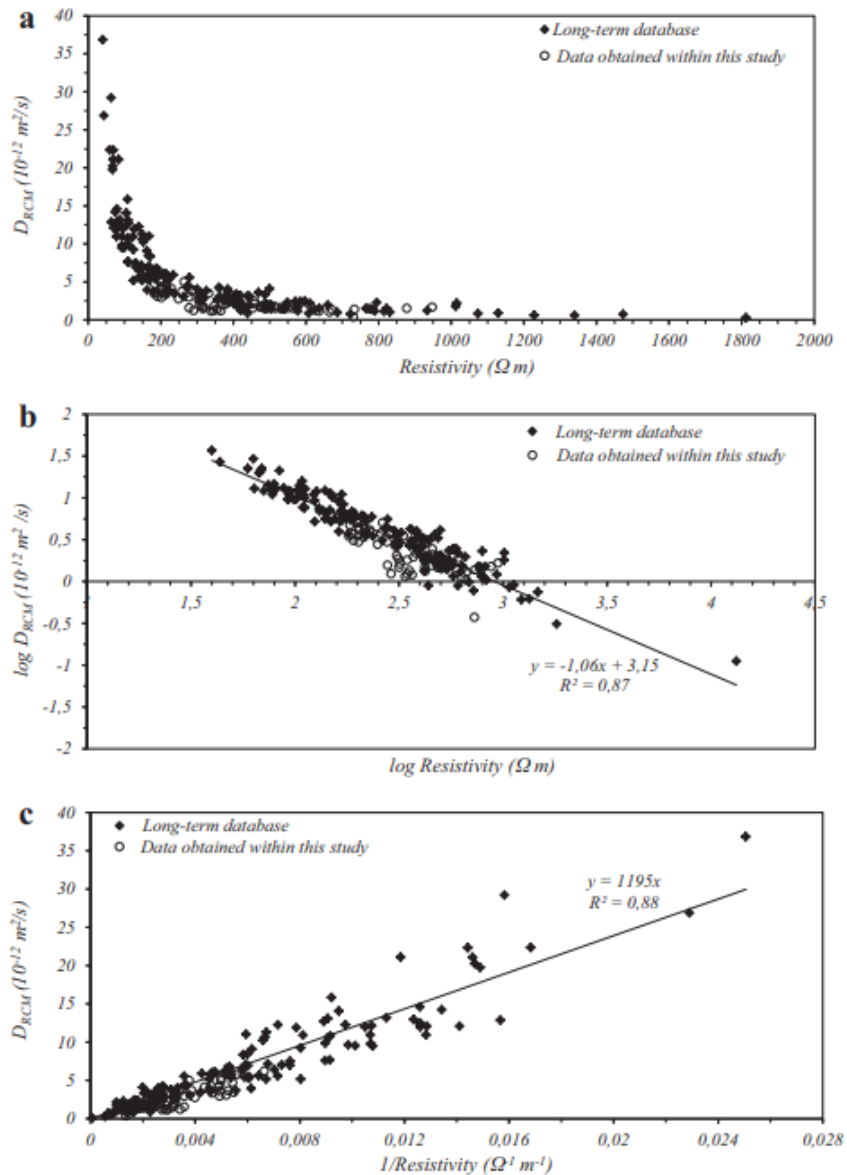


Figure 6.  $D_{RCM}$ -values and the associated resistivities measured in the ENCI concrete laboratory over the last few years. (a)  $D_{RCM}$  vs. resistivity; (b)  $\log(D_{RCM})$  vs.  $\log$ (resistivity), and (c) A linear correlation between  $D_{RCM}$  and  $1/resistivity$  (i.e. conductivity). (Noort et al., 2016).

Fewer studies have been performed to evaluate the correlation of the surface resistivity (four-point electrical resistivity) with the chloride diffusion coefficient in different types of concrete.

However, a linear relationship was also observed between conductivity from surface resistivity tests and chloride diffusion coefficient.

Hamilton et al. (2007) studied the permeability of concrete, especially the comparison of conductivity and diffusion methods. In the lab study, the surface resistivity test was conducted using moist curing (100% humidity) and lime curing (saturated  $\text{Ca}(\text{OH})_2$  solution) at 14, 28, 56, 91, 182, 364, 454, and 544 days. Then the results were compared to their respective 364-Day bulk diffusion results. Figure 7 and Figure 8 show the correlation between SR conductivity results and bulk diffusion results at 28 days and 91 days for moist cured and lime cured, respectively. Power regression was selected to mathematically fit the curve. Lime cured specimens showed a slightly better correlation with the conductivity results.

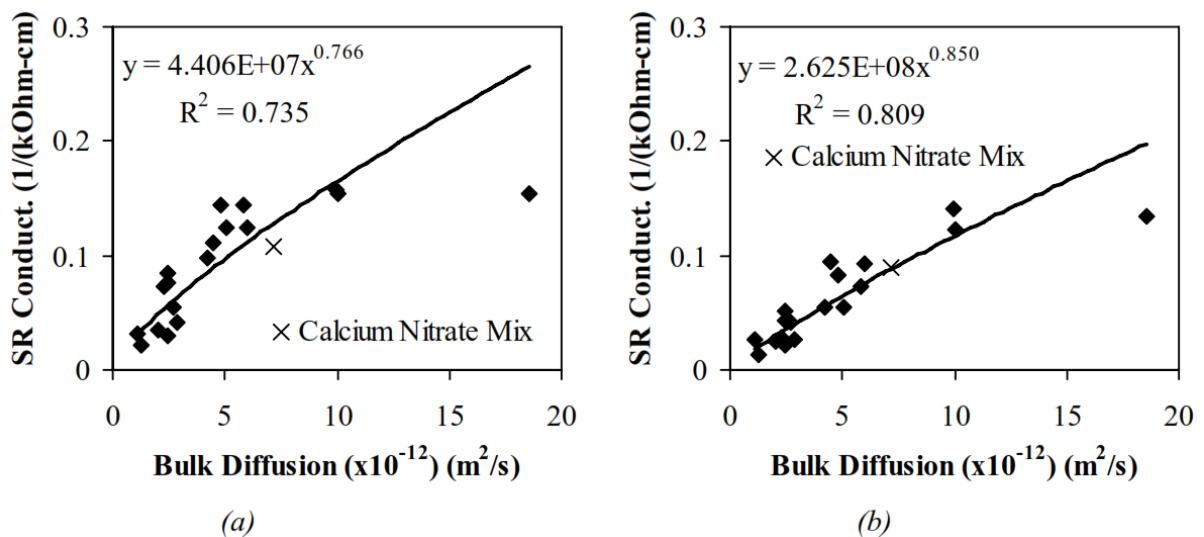


Figure 7. 364-day bulk diffusion vs. SR (moist cured) conductivity at (a) 28 days and (b) 91 days (Hamilton et al., 2007).

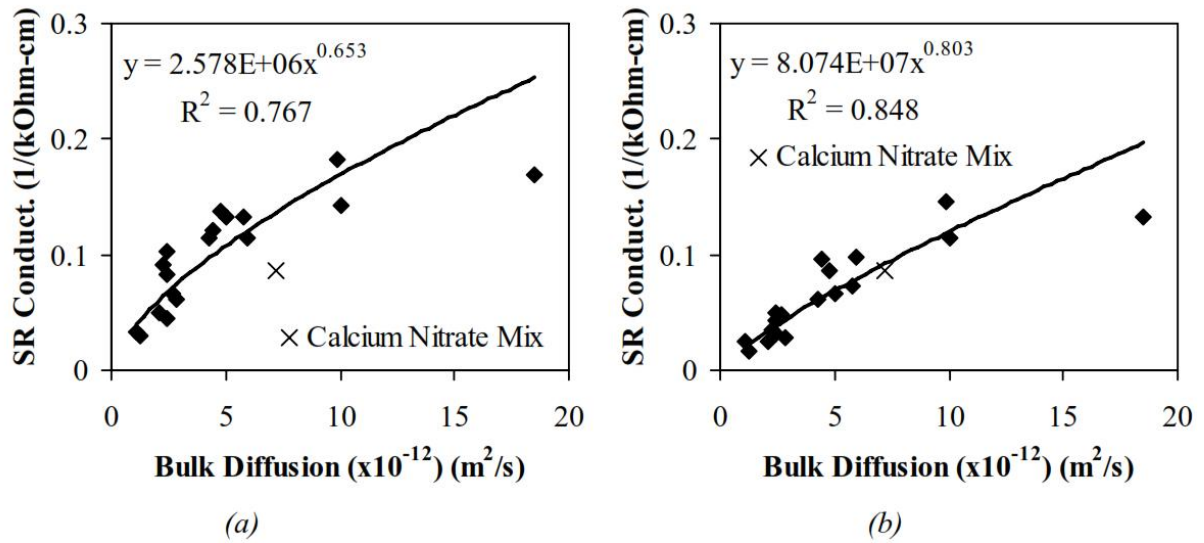


Figure 8. 364-day bulk diffusion vs. SR (lime cured) conductivity at (a) 28 days and (b) 91 days (Hamilton et al., 2007).

The correlation between RMT diffusion and bulk diffusion was examined. As shown in Figure 9, linear regression analysis was performed and the correlation at 91 days was better than the correlation at 28 days.

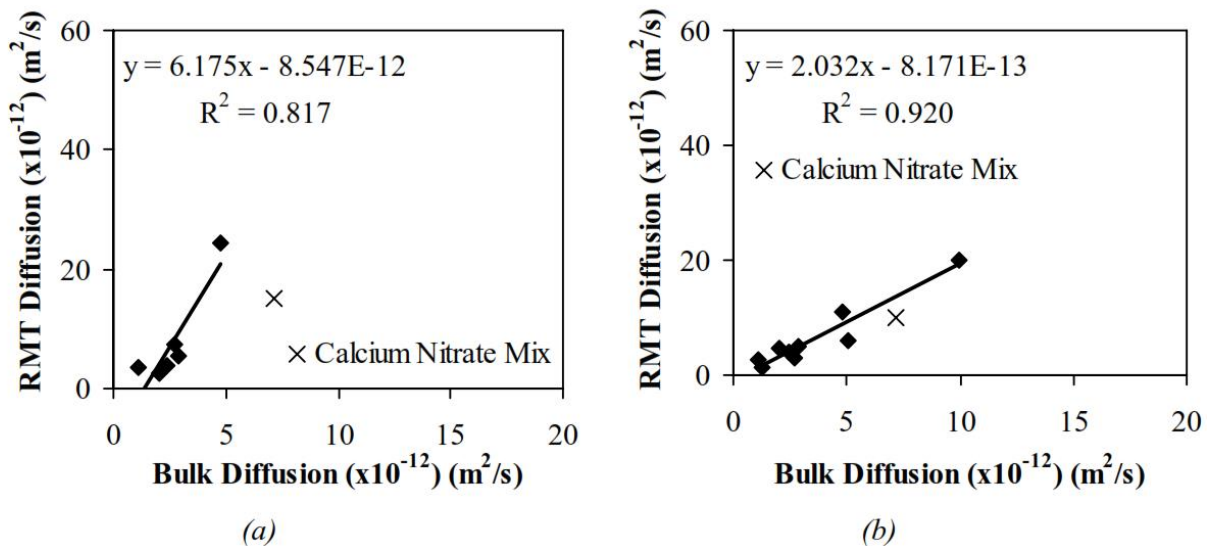


Figure 9. 364-day bulk diffusion vs. RMT diffusion at (a) 28 days and (b) 91 days (Hamilton et al., 2007).

Using a modified Nernst-Einstein equation, Backe et al. (2001), Sengul and Gjrrv (2008), and Baroghel-Bouny et al. (2011) used the following equations to describe the correlation between effective chloride diffusion coefficient ( $D_{eff}$ ) and the electrical resistivity ( $\rho'$ ):

$$F = \frac{D_0}{D_{eff}} = \frac{\sigma_0}{\sigma} \quad \text{Eq. 5}$$

$$D_{eff} = \frac{D_0}{\sigma_0} \sigma = \frac{D_0}{\sigma_0} \frac{1}{\rho'} = \frac{B}{\rho'} \quad \text{Eq. 6}$$

where  $F$  is the formation factor,  $D_0$  is free chloride ion diffusion coefficient in the pore solution,  $\sigma_0$  is the conductivity of the pore solution,  $\sigma$  is the conductivity of the porous material, and the coefficient  $B$  is dependent on the ionic concentrations of the pore solution. Using the value of  $\sigma_0 = 11.87 \Omega^{-1} m^{-1}$  and  $D_0 = 1.484 \times 10^{-9} m^2 s^{-1}$ , Baroghel-Bouny et al. (2011) derived the following equation:

$$D_{eff} \approx \frac{125}{\rho'} \quad \text{Eq. 7}$$

Liu (2012) conducted a comprehensive study on the effects of accelerated curing on the resistivity, diffusivity, and compressive strength of concrete with high volume pozzolans. In this study, he investigated the correlation between the non-steady-state migration coefficients ( $D_{nssm}$ ) from the RCM TEST (NT Build 492) and the surface resistivity results. Figure 10 shows the correlation between  $D_{nssm}$  and the 21 °C resistivity ( $\rho_{21}$ ). Based on the testing results, the following equation was derived:

$$D_{nssm} \approx 105.5 \frac{1}{\rho_{21}} \quad \text{Eq. 8}$$

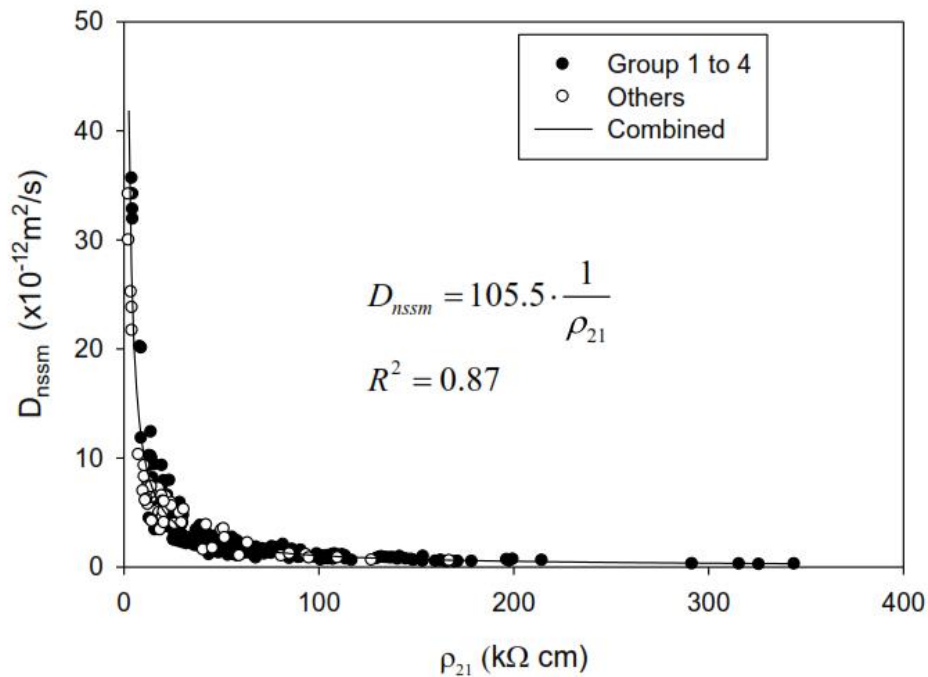


Figure 10. Correlation between and on specimens from Group 1 and Group 4 and other projects (Liu, 2012).

Similarly, Presuel-Moreno (2016), and Homayoonmehr et al. (2022) also observed a linear correlation between the chloride migration test results and the surface conductivity results.

Azarsa & Gupta (2017) performed a review on the electrical resistivity of concrete for durability evaluation. In their review, they reviewed the relationship between concrete electrical resistivity and its durability characteristics including chloride diffusivity and corrosion of reinforcement. They discussed the correlation between electrical resistivity and various durability characteristics of concrete, with a specific focus on chloride diffusion. It highlights that the transport of aggressive ions, such as chloride, through the concrete microstructure plays a crucial role in the degradation of reinforced concrete structures. The ability of concrete to resist the transfer of ions is closely related to its electrical resistivity. The paper summarizes findings from different studies that demonstrate the inverse relationship between concrete resistivity and chloride ingress. Lower resistivity values indicate areas where chloride penetration is expected to be faster. It also mentions the proposed relationship between resistivity and chloride threshold values, indicating the risk of chloride penetrability based on the charge passed.

## 2.2 Concrete Service Life Models based on Chloride Diffusion Rates

Concrete is a porous material with pores of various diameters, sizes, and lengths. Through the connected pores, chloride ions, water, gases, and other aggressive ions can penetrate into the concrete. From the viewpoint of structural design, service life is often defined as the time of damage initiation, especially in the case of chloride initiated pitting corrosion. In the AASHTO LRFD Bridge Design Specifications, service life is defined as the period of time on which the statistical derivation of transient loads is based. In ASTM E632-96, service life is defined as the period of time after installation during which all properties exceed the minimum acceptable values when routinely maintained.

Figure 11 illustrates the relationship between environmental load and concrete resistance. Concrete resistance decreases along with time due to concrete cracks, spalling, and strength degradation. The penetration of sulfate and chloride increases the environmental load. Therefore, the possibility of concrete environmental resistance failure increases over time. The end of the service life is when the accumulated damage in the bridge materials exceeds the tolerance limit.

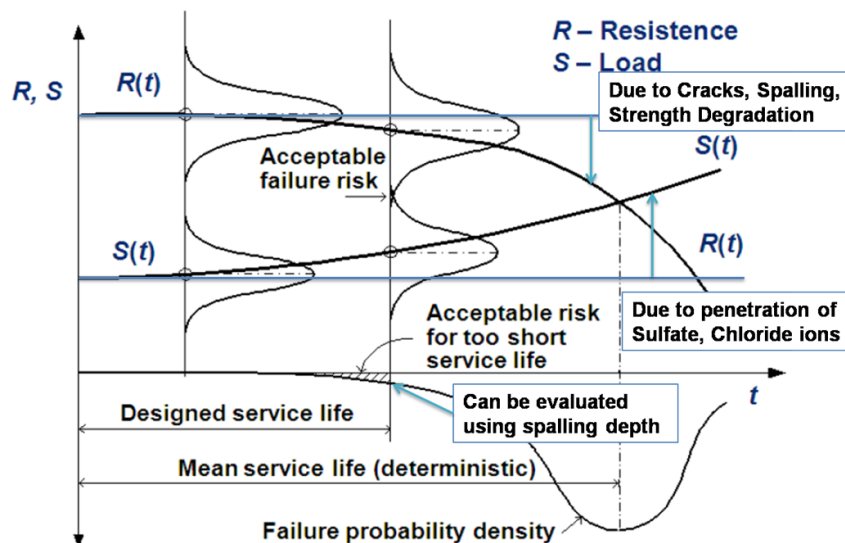


Figure 11. Relation between environmental load and concrete resistance.

To assess that whether the proposed threshold SR/BR values are appropriate for concrete in an extremely aggressive environment for 75- and 100-year designs, the service life needs to be predicted. To predict concrete service life, the end of service life should be connected with concrete performances and defined as a specific value. For example, in the case of chloride-initiated pitting corrosion, concrete service life depends on the time of chloride penetrated to reinforced steel, which is known as the Time to Initiation of Corrosion (TIC) (Figure 12). TIC depends on many factors, such as concrete diffusivity, concrete cover, and concrete mix design. It also depends on the environmental condition and protective strategies.

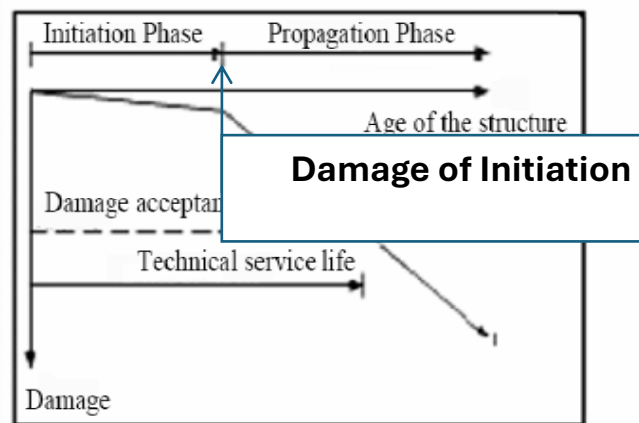


Figure 12. The service life of concrete structures. Two-phase modeling of deterioration (Tuutti, 1982).

Although carbonation and chloride ingress are not detrimental to the concrete itself, they impact the corrosion of steel reinforcement. Thus, most service life prediction models focus on carbonation and/or chloride ingress. Alexander and Beushausen (2019) conducted a review on durability, service life, and modeling for reinforced concrete structures. Various service life models were reviewed as shown in Table 1. Most models cover both chloride and carbon dioxide ingress into concrete including the European model “DuraCrete” and the North American “Life-365”.

Table 1. Summary of selected service life models for corrosion in RC structures (adapted from Alexander and Beushausen, 2019)

Model	Characteristics	Reference
<b>DuraCrete</b>	Probabilistic modelling of carbonation and chloride ingress. Performance-based approach for material characterization.	DuraCrete (2000)
<b>Life-365®</b>	Chloride diffusion model, based on Fick's law. Semi-probabilistic. Provides life-cycle cost analysis.	Free software ( <a href="http://www.Life365.org">www.Life365.org</a> ), Elhen, et.al (2009)
<b>Stadium®</b>	Multi-ionic model, based on Nernst-Planck equation. Provides chloride ingress rate and corrosion initiation. Also provides carbonation and sulphate profiles. Full probabilistic.	Proprietary software ( <a href="http://www.simcotechologies.com">www.simcotechologies.com</a> )
<b>fib Bulletin 56, Model Code 2010</b>	Based on Fick's 2nd law. Deals primarily with chloride ingress and carbonation. Full probabilistic. Performance-based approaches are included.	fib (2012)
<b>Concrete Works</b>	Based on Fick's law. Predicts strength, chloride ingress, thermal cracking. Variant of Life-365®	<a href="http://www.texasconcreteworks.com">www.texasconcreteworks.com</a> , Folliard, et al. (2008)
<b>LIFEPRED</b>	Based on Fick's Law.	Andrade and Tavares (2012)
<b>ClinConc</b>	Chloride diffusion model.	Tang (1996)
<b>DuraCon</b>	Based on Fick's law and the RCMT; also used for quality control and quality assurance, and condition assessment during operational life.	Gjørsv (2014)
<b>Durability Index Approach</b>	Performance-based design and quality control based on durability indicators for carbonation and chloride diffusion	Mackechnie (1995) Alexander et al. (1999)

There are some other software that are available for this project. Bentz et al. (2001) at the National Institute of Testing Standard (NIST) have developed a commercially available program (CONCLIFE) that allows one to predict the service life by examining concrete degradation. The user can input environmental and microstructure data into the program, which will access the degradation mechanism and then predict the service life (Figure 13).

In the absence of physical specimens, especially for HPC mixtures that are not typically used such as metakaolin, CEMHYD3D (another software available through NIST) could be used to model the cement hydration model. These software packages will be used for determining the service life of HPC in low pH and sulfate environments for this study.

## ***SERVICE LIFE OF CONCRETE STRUCTURES***

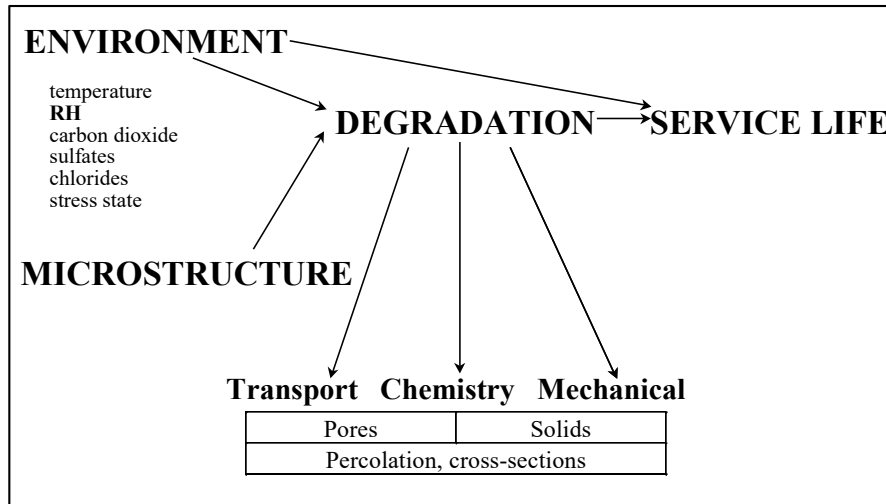


Figure 13. Diagram illustrating relationships between material properties and the environment in determining degradation and service life of concrete structures [Bentz et al., 2001)

Assumptions that need to be made:

- Sulfates enter only by sorption and not by diffusion.
- The concrete specimen is dry or only partially saturated and therefore the solution can enter by sorption.
- As soon as sulfate enters the concrete it will react with the cement hydration products and be transformed into ettringite. The composition of the cement is not taken into account.
- Wet-dry cycles are considered by a gradual accumulation of sulfate in the pores of the concrete. As the amount of sulfate increases in the concrete, more ettringite is formed. It is assumed that all the sulfate ions will react with the cement and form ettringite.
- The crystallization pressure is not taken into account.

$$X_{spall} = (2\alpha\gamma_f(1-\nu)) / (E(\beta C_E)^2) \quad \text{Eq. 9}$$

where,

$C_E$  = concentration of reacted sulfate as ettringite (mol/m<sup>3</sup>)

$E$  = Young's modulus (GPa)

$X_{spall}$  = Spalling depth (m)

$\alpha$  = roughness factor for fracture path

$\beta$  = linear strain caused by one mole of sulfate reacted ( $\text{m}^3/\text{mol}$ )

$\gamma_f$  = fracture surface energy of concrete (N/m)

$\nu$  = Poisson's ratio

Although ConcLife is designed for calculating pavement and concrete deck service life, it could also be used in calculating concrete piles service life based on the same sulfate attack theory (see Figure 14). The distinction between them is the differences in their geometries and thermal boundary conditions. The temperature and time-of-wetness behavior of concrete pavements and bridge decks will be different, as well as concrete piles.

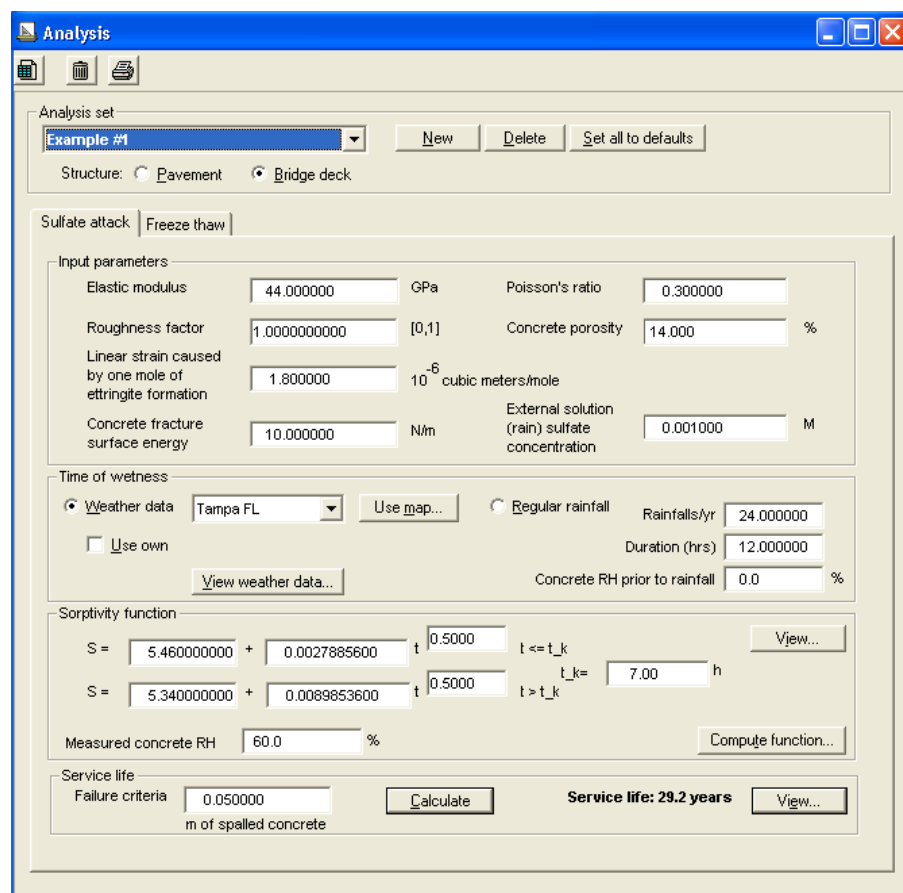


Figure 14. The interface of ConcLife.

The sorptivity function can be obtained by a water absorption test. In the use of ConcLife, local water level static should be used instead of rainy conditions. External solution sulfate concentration can be obtained by sampling local water bodies or from close bridges in FDOT Bridge Environment. The results from this model can be used to determine if the proposed thresholds are acceptable.

Assuming the chloride ion diffusion is one dimensional and semi-infinite, Fick's second law can be used to describe the chloride diffusion process (Liu, 2012):

$$\frac{\partial C}{\partial t} = D_{app} \frac{\partial^2 C}{\partial x^2} \quad \text{Eq. 10}$$

where  $t$  is time;  $x$  is the depth in the chloride diffusion path;  $D_{app}$  ( $\text{m}^2/\text{s}$ ) is the apparent diffusion coefficient;  $C$  is the chloride concentration at a specific exposure time and concrete depth.

To calculate TIC, Fick's Second Law is widely used in diffusion-based models. For example, a modified Fick's Second Law is used in DuraCrete Model (2000).

$$C_{(x,t)} = C_0 \left(1 - \text{erf} \frac{x}{2\sqrt{D_c t}}\right) \quad \text{Eq. 11}$$

where:

$C_{(x,t)}$  = chloride concentration at depth and time,

$C_0$  = surface chloride concentration,

$D_c$  = apparent diffusion coefficient,

$t$  = time for diffusion,

$x$  = depth

$\text{erf}$  = statistical error function.

The probabilistic service life of a reinforced concrete system can be estimated in terms of the time taken for the initiation of rebar corrosion or when the concentration of chloride at the level of embedded steel reaches the chloride threshold ( $C_{th}$ ). The chloride threshold is highly dependent

on many factors including the type of steel and binder, environmental, and material factors (Pillai et al., 2019).

Diffusion-based Models ignore cracks within the cement. However, cracks are inevitable in concrete. It interacts with sodium content and results in concrete spalling (see Figure 15). Therefore, it cannot be ignored.

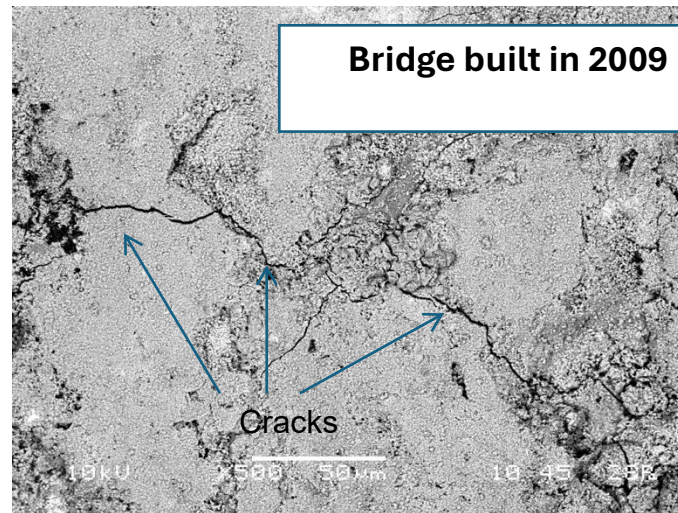


Figure 15. Cracks in a 2009-built bridge.

For concrete under sulfate-acid combination attack, the definition of concrete service life could be adopted as the percentage of sulfate reach a certain amount at the level of reinforced steel. However, due to the different transit mechanisms, the sorption-based sulfate-acid attack could result in an increase inside the concrete. It also depends on internal factors, such as concrete properties, concrete cover, and external factors, such as degree of exposure, and sulfate concentration. The model developed by Atkinson et al. (1985) and Atkinson and Hearne (1989) is widely adopted in sorptivity-based models. It evaluates the concrete performance by calculating spalling depth. The sorptivity-based models need to be investigated in this project as well.

## 2.3 Key Findings

Based on the literature review performed on the two topics as presented in this report, the following key findings can be concluded:

- (1) There are a number of research have been done regarding the correlation between BR test results and RMT results. The consensual conclusion is BR conductivity and RMT diffusion are linearly correlated.
- (2) Compared with the BR test, there are fewer research studied the relationship between SR test results and RMT results or BD results. Most of the researchers concluded that the SR conductivity is linearly correlated with the RMT result. However, Hamilton (2007) used power functions to describe the relationship between SR conductivity and BD result. Thus, the research team will consider different functions when aiming to establish the correlations.
- (3) All the correlation equations derived for diffusion coefficient and resistivity/ conductivity are empirical equations depending on the dataset used and they vary from one study to another study. Thus, based on the new testing results from this project combined with the existing results from the literature, the research team will derive a series of functions taking into consideration the influence of various parameters such as the use of pozzolan materials, c/m ratios, etc.
- (4) There are many service life models based on chloride diffusion. These service life models will be evaluated and validated with the new testing results from this project. Also, in order to achieve one of the objectives of BEE02 regarding the threshold for sulfate durability, a sulfate-acid combination attack needs to be considered, and the sorptivity-based models need to be investigated in this project as well.

## 3 Development of Testing Plan

In the process of developing a testing plan, there are two main subtasks. One is to select mix designs that FDOT approved for extreme aggressive environment for lab sampling and testing. In order to evaluate the effects of various materials and parameters, three different groups of mix designs are proposed. One of them only uses limestone for coarse aggregate. The other two uses both limestone and granite for a number of mix designs with the same mix proportions. After in-depth discussion with PM and FDOT SMO, there are 31 mixes have been chosen for further investigation.

The research team also have developed a specific testing plan designed to provide the data to meet the objectives of this research project. The plan includes the minimum testing at 28- and 56-days of standard curing and consists of four tests: surface resistivity (SR) test (AASHTO T358), bulk resistivity (BR) test (AASHTO T119), rapid sulfate permeability test (RSPT based on modified ASTM C1202), and length change test (ASTM C1012).

### 3.1 Selection of Mixes

The chloride and sulfate durability highly depends on the mix design and mix proportions. Therefore, the research team worked with the FDOT project manager to select a minimum of 17 FDOT-approved design mixes from the mix designs proposed in this report. The reason for having a minimum of 17 mixes is to evaluate the effect of various parameters. In order to identify unbiased thresholds for SR/BR as well as sulfate rapid electrochemical test, the selected mix designs cover various parameters. To limit the number of parameters to be considered, it is proposed that all concrete made using Type II (MH) and No. 57 coarse aggregate as a baseline. FDOT standard specification section 346 and FDOT Structures Design Guidelines (SDG) were used as a general guideline for developing these mix designs.

The research team first investigated the concrete mix designs with SR data that provided by FDOT. In order to identify the mixes that suitable for this study, several filters were applied including:

- (1) Only Class IV, Class IV (Drilled Shafts), Class V, Class V (Special), Class VI, and Class VII considered (based on Table 1.4.3-1 of FDOT SDG)
- (2) Only Type II (MH) cement
- (3) w/cm ratio ranging from 0.35 to 0.41

(4) SR larger or equal to 29 kΩ-cm

There are 43 mixes with highly reactive pozzolans (i.e. silica fume, metakaolin, and ultrafine fly ash) and 36 mixes without highly reactive pozzolans that satisfy these 4 criteria. Figure 16 and Figure 17 show the histogram of weight of different materials used in FDOT mix designs with and without highly reactive pozzolan, respectively. It shows that there is no clear distribution for cementitious material for mixes with or without highly reactive pozzolan. However, the weight of the coarse aggregate shows an approximate normal distribution. Per cubic yard, the average weight of coarse aggregate, fine aggregate, cementitious material, and water are 1672 lbs, 1143 lbs, 754 lbs, and 271 lbs for mixes with highly reactive pozzolans. On the other hand, the average weight of coarse aggregate, fine aggregate, cementitious material, and water are 1717 lbs, 1163 lbs, 708 lbs, and 272 lbs for mixes without highly reactive pozzolans. Thus, two baseline designs were selected and are summarized in

Table 2.

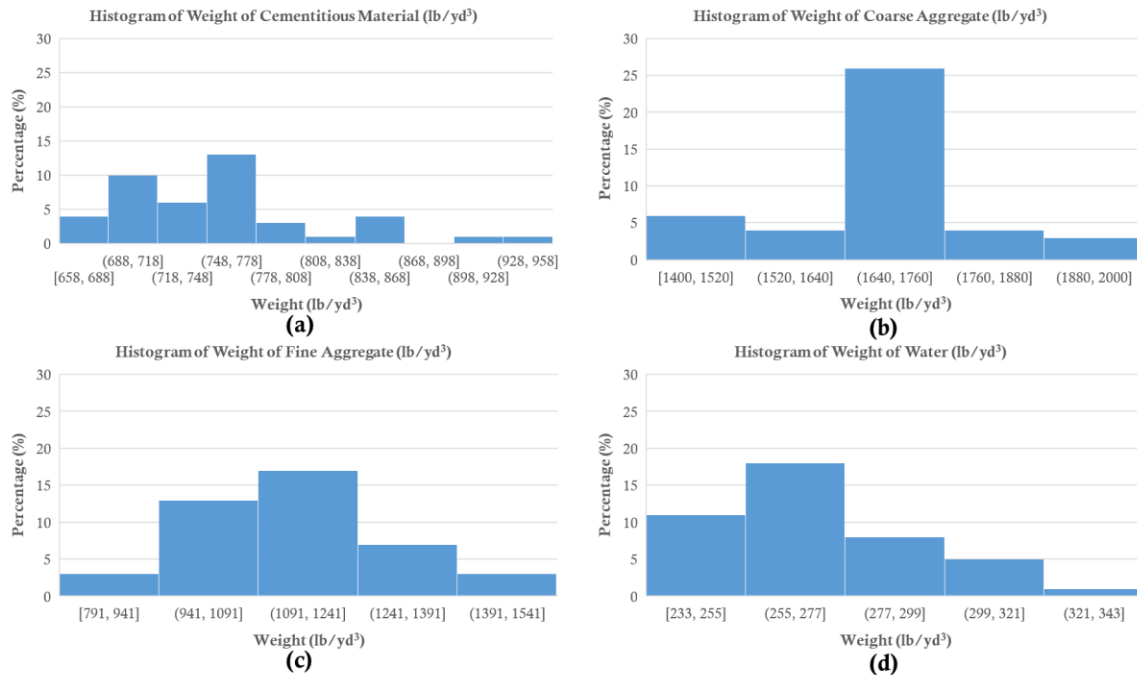


Figure 16. Histogram of weight of material from FDOT mix design database after filtering (with highly reactive pozzolan): (a) cementitious material, (b) coarse aggregate, (c) fine aggregate, and (d) water.

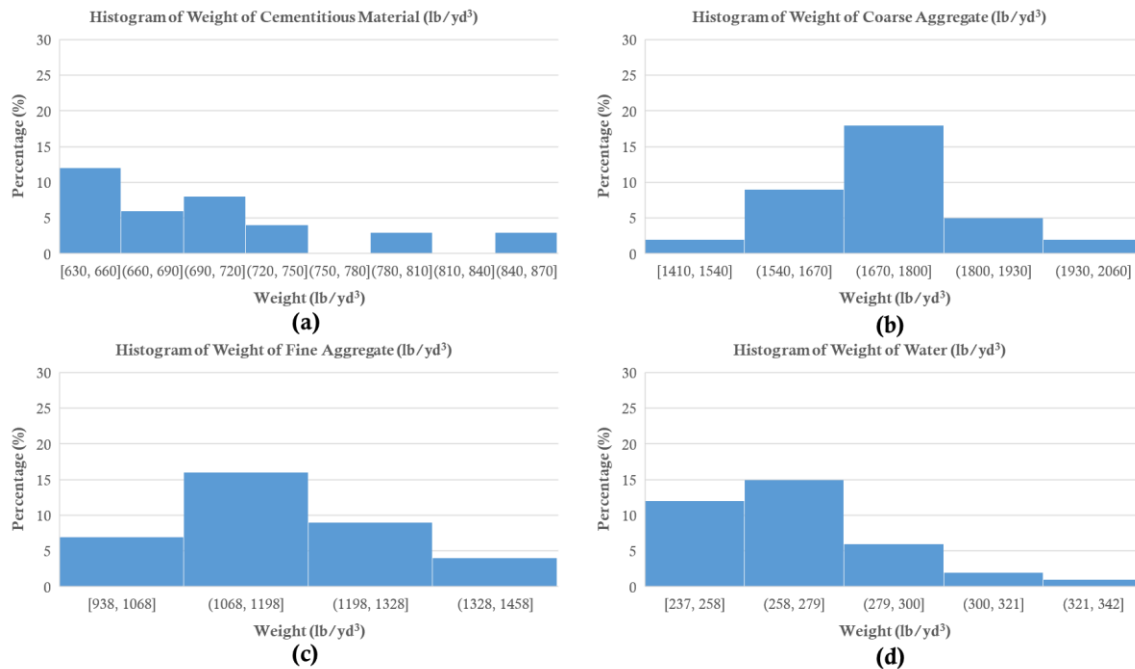


Figure 17. Histogram of weight of material from FDOT mix design database after filtering (without highly reactive pozzolan): (a) cementitious material, (b) coarse aggregate, (c) fine aggregate, and (d) water.

Table 2. Summary of the baseline mix designs

Use Highly Reactive Pozzolan	Coarse Aggregate (lbs/yd³)	Fine Aggregate (lbs/yd³)	Total Cementitious (lbs/yd³)	Water (lbs/yd³)	w/cm
No	1720	1160	710	270	0.38
Yes	1670	1140	770	270	0.35*

\*Based on FDOT section 346 Table 346-3, the maximum w/cm will be 0.35 if silica fume or metakaolin is used. The maximum w/cm will be 0.3 if ultrafine fly ash is used.

The research team focused on concrete mix designs approved for an extremely aggressive environment. To this end, Table 3 provides a list of approved cement replacements specified by FDOT, and Figure 18 shows the parameters that were proposed to be considered when selecting mix designs. It is anticipated that all concrete made using Type II (MH) and No. 57 coarse aggregate as a baseline.

Although a minimum of 17 FDOT mixes was proposed, the actual number of mixes considered should be determined based on how many mixes are needed to cover the spectrum of various parameters.

Table 3. Comparison of cement replacement materials in an extremely aggressive environment

Cement Replacement Materials	FDOT Section 346	Literature
Fly Ash Class F - Drilled Shaft - Precast Concrete - Other Concrete	33% to 37% >18% 18% to 30%	<30% 70% for action on C-S-H
Slag Cement - Drilled Shaft - Precast Concrete - Other Concrete	58% to 62% >50% 50% to 70%	>60% Not recommended for action on C-S-H
Fly Ash Class F and Slag	10% to 20% fly ash, 50% to 60% slag, 30% cement	>10% fly ash, >50% slag
Silica Fume	3% to 9%	>10%
Metakaolin	8% to 12%	~20%
Ultrafine Fly Ash	8% to 12%	

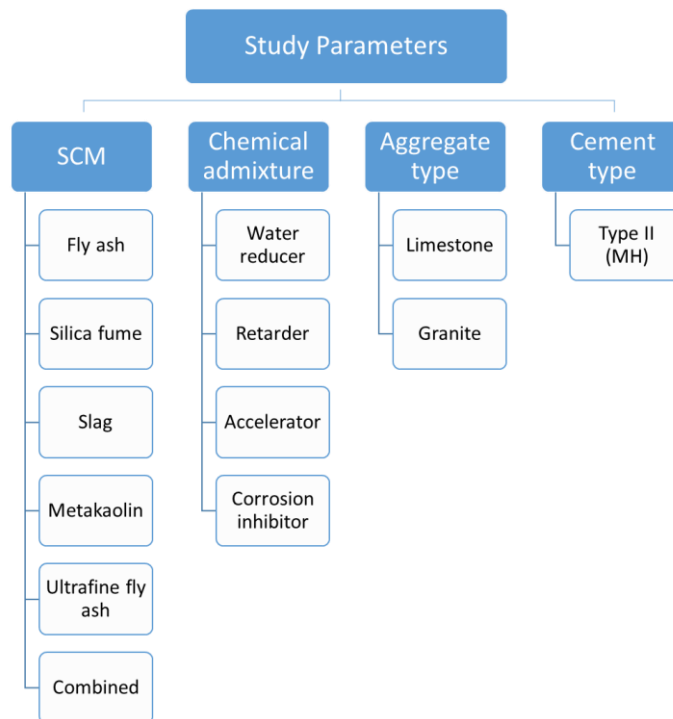


Figure 18. Parameters for mix design selection

In light of aforementioned information, the research team developed three sets of mix designs.

### 3.1.1 Mix Design Set A

Mix design set A didn't include aggregate type as a variable and only considered limestone as a coarse aggregate. The following parameters will be evaluated:

Seven different percentages of cement, i.e., 30%, 36%, 40%, 63%, 67%, 70%, and 100%.

- Six different percentages of fly ash, i.e., 10%, 18%, 20%, 22%, 33%, and 37%.
- Five different percentages of slag, i.e., 50%, 52%, 55%, 60%, 70%.
- Two different percentages of a ternary blend of fly ash and slag, i.e., 10%/60% and 20%/50% fly ash and slag combinations.
- Two silica fume percentages, i.e., 8% and 9%
- Three metakaolin percentages, i.e., 8%, 9%, and 12%
- Three ultrafine fly ash percentages, i.e., 8%, 9%, and 12%
- Three different w/cm ratios, i.e., 0.30, 0.35, and 0.38
- Total of 19 mix designs. A18 and A19 are designed with w/cm of 0.38 for comparison purposes.

Table 4 shows a summary of mix design set A.

Table 4. Summary of mix design set A

Mix Design #	Coarse Aggregate	Fine Aggregate	Cement	Fly Ash	Slag	Metakaolin	Silica Fume	Ultra Fine Fly Ash	w/cm
	lb/yd <sup>3</sup>	lb/yd <sup>3</sup>	lb/yd <sup>3</sup>	lb/yd <sup>3</sup>	lb/yd <sup>3</sup>	lb/yd <sup>3</sup>	lb/yd <sup>3</sup>	lb/yd <sup>3</sup>	
A1	1720	1160	710						0.38
A2	1720	1160	213	142	355				0.38
A3	1720	1160	213	71	426				0.38
A4	1720	1160	475.7	234.3					0.38
A5	1720	1160	284		426				0.38
A6	1720	1160	447.3	262.7					0.38
A7	1720	1160	213		497				0.38
A8	1670	1140	539	169.4			61.6		0.35
A9	1670	1140	539	169.4		61.6			0.35
A10	1670	1140	539	169.4				61.6	0.30
A11	1670	1140	277.2		423.5		69.3		0.35
A12	1670	1140	277.2		423.5	69.3			0.35
A13	1670	1140	277.2		423.5			69.3	0.30
A14	1670	1140	539	138.6		92.4			0.35
A15	1670	1140	539	138.6				92.4	0.30
A16	1670	1140	277.2		400.4	92.4			0.35
A17	1670	1140	277.2		400.4			92.4	0.30
A18	1670	1140	539	169.4		61.6			0.38
A19	1670	1140	539	169.4				61.6	0.38

### 3.1.2 Mix Design Set B

Mix design set B include seven mix designs with granite aggregate. The following parameters will be evaluated:

- Six different percentages of cement, i.e., 30%, 36%, 40%, 67%, 70%, and 100%.
- Four different percentages of fly ash, i.e., 18%, 20%, 22%, and 33%.
- Three different percentages of slag, i.e., 50%, 52%, and 60%.
- One silica fume percentages, i.e., 8%
- Two metakaolin percentages, i.e., 8% and 12%
- Two ultrafine fly ash percentages, i.e., 8% and 12%
- Three different w/cm ratios, i.e., 0.30, 0.35, and 0.38
- Total of 20 mix designs

Table 5 shows a summary of mix design set B.

Table 5. Summary of mix design set B

Mix Design #	Type of Coarse Aggregate	Coarse Aggr.	Fine Aggr.	Cement	Fly Ash	Slag	Metakaolin	Silica Fume	Ultra Fine Fly Ash	w/c m
		lb/yd <sup>3</sup>	lb/yd <sup>3</sup>	lb/yd <sup>3</sup>	lb/yd <sup>3</sup>	lb/yd <sup>3</sup>	lb/yd <sup>3</sup>	lb/yd <sup>3</sup>	lb/yd <sup>3</sup>	
B1	Granite	1720	1160	710						0.38
B2	Limestone	1720	1160	710						0.38
B3	Granite	1720	1160	213	142	355				0.38
B4	Limestone	1720	1160	213	142	355				0.38
B5	Granite	1720	1160	475.7	234.3					0.38
B6	Limestone	1720	1160	475.7	234.3					0.38
B7	Granite	1720	1160	284		426				0.38
B8	Limestone	1720	1160	284		426				0.38
B9	Granite	1670	1140	539	169.4			61.6		0.35
B10	Granite	1670	1140	539	169.4		61.6			0.35
B11	Granite	1670	1140	539	169.4				61.6	0.30
B12	Limestone	1670	1140	539	169.4			61.6		0.35
B13	Limestone	1670	1140	539	169.4		61.6			0.35
B14	Limestone	1670	1140	539	169.4				61.6	0.30
B15	Limestone	1670	1140	539	138.6		92.4			0.35
B16	Limestone	1670	1140	539	138.6				92.4	0.30
B17	Limestone	1670	1140	277.2		400.4	92.4			0.35
B18	Limestone	1670	1140	277.2		400.4			92.4	0.30
B19	Limestone	1670	1140	539	169.4		61.6			0.38
B20	Limestone	1670	1140	539	169.4				61.6	0.38

### 3.1.3 Mix Design Set C

Mix design set C include three mix designs with granite aggregate. The following parameters will be evaluated:

- Seven different percentages of cement, i.e., 30%, 36%, 40%, 63%, 67%, 70%, and 100%.
- Six different percentages of fly ash, i.e., 10%, 18%, 20%, 22%, 33%, and 37%.
- Three different percentages of slag, i.e., 55%, 60%, and 70%.
- Two silica fume percentages, i.e., 8% and 9%
- Three metakaolin percentages, i.e., 8%, 9%, and 12%

- Three ultrafine fly ash percentages, i.e., 8%, 9%, and 12%
- Three different w/cm ratios, i.e., 0.30, 0.35, and 0.38
- Total of 19 mix designs

Table 6 shows a summary of mix design set C.

Table 6. Summary of mix design set C

Mix Design #	Type of Coarse Aggregate	Coarse Aggr.	Fine Aggr.	Cement	Fly Ash	Slag	Metakaolin	Silica Fume	Ultra Fine Fly Ash	w/cm
		lb/yd <sup>3</sup>	lb/yd <sup>3</sup>	lb/yd <sup>3</sup>	lb/yd <sup>3</sup>	lb/yd <sup>3</sup>	lb/yd <sup>3</sup>	lb/yd <sup>3</sup>	lb/yd <sup>3</sup>	
C1	Granite	1720	1160	710						0.38
C2	Limestone	1720	1160	710						0.38
C3	Granite	1720	1160	213	71	426				0.38
C4	Limestone	1720	1160	213	71	426				0.38
C5	Granite	1670	1140	539	169.4			61.6		0.35
C6	Limestone	1670	1140	539	169.4			61.6		0.35
C7	Limestone	1720	1160	475.7	234.3					0.38
C8	Limestone	1720	1160	284		426				0.38
C9	Limestone	1720	1160	447.3	262.7					0.38
C10	Limestone	1720	1160	213		497				0.38
C11	Limestone	1670	1140	539	169.4		61.6			0.35
C12	Limestone	1670	1140	539	169.4				61.6	0.30
C13	Limestone	1670	1140	277.2		423.5		69.3		0.35
C14	Limestone	1670	1140	277.2		423.5	69.3			0.35
C15	Limestone	1670	1140	277.2		423.5			69.3	0.30
C16	Limestone	1670	1140	539	138.6		92.4			0.35
C17	Limestone	1670	1140	539	138.6				92.4	0.30
C18	Limestone	1670	1140	539	169.4		61.6			0.38
C19	Limestone	1670	1140	539	169.4				61.6	0.38

### 3.1.4 Final Selected Mix Designs

The selected concrete mix designs are categorized into several groups based upon the type and proportion of cementitious materials used. Type II cement was used in all mixes. There are a total of 10 groups with each being made up of three different mix designs, except for group 10 which includes four. Table 7 provides the mix proportions for each of the 31 concrete mix designs. Also,

descriptions have been provided below about the proportion and water-cement ratios of the mix designs within each group.

1. Baseline Mixes (Group 1):

- Composed solely of cement.
- Consists of three mixes with a fixed w/c ratio of 0.44 but varying amounts of cement which results in different cement paste to aggregate ratios.

2. Fly Ash Mixes (Group 2):

- Composed of 20% fly ash and 80% cement.
- Consists of three mixes with varying w/c ratios of 0.30, 0.40, and 0.50.

3. Slag Mixes (Group 3):

- Composed of 70% slag and 30% cement.
- Consists of three mixes with varying w/c ratios of 0.30, 0.40, and 0.50.

4. Slag and Fly Ash Mixes (Group 4):

- Composed of 50% slag, 20% fly ash, and 30% cement.
- Consists of three mixes with varying w/c ratios of 0.25, 0.35, and 0.45.

5. Fly Ash and Silica Fume Mixes (Group 5):

- Composed of 20% fly ash, 8% silica fume, and 72% cement.
- Consists of three mixes with varying w/c ratios of 0.25, 0.34, and 0.44.

6. Slag and Silica Fume Mixes (Group 6):

- Composed of 55% slag, 6% silica fume, and 39% cement.
- Consists of three mixes with varying w/c ratios of 0.22, 0.34, and 0.46.

7. Fly Ash and Metakaolin Mixes (Group 7):

- Composed of 18% fly ash, 12% metakaolin, and 70% cement.
- Consists of three mixes with varying w/c ratios of 0.24, 0.30, and 0.36.

8. Slag and Metakaolin Mixes (Group 8):

- Composed of 55% slag, 8% metakaolin, and 37% cement.
- Consists of three mixes with varying w/c ratios of 0.22, 0.30, and 0.38.

9. South Florida Mixes (Group 9):

- Composed of 42% fly ash and 58% cement.

- Consists of three mixes with varying w/c ratios of 0.19, 0.23, and 0.27.

#### 10. Alternative Slag Source Mixes (Group 10):

These mixes used slag from another source which has a different pozzolan content. The mix designs also included differing proportions of cementitious materials.

- Mix 28: 70% slag, 30% cement with a w/c ratio of 0.40.
- Mix 29: 50% slag, 20% fly ash, and 30% cement with a w/c ratio of 0.35.
- Mix 30: 55% slag, 6% silica fume, and 39% cement with a w/c ratio of 0.34.
- Mix 31: 55% slag, 8% metakaolin, and 37% cement with a w/c ratio of 0.30.

The first group of mix designs provides a baseline by keeping the water-cement ratio fixed and using cement as the only cementitious material in the mix. The mixes included in groups two through eight provide a large spectrum of proportions and water-cement ratios. The South Florida mixes were developed for mass concrete production and to explore regional variations in concrete performance. Last, the alternative slag source mix designs were introduced to assess the impact of different pozzolan contents on concrete durability. By providing such a significant sample size of concrete mix designs, the analysis and results of the testing methods to be discussed in the upcoming sections should adequately represent concrete mixes developed for extremely aggressive environments.

Table 7. Concrete mix design proportions

Batch #	Cement		Fly Ash		Slag1 <17%		Slag2 >17%		Silica Fume		Metakaolin		w/cm
	lb/yd <sup>3</sup>	%	lb/yd <sup>3</sup>	%	lb/yd <sup>3</sup>	%	lb/yd <sup>3</sup>	%	lb/yd <sup>3</sup>	%	lb/yd <sup>3</sup>	%	
1	450	100%	0	0%	0	0%	0	0%	0	0%	0	0%	0.44
2	750	100%	0	0%	0	0%	0	0%	0	0%	0	0%	0.44
3	600	100%	0	0%	0	0%	0	0%	0	0%	0	0%	0.44
4	520	80%	130	20%	0	0%	0	0%	0	0%	0	0%	0.3
5	520	80%	130	20%	0	0%	0	0%	0	0%	0	0%	0.4
6	520	80%	130	20%	0	0%	0	0%	0	0%	0	0%	0.5
7	195	30%	0	0%	455	70%	0	0%	0	0%	0	0%	0.3
8	195	30%	0	0%	455	70%	0	0%	0	0%	0	0%	0.4
9	195	30%	0	0%	455	70%	0	0%	0	0%	0	0%	0.5
10	210	30%	140	20%	350	50%	0	0%	0	0%	0	0%	0.25
11	210	30%	140	20%	350	50%	0	0%	0	0%	0	0%	0.35
12	210	30%	140	20%	350	50%	0	0%	0	0%	0	0%	0.45
13	576	72%	160	20%	0	0%	0	0%	64	8%	0	0%	0.25
14	576	72%	160	20%	0	0%	0	0%	64	8%	0	0%	0.34
15	576	72%	160	20%	0	0%	0	0%	64	8%	0	0%	0.44
16	312	39%	0	0%	440	55%	0	0%	48	6%	0	0%	0.22
17	312	39%	0	0%	440	55%	0	0%	48	6%	0	0%	0.34
18	312	39%	0	0%	440	55%	0	0%	48	6%	0	0%	0.46
19	665	70%	171	18%	0	0%	0	0%	0	0%	114	12%	0.24
20	665	70%	171	18%	0	0%	0	0%	0	0%	114	12%	0.3
21	665	70%	171	18%	0	0%	0	0%	0	0%	114	12%	0.36
22	277.5	37%	0	0%	412.5	55%	0	0%	0	0%	60	8%	0.3
23	277.5	37%	0	0%	412.5	55%	0	0%	0	0%	60	8%	0.22
24	277.5	37%	0	0%	412.5	55%	0	0%	0	0%	60	8%	0.38
SFM1	634.7	59%	450.3	42%	0	0%	0	0%	0	0%	0	0%	0.19
SFM2	634.7	59%	450.3	42%	0	0%	0	0%	0	0%	0	0%	0.23
SFM3	634.7	59%	450.3	42%	0	0%	0	0%	0	0%	0	0%	0.27
SLAG 2-1	195	30%	0	0%	0	0%	455	70%	0	0%	0	0%	0.4
SLAG 2-2	210	30%	140	20%	0	0%	350	50%	0	0%	0	0%	0.35
SLAG 2-3	312	39%	0	0%	0	0%	440	55%	48	6%	0	0%	0.34
SLAG 2-4	277.5	37%	0	0%	0	0%	412.5	55%	0	0%	60	8%	0.3

## 3.2 Lab Test Plan

There are two tests will be conducted for each concrete batch. The bulk diffusion (BD) test according to ASTM C1556, “*Standard Test Method for Determining the Apparent Chloride Diffusion Coefficient of Cementitious Mixtures by Bulk Diffusion,*” and the rapid migration test (RMT) according to NT Build 492, “*Chloride Migration Coefficient from Non-Steady-State Migration Experiments.*” In this section, detailed testing setup and lab testing plan are presented to evaluate the chloride diffusion.

### 3.2.1 Bulk Diffusion (BD) Test Setup

The BD test will be set up based on ASTM C1556, “*Standard Test Method for Determining the Apparent Chloride Diffusion Coefficient of Cementitious Mixtures by Bulk Diffusion.*” Drilled cores, molded cylinders, or molded cubes are acceptable test specimens. Test specimens must be free of defects such as voids or cracks visible to the unaided eye. The minimum dimension across the exposed surface of each test specimen must be at least 75 mm but not less than three times the nominal maximum aggregate particle size. The depth of the specimen must be at least 75 mm. Twenty-eight days of laboratory standard moist curing in accordance with practices ASTM C31 or C192 shall be provided prior to sample preparation for immersion in the exposure liquid. As shown in Figure 19, the test specimen is prepared by cutting parallel to the finished surface. The top 50 mm is cut off and discarded. The next segment of at least 75-mm thickness is used as the test specimen. The 20-mm slice will be used to determine the initial chloride-ion content. The specimens shall be rinsed with tap water immediately after cutting. Then the specimens shall be rinsed again after scrubbing the surface with a stiff nylon brush. Air dry the specimen prior to sealing specimen surfaces for no more than 24 h in laboratory air maintained at  $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$  and  $50\% \text{ RH} \pm 3\% \text{ RH}$ . Seal all sides of the exposure specimen except for the exposed surface following the procedure described in ASTM C1202. The initial mass of the test specimen shall be determined when the coating has hardened. Immerse the test specimen in the saturated calcium hydroxide water bath at  $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$  in a tightly closed plastic container that filled to the top. After 24 h of immersion, the specimen needs to be removed and dried with a paper towel, and the mass

of the specimen is determined. The test specimen is immersed in a saturated calcium hydroxide water bath until the mass doesn't change by more than 0.1% in 24 h.

For exposure, remove the saturated test specimen from the calcium hydroxide water bath, rinse the specimen surface immediately with tap water, and place the specimen in the exposure container, fill the container with the exposure liquid, and then seal the container. The container should be placed in a temperature controlled chamber or room at  $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$ . Record the start date and time to the nearest hour. Multiple specimens are permitted to be placed in a single container. However, the entire exposure surface of the specimens cannot be obstructed. The concentration of the solution is  $165\text{g} \pm 1\text{ g NaCl}$  per L of solution. Maintain the exposed surface area to exposure liquid volume ratio within the range of  $50\text{ cm} \pm 30\text{ cm}^2/\text{L}$ . The exposure time is 6 months for this project.

Once the 6 months exposure time is completed, remove the test specimen from the exposure liquid, rinse with tap water, and dry for at least 24h in laboratory air at  $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$  and  $50\% \text{ RH} \pm 3\% \text{ RH}$ . Then obtain the powder samples by grinding off material in layers parallel to the exposed surface. For each layer, collect at least 10 g of powder for chloride analysis. The acid-soluble chloride-ion content of the powder samples shall be determined according to FM 5-516 for both exposed specimen and the 20-mm thick slice.

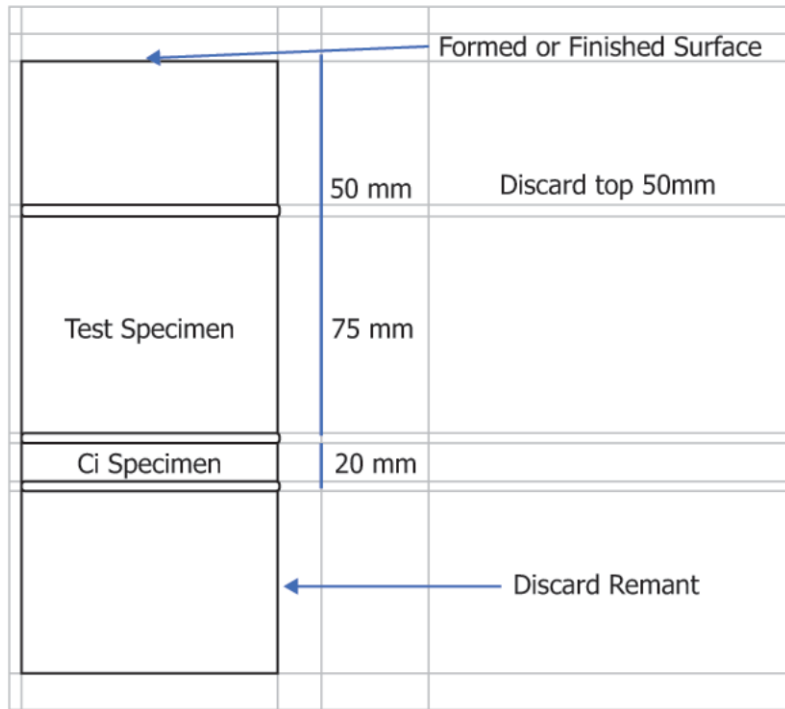


Figure 19. Sketch of specimens obtained from a typical sample not previously exposed to chlorides (ASTM, 2022)

### 3.2.2 Rapid Migration Test (RMT) Setup

The test setup will be based on NT Build 492, “Concrete, Mortar and Cement-based Repair Materials: Chloride Migration Coefficient from Non-Steady-State Migration Experiments”. This test is to determine the chloride migration coefficient, which is a measure of the resistance of the tested material to chloride penetration. Unlike BD test which tests the movement of chloride ions under a concentration gradient, this test measures the movement of ions under the action of an external electrical field.

For this test, cylindrical specimens sliced from cast cylinders will be used. These specimens shall have a diameter of 100 mm (4 in.) and a thickness of 50 mm (2 in.). Three specimens are needed. An external electrical potential is applied axially across the specimen and forces the chloride ions outside of the specimen to migrate into the specimen. After a certain test duration, the specimen is axially split, and a silver nitrate solution is sprayed on to the freshly split sections.

Then the chloride penetration depth will be measured from the visible white silver chloride precipitation as shown in Figure 20. The measured penetration depth will be used to calculate the chloride migration coefficient.

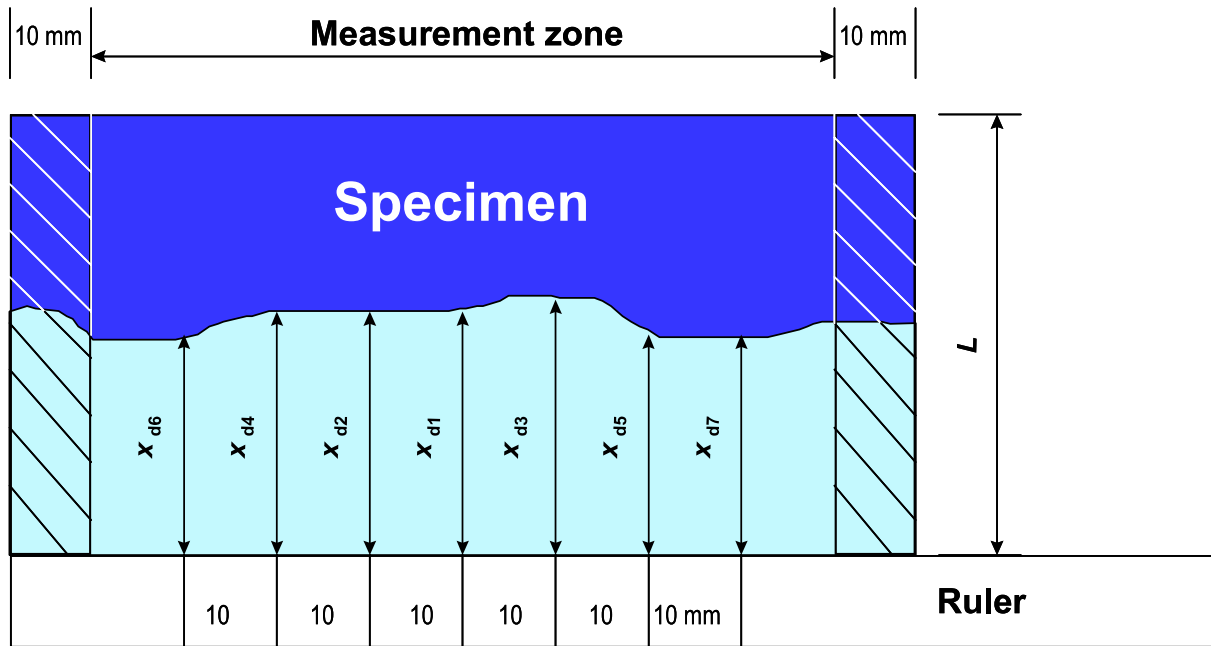
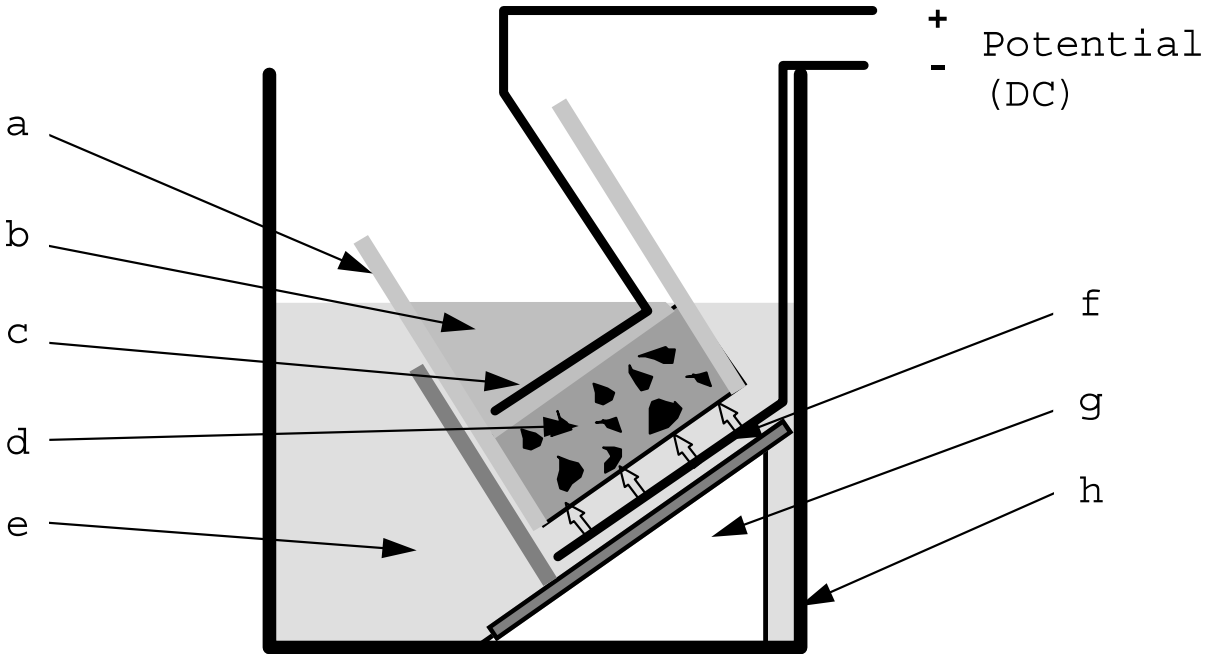


Figure 20. Illustration of measurement for chloride penetration depths (NT Build 492, 1999)

For this project, a cast cylinder will be cut into two halves equally, then a  $50 \pm 2$  mm thick slice will be cut from one half. The end surface that was nearer to the first cut will be exposed to the chloride solution (catholyte). Preconditioning is needed for this test. After specimens are taken and surface-dry, place them in a vacuum container for vacuum treatment with both end surfaces exposed. Reduce the absolute pressure in the vacuum container to a pressure in the range of 10-50 mbar (1-5 kPa) within a few minutes and maintain the vacuum for three hours. Then fill the container with the saturated  $\text{Ca}(\text{OH})_2$  solution while the vacuum pump steel running until all the specimens are immersed. Maintain the vacuum for a further hour before allowing air to enter the container. Keep the specimens in the solution for  $18 \pm 2$  hours.



- |                  |                    |
|------------------|--------------------|
| a. Rubber sleeve | e. Catholyte       |
| b. Anolyte       | f. Cathode         |
| c. Anode         | g. Plastic support |
| d. Specimen      | h. Plastic box     |

Figure 21. One arrangement of the migration set-up (NT Build 492, 1999)

As shown in Figure 21, the specimen will be placed into a rubber sleeve and then placed on a plastic support. The space in the sleeve above the specimen will be filled with 300 ml anolyte solution (0.3 M NaOH). The catholyte reservoir will be filled with about 12 L of 10 % NaCl solution. An anode and a cathode will be immersed in the anolyte and catholyte solution, respectively. Connect the cathode and the anode to the negative and positive pole of the power supply, respectively. With the power on and initial voltage set at 30 V, record the initial current for each specimen. The test duration and voltage will be determined based on the initial current (Table 8. Test voltage and duration for concrete specimen with normal binder content). After adjustment, record the initial current again as well as the initial temperature. Before terminating the test, record the final current and temperature. As mentioned earlier, once the test finished, the chloride penetration depth will be measured by spraying 0.1 M silver nitrate solution on the split

surface of the specimen and the non-steady-state migration coefficient will be calculated according to NT Build 492.

Table 8. Test voltage and duration for concrete specimen with normal binder content

Initial current $I_0$ (with 30 V) (mA)	Applied voltage U (after adjustment) (V)	Possible new initial current $I_0$ (mA)	Test duration $t$ (hour)
$I_0 < 5$	60	$I_0 < 10$	96
$5 \leq I_0 < 10$	60	$10 \leq I_0 < 20$	48
$10 \leq I_0 < 15$	60	$20 \leq I_0 < 30$	24
$15 \leq I_0 < 20$	50	$25 \leq I_0 < 35$	24
$20 \leq I_0 < 30$	40	$25 \leq I_0 < 40$	24
$30 \leq I_0 < 40$	35	$35 \leq I_0 < 50$	24
$40 \leq I_0 < 60$	30	$40 \leq I_0 < 60$	24
$60 \leq I_0 < 90$	25	$50 \leq I_0 < 75$	24
$90 \leq I_0 < 120$	20	$60 \leq I_0 < 80$	24
$120 \leq I_0 < 180$	15	$60 \leq I_0 < 90$	24
$180 \leq I_0 < 360$	10	$60 \leq I_0 < 120$	24
$I_0 \geq 360$	10	$I_0 \geq 120$	6

*Note: For specimens with a special binder content, such as repair mortars or grouts, correct the measured current by multiplying by a factor (approximately equal to the ratio of normal binder content to actual binder content) in order to be able to use the above table.*

### 3.2.3 Lab Sampling and Testing Plan

Since a large number of mixes will be investigated and a total of six tests will be conducted between BEE02 and BED 32, the lab testing need to be carefully scheduled especially many of the tests require preconditioning of the specimens with different procedure. Essentially, the lab plan should be developed to accommodate all the tests without any confliction. Figure 22 shows a flowchart of lab sampling and testing. Please note that although only 14 samples are needed for SR, BR, RSPT, BD and RMT, total 20 (twenty) 100 mm by 200 mm specimens will be taken. The ones that didn't use may be used to evaluate between-lab variability, conduct other tests, or reserve as back up specimens. Figure 22 shows the sampling and testing schedule for a typical mix. Please note the sampling and testing for BEE02 are integrated into this lab testing plan. The research team will coordinate closely with the PM during the process.

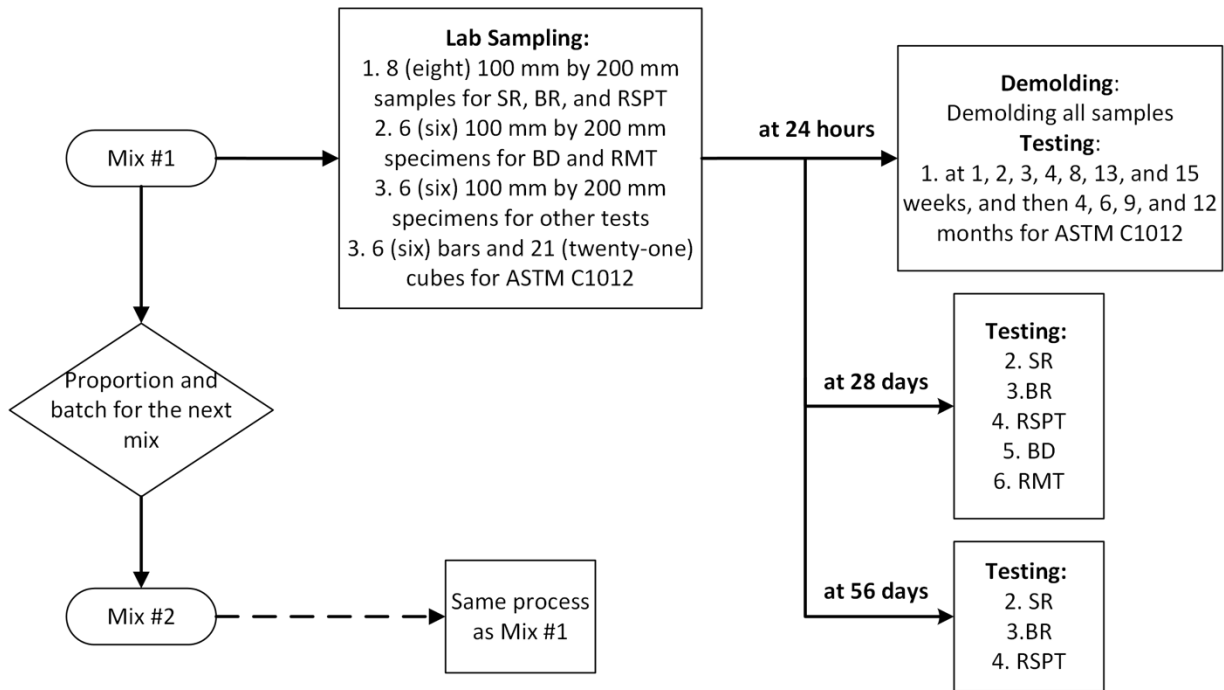


Figure 22. Flowchart of lab sampling and testing

Table 9. Sampling and testing schedule for a typical mix

Critical Dates	Action Item
<b>Before mix</b>	- Prepare and proportion materials - Prepare all the molds
<b>Day 1</b>	- Mix concrete - Record actual proportions and all the chemical and admixtures used - Cast samples
<b>Day 2</b>	- Demold all samples
<b>1 week</b>	- 1-week testing for ASTM C1012
<b>2 weeks</b>	- 2-week testing for ASTM C1012
<b>3 weeks</b>	- 3-week testing for ASTM C1012
<b>Day 27</b>	- Prepare and calibrate all equipment for testing
<b>Day 28</b>	- 4-week testing for ASTM C1012 - SR and BR tests - Coating for RSPT and BD - Conditioning for RMT
<b>Day 29</b>	- RMT test - Conditioning for RSPT - Immersion for BD
<b>Day 30</b>	- RSPT test - Place BD samples into the exposure tank
<b>Day 55</b>	- Prepare and calibrate all equipment for testing
<b>Day 56</b>	- 8-week testing for ASTM C1012 - SR and BR tests - Coating for RSPT
<b>Day 57</b>	- Conditioning for RSPT
<b>Day 58</b>	- RSPT test
<b>Week 13</b>	- 13-week testing for ASTM C1012
<b>Week 15</b>	- 15-week testing for ASTM C1012
<b>4 Months</b>	- 4-month testing for ASTM C1012
<b>6 Months</b>	- 6-month testing for ASTM C1012
<b>7 Months (6 months exposure)</b>	- BD Test
<b>9 Months</b>	- 9-month testing for ASTM C1012
<b>12 Months</b>	- 12-month testing for ASTM C1012

For all the testing data, if possible, the mean, standard deviation, distribution, and percentiles will be calculated. The results from the statistical analysis will provide insight on the significance of between-lab variance. The research team will analyze the data and will try to establish the correlations between BD, RMT, SR, and BR results. These correlations are critical to find the appropriate thresholds for SR and BR tests.

## 4 Testing Results and Analysis

In this project, four tests were conducted for the mix designs chosen in Task 2. These tests are surface resistivity (SR) test (AASHTO T358), bulk resistivity (BR) test (AASHTO T119), rapid sulfate permeability test (RSPT based on modified ASTM C1202), and length change test (ASTM C1012). SR test, BR test, and RSPT test were performed at both 28 days and 56 days while the length change test performed at various weeks according to ASTM C1012. In this report, testing results are presented and analyzed in sections 4.1 through 4.5.

### 4.1 BD Test Results

The bulk diffusion (BD) test is performed according to ASTM C1556, “Standard Test Method for Determining the Apparent Chloride Diffusion Coefficient of Cementitious Mixtures by Bulk Diffusion.” This test is designed to determine the chloride diffusion coefficient in concrete, providing insights into the long-term durability of cementitious materials when exposed to chloride environments.

#### 4.1.1 Specimen Preparation

##### 4.1.1.1 *Sample Description:*

- Drilled cores, molded cylinders, or molded cubes are acceptable test specimens. Specimens must be free of defects such as voids or cracks visible to the unaided eye.

##### 4.1.1.2 *Dimensions:*

- The minimum dimension across the exposed surface of each test specimen must be at least 75 mm but not less than three times the nominal maximum aggregate particle size. The depth of the specimen must be at least 75 mm.

##### 4.1.1.3 *Curing*

- The specimens undergo 28 days of standard moist curing in accordance with ASTM C31 or C192 as shown in Figure 23.
- **Cutting:** The test specimen is prepared by cutting parallel to the finished surface. The top 50 mm is cut off and discarded. The next segment of at least 75 mm thickness is used as

the test specimen. A 20 mm slice is used to determine the initial chloride-ion content as shown in Figure 24.

- **Rinsing:** The specimens are rinsed with tap water immediately after cutting, then scrubbed with a stiff nylon brush and rinsed again.
- **Air Drying:** Specimens are air-dried for no more than 24 hours in laboratory air at  $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$  and  $50\% \text{RH} \pm 3\% \text{RH}$ .
- **Sealing:** All sides of the exposure specimen, except for the exposed surface, are sealed following the procedure described in ASTM C1202 as shown in Figure 25 . The initial mass of the test specimen is determined once the coating has hardened.

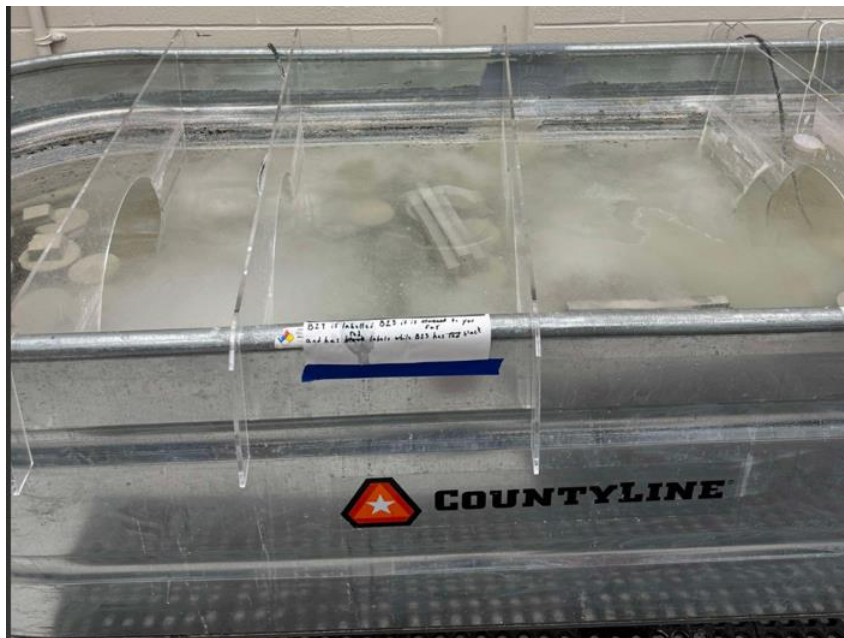


Figure 23. Curing tank

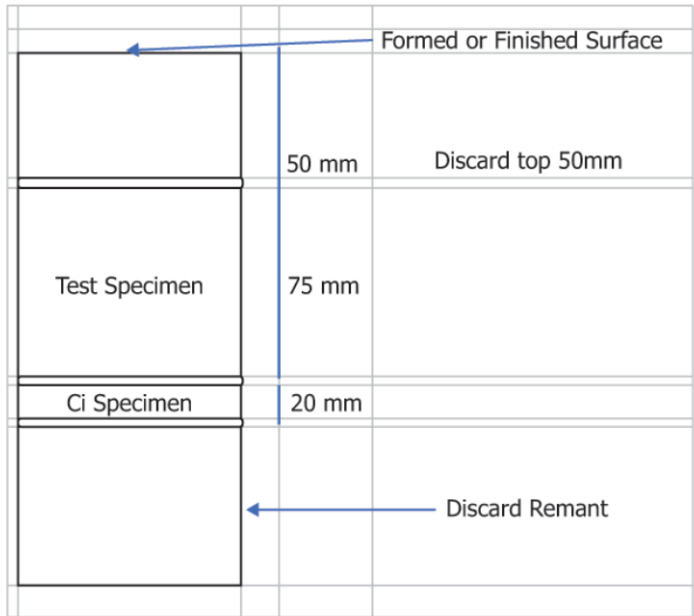


Figure 24. Sketch of specimens obtained from a typical sample not previously exposed to chlorides (ASTM, 2022)



Figure 25. Samples coated with epoxy

#### 4.1.1.4 Saturation

- **Immersion:** The test specimen is immersed in a saturated calcium hydroxide water bath at  $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$  in a tightly closed plastic container filled to the top.
- **Mass Stabilization:** After 24 hours of immersion, the specimen is dried with a paper towel, and its mass is determined. The specimen remains in the water bath until its mass does not change by more than 0.1% in 24 hours.

#### 4.1.1.5 Exposure

- **Chloride Solution:** Once saturation is achieved, the specimen is removed from the calcium hydroxide water bath, rinsed with tap water, and placed in the exposure container. The container is filled with an exposure liquid of  $165 \text{ g} \pm 1 \text{ g}$  NaCl per L of solution and sealed. The specimen is exposed for six months at  $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$  as shown in Figure 26 .
- **Exposure Details:** Multiple specimens can be placed in a single container as long as the entire exposure surface of each specimen is not obstructed. The surface area to liquid volume ratio is maintained within  $50 \text{ cm} \pm 30 \text{ cm}^2/\text{L}$ .



Figure 26. Samples inside the exposure tank

#### 4.1.1.6 Post-Exposure Processing

- **Drying:** After the six-month exposure period, the specimen is removed from the exposure liquid, rinsed with tap water, and dried for at least 24 hours in laboratory air at  $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$  and  $50\% \text{RH} \pm 3\% \text{RH}$ .
- **Grinding:** The specimens are ground off in layers parallel to the exposed surface using the concrete milling machine as shown in Figure 27 , and at least 12 g of powder is collected from each layer for chloride analysis. The grinding was conducted by the project team with the use of a mill machine equipped with a rotary table and digital caliper. The specimen should be placed at the center of the stage within the clamps, and these clamps should then

be tightened down with an Allen wrench to ensure the sample does not move during grinding. A 4-inch rubber coupling with a metal clamp should be placed onto the specimen that can be tightened to the diameter of the concrete specimen, as shown in Figure 28. This is to contain and capture any powder that would otherwise be lost during grinding. For a layer of the specimen to be grinded, the stage should be centered at the origin of (0,0), but the stage can be moved horizontally along the x-axis as needed. The mill machine has a diamond core drill bit that is a circular, hollow bit with an outer diameter of 35 mm and a thickness of 2.75 mm. The diamond grit extends from the bottom of the bit to a height of 15 mm along its side, and it also has a U-shaped chip flute which reduces the cutting resistance and improves drilling speed. Yet, the bit only covers a portion of the area of the specimen surface which is about 100 mm in diameter. So, this is the case where the stage would be moved in the horizontal direction to cover the remaining area of the exposed surface to be grinded. The bit is moved vertically up and down with a lever, and once the specimen is in position, the lever should be moved allowing the bit to be lowered to the sample. At the point where the bit touches the surface of the specimen, the electronic caliper should be set to zero.



Figure 27. Grinding with concrete milling machine



Figure 28. Concrete sample in position on milling machine stage

The concrete powder samples were obtained by grinding material from the specimen in layers, parallel to the exposed surface. The ASTM standard prescribes a minimum of eight layers for the test, with depth increments specified in the accompanying table shown in Table 10. However, if the chloride content did not reach the initial chloride content value after grinding the prescribed layers, additional layers were ground in increments, ensuring that the chloride profile was extended as needed. The goal of the grinding process is to obtain at least six data points from just below the exposed surface to a depth where the chloride concentration equals or slightly exceeds the initial chloride content. This ensures a thorough profile of chloride penetration into the concrete. If necessary, grinding continued in smaller increments, such as quarter-inch layers (approximately 6.35 mm), until the chloride concentration stabilized. Table 11 shows the basis for the grinding depths for each concrete mix and its layers. For example, mix design 9 has a water-cement ratio of 0.5, but because it contains slag, its classification for w/c ratio should be 0.4 based upon the criteria of Table 1 of ASTM C1556. Also, the default lowest w/c ratio to be used is 0.25, so any mix design with a lower value must be updated to this value.

Table 10. Recommended depth intervals (in mm) for powder grinding

w/cm	0.25	0.30	0.35	0.40	0.50	0.60	0.70
Depth 1	0-1	0-1	0-1	0-1	0-1	0-1	0-1
Depth 2	1-2	1-2	1-2	1-3	1-3	1-3	1-5
Depth 3	2-3	2-3	2-3	3-5	3-5	3-6	5-10
Depth 4	3-4	3-4	3-5	5-7	5-8	6-10	10-15
Depth 5	4-5	4-6	5-7	7-10	8-12	10-15	15-20
Depth 6	5-6	6-8	7-9	10-13	12-16	15-20	20-25
Depth 7	6-8	8-10	9-12	13-16	16-20	20-25	25-30
Depth 8	8-10	10-12	12-16	16-20	20-25	25-30	30-35
<i>A Luping, Tang and Sørensen, Henrik, "Evaluation of the Rapid Test Methods for Measuring the Chloride Diffusion Coefficients of Concrete," NORDTEST Project No. 1388-98, Swedish National Testing and Research Institute, SP Report 1998:42.</i>							
NOTE 1—For cementitious mixtures with pozzolan or slag, the depth intervals in the column one place to the left should be applied. For example, use the depth intervals for w/cm = 0.35 for silica fume concrete with w/cm = 0.40.							

Once the depth of a layer to be grinded is determined, the lever should be moved to ensure that the bit is raised up to where it is not touching the sample. The mill machine can then be turned on and the bit can be lowered onto the surface of the specimen. As the specimen is grinded into powder, the bit can be lowered slowly until the desired depth is reached by looking at the electronic caliper. Once it reaches this value, the height of the bit can be locked in place to ensure the depth stays uniform across the surface. Again, the powder samples are obtained by grinding off material in layers parallel to the exposed surface. The stage can then be slowly moved horizontally to a specified position from the origin. The rotary table can then be used to slowly rotate the stage about its axis in the clockwise direction until the area has been entirely grinded to a powder. Often, this can be verified by the sound of the bit while grinding and the restriction of rotating the stage. Upon the completion of grinding a specific area of the specimen, the stage can once again slowly be moved horizontally to another position. The purpose of this again is to cover the desired area of the surface to produce the required amount of powder per layer. The procedure can then be repeated for the use of the rotary table. It is important to note that the specimen should not be grinded closer than 5mm from its edge to avoid any negative impacts or disturbance of the coating. The project team typically grinded to a distance of about 10 mm from its edge to entirely avoid this issue.

Table 11. Depth intervals grinded for each concrete mix design (mm)

Batch #	w/cm	Slag/Silica	Depth 1	Depth 2	Depth 3	Depth 4	Depth 5	Depth 6	Depth 7	Depth 8	Depth 9	Depth 10	Depth 11	Depth 12	Depth 13	Depth 14	Total Depth
1	0.44	n	1	2	2	2	3	3	3	4	6.35	6.35	6.35	6.35	6.35	6.35	55.25
2	0.44	n	1	2	2	2	3	3	3	4	6.35	6.35	6.35	6.35	6.35	3.4	55.15
3	0.44	n	1	2	2	2	3	3	3	4	6.35	6.35	6.35	6.35	6.35	-	51.75
4	0.3	n	1	1	1	1	1	2	2	2	6.35	6.35	6.35	-	-	-	30.05
5	0.4	n	1	2	2	2	3	3	3	4	6.35	6.35	-	-	-	-	32.7
6	0.5	n	1	2	2	3	4	4	4	5	6.35	6.35	6.35	-	-	-	44.05
7	0.3	y	1	2	1	1	1	1	2	2	-	-	-	-	-	-	10
8	0.4	y	1	1	1	2	2	2	3	4	6.35	-	-	-	-	-	22.35
9	0.5	y	1	2	2	2	3	3	3	4	6.35	-	-	-	-	-	26.35
10	0.25	y	1	1	1	1	1	1	2	2	-	-	-	-	-	-	10
11	0.35	y	1	1	1	1	2	2	2	2	-	-	-	-	-	-	12
12	0.45	y	1	2	2	2	3	3	3	4	-	-	-	-	-	-	20
13	0.25	y	1	1	1	1	1	1	2	2	-	-	-	-	-	-	10
14	0.34	y	1	1	1	1	1	2	2	2	-	-	-	-	-	-	11
15	0.44	y	1	1	1	1	2	2	3	4	6.35	6.35	6.35	6.35	-	-	40.4
16	0.22	y	1	1	1	1	1	1	2	2	-	-	-	-	-	-	10
17	0.34	y	1	1	1	1	2	2	2	2	-	-	-	-	-	-	12
18	0.46	y	1	2	2	2	3	3	3	4	6.35	6.35	-	-	-	-	32.7
19	0.24	y	1	1	1	1	1	2	2	2	-	-	-	-	-	-	11
20	0.3	n	1	1	1	1	1	2	2	2	-	-	-	-	-	-	11
21	0.36	n	1	1	1	2	2	2	3	4	6.35	-	-	-	-	-	22.35
22	0.3	y	1	1	1	1	1	1	2	2	-	-	-	-	-	-	10
23	0.22	y	1	1	1	1	1	1	2	2	-	-	-	-	-	-	10
24	0.3	y	1	1	1	1	1	1	2	2	-	-	-	-	-	-	10
SFM1	0.19	n	1	1	1	1	1	1	2	2	-	-	-	-	-	-	10
SFM2	0.23	n	1	1	1	1	1	1	2	2	-	-	-	-	-	-	10
SFM3	0.27	n	1	1	1	1	1	1	2	2	-	-	-	-	-	-	10
SLAG2-1	0.4	y	1	1	1	2	2	2	3	4	-	-	-	-	-	-	16
SLAG2-2	0.35	y	1	1	1	1	2	2	2	2	-	-	-	-	-	-	12
SLAG2-3	0.34	y	1	1	1	1	2	2	2	2	-	-	-	-	-	-	12
SLAG2-4	0.3	y	1	1	1	1	1	1	2	2	-	-	-	-	-	-	10

To remove the grinded layer and specimen from the mill machine, the stage should be moved back to its original origin, and the equipment can be turned off to stop the rotation of the bit. Then, the lever can be unlocked to allow the bit to be raised from the specimen, and any tool can be used to tap the side of the bit, allowing any remaining powder to fall from it onto the sample surface contained within the rubber coupling. The clamps on the stage can then be loosened using the Allen wrench to release the specimen, which can then be removed and brought to another surface. A paper bowl was then placed on a scale on that surface where it was tared to zero. The rubber coupling attached to the specimen was held on its side to pour the powder into the bowl, and a metal brush was used to remove the remaining powder stuck to the rubber or the surface of the specimen and brush it into the paper bowl, as illustrated in Figure 29. The powder obtained was then weighed on the scale to ensure that at least 12 grams of powder were collected which is the requirement for the chloride analysis per FM5-516. The concrete powder sample was then transferred to an aluminum metal tin for storage and sealed with a lid. Using a marker, the metal tin was labeled with the mix design and layer number as well as the depth of the layer to ensure organization and prevent confusion.



Figure 29. Transferring the powder to the scale

In transitioning to grinding another layer, the rubber coupling was removed from the concrete specimen to be cleaned off with a rag and compressed air. Also, the surface of the specimen was scrubbed with the brush to remove any excess powder. These steps are key to ensure that there is no contamination between layers of a concrete specimen. Once clean, the rubber coupling can be reattached to the concrete specimen and the procedures outlined above can be repeated to grind additional layers for the concrete specimen. If grinding is stopped for a particular concrete sample before completion, it should be returned to its watertight resealable polyethylene bag until the resumption of grinding.

## 4.1.2 Chloride Analysis

The acid-soluble chloride ion content of the powder samples was determined in accordance with FM 5-516 for both the exposed specimen and the 20-mm thick slice. To ensure accuracy, we prepared and tested blank samples, Chloride Check Samples (CCS), and Laboratory Control Samples (LCS) following specified guidelines, conducting 25 repetitions to establish upper and lower control limits. Additionally, daily control tests were performed to verify the validity of the solutions used and ensure proper electrode maintenance.

### 4.1.2.1 *Blank Samples*

- End points for deionized water with ISA were collected for 25 samples to establish the upper and lower control limits for both 0.01 & 0.1 concentrations of silver nitrate. The blank samples control limits are shown in Table 12.



- Dispensing 5.0 mL of deionized water to each beaker, then slowly adding 35.0 mL of 5% HNO<sub>3</sub> using a dispenser and mixing thoroughly with a clean glass rod to avoid excessive frothing.
- Covering the beakers with watch glasses and placing them on a hot plate, heating to a minimum of 250°C (482°F) until boiling as shown in Figure 30.
- 1-2 mL of concentrated HNO<sub>3</sub> should be added to drive off sulfide ions as hydrogen sulfide if the emission of a sulfur smell was observed. The samples are then boiled for 10-12 minutes.

#### *4.1.2.4 Filtering and Sample Preparation*

- Beakers were removed from the hot plate.
- A No. 41 filter paper was placed in a glass funnel to filter the extract from each beaker into a 100-mL volumetric flask as shown in Figure 31.
- Residues from the beakers were rinsed into the filter using a wash bottle.
- Residues on the filter sides were rinsed with small portions of hot deionized water, ensuring the final mark volume was not exceeded.
- The samples were allowed to cool to room temperature (about 1 hour) and then diluted to volume with deionized water as shown in Figure 32.

Table 13. Results for CCS and LCS

	CCS		LCS	
Sample No.	End Point	ppm	End Point	lb/yd <sup>3</sup>
1	0.8527	3.0569	0.4658	0.1601
2	0.9048	2.9731	0.4278	0.1218
3	0.8493	3.1461	0.4525	0.1652
4	0.8742	3.0141	0.2674	0.0820
5	0.8359	3.0608	0.4540	0.1622
6	0.8527	3.0569	0.3025	0.1051
7	0.8351	2.9622	0.4364	0.1471
8	0.8230	2.9859	0.5633	0.1962
9	0.8416	2.9107	0.6060	0.1972
10	0.8116	2.9542	0.5647	0.1995
11	0.8000	2.8700	0.5342	0.1831
12	0.8327	3.0267	0.3388	0.1212
13	0.8311	2.8784	0.3740	0.1195
14	0.8278	2.8653	0.2477	0.0768
15	0.8787	2.9100	0.5387	0.1620
16	0.8200	2.8804	0.4402	0.1457
17	0.8373	2.8743	0.4990	0.1591
18	0.8941	2.8510	0.4512	0.1217
19	0.8156	2.8560	0.3022	0.0984
20	0.8487	2.9784	0.3954	0.1303
21	0.9132	3.0654	0.3353	0.0966
22	0.8623	2.8748	0.4374	0.1300
23	0.8056	2.8672	0.4329	0.1469
24	0.8599	2.8556	0.2576	0.0684
25	0.8527	3.0569	0.3813	0.1316
<b>Maximum</b>	0.9132	3.1461	0.6060	0.1995
<b>Average</b>	0.8464	2.9533	0.4203	0.1371
<b>Minimum</b>	0.8000	2.8510	0.2477	0.0684

#### 4.1.2.5 *Sample Chloride Determination summary*

- The electrode was rinsed with deionized water and patted dry with a lint-free tissue.
- The contents of the 100-mL volumetric flasks were transferred to a 250-mL beaker.
- The beaker was placed on a magnetic stirrer, a stir bar was inserted, and the solution was stirred at a moderate and constant rate.
- The electrode was immersed in the solution. If the potential was less than that of a 10-ppm chloride solution, 0.01 N AgNO<sub>3</sub> was substituted with 0.1 N AgNO<sub>3</sub>.
- 0.01 N AgNO<sub>3</sub> titrant was added in 0.50 mL increments until the scaling potential was reached or just exceeded. The total volume of titrant and the potential were recorded as shown in Figure 33.
- Four additional 0.50 mL titrant additions were made, and the potential was recorded after each addition.
- The electrode and stir bar were removed, rinsed with deionized water, and patted dry with a lint-free tissue. The electrode was kept in a beaker of clean deionized water when not in use.
- The end point was determined, and the blank end point was subtracted to obtain the sample end point.
- This procedure was repeated for the remaining replicates.



Figure 30. Samples boiling



Figure 31. Filtration process



Figure 32. Samples ready to be tested

#### 4.1.2.6 Chloride Content Results and Analysis

As shown in Figure 33, the determination of the initial chloride content and the chloride diffusion profiles of the tested concrete mixes was carried out in accordance with ASTM C1556-22 *Standard Test Method for Determining the Apparent Chloride Diffusion Coefficient of Cementitious Mixtures by Bulk Diffusion*.

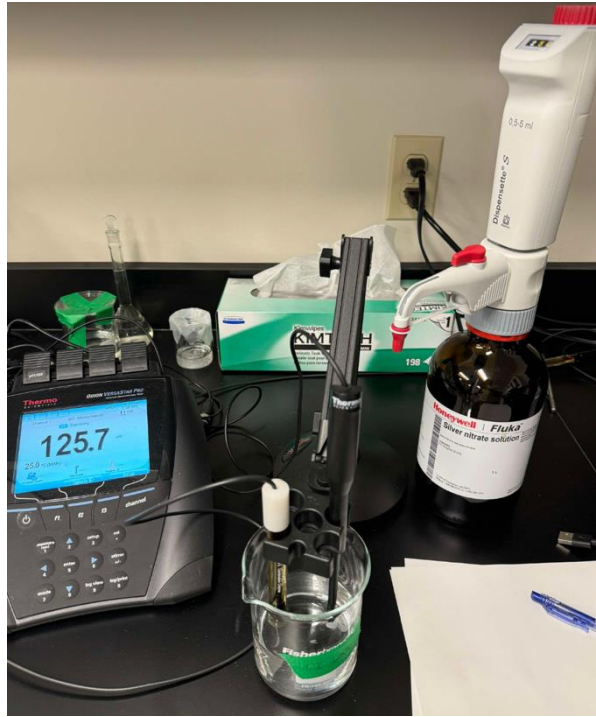


Figure 33. Ion-selective electrode and ion specific electrode/mV meter

### 4.1.3 Chemical Analysis

#### 4.1.3.1 Background

After collecting the powder for each layer of a batch of concrete in metal tins, they must be dried in an oven for 24 hours before chloride analysis can be performed on the samples. It is important to remove the lid from the metal tin to expose the powder for proper drying. Both the metal tin and its lid should be labeled to ensure that the powder is not misplaced. After the time has passed, the metal tins can be removed from the oven and sealed once again with the lid. The powder is then ready for analysis, so the acid-soluble chloride-ion content of the powder samples shall be determined according to FM 5-516 for both the exposed specimen and the initial chloride sample.

To provide a brief overview of the project, after acquiring all of the necessary equipment, materials, reagents, etc. to complete the project, 25 sets of control samples were conducted for blank samples containing different level concentrations of silver nitrate,  $AgNO_3$ , chloride check samples (CCS), and laboratory control samples (LCS). The reasoning to do these control tests

before even initiating the actual specimens to be analyzed was to validate that the project laboratory as well as its equipment and resources were effective and capable of performing such a baseline. The values determined for each type of control sample were gathered to create upper and lower limits that the future baseline checks should range within. Then, the initial chloride samples were tested to determine the initial chloride content within the concrete mixes without any exposure to the aggressive environment to which the exposed specimens were subjected. The layers for each of the exposure samples were tested to determine the chloride content leading to the chloride diffusion profile. The details behind the production of the necessary solutions, preparation and maintenance of the electrode, baseline check, and procedures for preparation, digestion, and analysis of samples are outlined in the following sections.

#### 4.1.3.2 *Preparation of Solutions*

In order to conduct the chemical analysis of the specimens, numerous solutions must be prepared for use during experimentation. These solutions are often used up within a reasonable time depending on the amount of testing conducted. Silver nitrate,  $AgNO_3$ , is the titrant to be used during the titration of chemical analysis, and it can be prepared with two different concentrations. It is important to wear gloves at all times when handling reagents and solutions for safety. The first concentration is 0.1 N which is produced with reagent grade silver nitrate particles. The particles should be weighed on a scale using a wash glass large enough to hold the required mass of 16.987 g, as shown in Figure 34. The procedures for weighing will be outlined in the following section.



Figure 34. Silver nitrate particles weighed on analytical balance

Once the correct mass is weighed, a glass funnel can be placed in a 1,000 mL flask, and then the particles can be carefully poured from the wash glass into the funnel leading to the flask. To ensure, all particles are removed from the wash glass and the funnel, a wash bottle filled with deionized water, DI, can be used to rinse the excess into the flask. Once complete, the funnel and wash glass can be removed, and the flask can be filled with enough deionized water to dissolve the  $AgNO_3$  particles. It may require swirling the solution inside the flask to aid in dissolving the particles. The flask can then be filled to its volume of 1,000 mL by using either the DI water wash bottles or directly from the DI water machine dispenser. It is important to note that the DI water is a key element to the chemical analysis process as it is used in every solution and digestion of samples. The DI water quality should be validated before initiating the chemical analysis portion of the bulk diffusion test. The DI water machine has filters inside that remove the contaminations and ions from the water. This is especially critical for the chloride ions the project team is testing.

Once the 0.1 N  $AgNO_3$  has been prepared, it should be transferred to a brown, chemically resistant bottle and stored in a dark cabinet away from any light. The reasoning is because light deteriorates the solution over time, so this should be considered when the solution is not in use. The 0.1 N silver nitrate is used often throughout the testing procedures, so a dispenser with a maximum output

of 5 mL can be screwed onto the top of the bottle. The dispenser must be calibrated before it is used to dispense the silver nitrate for the preparation of other solutions. So, first the valve on the dispensing nozzle of the dispenser should be closed and the body of the dispenser should be lifted up and pushed down repeatedly to prime the inlet tube and remove air. Once complete, ensure that the volume adjustment mechanism is set to 0.5 mL, and the valve can be opened. It may be necessary to pump a few increments into a beaker or the sink to remove any existing air from the line. Then, a 5 mL volumetric flask should be obtained and filled with 10 increments of 0.5 mL to reach the expected volume, as shown in Figure 35. The project team member should verify that the dispenser is in fact dispensing the correct volume of 5 mL. If so, the dispenser has been calibrated and is ready for use. The dispensing volume should be checked each day of use as a baseline for any solutions that is equipped with a dispenser.

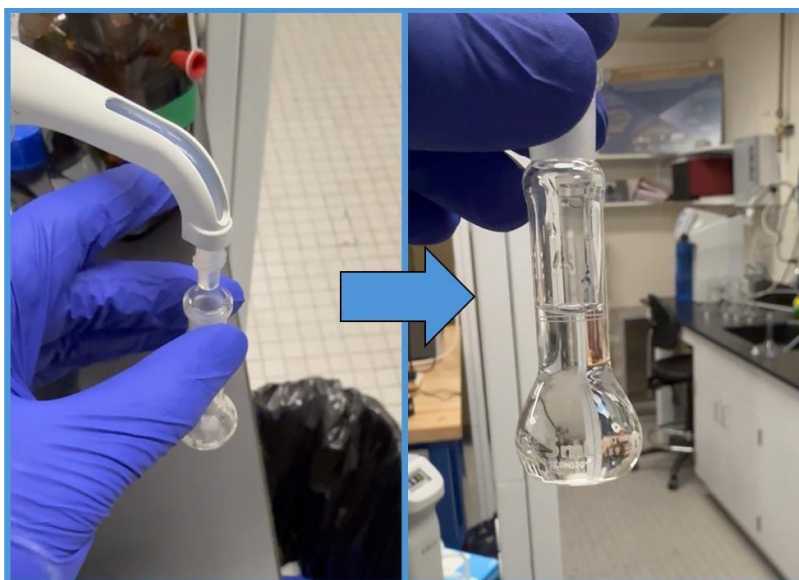


Figure 35. Calibration of dispensers with 5-mL volumetric flask

After verifying the calibration of the dispenser for the 0.1 N silver nitrate, 50 mL of the solutions can be dispensed into a 500 mL volumetric flask to prepare the 0.01 N solution. The flask should be filled to volume with DI water. Again, the solution should also be stored in the same type of bottle and environment as the 0.1 N concentration. A dispenser should also be attached to the bottle

and calibrated as previously explained. The 0.01N  $AgNO_3$  should be prepared fresh each week for testing, while the 0.1  $AgNO_3$  can be used for two weeks.

Next, a 5% nitric acid,  $HNO_3$ , solution should be prepared under a chemical fume hood using concentrated, about 70%, nitric acid and DI water, as shown in Figure 36. It is encouraged to wear a face mask to prevent breathing in the fumes as well. It is crucial that water is not added to the acid, so first fill about half of a 2,000 mL volumetric flask with DI water. Then, the concentrated nitric acid, stored in the fume hood, can be poured into a graduated cylinder to reach a volume of 250 ml. From there, the acid can be carefully poured into the volumetric flask which will be filled to volume with DI water. The solution should be mixed well and sealed with a stopper. The 5% nitric acid will be used often during sample digestion, so the solution can be poured into a brown, chemical resistant bottle and equipped with a 50mL maximum output dispenser. Any remaining nitric acid solution can be stored in its original flask for future use.

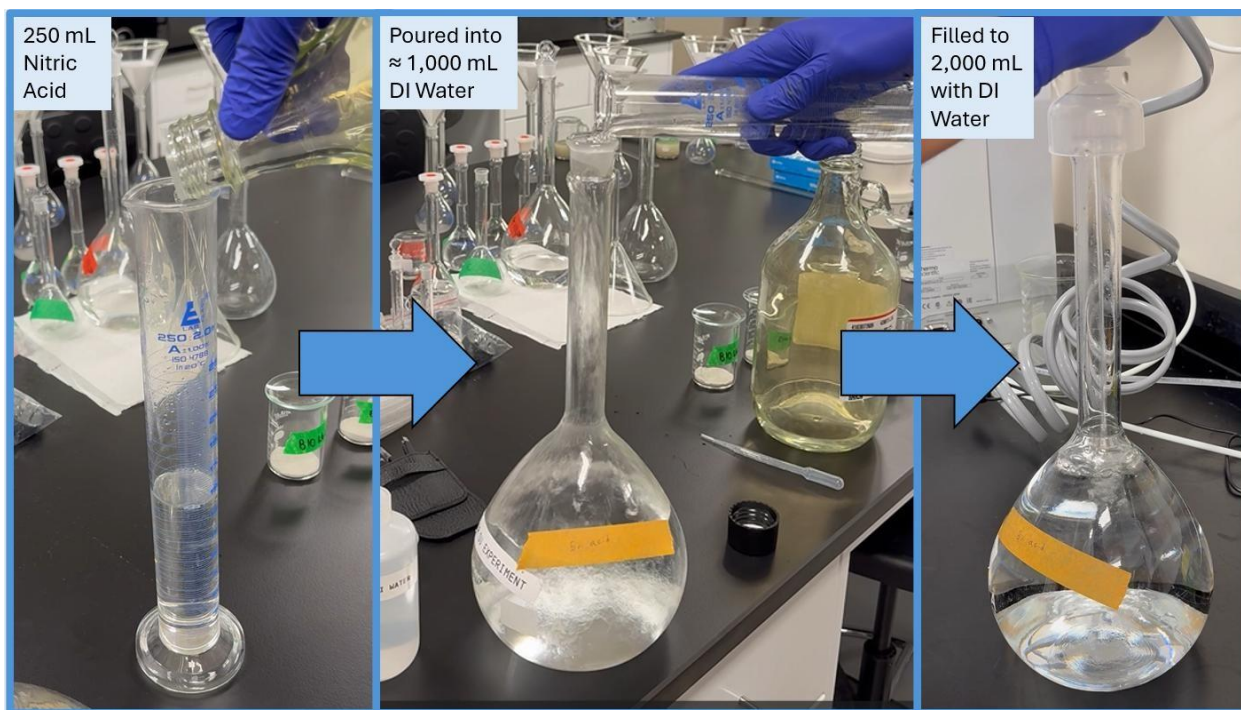


Figure 36. Preparation of 5% nitric acid

A low-level ionic strength adjuster (ISA) solution should also be produced with reagent grade potassium nitrate,  $\text{KNO}_3$ , and DI water. The  $\text{KNO}_3$  particles should be weighed on a scale using a wash glass large enough to hold the required mass of 15 g. The procedures for weighing will be outlined in the following section. Once the correct mass is weighed, a glass funnel can be placed in a 100-mL flask, and then the particles can be carefully poured from the wash glass into the funnel leading to the flask. To ensure all particles are removed from the wash glass and the funnel, use a wash bottle filled with deionized water (DI) to rinse the excess into the flask. Once complete, the funnel and wash glass can be removed, and the flask can be filled with enough deionized water to dissolve the  $\text{KNO}_3$  particles. It may require swirling the solution inside the flask to aid in dissolving the particles. The flask can then be filled to its volume of 100 mL with DI water. The solution should be mixed well and sealed with a stopper.

The final solution preparation to be outlined in this section is the chloride check sample (CCS) as previously discussed. The CCS is produced by transferring 30 mL of 100-ppm chloride standard solution using a transfer pipet or into a graduated cylinder to ensure the correct volume has been obtained. The verified 30 mL of the solution can then be transferred to a 1,000-mL volumetric flask and then filled to volume with DI water. The solution should be mixed well and sealed with a stopper. All of the solutions used for the digestion and chemical analysis of concrete powder samples are shown in Figure 37 below.



Figure 37. Prepared solutions needed for chemical analysis

#### 4.1.3.3 Preparation of Samples

To prepare specimens for testing whether they are initial chloride samples or exposure samples, the concrete powder must be weighed out in triplicate to obtain 4 grams to be tested, which means that three samples of 4 g must be prepared. The mass must be measured to a precision of 0.0001 g, so it can be difficult to get exactly 4 g of mass. The FM5-516 provides that 4 g can be measured to a tolerance of  $\pm 0.0005$  g, which indicates that when weighing the range should be 3.9995 to 4.0005 g per sample, as shown in Figure 38.

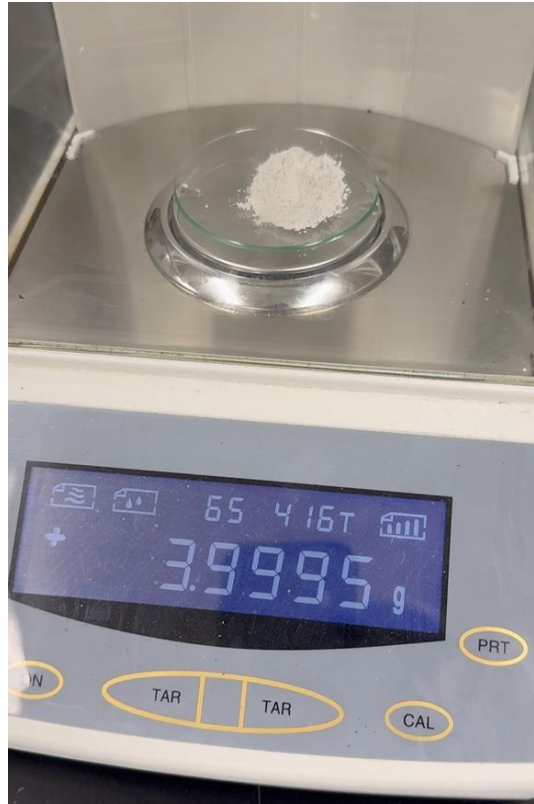


Figure 38. Weighing a concrete powder sample within tolerance

The weighing should be conducted using an analytical balance that measures with high precision and accuracy with glass doors on the top as well as both sides. These doors can be opened while placing material on the scale and closed for the reading to take place. The balance should also have a leveling bubble to ensure that the balance is level while weighing samples. These features can be depicted in Figure 39 below which provides insight into the equipment and tools used at the weighing station of the research project.

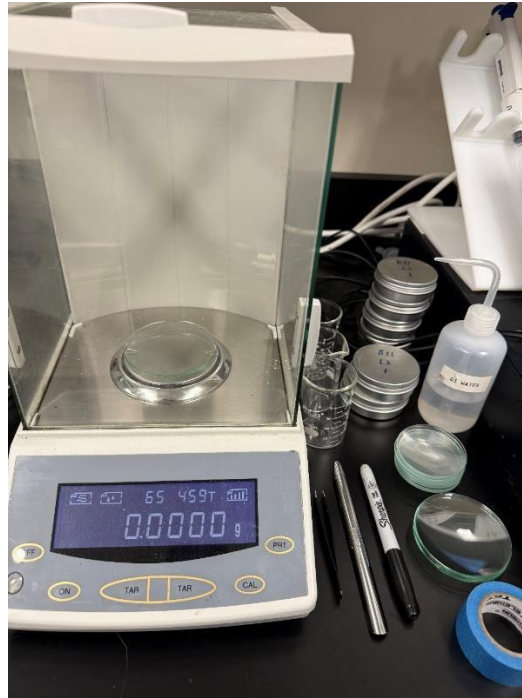


Figure 39. Analytical balance and tools used to weigh concrete powder samples

To weigh a sample, a door should be opened and a wash glass large enough to hold the mass of the specimen should be placed on the balance. The balance should then be tared to zero. The metal tin for the desired specimen to be tested can be opened, and a metal scoopula should be used to carefully collect some of the powder and place it onto the wash glass. While doing so, the project team member must be mindful to remove any larger particles from the powder with tweezers. After putting sufficient mass of the powder onto the scale close to the required 4 g, all of the doors should be closed to gather an accurate reading. After iteratively adding and removing powder until the correct range of mass is achieved, the wash glass can be removed from the balance, and the powder can be transferred into a 100 mL beaker. To prevent losing powder from spillage, a piece of paper or filter paper can be placed underneath the transfer to collect any small traces and pour them back into the beaker.

A piece of painter's tape should be placed on the beaker and labelled with a marker to indicate which concrete mix, layer, and sample it belongs to. It is also common practice to cover the beaker with a wash glass to ensure no contaminants impact the sample. The procedures outlined should be repeated in triplicate for each metal tin of powder which would be a layer for an exposure

sample and a concrete mix for an initial chloride sample. It is important to note that the first layer of each exposure sample can be neglected, so it should not be weighed out or digested as described in the following section. When transitioning from weighing one layer to the next or one concrete mix to another, the metal scoopula, tweezers, and wash glass should be rinsed with DI water and dried to ensure that powder samples are not being mixed. A laboratory control sample, LCS, as previously mentioned, would be the only exception to the triplicate requirement or cleaning between weighing because the powder is only tested once as a baseline. Also, the number of LCS samples prepared at any time can be done as desired or needed to fulfill the baseline check. The LCS powder has been provided by the FDOT SMO in Gainesville, Florida. Once the samples have been weighed, they are ready for sample digestion as detailed in the following section.

#### *4.1.3.4 Sample Digestion*

The prepared sample can be used for the digestion process which includes dispensing DI water and nitric acid into the specimens, boiling them on a hot plate, and filtering the boiled samples. The process is the same for LCS, exposure, and initial chloride samples to be tested. First, a bottle should be filled with DI water and equipped with a dispenser to be calibrated, and 5 mL of DI water should be dispensed into the 100 mL beaker containing the powder sample. The 5%  $HNO_3$  previously produced and stored in a bottle with a dispenser should be used to slowly dispense 35 mL into the beaker. It is important to note that adding the acid causes the sample solution to froth and react upward in the beaker. This step requires the project team member to be very careful to ensure that the froth does not rise up out of the beaker which would lose some of its contents. The situation would require the user to reprepare the sample. So, to prevent the issue, the 35 mL should be dispensed in several smaller increments and stirred carefully with a glass stir rod after each one, as shown in Figure 40. After dispensing the total 35 mL, the project team member should continue stirring the solution until there is no more frothing. A clean stir rod should be used for each layer and concrete mix.



Figure 40. Dispensing 5% nitric acid and stirring until minimal frothing

If a specimen's mix design contains slag, a cementitious material discussed in the concrete mix design section, 1 to 2 mL of the concentrated nitric acid should be dispensed into the solution. The reasoning behind this is samples that contain slag have significant levels of sulfides which will interfere with the operation of the electrode and may cause erroneously high chloride results.

By adding the concentrated nitric acid, it drives the sulfide ions off as hydrogen sulfide. So, a small amount of concentrated nitric acid can be poured into a small beaker, and typically, the project research team dispensed 1 mL of the acid into the sample solution using a transfer pipet.

After producing the sample solution depending on the concrete mix, the sample beaker can be placed on the hot plate with a wash glass on top matching the size of the beaker diameter. The hot plate should be turned on in advance of boiling samples to ensure that it reaches the appropriate temperature before beginning. The hot plate should be set to a minimum of 250°C, while the project team typically used a temperature of about 300°C. It is important not to set the temperature too high where a sample solution may boil out of its beaker. The length of time that samples should be boiled again depends on whether the concrete mix contains slag or not. If a sample does contain slag, it should be boiled for 10 to 12 minutes, otherwise, the time of boiling should be 3 to 5

minutes. Once a sample has begun boiling, a timer can be set for it depending on the time required. It is worth noting that keeping track of many different samples on the hot plate can be difficult, so the project team typically has a maximum of 9 beakers boiling at any given time. Also, placing the triplicate samples on the hot plate at one time makes it easier to track their boiling together rather than individually. Project team members must also stay aware of the placement of beakers as they boil because sometimes they move around which could lead them to falling off if not monitored.

Once the time for boiling has elapsed for a particular sample, the beaker and wash glass can be removed from the hot plate using heat resistant tongs or gloves, as shown in Figure 41. The filtering setup can be prepared by placing a 100 mL flask on a surface and putting a funnel into it. Then, a No. 41 filter paper disc with a diameter of 125 mm should be handled with gloves to avoid contamination and folded in half twice to make a cone-like shape. The filter should be put into the funnel and formed to its shape, and DI water can be put on it to ensure that it stays open by sticking to the funnel surface. Also, it is important to make sure that there is no gap between the filter and the funnel to avoid spillage of the sample solution directly into the flask.

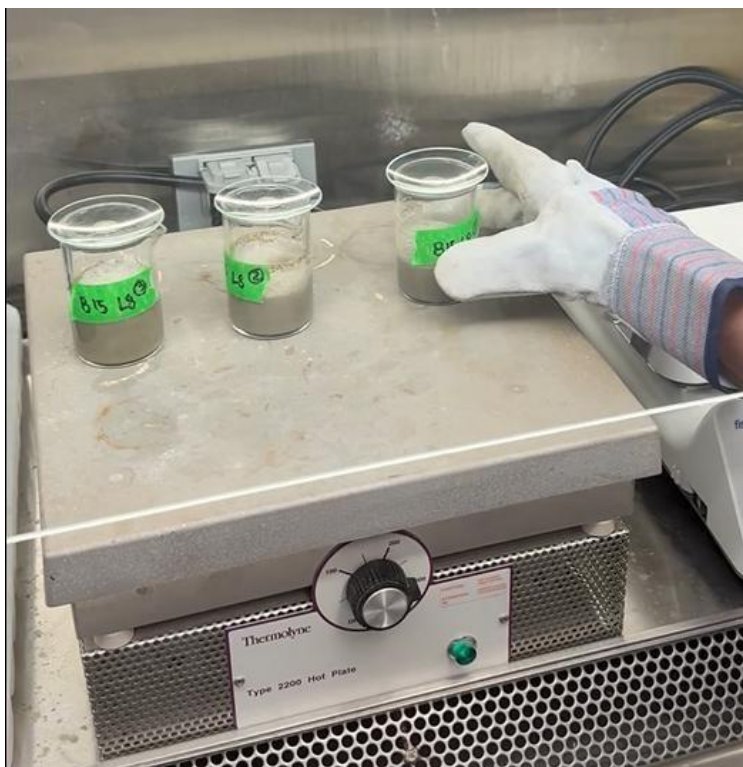


Figure 41. Removing a triplicate set of samples from hot plate

Once the beaker has cooled off enough to be handled by the touch, the sample solution can be carefully poured into the filter paper within the funnel. By the solution still being warm, it allows it to be filtered faster which is beneficial. In addition, a larger beaker with DI water can be placed on the hot plate while boiling samples, so it can be heated for use when filtering. The heated DI water can be poured into a wash bottle that can be used to rinse residue from the sides of the filter paper towards the bottom, as shown in Figure 42 below, because the residue is left behind when the solution level lowers while filtering. Once most of the contents of the beaker have been emptied into the filter, the remaining contents can be rinsed out with the wash bottle of DI water into the filter paper to ensure no solution is left in the beaker. It is important to note that project team members should be very careful not to overfill the flask with DI water beyond the 100 mL volume line because the sample will not be valid for testing. The painter's tape used to identify the beaker can then be placed on the flask to ensure that the samples are not misidentified.

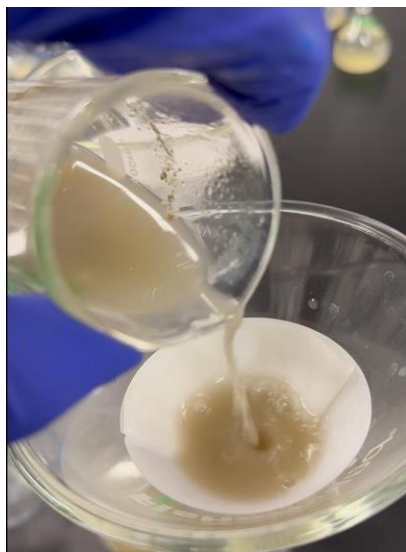


Figure 42. Pouring the boiled solution into the filter paper–filled funnel



Figure 43. Rinsing residue from sides of filter paper

After all the residue in the filter paper has been rinsed to the bottom and the solution has finished filtering, the flask should be left for about an hour or until the solution within the flask has returned to room temperature. Upon cooling to room temperature, the filter paper and funnel can be removed from the flask. The flask can then be filled to volume with room temperature DI water and sealed with a stopper, as shown in Figure 44 for concrete batch 19. The sample is then ready for analysis.

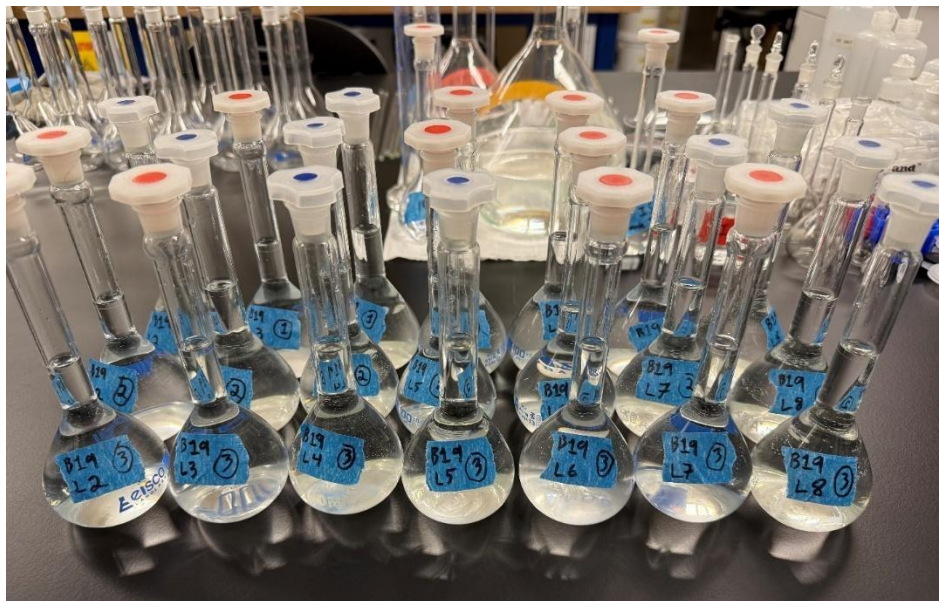


Figure 44. Samples of a concrete batch prepared for analysis

#### *4.1.3.5 Electrode Preparation and Maintenance*

To chemically analyze the concrete specimens prepared and digested in the previous sections, an electrode is used to determine the chloride content of the sample through titration. The electrode should be specific to testing for chloride ions with its recommended filling solution. In addition, the electrode should be connected to an ion specific electrode meter with a 0.1 mV resolution compatible with the electrode to be used. The project research team has used a Fisher brand Accumet Chloride Combination electrode for testing which includes its recommended filling solution. The meter used is a Thermo Scientific Orion Star which includes an automatic stirrer and electrode stand. The project team also has an Oakton Ion 700 meter which includes an electrode stand, but this unit does not have an integrated stirrer, so testing will be conducted with a stir plate and magnetic stir bars. The use of these two meters depends on availability and the need to operate in tandem.

The electrode should be prepared by filling it with its filling solution, flushing the electrode, refilling the electrode, polishing the tip with electrode polishing paper, and conditioning it with a solution. So, the criteria for restoring and conditioning the electrode are prior to using the electrode for the first time, prior to testing, and after every 36 analyses or a lapse in use of more than an hour. For the electrode's first use, there will be no filling solution in the electrode, so the nozzle of the small bottle of the solution should be inserted into the small hole on the side, towards the top of the electrode. By squeezing the bottle, the solution should be dispensed into the electrode until the level is just below the hole. For an actively used electrode, the procedure will start at this point. While holding the electrode over a sink, the white cap at the top of the electrode where the cable is attached should be pushed down to release the filling solution or flush the electrode. The electrode should then be refilled with the filling solution using the same procedure. Once complete, the sides and bottom of the electrode should be rinsed with DI water, while covering the hole, to ensure no filling solution remains on the outside of it nor any DI water entering the electrode. A piece of the pink polishing paper strips can then be cut off into a size just larger than the diameter of the tip of the electrode using scissors. The polishing paper has both a shiny and dull side, so a bit of DI water can be dispensed on the dull side of the paper and then rubbed on the tip of the electrode for about 30 seconds to polish it. This process can be reflected in Figure 45 below.

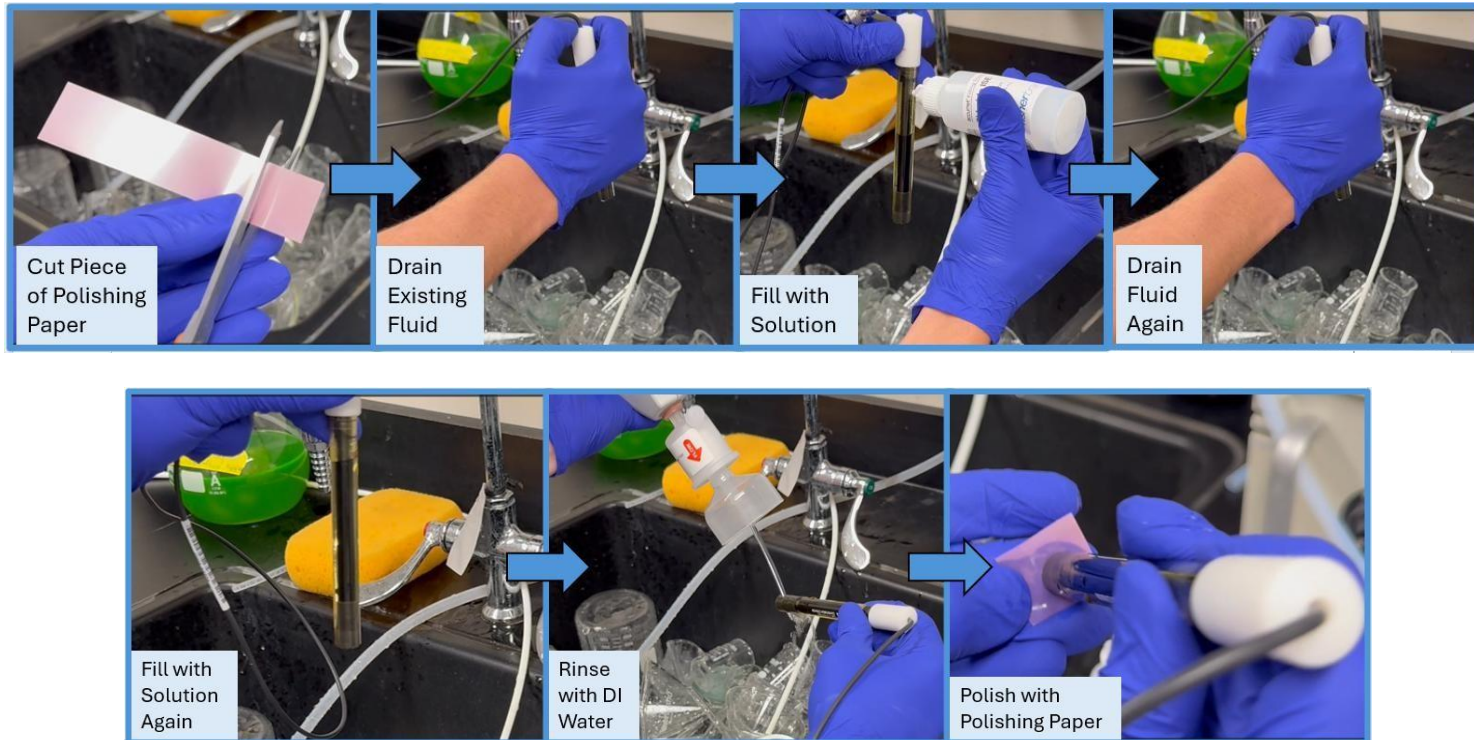


Figure 45. Flushing and polishing procedures for electrode

Maintenance The electrode should be placed in its stand and connected to the meter with its cable which then makes it ready to be conditioned in a solution. The conditioning solution is prepared in a 250 mL beaker with 100 mL of DI water and 2.5 mL of 0.01 N  $AgNO_3$  using the previously produced and stored solutions with dispensers. Also, 1 mL of ISA should be dispensed into the solution using a transfer pipet. The beaker should be placed under the stirrer and electrode situated in the stand, and they can then both be lowered into the solution. The stirrer can be turned on at a moderate constant rate for 10 minutes to condition the electrode, as shown in Figure 46. Once the time has elapsed, the electrode and stirrer can be raised from the solution and rinsed with DI water into the now used solution. Finally, the electrode can be dried off with lint-free tissue paper and lowered into a beaker of clean DI water when not in use. If the electrode is not anticipated to be used for a long period of time, it can be removed, drained of its filling solution, dried, capped with its rubber covering on the tip, and stored in a dry cabinet.

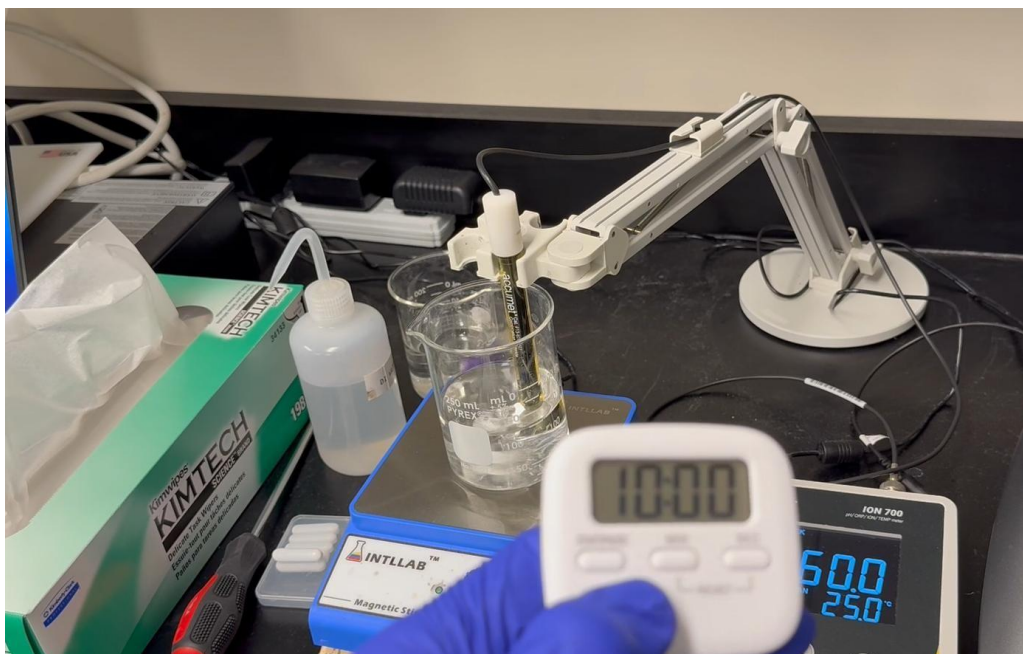


Figure 46. Conditioning the electrode in its solution

Before using the electrode for the first time, it is necessary to check the slope of the calibration curve to see if it is within the manufacturer's suggested mV range. This process should most likely only be done once, but it may be necessary if at some point the electrode performance seems strange. The slope is defined as the change in mV observed when the concentration changes by a factor of 10. So, a project team member should add 100 mL of DI water and 1 mL of ISA to a 250 mL beaker. The beaker should be placed under the stirrer and electrode situated in the stand, and they can then both be lowered into the solution. A project team member should then use a transfer pipet to add 1 mL of 1,000 mg/L chloride standard solution to the beaker. With the meter in mV mode, the reading can then be taken using the meter with the stirrer automatically being activated to stir at a moderate and constant rate, and once the reading is stable, it can be recorded.

Again, using a transfer pipet, 10 mL of 1,000 mg/L chloride standard solution should be added to the beaker. Once the reading is stable, the reading is recorded and the difference in readings is verified to be within the manufacturer's suggested range. If not, troubleshooting should take place using the instructions in the electrode manual and contacting support. Another check that should most likely be conducted just once is to identify the voltage potential threshold for determining which  $AgNO_3$  titrant concentration should be used to analyze a sample. The required solution is

produced by transferring 10 mL of 100 ppm chloride standard solution into a 100 mL volumetric flask and bringing it to volume with DI water. With a conditioned electrode, the solution should be measured to obtain the mV reading, which was 106.5 mV for the research conducted in this study. The reading calculated is the threshold at which point the project team member would transition from a 0.1 N concentration to a 0.01 N concentration. For example, if a sample had a reading lower than the threshold before titration had begun, a 0.1 concentration would be used for titration, and a reading higher than the threshold would be the contrary.

#### *4.1.3.6 Baseline Check*

As previously mentioned, 25 sets of control samples were conducted for blank samples with normality of 0.01 and 0.1 silver nitrate, chloride check samples (CCS), and laboratory control samples (LCS). Again, the reasoning to do these control tests before even initiating the analysis of the actual specimens was to validate that the project laboratory as well as its equipment and resources are effective and capable of performing such a baseline. The baseline should be tested in a specific sequence by starting with the 0.01 blank and followed by the 0.1 blank, CCS, and LCS. When conducting the 25 sets of control samples, this sequence was simply repeated 25 times. Yet, for normal testing of initial chloride and exposure samples the sequence must only be conducted each time the electrode is restored and conditioned. So, each of the baseline tests will be detailed below as well as the use of an FDOT spreadsheet which validates the test results allowing for the continuation of the baseline sequence. First, the 0.01 blank solution can be prepared by dispensing 100 mL of DI water into a 250 mL beaker as well as 1 mL of ISA using a transfer pipet. The beaker should be placed under the stirrer and conditioned electrode situated in the stand, and they can then both be lowered into the solution. Then, 1 mL of 0.01 N  $AgNO_3$  can be dispensed into the solution, and the first reading can be taken using the electrode meter, as shown in Figure 47. The recorded value is the scaling potential for the 0.01 N concentration. Next, 0.5 mL of 0.01 N  $AgNO_3$  should be dispensed and the reading recorded for four increments. The result will be 5 recorded readings in mV with the first being the scaling potential and the last four data points.



Figure 47. Baseline check for 0.01 blank sample

The values should be entered into the FDOT spreadsheet along with the normality and electrode slope previously identified to calculate the end point which should be  $0 \pm 0.1$ , as shown in Figure 48. The project team rarely experienced invalid blank sample end points, but if the endpoint were to be out of tolerance, there could be several causes to consider such as the quality of the DI water, preparation of the 0.01 N  $AgNO_3$  and/or ISA, or the maintenance of the electrode. It may require some thought to conclude what the issue might be, but it should be resolved before continuing to the 0.1 blank solution. As a note, the only time the project team experienced an invalid blank endpoint was due to a filter going bad in the DI water machine which led to the possible contamination of all solutions and DI water. Once the 0.01 blank test was complete and validated, the same process could be conducted for the 0.1 blank test except with the use of the 0.1 N  $AgNO_3$  solution. Most likely, if the 0.01 blank test was valid, the 0.1 blank should be as well.

<b>Normality:</b>	0.01		<b>Normality:</b>	0.1
<b>Elect. Slope:</b>	25.879		<b>Elect. Slope:</b>	25.879
<b>AgNO<sub>3</sub> (ml)</b>	<b>Potential (mV)</b>		<b>AgNO<sub>3</sub> (ml)</b>	<b>Potential (mV)</b>
1.00	233.0		1.00	293.3
1.50	243.8		1.50	303.6
2.00	251.3		2.00	310.7
2.50	257.0		2.50	316.3
3.00	261.7		3.00	320.7
	<b>End Point</b>		<b>End Point</b>	
	0.0412		-0.0366	

Figure 48. Baseline check for 0.01 and 0.1 blanks

Once the 0.1 blank test was complete and validated, the CCS solution could be prepared by first gathering the 1,000 mL volumetric flask filled with CCS and utilizing a funnel to pour it into a 100 ml volumetric flask. It is preferred to slightly overfill the flask above the volume line and use a clean pipet to precisely bring the CCS to volume. Then, the 100 mL of CCS can be transferred from the flask to a 250 mL beaker as well as 1 mL of ISA using a transfer pipet. In this control test, 0.5 mL increments of 0.01 *AgNO<sub>3</sub>* are dispensed into the solution until the scaling potential for the 0.01 concentration is reached or just exceeded. Then, the four increments of 0.5 mL can be conducted with the same procedure as the blanks. The readings should again be input into the correct section of the FDOT spreadsheet, but the only other inputs should be the volume of 0.01 *AgNO<sub>3</sub>* required to reach the scaling potential and the volume of the CCS used in the solution which should be 100 ml. Upon determining the end point, the blank end point can be subtracted to calculate the CCS end point which is displayed as a concentration with a tolerance of 3 ppm ± 0.15 ppm. If the CCS control test is not valid, the reasoning would most likely be due to the preparation of the CCS or the filling solution of the electrode. The CCS should not expire, but the project team has experienced that over time that CCS can become ineffective in producing a valid concentration in tolerance. The issue can often be resolved by reparing the CCS solution, but if not, the electrode may need to be reconditioned, and the sequence must be restarted with the blanks.

Once the CCS test is complete and validated, the LCS solution can be prepared by simply gathering the 100 mL volumetric flask produced from digestion. Before the solution is transferred to a 250 mL beaker, it should first be shaken up while holding the stopper firmly to ensure that the contents

have not settled and become stuck to the inside of the flask. It may require pouring a small amount of the LCS solution into the beaker and then recovering the top of the flask to shake it up with less volume inside to disturb the surface. Ultimately, the 100 mL of LCS should be transferred into the beaker to be analyzed. Each of the solutions used for the baseline check for chemical analysis can be shown in Figure 49.



Figure 49. Prepared baseline check solutions

In continuation, once the electrode is lowered into the solution, an initial reading can be taken to determine if the potential is less than what would be measured in a 10-ppm chloride solution. If the reading is in fact less than that value, the 0.01  $AgNO_3$  should be used for titration as discussed in the electrode maintenance section. Then the same process as the CCS test should be conducted to determine the endpoint and subtract the selected blank's endpoint to calculate the LCS endpoint which can be displayed as a density in  $lb/yd^3$ . The unit weight of the LCS as determined by the FDOT as well as the mass of the sample being about 4 g should be input into the FDOT spreadsheet, as shown in

Figure 50. The project team has very rarely had an LCS control sample be invalid, but if it were to occur, the LCS solution should be reproduced. It is beneficial to prepare and digest multiple LCS samples to have on hand if this were to arise in order to prevent a significant delay. Once the LCS test was completed and validated, the analysis of the initial chloride and exposure samples can be conducted.

CCS		LCS	
Volume (mL):	100	Unit Weight:	3800
		Mass (g):	4
AgNO <sub>3</sub> (ml)	Potential (mV)	AgNO <sub>3</sub> (ml)	Potential (mV)
2.00	238.7	1.50	234.9
2.50	247.6	2.00	243.6
3.00	254.1	2.50	250.3
3.50	259.3	3.00	255.3
4.00	264.2	3.50	259.6
Intercp:	0.00845	Intercp:	0.00288
End Pt:	0.852	End Pt:	0.288
ppm	2.876	lb/yd <sup>3</sup>	0.083
ppm	good	lb/yd <sup>3</sup>	good

Figure 50. Baseline check for CCS and LCS

#### 4.1.3.7 Analysis of Samples

The initial chloride samples and exposure samples can both be tested with the same procedures as the LCS. Once the required normality of silver nitrate is determined, it should be entered as an input into the FDOT spreadsheet as well, it is important to note that, while the initial chloride sample usually needs a smaller volume of titrant to reach the scaling potential, the first few layers of the exposure samples may require a much higher volume of silver nitrate to reach the scaling potential. So, with this being the case, rather than dispensing a single 0.5 mL increment and waiting for the reading, the common practice used by the project team is to apply several increments at a time to speed up the process. Yet, if this procedure is utilized, the project team member conducting the test should be very careful to record how many increments have been used with a simple tally or even changing the value in the spreadsheet iteratively. In addition, the team members must be very familiar with the trend and pattern of the increasing mV with added increments of titrant. The potential often increases in small increments until it reaches closer to its peak and then the value jumps significantly without expectation, so to avoid greatly surpassing the scaling potential, the user must be organized, attentive, and patient to add several increments at a time.

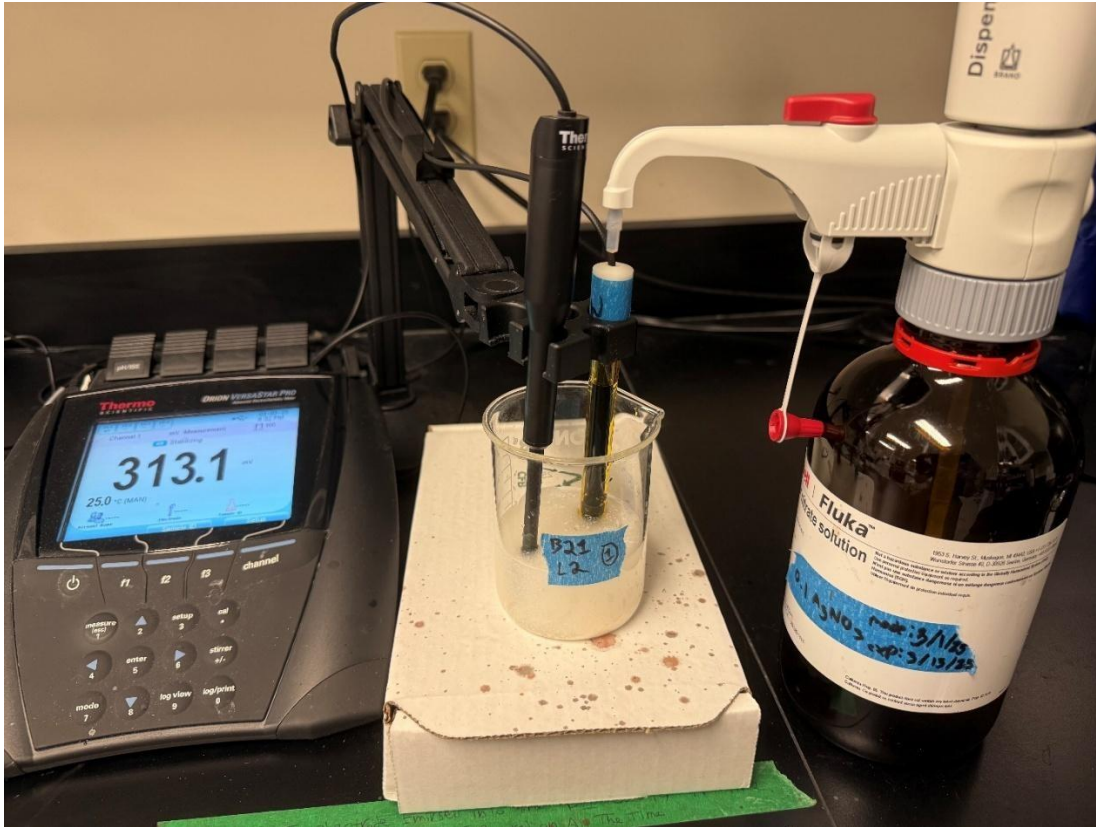


Figure 51. Chemical analysis of exposure sample using 0.1 N silver nitrate

Once each of the three samples of the triplicate have been tested, the values are provided in several different units including percent mass, ppm,  $lb/yd^3$ , and  $kg/m^3$ . Based upon FM5516, the chloride concentration for the triplicate values must be within a specified range of no more than  $0.08 lb/yd^3$  when using 0.01 N  $AgNO_3$  and no greater than  $0.8 lb/yd^3$  with 0.1 silver nitrate. If the range is larger than these values, the test is invalid which requires the layer to be reanalyzed as indicated by red highlighting in the FDOT spreadsheet. The individual triplicate values are then averaged into a single value representing the concrete mix for initial chloride sample or the layer for exposure samples. Although the percentage mass will be used in the following section for developing the chloride diffusion profile, the density of chloride in the concrete sample is beneficial to visualize how much chloride is present in the specific concrete mix sample. With the first layer of the exposure sample being neglected, the expected trend for a sample with increasing depth would be a decrease in chloride content, and this has been widely observed by the project team. To reflect these findings further, the results are provided in the following section.

## 4.1.4 Results

### 4.1.4.1 Chloride Content

After completing the preparation and testing procedures for each concrete powder sample, the immediate results from the bulk diffusion (BD) test method were obtained. Concrete powder samples were collected from each mix prior to exposure to the chloride solution to determine the *initial chloride content*. This step is essential for establishing the baseline chloride concentration in the concrete, which is later used to calculate the chloride diffusion coefficient.

The titration process was performed on triplicate samples using the FDOT chloride determination spreadsheet, ensuring consistency and accuracy across all mixes. Figure 52 presents a sample of the calculations used to determine the chloride content. The summarized results for the initial chloride content of all concrete mixes are presented in Table 7, with corresponding values reported in both pounds per cubic yard (lb/yd<sup>3</sup>) and percentage by weight of concrete.

Replicate A		Replicate B		Replicate C	
MAC #	CI B2	MAC #	CI B2	MAC #	CI B2
Unit Weight:	3846.474	Unit Weight:	3846.474	Unit Weight:	3846.474
Mass (g)	4.0000	Mass (g)	4.0000	Mass (g)	4.0000
Normality:	0.01	Normality:	0.01	Normality:	0.01
AgNO <sub>3</sub> (ml)	Potential (mV)	AgNO <sub>3</sub> (ml)	Potential (mV)	AgNO <sub>3</sub> (ml)	Potential (mV)
2.0	238.8	2.0	240.4	2.0	240.8
2.50	245.7	2.50	247.2	2.50	247.2
3.00	251.2	3.00	252.5	3.00	252.8
3.50	255.6	3.50	256.8	3.50	257.4
4.00	259.4	4.00	260.6	4.00	261.2
Intercp:	0.00413	Intercp:	0.00363	Intercp:	0.00420
End Pt:	0.415	End Pt:	0.364	End Pt:	0.421
%	0.0040	%	0.0035	%	0.0041
ppm	40	ppm	35	ppm	41
lb/yd <sup>3</sup>	0.154	lb/yd <sup>3</sup>	0.136	lb/yd <sup>3</sup>	0.156
kg/m <sup>3</sup>	0.091	kg/m <sup>3</sup>	0.081	kg/m <sup>3</sup>	0.092
			Average	Range	
		%	0.0039	0.0005	
		ppm	38.6	5.0766	
		lb/yd <sup>3</sup>	0.1485	0.0195	
		kg/m <sup>3</sup>	0.0881	0.0116	

Figure 52. Sample for the calculations of the initial chloride content

Table 7. Summary of Initial Chloride Content for Concrete Mixes

<b>BD Summary of Initial Chloride Content</b>		
<b>Mix Number</b>	<b>Chloride content (lb/yd3)</b>	<b>% weight of concentration</b>
<b>1</b>	0.1515	0.0038
<b>2</b>	0.1485	0.0039
<b>3</b>	0.1599	0.0041
<b>4</b>	0.2972	0.0074
<b>5</b>	0.2476	0.0063
<b>6</b>	0.1963	0.0052
<b>7</b>	1.2015	0.0299
<b>8</b>	0.1658	0.0042
<b>9</b>	0.1112	0.0029
<b>10</b>	0.1802	0.0045
<b>11</b>	0.1777	0.0045
<b>12</b>	0.1688	0.0044
<b>13</b>	0.3691	0.0092
<b>14</b>	0.3449	0.0089
<b>15</b>	0.3239	0.0086
<b>16</b>	0.2576	0.0063
<b>17</b>	0.1836	0.0047
<b>18</b>	0.2314	0.0062
<b>19</b>	0.2079	0.0052
<b>20</b>	0.2994	0.0077
<b>21</b>	0.3398	0.009
<b>22</b>	0.071	0.0018
<b>23</b>	0.0853	0.0021
<b>24</b>	0.1557	0.0039
<b>SFM1</b>	0.2714	0.0068
<b>SFM2</b>	0.2794	0.0072
<b>SFM3</b>	0.3571	0.0087
<b>Slag 2-1</b>	0.1693	0.0043
<b>Slag 2-2</b>	0.1685	0.0043
<b>Slag 2-3</b>	0.1421	0.0038
<b>Slag 2-4</b>	0.0731	0.0018

The chloride content for exposure samples from each batch of concrete was also recorded, but unlike the initial chloride samples, a batch of concrete had each of its layers tested in triplicate. The discussion of the chloride content results will be continued in the following section for better visual illustration of the data.

#### 4.1.4.2 Chloride Diffusion Coefficient Calculation:

After determining chloride concentrations at different depths using ASTM C1556, we applied Fick's Second Law shown in Eq. 1 to calculate the apparent chloride diffusion coefficient after 180 days of exposure to a chloride solution.

$$C(x, t) = C_s - (C_s - C_i) \cdot \operatorname{erf}\left(\frac{x}{\sqrt{4 \cdot D_a \cdot t}}\right) \quad \text{Eq. 12}$$

where,

$C(x, t)$  = chloride concentration, measured at depth  $x$  and exposure time  $t$ , mass %,

$C_s$  = projected chloride concentration at the interface between the exposure liquid and test specimen that is determined by the regression analysis, mass %,

$C_i$  = initial chloride-ion concentration of the cementitious mixture prior to submersion in the exposure solution, mass %

$\operatorname{erf}$  = the error function,

$D_a$  = apparent chloride diffusion coefficient,  $\text{m}^2/\text{s}$ ,

$x$  = depth below the exposed surface (to the middle of the layer),  $\text{m}$ , and

$t$  = the exposure time, second.

##### 4.1.4.2.1 Chloride Diffusion Profile Results After 180 Days

After 180 days (six months) of exposure in the chloride solution, thirty-one (31) concrete mixes were tested to assess the chloride diffusion profiles. Each specimen was subjected to profile grinding, and powder samples were obtained at increasing depths from the exposed surface. For each mix, grinding continued until the chloride content at a specific depth reached the initial chloride content value. This often required grinding more than the recommended eight layers, sometimes extending to ten or more layers.

The chloride content at each depth was measured and recorded. The following Tables (Table 6 through Table 35) and Figures (Figure 31 through Figure 61) present the chloride content results for all mixes, plotted as a function of depth. Each figure provides a visual representation of chloride penetration into the concrete mix over the six-month exposure period. The apparent chloride diffusion rates for the different mixes are presented in Table 36. Since all 31 mixes have now been included in this analysis, complete trends and correlations have been established and are being evaluated. The research team is currently analyzing the chloride diffusion data and comparing results with the RMT findings to develop correlations and assess performance differences among all mixes.

4.1.4.2.2 Mix 01:

Table 14. Chloride content results for mix 01

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	2	3	0.777	31.17
3	2	5	0.679	27.24
4	2	7	0.64	25.68
5	3	10	0.547	21.95
6	3	13	0.49	19.67
7	3	16	0.464	18.6
8	4	20	0.318	12.75
9	6.35	26.35	0.255	10.23
10	6.35	32.7	0.194	7.78
11	6.35	39.05	0.134	5.39
12	6.35	45.4	0.097	3.89
13	6.35	51.75	0.067	2.67
14	4.5	56.25	0.03	1.21

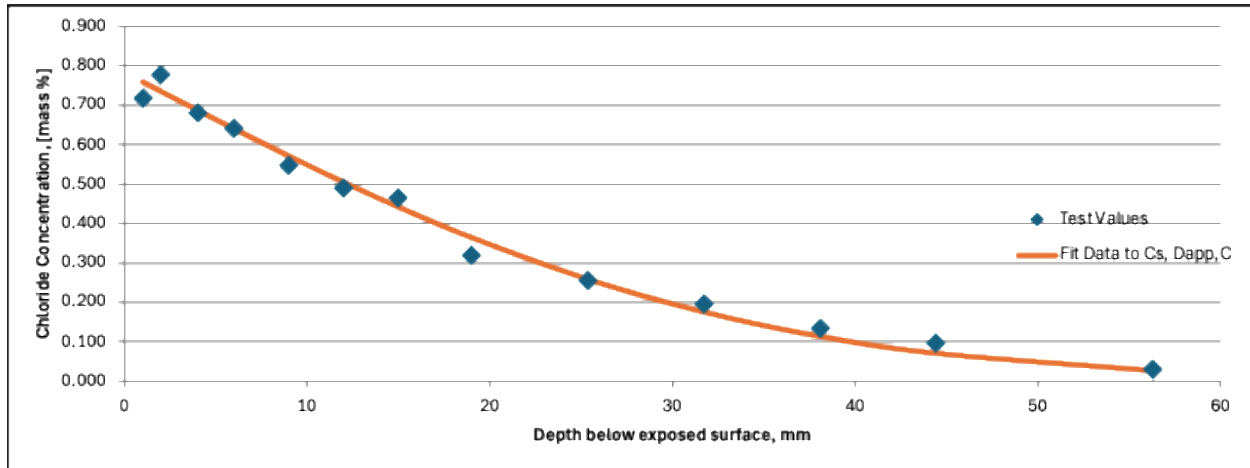


Figure 53. Chloride profile for mix 01

4.1.4.2.3 Mix 02:

Table 15. Chloride content results for mix 02

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	2	3	1.198	46.06
3	2	5	1.066	41.01
4	2	7	0.875	33.64
5	3	10	0.776	28.44
6	3	13	0.61	23.47
7	3	16	0.576	22.14
8	4	20	0.432	16.62
9	6.35	26.35	0.249	9.59
10	6.35	32.7	0.125	4.79
11	6.35	39.05	0.0334	1.28
12	6.35	45.4	0.0071	0.278
13	3.4	48.8	0.006	0.23

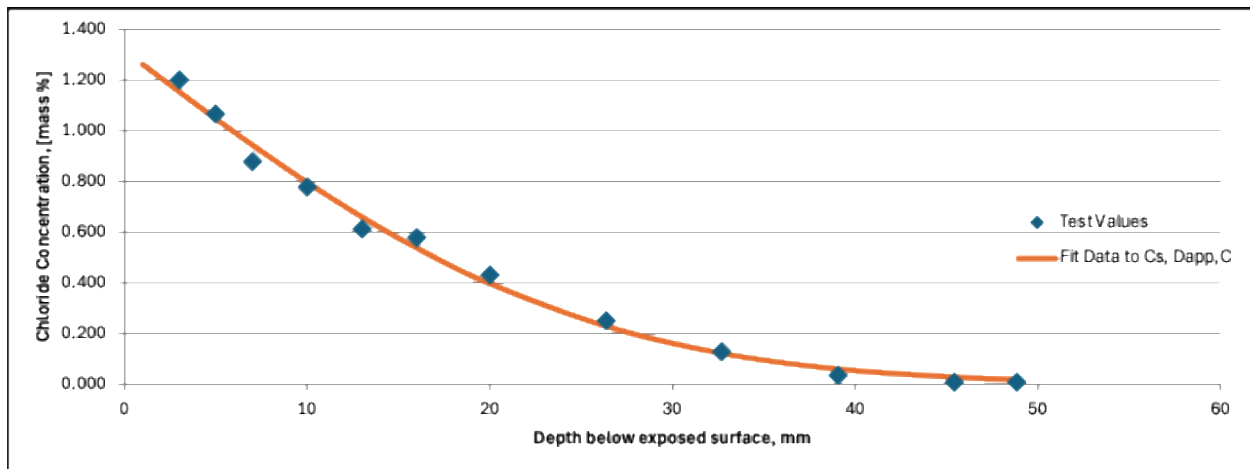


Figure 54. Chloride profile for mix 02

4.1.4.2.4 Mix 03:

Table 16. Chloride content results for mix 03

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	2	3	1.03	40.47
3	2	5	0.877	34.47
4	2	7	0.72	28.3
5	3	10	0.55	21.74
6	3	13	0.475	18.66
7	3	16	0.455	17.9
8	4	20	0.36	14.17
9	6.35	26.35	0.262	10.29
10	6.35	32.7	0.161	6.32
11	6.35	39.05	0.0969	3.81
12	6.35	45.4	0.0403	1.585
13	6.35	51.75	0.0081	0.318

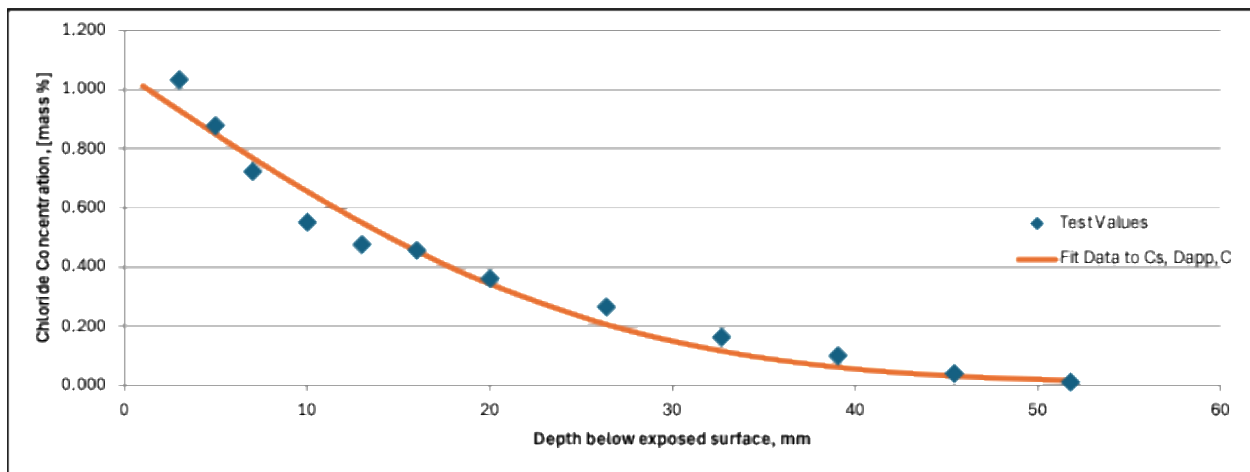


Figure 55. Chloride profile for mix 03

4.1.4.2.5 Mix 04:

Table 17. Chloride content results for mix 04

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	1	2	0.777	31.52
3	1	3	0.679	27.7
4	1	4	0.64	24.84
5	2	6	0.547	23.14
6	2	8	0.49	19
7	2	10	0.464	16.52
8	2	12	0.318	13.03
9	6.35	18.35	0.255	9.93
10	6.35	24.7	0.194	4.12
11	6.35	31.05	0.0049	0.1973

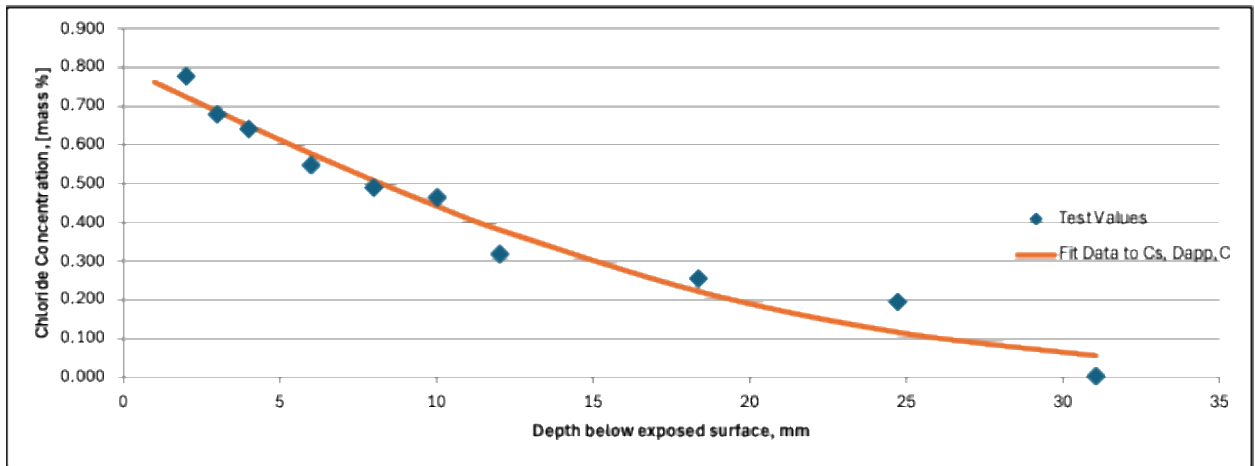


Figure 56. Chloride profile for mix 04

4.1.4.2.6 Mix 05:

Table 18. Chloride content results for mix 05

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	2	3	0.959	37.51
3	2	5	0.808	31.58
4	2	7	0.706	27.6
5	3	10	0.683	26.69
6	3	13	0.476	18.61
7	3	16	0.381	14.7
8	4	20	0.35	13.72
9	6.35	26.35	0.27	10.65
10	6.35	32.7	0.14	5.49

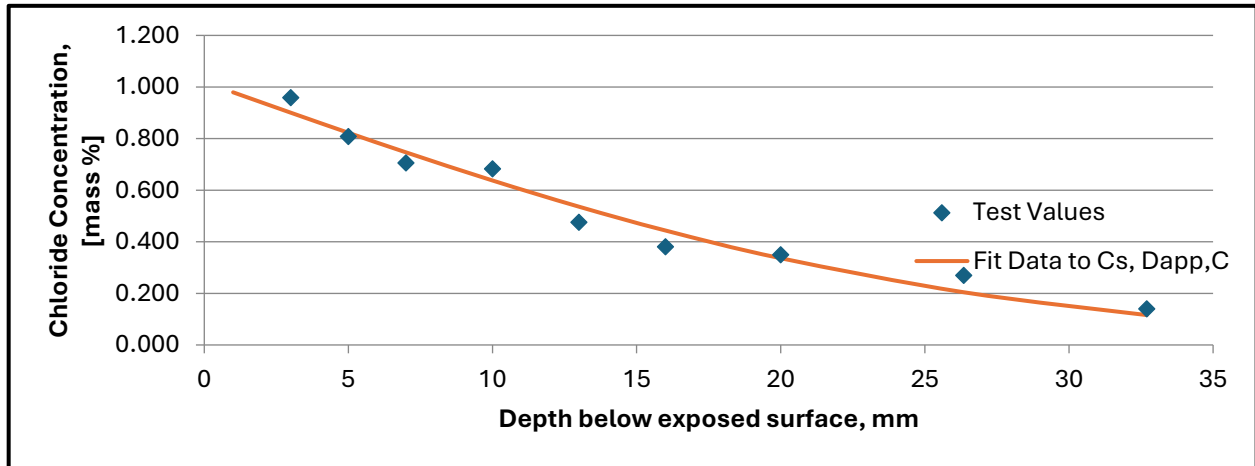


Figure 57. Chloride profile for mix 05

4.1.4.2.7 Mix 06:

Table 19. Chloride content results for mix 06

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	2	3	0.81	30.97
3	2	5	0.7	26.73
4	3	8	0.56	21.41
5	4	12	0.49	18.63
6	4	16	0.35	13.35
7	4	20	0.21	8.02
8	5	25	0.086	3.3
9	6.35	31.35	0.0243	0.932
10	6.35	37.7	0.009	0.342

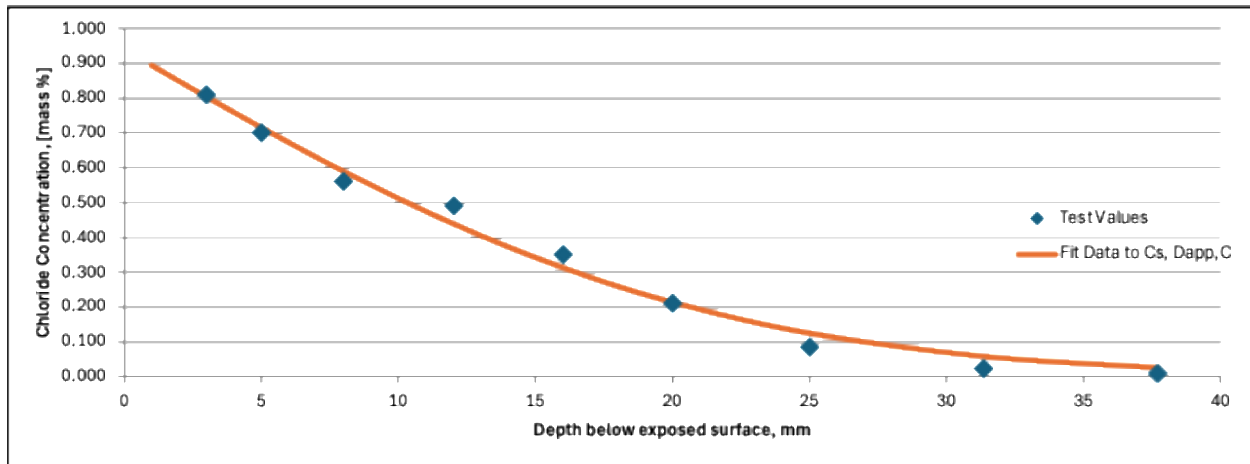


Figure 58. Chloride profile for mix 06

4.1.4.2.8 Mix 07:

Table 20. Chloride content results for mix 07

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	1	2	0.604	30.98
3	1	3	0.814	24.21
4	1	4	0.506	19.24
5	1	5	0.425	16.18
6	1	6	0.347	13.22
7	1	7	0.322	12.25
8	2	9	0.205	7.81
9	2	11	0.09	3.6

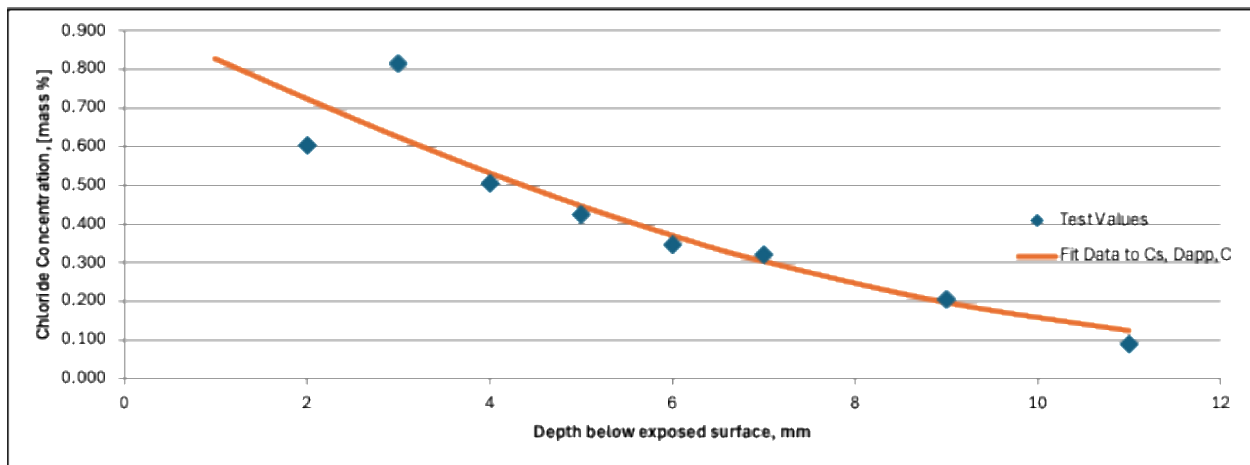


Figure 59. Chloride profile for mix 07

4.1.4.2.9 Mix 08:

Table 21. Chloride content results for mix 08

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	1	2	0.9068	35.4551
3	1	3	0.7031	27.4923
4	2	5	0.5262	20.5729
5	2	7	0.3882	15.1779
6	2	9	0.2651	10.3665
7	3	12	0.1431	5.5939
8	4	16	0.0544	2.1278
9	5	21	0.0097	0.381

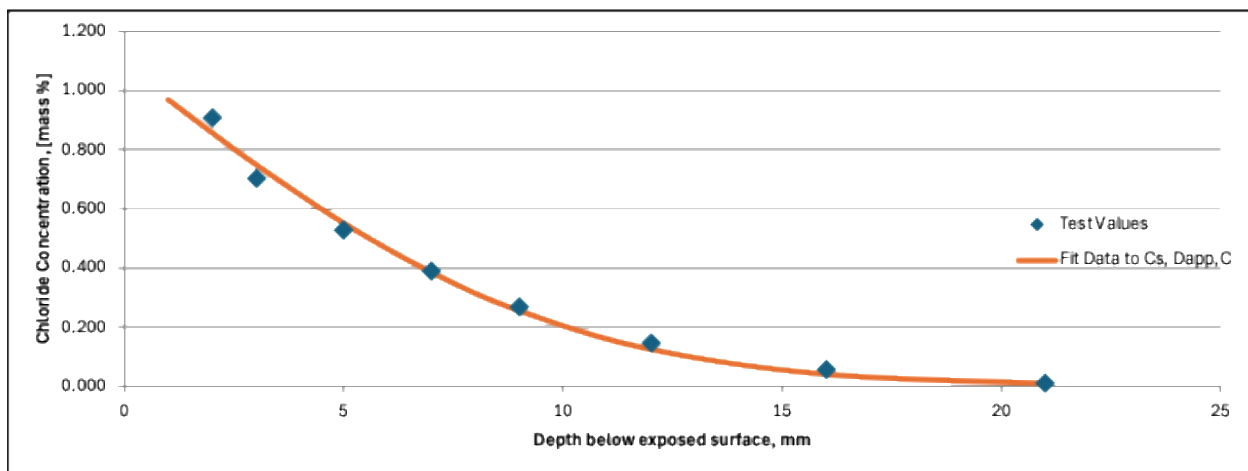


Figure 60. Chloride profile for mix 08

4.1.4.2.10 Mix 09:

Table 22. Chloride content results for mix 09

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	2	3	1.1949	45.5009
3	2	5	1.0104	38.4767
4	2	7	0.8267	31.4792
5	3	10	0.5968	22.7275
6	3	13	0.3911	14.8941
7	3	16	0.1716	6.5352
8	4	20	0.0893	3.4013

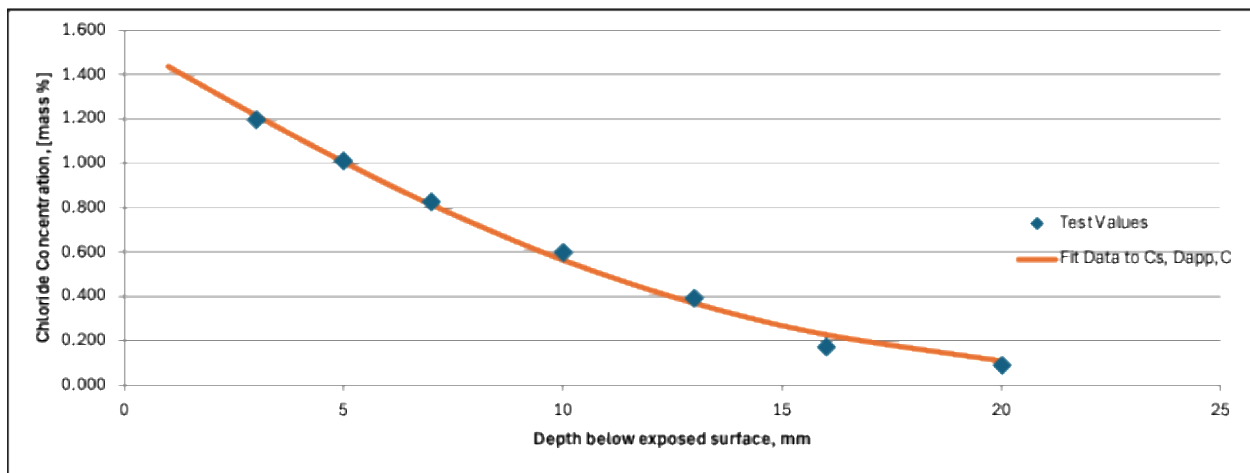


Figure 61. Chloride profile for mix 09

4.1.4.2.11 Mix 10:

Table 23. Chloride content results for mix 10

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	1	2	0.7466	30.1794
3	1	3	0.616	24.8998
4	1	4	0.5134	20.7507
5	1	5	0.413	16.6923
6	1	6	0.36	14.5509
7	2	8	0.3057	12.3567
8	2	10	0.1999	8.0801

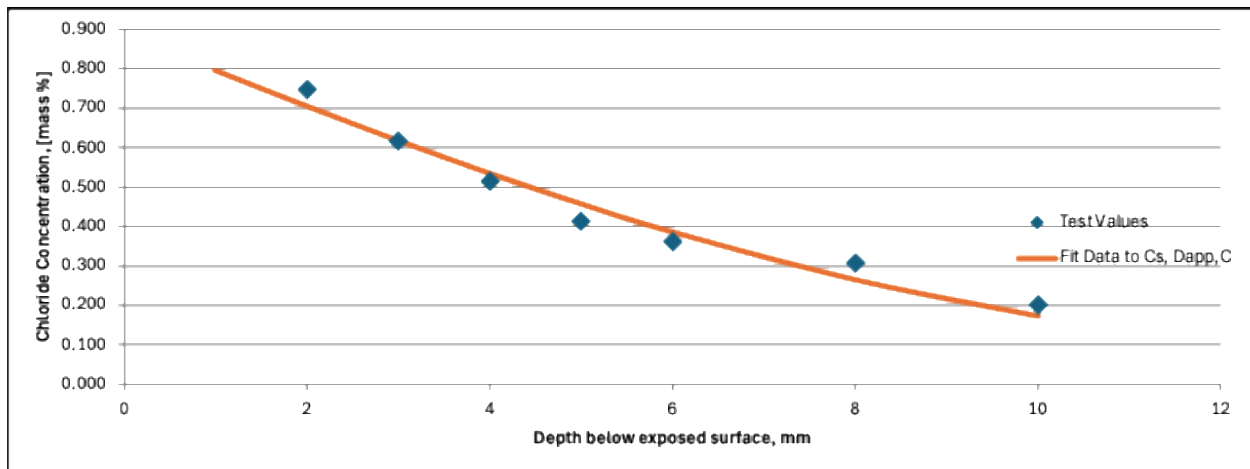


Figure 62. Chloride profile for mix 10

4.1.4.2.12 Mix 11:

Table 24. Chloride content results for mix 11

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	1	2	0.8823	34.6537
3	1	3	0.7896	31.0132
4	1	4	0.6153	24.164
5	2	6	0.4364	17.1383
6	2	8	0.2884	11.3252
7	2	10	0.205	8.0504
8	2	12	0.1303	5.1175

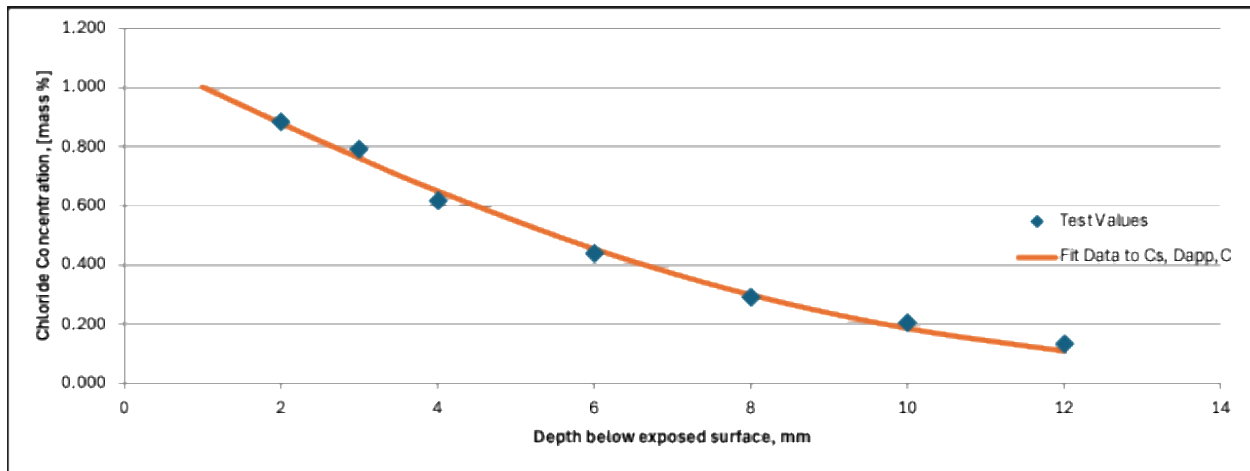


Figure 63. Chloride profile for mix 11

4.1.4.2.13 Mix 12:

Table 25. Chloride content results for mix 12

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	2	3	0.9884	37.7061
3	2	5	0.7529	28.7218
4	2	7	0.5493	20.9549
5	3	10	0.3283	12.5252
6	3	13	0.1584	6.0411
7	3	16	0.0656	2.504
8	4	20	0.0235	0.8949

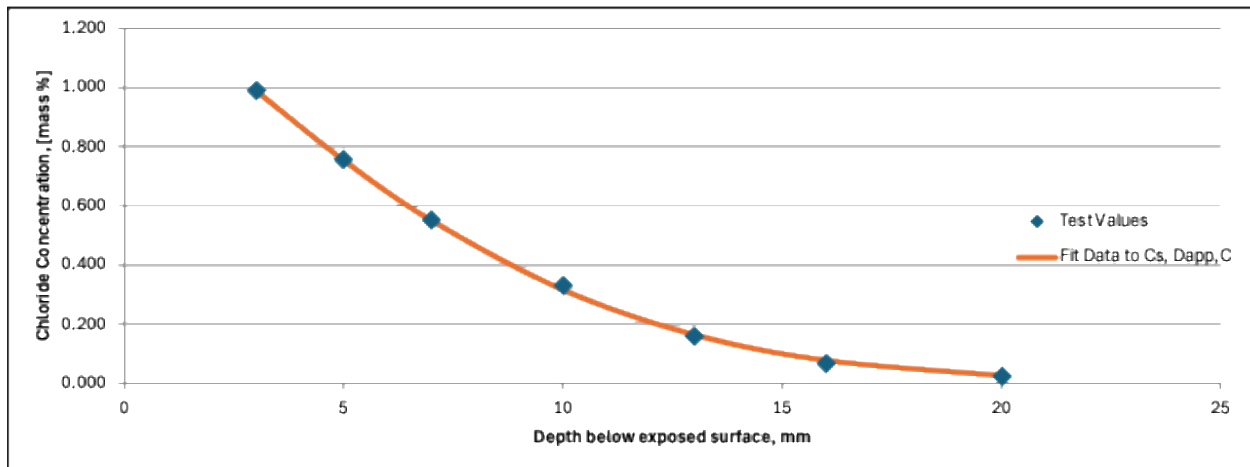


Figure 64. Chloride profile for mix 12

4.1.4.2.14 Mix 13:

Table 26. Chloride content results for mix 13

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	1	2		
3	1	3	1.1463	45.8535
4	1	4	1.0542	42.1664
5	1	5	0.928	37.119
6	1	6	0.7924	31.6976
7	2	8	0.6428	25.7136
8	2	10	0.511	20.442

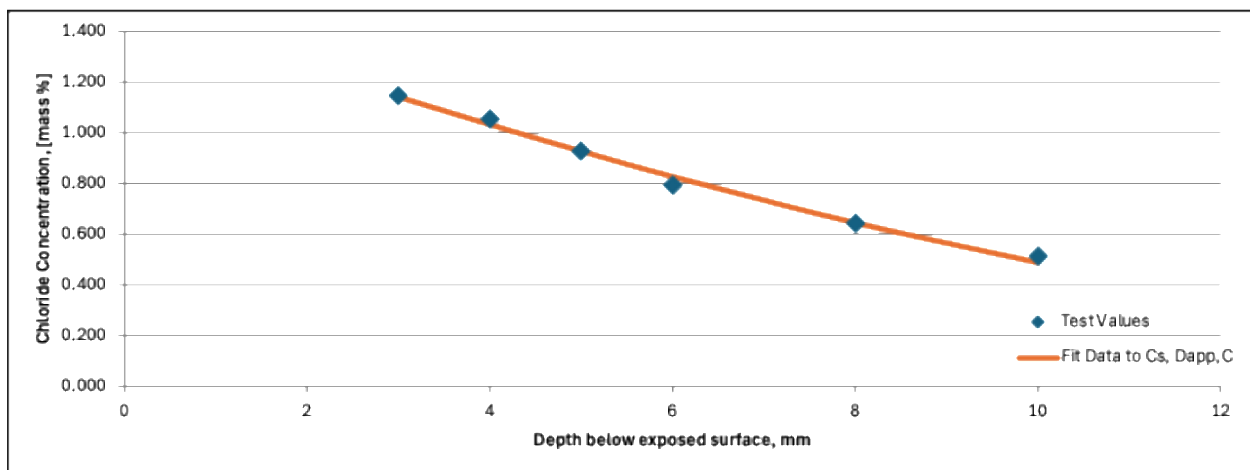


Figure 65. Chloride profile for mix 13

4.1.4.2.15 Mix 14:

Table 27. Chloride content results for mix 14

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	1	2	0.9333	36.2951
3	1	3	0.8364	32.529
4	1	4	0.7845	30.5096
5	2	6	0.6935	26.9693
6	2	8	0.5275	20.516
7	2	10	0.4505	17.5189
8	2	12	0.331	12.872

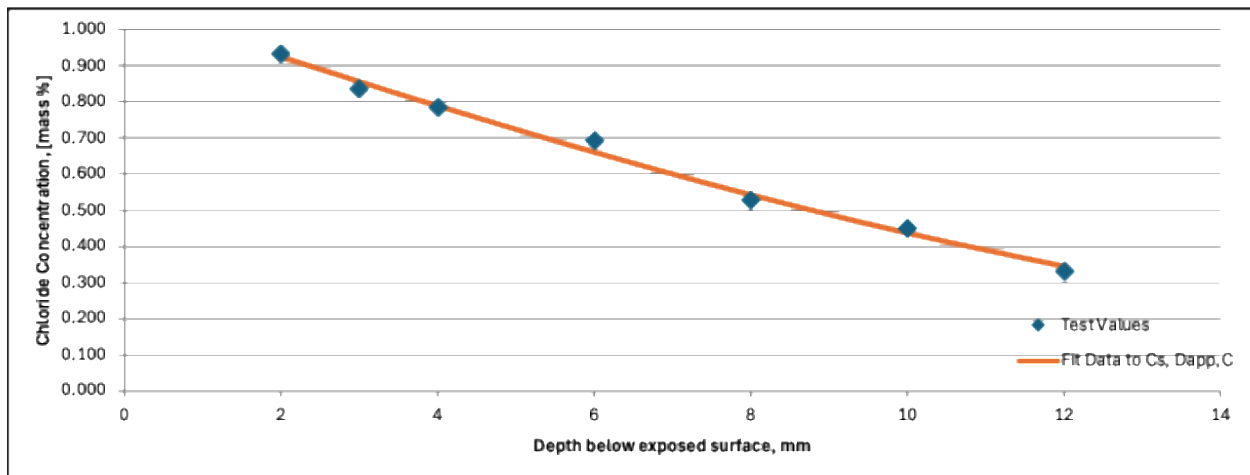


Figure 66. Chloride profile for mix 14

4.1.4.2.16 Mix 15:

Table 28. Chloride content results for mix 15

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	1	2	1.0539	39.722
3	1	3	1.126	42.4372
4	2	5	1.0703	40.339
5	2	7	0.8993	33.8949
6	2	9	0.7662	28.8789
7	3	12	0.7081	26.69
8	4	16	0.4954	18.6732
9	5	21	0.2306	8.6927
10	5	26	0.1286	4.8452

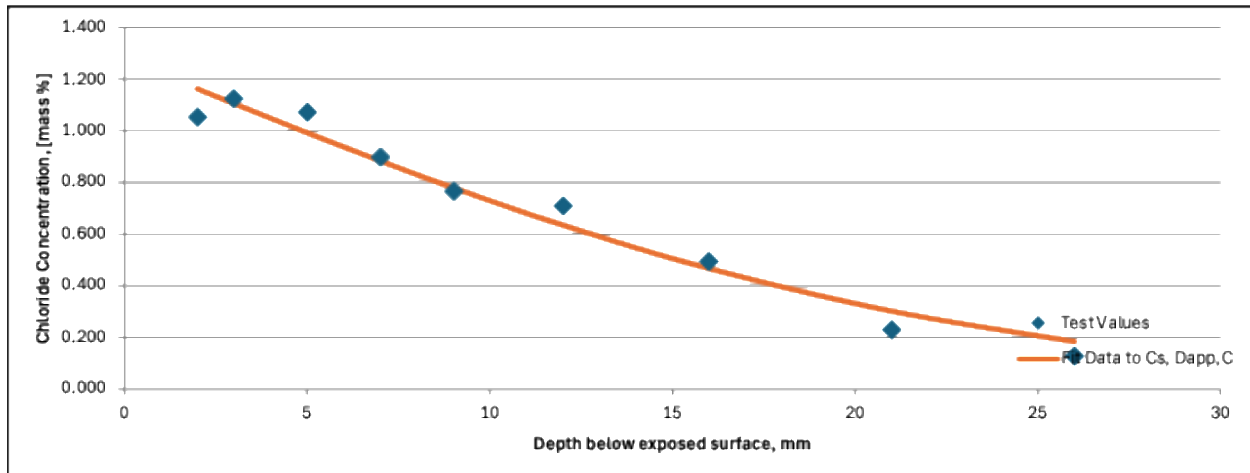


Figure 67. Chloride profile for mix 15

4.1.4.2.17 Mix 16:

Table 29. Chloride content results for mix 16

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	1	2	0.8642	35.0781
3	1	3	0.6658	27.0258
4	1	4	0.5143	20.8748
5	1	5	0.3984	16.1727
6	1	6	0.3236	12.1352
7	2	8	0.2182	8.8565
8	2	10	0.1147	4.6563

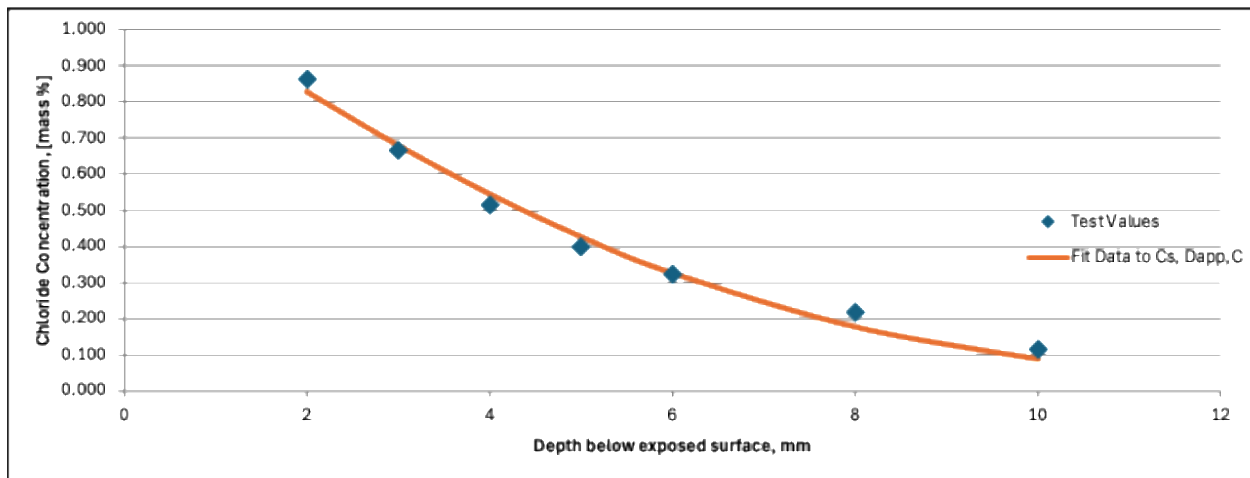


Figure 68. Chloride profile for mix 16

4.1.4.2.18 Mix 17:

Table 30. Chloride content results for mix 17

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	1	2	0.8039	31.3908
3	1	3	0.666	26.0062
4	1	4	0.5672	22.1506
5	2	6	0.4483	17.5074
6	2	8	0.344	13.4351
7	2	10	0.2655	10.3663
8	2	12	0.1895	7.3998

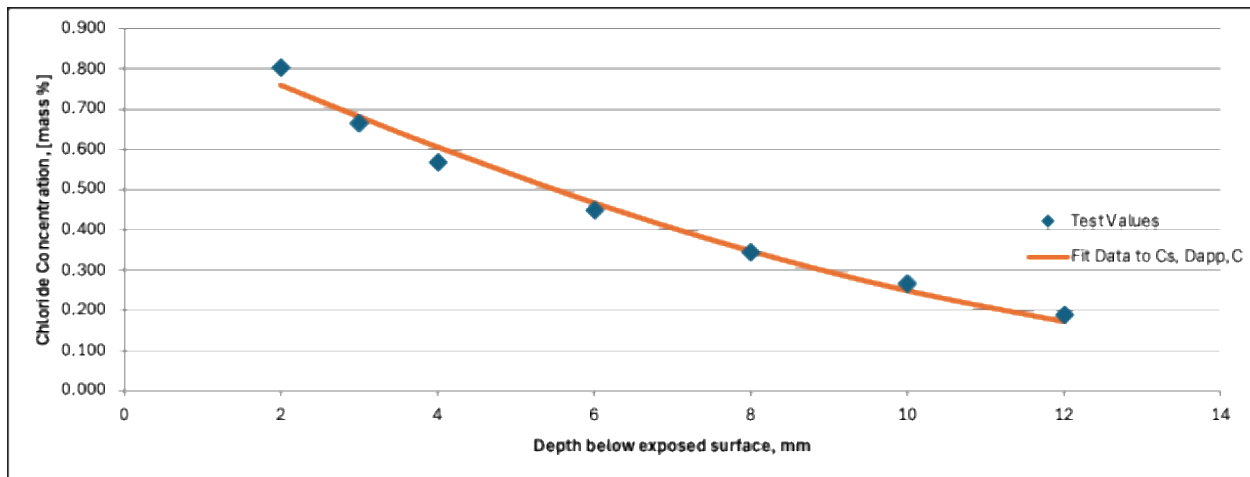


Figure 69. Chloride profile for mix 17

4.1.4.2.19 Mix 18:

Table 31. Chloride content results for mix 18

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	2	3	1.0539	39.5307
3	2	5	0.7387	27.7087
4	2	7	0.5665	21.2494
5	3	10	0.2946	11.0505
6	3	13	0.1084	4.0645
7	3	16	0.0407	1.5261
8	4	20	0.0171	0.6421

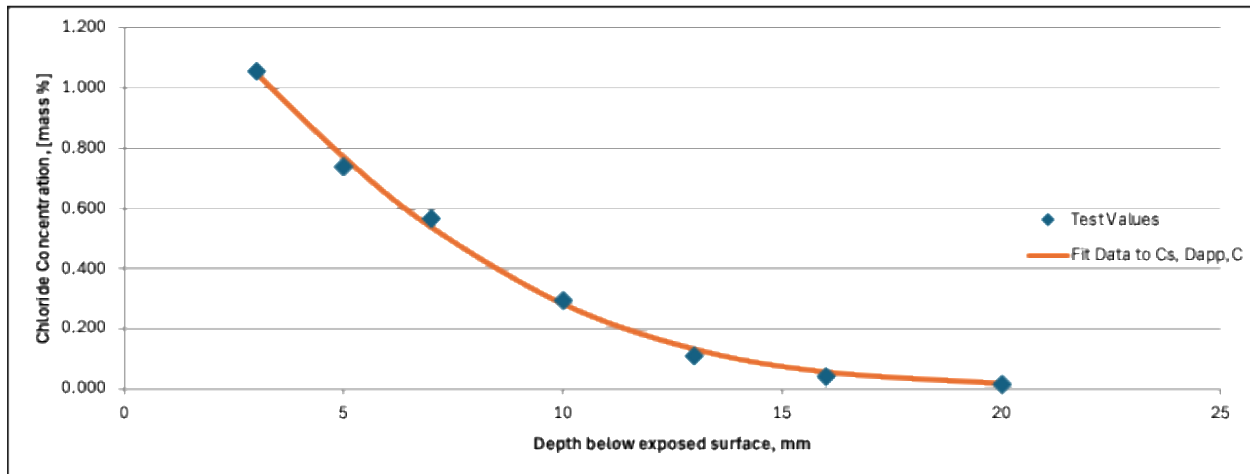


Figure 70. Chloride profile for mix 18

4.1.4.2.20 Mix 19:

Table 32. Chloride content results for mix 19

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	1	2	0.732	29.1181
3	1	3	0.5513	21.9308
4	1	4	0.4365	17.3621
5	1	5	0.3591	14.2833
6	1	6	0.2995	11.9151
7	2	8	0.2206	8.7771
8	2	10	0.1546	6.15

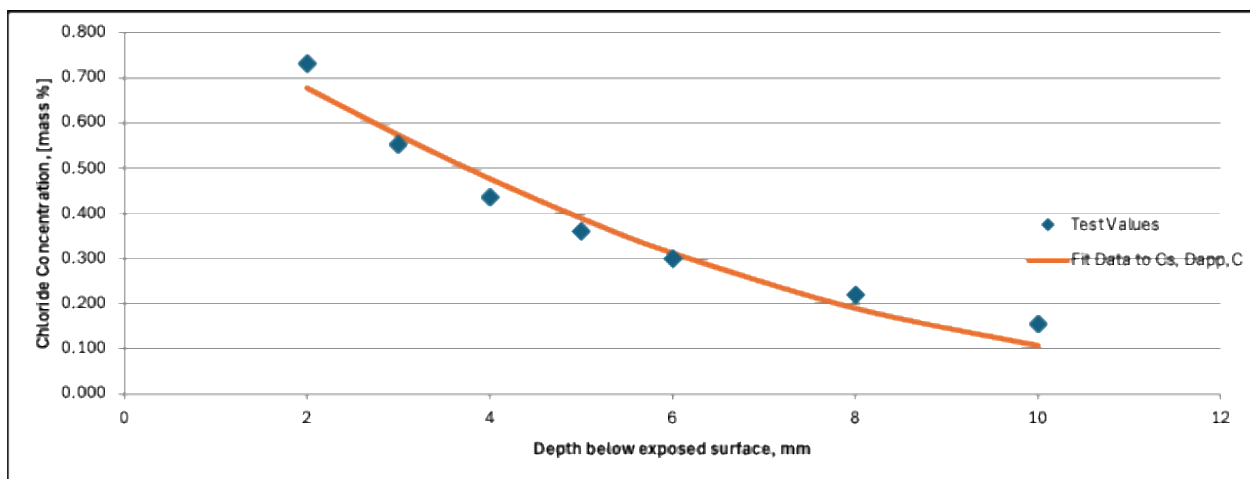


Figure 71. Chloride profile for mix 19

4.1.4.2.21 Mix 20:

Table 33. Chloride content results for mix 20

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	1	2	1.1744	45.8268
3	1	3	0.978	38.1614
4	1	4	0.8214	32.0503
5	2	6	0.656	25.5987
6	2	8	0.4628	18.0587
7	2	10	0.3481	13.5811
8	2	12	0.262	10.222

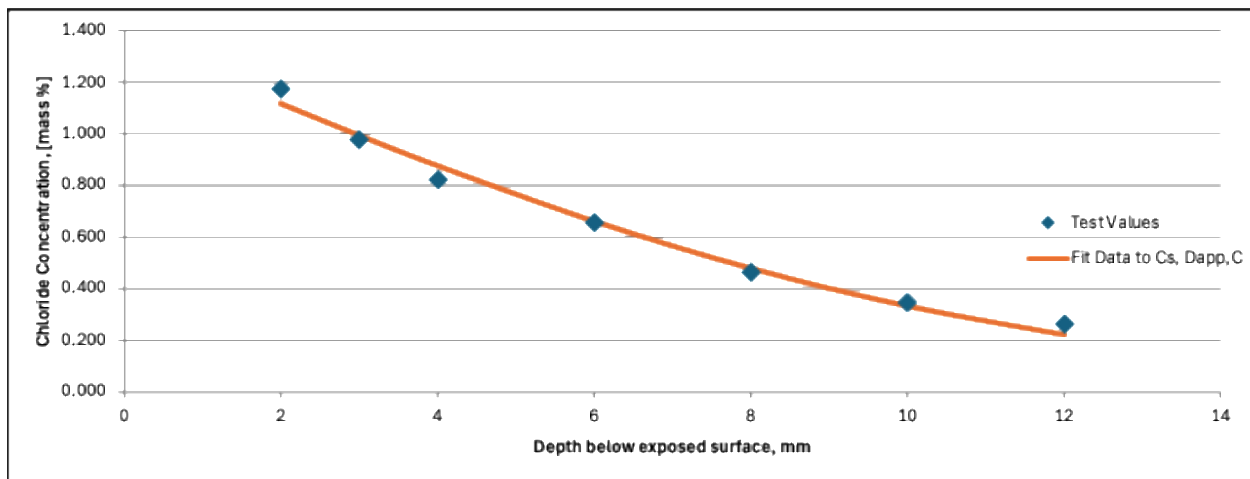


Figure 72. Chloride profile for mix 20

4.1.4.2.22 Mix 21:

Table 34. Chloride content results for mix 21

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	1	2	1.198	45.4288
3	1	3	1.0743	40.737
4	2	5	0.8619	32.684
5	2	7	0.6411	24.3106
6	2	9	0.4967	18.8359
7	3	12	0.3728	14.1365
8	4	16	0.217	8.2305
9	5	21	0.017	0.6435

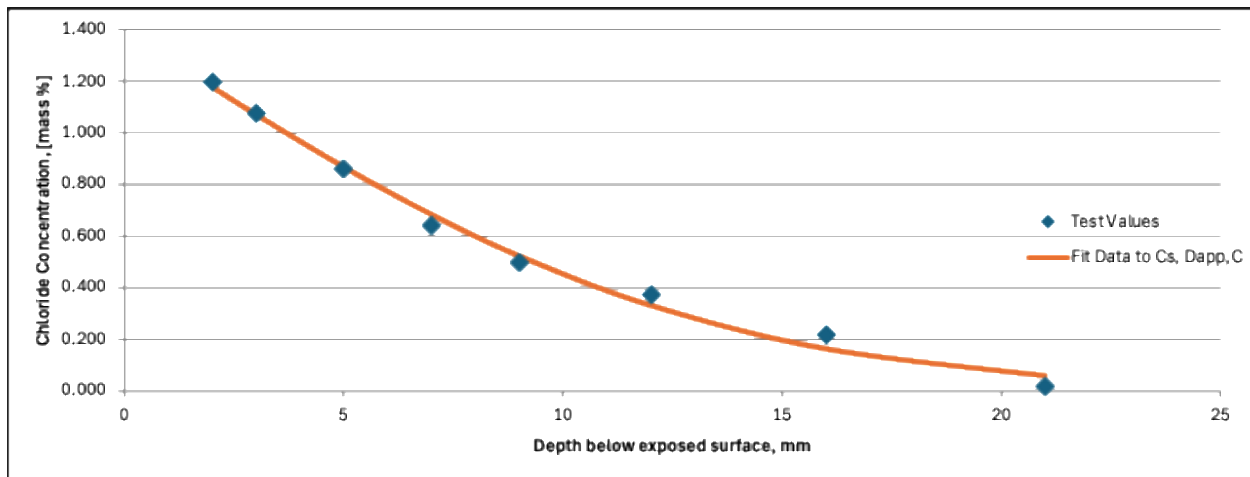


Figure 73. Chloride profile for mix 21

4.1.4.2.23 Mix 22:

Table 35. Chloride content results for mix 22

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	1	2	0.6755	26.8669
3	1	3	0.5332	21.2075
4	1	4	0.529	21.0418
5	1	5	0.4658	18.5253
6	1	6	0.4058	16.1395
7	2	8	0.3187	12.6768
8	2	10	0.2247	8.9379

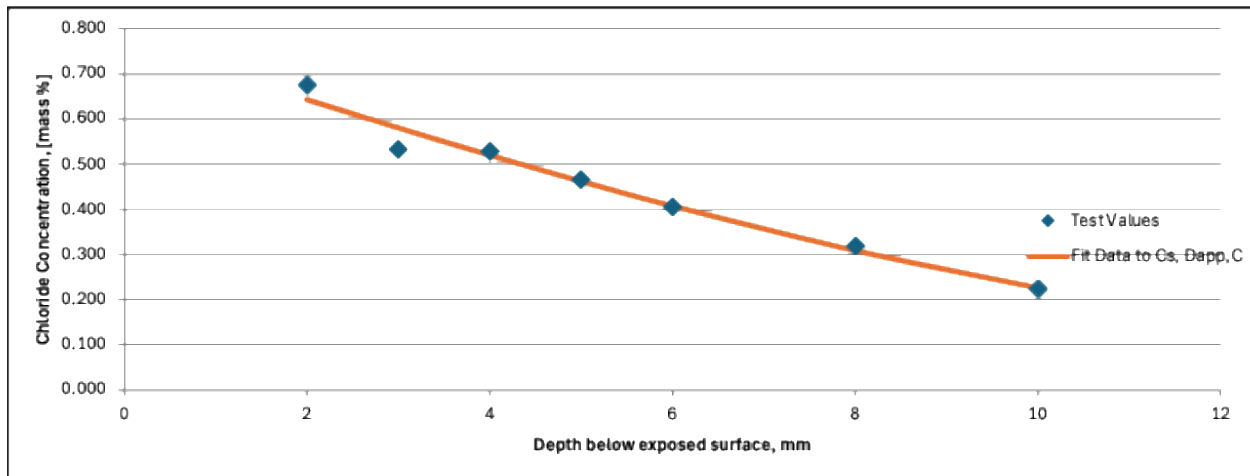


Figure 74. Chloride profile for mix 22

4.1.4.2.24 Mix 23:

Table 36. Chloride content results for mix 23

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	1	2	0.6417	26.1949
3	1	3	0.526	21.4698
4	1	4	0.4331	17.6789
5	1	5	0.3663	14.9543
6	1	6	0.2822	11.5174
7	2	8	0.2116	8.6377
8	2	10	0.1491	6.0863

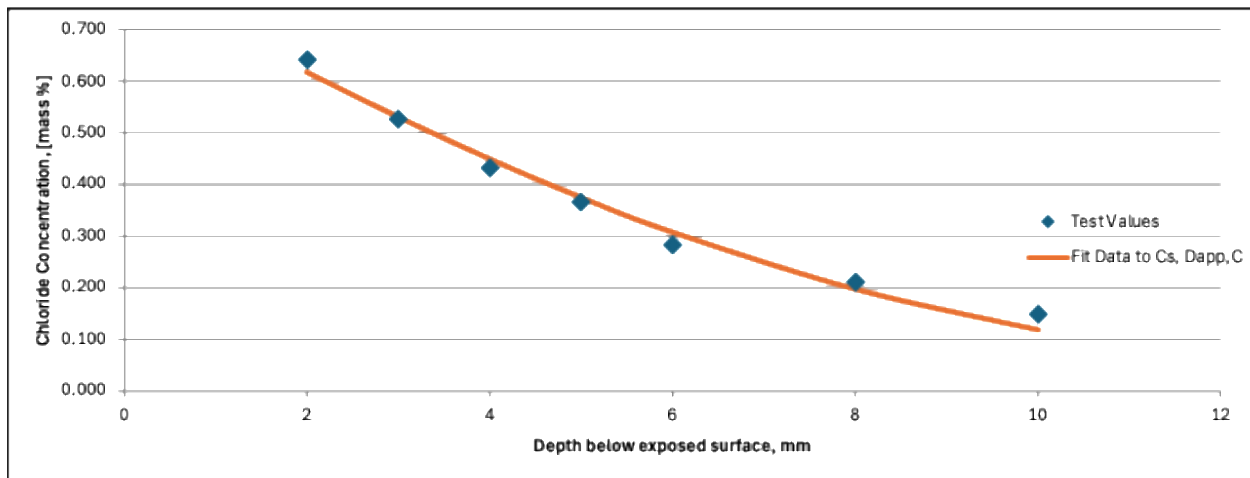


Figure 75. Chloride profile for mix 23

4.1.4.2.25 Mix 24:

Table 37. Chloride content results for mix 24

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	1	2	0.7529	29.9282
3	1	3	0.5637	22.4081
4	1	4	0.4128	16.4099
5	1	5	0.3303	13.1297
6	1	6	0.2701	10.7369
7	2	8	0.1913	7.6039
8	2	10	0.1071	4.257

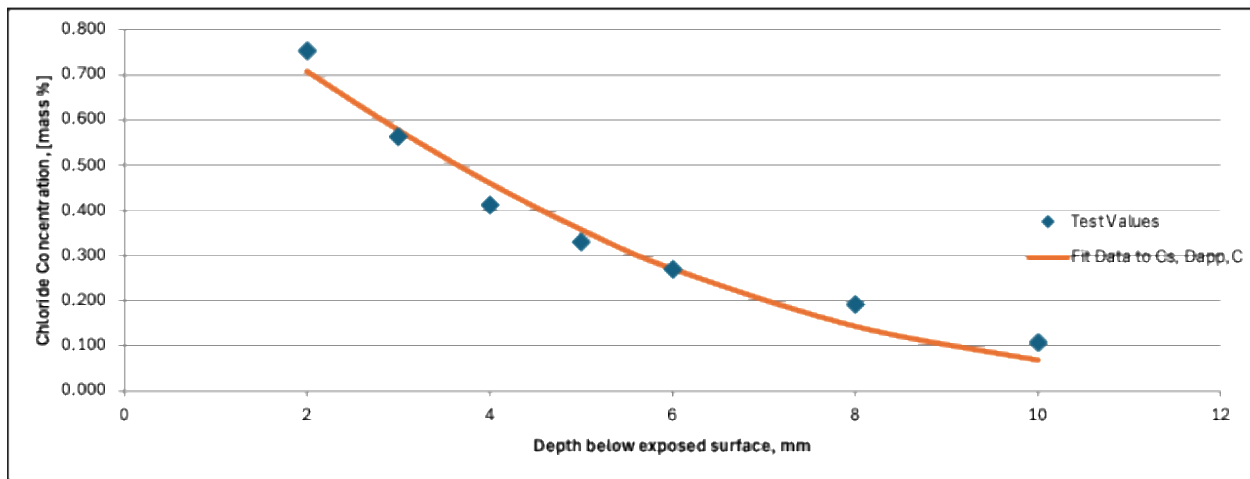


Figure 76. Chloride profile for mix 24

4.1.4.2.26 Mix SFM1:

Table 38. Chloride content results for mix SFM1

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	1	2	0.8448	33.716
3	1	3	0.7324	29.228
4	1	4	0.6356	25.367
5	1	5	0.5438	21.703
6	1	6	0.4536	18.102
7	2	8	0.3802	15.176
8	2	10	0.2464	9.835

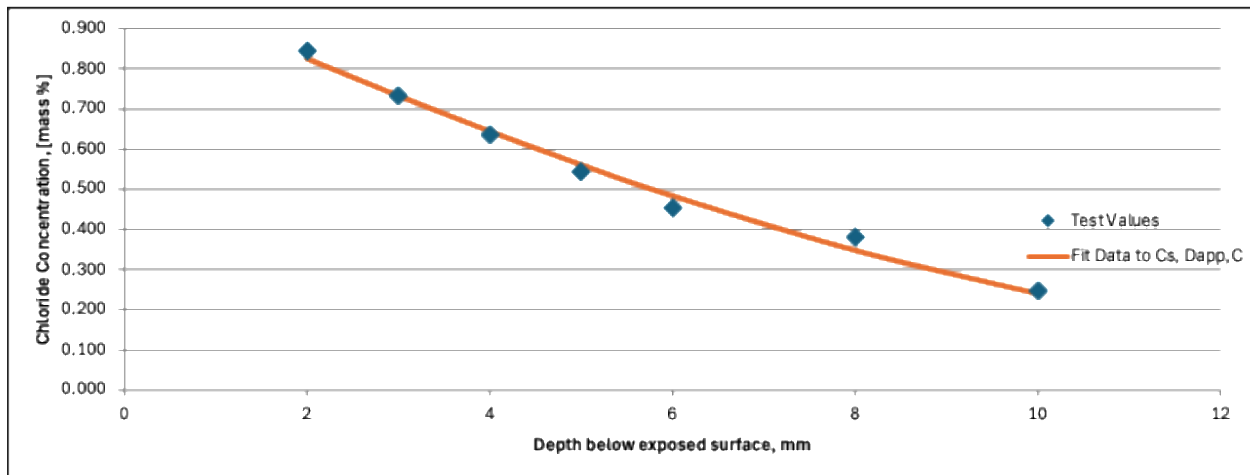


Figure 77. Chloride profile for mix SFM1

4.1.4.2.27 Mix SFM2:

Table 39. Chloride content results for mix SFM2

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	1	2	0.7461	29.775
3	1	3	0.6868	27.41
4	1	4	0.5882	23.474
5	1	5	0.5015	20.013
6	1	6	0.4393	17.531
7	2	8	0.3787	15.115
8	2	10	0.3118	12.442

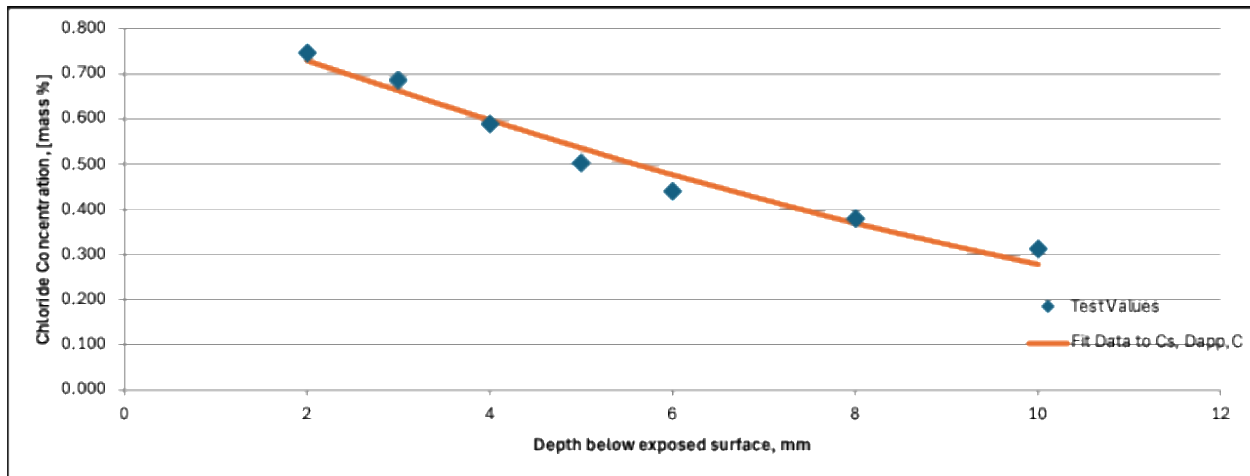


Figure 78. Chloride profile for mix SFM2

4.1.4.2.28 Mix SFM3:

Table 40. Chloride content results for mix SFM3

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	1	2	1.132	44.998
3	1	3	1.0171	40.431
4	1	4	0.8866	35.244
5	1	5	0.7544	29.988
6	1	6	0.6471	25.724
7	2	8	0.4695	18.662
8	2	10	0.2611	10.377

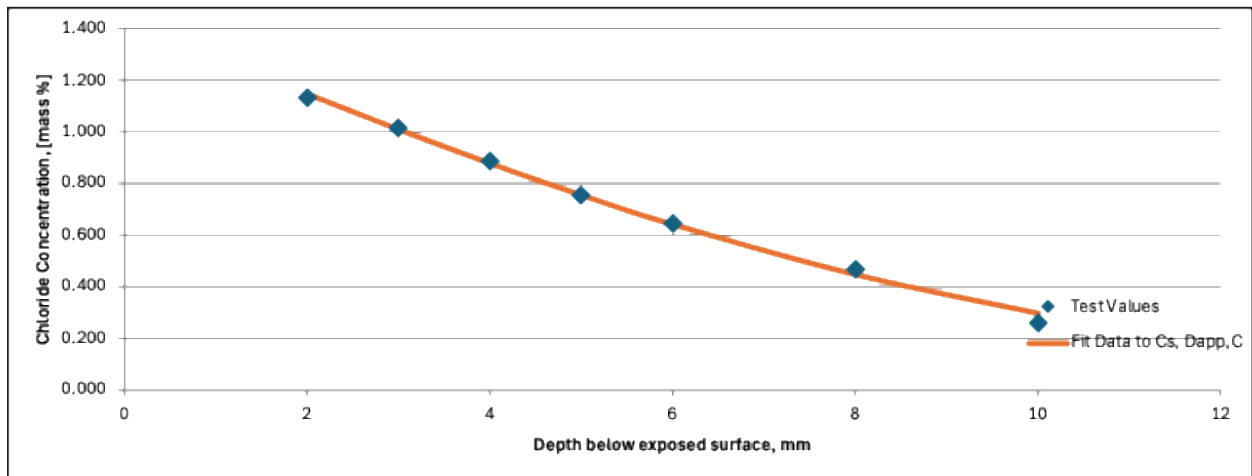


Figure 79. Chloride profile for mix SFM3

4.1.4.2.29 Mix SLAG2-1:

Table 41. Chloride content results for mix SLAG2-1

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	1	2	0.8565	34.047
3	1	4	0.8181	32.518
4	2	6	0.7328	29.13
5	2	8	0.5662	22.505
6	2	11	0.4136	16.44
7	3	14	0.2481	9.862
8	4	18	0.0959	3.814

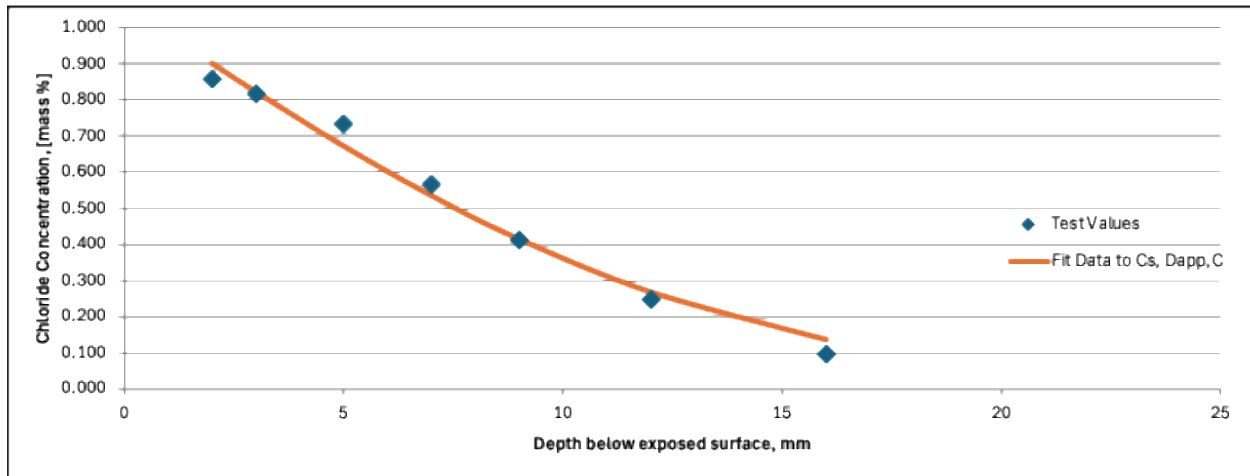


Figure 80. Chloride profile for mix SLAG2-1

4.1.4.2.30 Mix SLAG2-2:

Table 42. Chloride content results for mix SLAG2-2

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	1	2	0.7221	28.705
3	1	3	0.6772	26.92
4	1	4	0.6556	26.06
5	2	5	0.5161	20.513
6	2	7	0.3042	12.09
7	2	9	0.2072	8.236

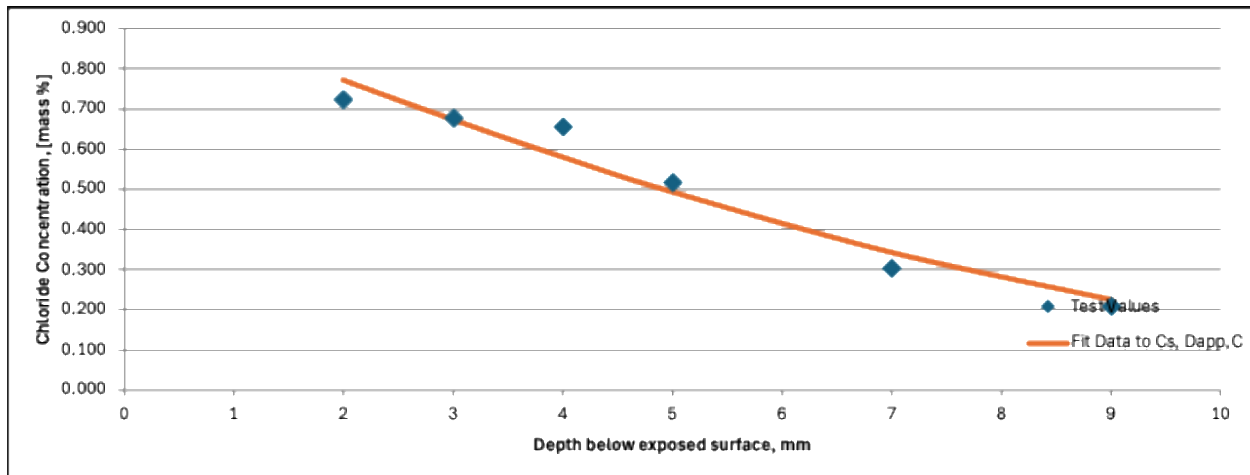


Figure 81. Chloride profile for mix SLAG2-2

4.1.4.2.31 Mix SLAG2-3:

Table 43. Chloride content results for mix SLAG2-3

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	1	2	0.8656	34.406
3	1	3	0.8681	34.507
4	1	4	0.8284	32.927
5	2	5	0.6153	24.457
6	2	7	0.3867	15.37
7	2	9	0.2006	7.973
8	2	11	0.1036	4.118

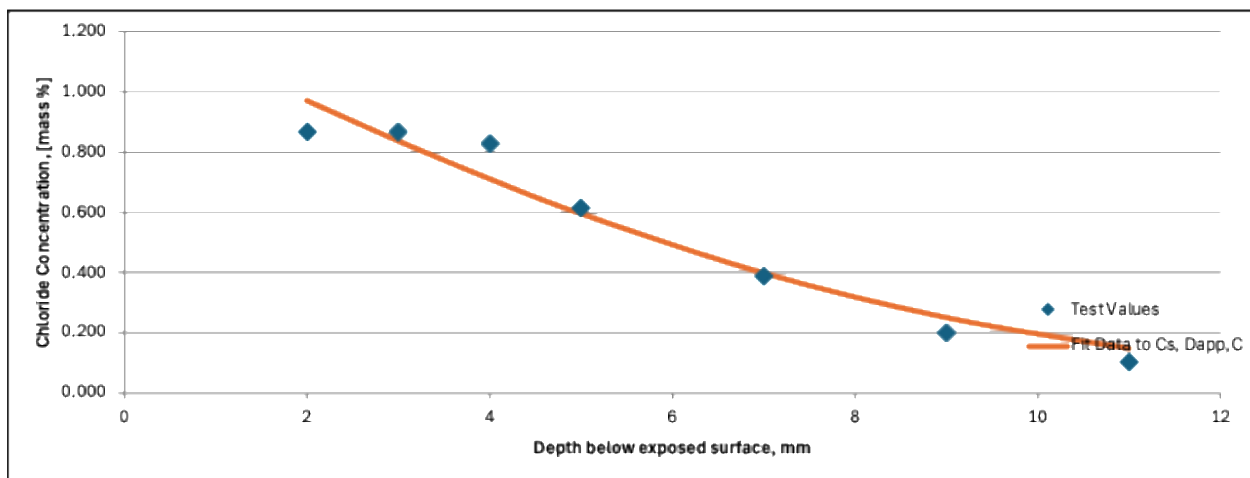


Figure 82. Chloride profile for mix SLAG2-3

#### 4.1.4.2.32 Mix SLAG2-4:

Table 44. Chloride content results for mix SLAG2-4

Layer no	Thickness (mm)	Depth (mm)	% wt. of concentration	Chloride content lb/yd <sup>3</sup>
2	1	2	0.7611	30.254
3	1	3	0.6236	24.788
4	1	4	0.5543	22.034
5	1	5	0.422	16.776
6	1	6	0.3315	13.176
7	2	8	0.2325	9.242
8	2	10	0.1578	6.271

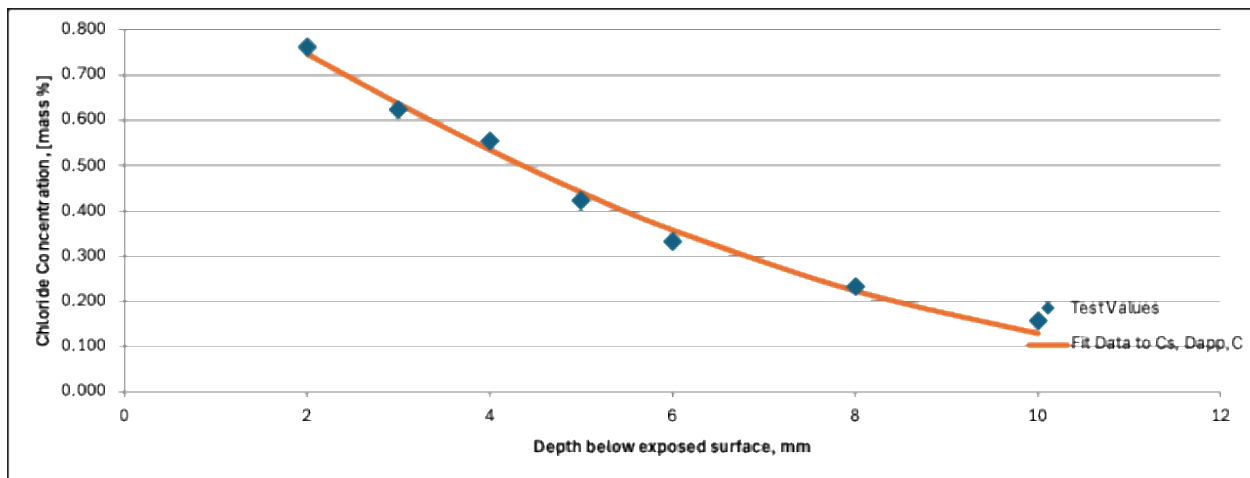


Figure 83. Chloride profile for mix SLAG2-4

#### 4.1.4.2.33 Apparent diffusion rate for the different mixes

The obtained diffusion coefficients ranged between approximately  $0.9 \times 10^{-12}$  and  $20.8 \times 10^{-12}$  m<sup>2</sup>/s, covering a broad spectrum of mix designs with different supplementary cementitious materials (SCMs) and water-to-cement ratios (w/c). A clear trend was observed in which mixes incorporating pozzolanic or latent hydraulic materials (fly ash, slag, silica fume, or metakaolin) exhibited significantly lower diffusion coefficients compared to plain Portland cement mixes. Additionally, decreasing the w/c ratio consistently reduced the apparent diffusion rate.

Table 45. Apparent diffusion rate for the different mixes

Mix No	Description	D180 m <sup>2</sup> /sec x10 <sup>-12</sup>
1	100% Portland cement w/ 0.44 w/c	21.2
2	100% Portland cement w/ 0.44 w/c	11.8
3	100% Portland cement w/ 0.44 w/c	12.9
4	20% Fly Ash with 0.3 w/c	8.88
5	20% Fly Ash with 0.4 w/c	13.1
6	20% Fly Ash with 0.5 w/c	8.54
7	70% Slag with 0.3 w/c	1.47
8	70% Slag with 0.4 w/c	1.81
9	70% Slag with 0.5 w/c	3.83
10	20% Fly Ash and 50% Slag	1.85
11	20% Fly Ash and 50% Slag	1.61
12	20% Fly Ash and 50% Slag	2.17
13	20% Fly Ash and 8% Silica Fume	3.28
14	20% Fly Ash and 8% Silica Fume	4.59
15	20% Fly Ash and 8% Silica Fume	9.7
16	55% Slag & 6% Silica Fume with 0.22 w/c	0.978
17	55% Slag & 6% Silica Fume with 0.34 w/c	2.56
18	55% Slag & 6% Silica Fume with 0.46 w/c	1.78
19	18% Fly Ash & 12% Metakaolin with 0.24 w/c	1.27
20	18% Fly Ash & 12% Metakaolin with 0.3 w/c	2.29
21	18% Fly Ash & 12% Metakaolin with 0.36 w/c	3.22
22	55% Slag & 8% Metakaolin with 0.3 w/c	2.85
23	55% Slag & 8% Metakaolin with 0.22 w/c	1.5
24	55% Slag & 8% Metakaolin with 0.38 w/c	0.944
SFM1	42% Fly Ash with 0.19 w/c	2.22
SFM2	42% Fly Ash with 0.23 w/c	3.16
SFM3	42% Fly Ash with 0.27 w/c	1.96
SLAG2-1	70% Slag 2 with 0.4 w/c	4.78
SLAG2-2	50% Slag 2 & 20% Fly Ash with 0.35 w/c	1.77
SLAG2-3	55% Slag 2 & 6% Silica Fume with 0.3 w/c	1.54
SLAG2-4	55% Slag 2 & 8% Metakaolin with 0.3 w/c	1.38

## 4.2 RMT Test Results

The rapid migration test (RMT) aims to determine the chloride migration coefficient in concrete, mortar, or cement-based repair materials from non-steady-state migration experiments. The procedure is based on NT Build 492, “Concrete, Mortar and Cement-based Repair Materials: Chloride Migration Coefficient from Non-Steady-State Migration Experiments.” This test measures the resistance of the tested material to chloride penetration under the action of an external electrical field, unlike the bulk diffusion (BD) test, which tests the movement of chloride ions under a concentration gradient.

### 4.2.1 Specimen Preparation

#### 4.2.1.1 *Sample Description:*

- Cylindrical specimens with a diameter of 100 mm (4 in.) and a thickness of 50 mm (2 in.) are sliced from cast cylinders or drilled cores. Three specimens are needed for each test.

#### 4.2.1.2 *Preconditioning:*

- The specimens are surface-dried and placed in a vacuum container for vacuum treatment with both end surfaces exposed.
- The absolute pressure in the vacuum container is reduced to a range of 10-50 mbar (1-5 kPa) within a few minutes and maintained for three hours.
- While the vacuum pump is still running, the container is filled with a saturated calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ) solution until all specimens are immersed.
- The vacuum is maintained for an additional hour before allowing air to enter the container.
- The specimens are kept in the solution for  $18 \pm 2$  hours.

### 4.2.2 Testing Procedure

#### 4.2.2.1 *Setup:*

- An external electrical potential is applied axially across the specimen to force chloride ions to migrate into the specimen (Figure 84 & Figure 85).
- The specimen is placed into a rubber sleeve and then positioned on a plastic support. The space in the sleeve above the specimen is filled with 300 ml of anolyte solution (0.3 M

NaOH). The catholyte reservoir is filled with about 12 L of 10% NaCl solution.

- An anode and a cathode are immersed in the anolyte and catholyte solution, respectively. The cathode and anode are connected to the negative and positive poles of the power supply, respectively.

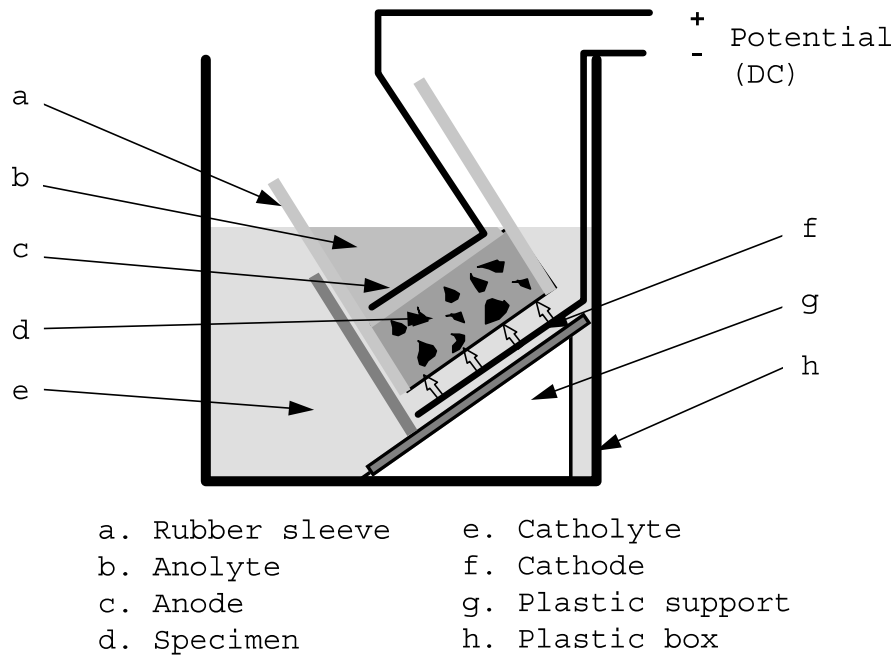


Figure 84. One arrangement of the migration set-up (NT Build 492, 1999)

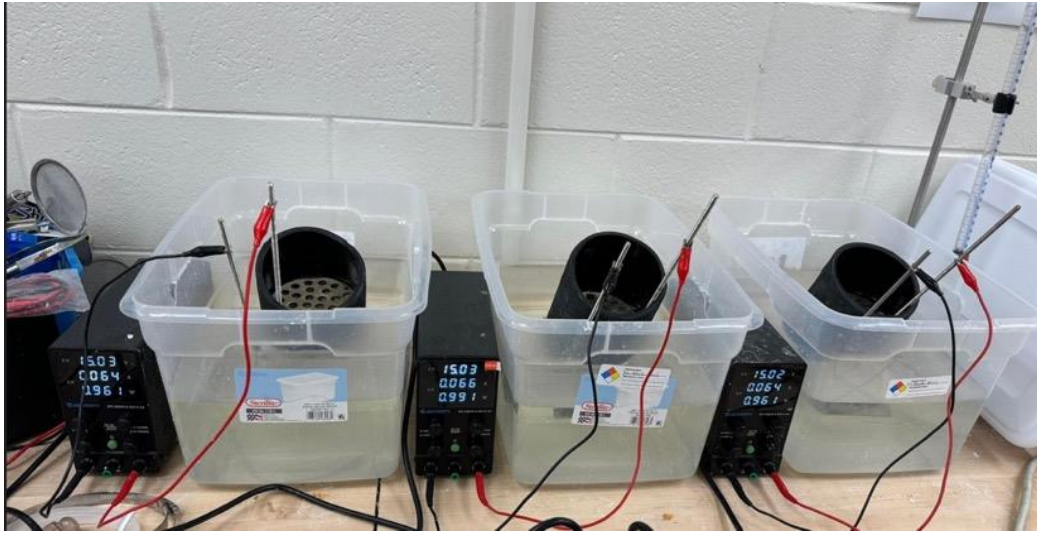


Figure 85. RMT setup

#### 4.2.2.2 Test Execution:

- The power supply is turned on, with the initial voltage set at 30 V, and the initial current for each specimen is recorded.
- The test duration and voltage are determined based on the initial current, as outlined in Table 46 of NT Build 492.
- The initial current and temperature are recorded, and the final current and temperature are documented before terminating the test.

Table 46. Test voltage and duration for concrete specimen with normal binder content

Initial current $I_0$ (with 30 V) (mA)	Applied voltage U (after adjustment) (V)	Possible new initial current $I_0$ (mA)	Test duration t (hour)
$I_0 < 5$	60	$I_0 < 10$	96
$5 \leq I_0 < 10$	60	$10 \leq I_0 < 20$	48
$10 \leq I_0 < 15$	60	$20 \leq I_0 < 30$	24
$15 \leq I_0 < 20$	50	$25 \leq I_0 < 35$	24
$20 \leq I_0 < 30$	40	$25 \leq I_0 < 40$	24
$30 \leq I_0 < 40$	35	$35 \leq I_0 < 50$	24
$40 \leq I_0 < 60$	30	$40 \leq I_0 < 60$	24
$60 \leq I_0 < 90$	25	$50 \leq I_0 < 75$	24
$90 \leq I_0 < 120$	20	$60 \leq I_0 < 80$	24
$120 \leq I_0 < 180$	15	$60 \leq I_0 < 90$	24
$180 \leq I_0 < 360$	10	$60 \leq I_0 < 120$	24
$I_0 \geq 360$	10	$I_0 \geq 120$	6

*Note: For specimens with a special binder content, such as repair mortars or grouts, correct the measured current by multiplying by a factor (approximately equal to the ratio of normal binder content to actual binder content) in order to be able to use the above table.*

#### 4.2.2.3 Post-Test Procedure:

- After the predetermined test duration, the specimen is axially split, and a silver nitrate solution (0.1 M) is sprayed onto the freshly split sections to measure the chloride penetration depth.
- Due to coarse aggregate distribution sometimes blocking the reading path, specimens are split into quarters to collect the required number of readings accurately (Figure 86).
- The chloride penetration depth is measured from the visible white silver chloride precipitation, as shown in Figure 87 and as illustrated in Figure 88 of NT Build 492. This measured penetration depth is used to calculate the chloride migration coefficient.



Figure 86. RMT samples after splitting

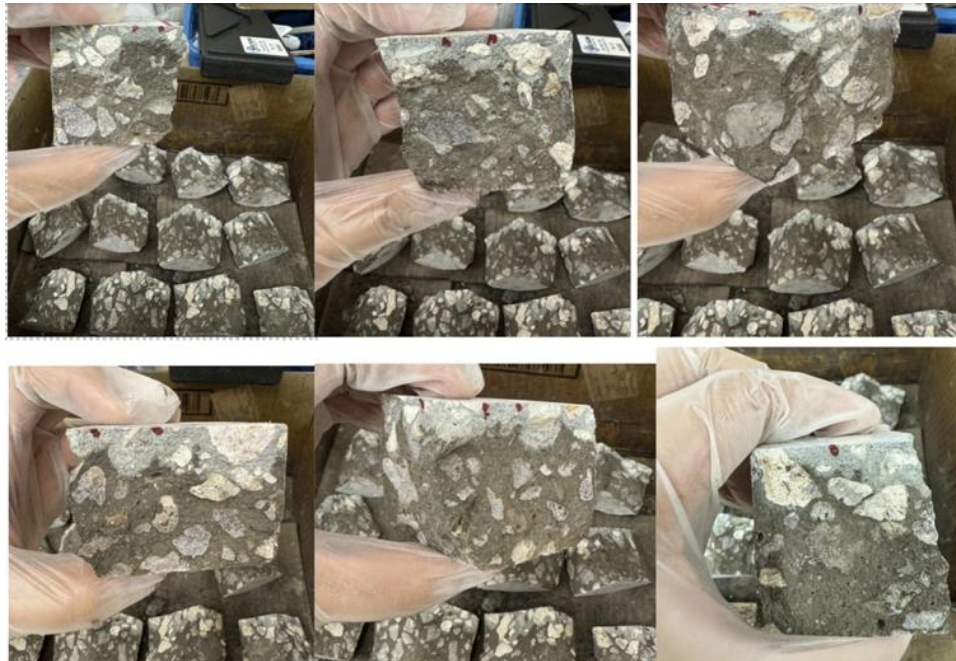


Figure 87. Measurement for chloride penetration depths

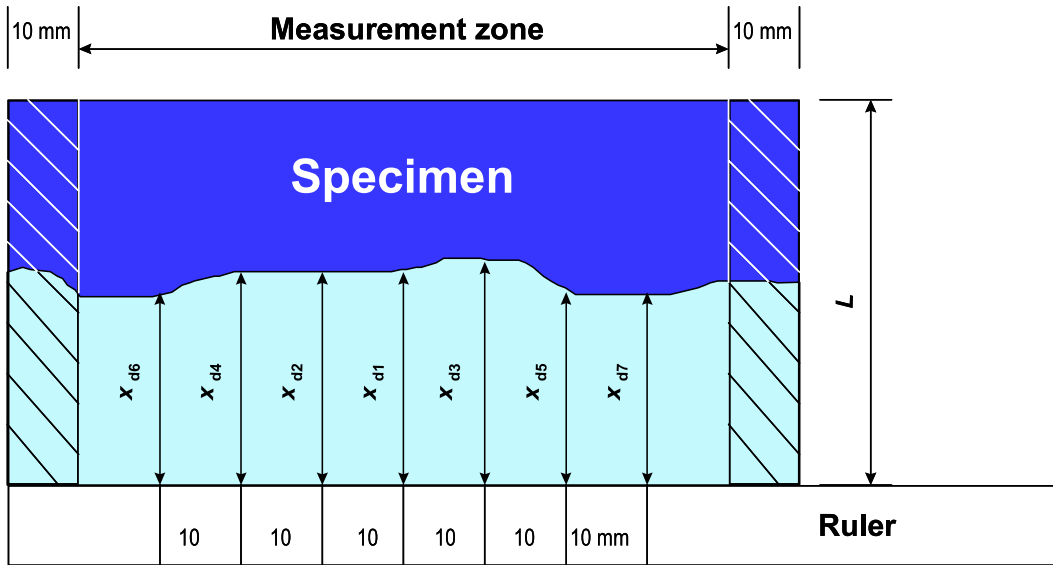


Figure 88. Illustration of measurement for chloride penetration depths (NT Build 492, 1999)

### 4.2.3 Results

The team has successfully completed the rapid migration test (RMT) for all 31 mixes, ensuring accurate specimen preparation and testing procedures. As shown in Table 47, the results provide valuable insights into the chloride migration characteristics of the tested samples, demonstrating the efficacy of different concrete mix designs in resisting chloride penetration.

Table 47. RMT results

<i>Mix Number</i>	<i>Non steady migration coefficient. <math>D_{nssm} \times 10^{-12} (m^2/s)</math></i>	<i>Type of mix</i>	<i>w/c ratio</i>
1	NA	100 % Portland cement w/ 0.44 w/c	0.44
2	17.61	100 % Portland cement w/ 0.44 w/c	0.44
3	24.60	100 % Portland cement w/ 0.44 w/c	0.44
4	11.88	20% fly ash with 0.3 w/c	0.3
5	19.80	20% fly ash with 0.4 w/c	0.4
6	19.89	20% fly ash with 0.5 w/c	0.5
7	2.65	70% slag with 0.3 w/c	0.3
8	2.35	70% slag with 0.4 w/c	0.4
9	2.78	70% slag 0.5w/c	0.5
10	3.96	20% fly ash and 50% slag	0.25
11	4.05	20% fly ash and 50% slag	0.35
12	3.62	20% fly ash and 50% slag	0.45
13	6.79	20% fly ash and 8% silica fume	0.25
14	8.18	20% fly ash and 8% silica fume	0.35
15	17.70	20% fly ash and 8% silica fume	0.44
16	1.77	55% slag & 6% silica fume	0.22
17	1.12	55% slag & 6% silica fume	0.34
18	2.17	55% slag & 6% silica fume	0.46
19	NA	18% fly ash& 12% Metakaolin	0.24
20	2.92	18% fly ash& 12% Metakaolin	0.3
21	4.65	18% fly ash& 12% Metakaolin	0.36
22	1.51	55% slag & 8% Metakaolin	0.3
23	1.20	55% slag & 8% Metakaolin	0.22
24	NA	55% slag & 8% Metakaolin	0.38
SFM1	3.19	42% Fly Ash	0.19
SFM2	6.51	42% Fly Ash	0.23
SFM3	3.54	70% SLAG 2	0.27
SLAG2-1	1.97	70% SLAG 2	0.4
SLAG2-2	1.65	50% SLAG 2, 20% FLY ASH	0.35
SLAG2-3	1.40	55% SLAG 2, 6% SILICA FUME	0.34
SLAG2-4	NA	55% SLAG 2, 8% Metakaolin	0.3

- **100% Portland Cement Mixes:** Mixes with 100% Portland cement exhibited higher average penetration depths, ranging from 18.94 mm to 24.69 mm, and relatively high  $D_{nssm}$  values ( $17.61 \times 10^{-12}$  to  $24.60 \times 10^{-12}$  m<sup>2</sup>/s). These values indicate higher permeability and lower resistance to chloride ion penetration compared to mixes containing SCMs.
- **20% Fly Ash Mixes:** Incorporating 20% fly ash showed a reduction in penetration depth and migration coefficient, particularly with lower w/c ratios. Mixes with w/c ratios of 0.3, 0.4, and 0.5 demonstrated  $D_{nssm}$  values of  $11.88 \times 10^{-12}$ ,  $19.80 \times 10^{-12}$ , and  $19.89 \times 10^{-12}$  m<sup>2</sup>/s.
- **70% Slag Mixes:** Mixes containing 70% slag showed significantly reduced penetration depths (7.23 mm to 9.97 mm) and  $D_{nssm}$  values ( $2.35 \times 10^{-12}$  to  $2.78 \times 10^{-12}$  m<sup>2</sup>/s). These results show the effectiveness of slag in improving the chloride ion resistance of concrete, making it a preferable option for structures exposed to harsh environmental conditions.
- **Combination of 20% Fly Ash and 50% Slag:** Mixes combining fly ash and slag also performed well, with penetration depths ranging from 11.16 mm to 14.84 mm and  $D_{nssm}$  values between  $3.62 \times 10^{-12}$  and  $4.05 \times 10^{-12}$  m<sup>2</sup>/s.
- **20% Fly Ash and 8% Silica Fume Mixes:** The addition of silica fume to fly ash mixes resulted in moderate improvement, with  $D_{nssm}$  values ranging from  $6.79 \times 10^{-12}$  to  $17.7 \times 10^{-12}$  m<sup>2</sup>/s. This blend shows enhanced durability properties, likely due to the pozzolanic activity of silica fume leading to a denser microstructure.
- **55% Slag and 6% Silica Fume Mixes:** This combination produced some of the best results, with very low penetration depths (5.29 mm to 8.37 mm) and  $D_{nssm}$  values ranging from  $1.12 \times 10^{-12}$  to  $2.17 \times 10^{-12}$  m<sup>2</sup>/s. These mixes demonstrate superior resistance to chloride penetration, making them highly suitable for environments with significant chloride exposure.

- **18% Fly ash and 12% Metakaolin Mixes:** These mixes showed low  $D_{nssm}$  values (2.92 to 4.65  $\times 10^{-12}$  m<sup>2</sup>/s) and penetration depths, further remarking the effectiveness of combining fly ash with metakaolin to reduce permeability and enhance durability.
- **55% Slag and 8% Metakaolin Mixes:** These mixes displayed similar low  $D_{nssm}$  values (1.20 to 1.77  $\times 10^{-12}$  m<sup>2</sup>/s) and penetration depths, further emphasizing the effectiveness of combining slag with metakaolin to reduce permeability and enhance durability.
- **SFM1:** With a w/c ratio of 0.19, this mix demonstrated an average penetration depth of 8.93 mm and a non-steady-state migration coefficient ( $D_{nssm}$ ) of  $3.18 \times 10^{-12}$  m<sup>2</sup>/s. These values indicate that the concrete has a moderate to low resistance to chloride ion penetration. The relatively low  $D_{nssm}$  value suggests an improvement in durability, especially when compared to mixes without supplementary cementitious materials (SCMs).
- **SFM2:** This mix, with a slightly higher w/c ratio of 0.23, showed a deeper penetration depth of 15.96 mm and a higher  $D_{nssm}$  value of  $6.51 \times 10^{-12}$  m<sup>2</sup>/s. The increased penetration depth and migration coefficient indicate a reduced resistance to chloride ion ingress compared to SFM1. However, the inclusion of 42% fly ash still offers a significant improvement over conventional Portland cement mixes.
- **SFM3:** This mix, with a higher w/c ratio of 0.27, showed a penetration depth of 10.35 mm and a  $D_{nssm}$  value of  $3.54 \times 10^{-12}$  m<sup>2</sup>/s.

# 5 Conclusions and Recommendations

The results obtained from this project are used to choose thresholds for SR and BR tests. In order to do that, it is important to investigate the correlation between concrete resistivity and chloride diffusion coefficient because the chloride diffusion coefficient is the parameter that directly affects the service life of the reinforced concrete structure. Aiming at selecting appropriate thresholds for SR and BR tests for an extremely aggressive environment, the research team developed the following program that correlates service life, chloride diffusion coefficient and concrete resistivity. Based on these correlations, as shown in Figure 89, a comprehensive approach was developed, and the appropriate thresholds are proposed.

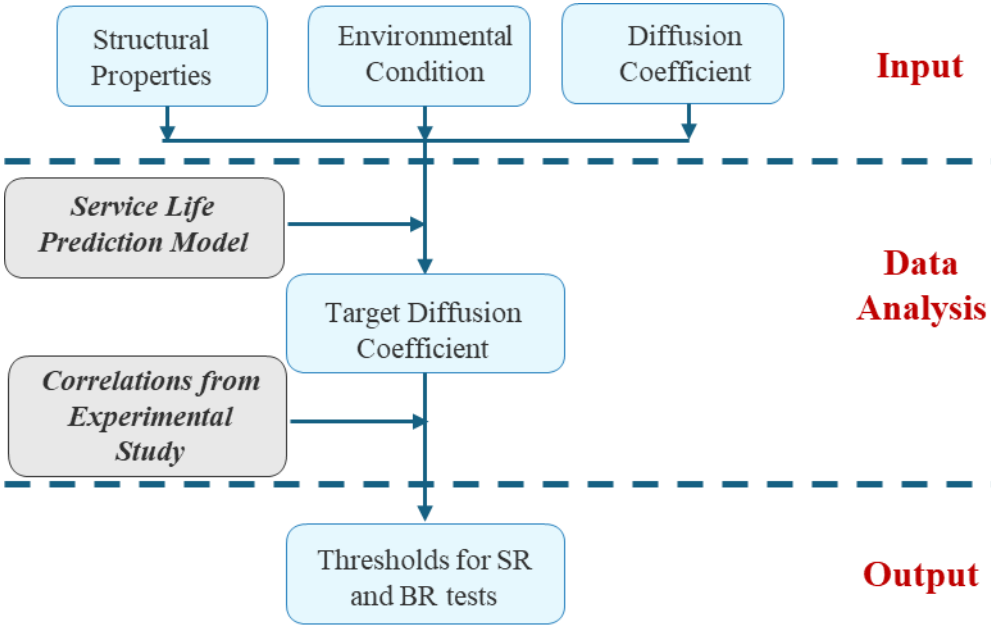


Figure 89. Comprehensive approach for thresholds selection

## 5.1 Target Diffusion Coefficient

Fick’s Second Law is widely used in diffusion-based service life models including ACI 365.1R-17 (ACI, 2017), fib model code 2010 (fib, 2012) and ISO 16204:2012 (ISO, 2012). A general Fick’s Second Law model can be expressed as:

$$C_{(x,t)} = C_0 \left(1 - \operatorname{erf} \frac{x}{2\sqrt{D_c t}}\right) \quad \text{Eq. 13}$$

where:

$C_{(x,t)}$  = chloride concentration at depth and time,

$C_0$  = surface chloride concentration,

$D_c$  = apparent diffusion coefficient,

$t$  = time for diffusion,

$x$  = depth

$\operatorname{erf}$  = statistical error function.

There are several key parameters in this model including the critical concentration at depth and time, surface chloride concentration, depth, time for diffusion, and apparent diffusion coefficient. For this study, the extremely aggressive environment condition is considered, and the surface chloride concentration is assumed to be 2000 ppm as specified in FDOT SDG. The service life of a reinforced concrete structural element can be divided into two periods: initiation period and propagation period (Life 365, 2020). The propagation period is the period starting from initiation of steel corrosion to the time to repair. It depends on the rebar type and the moisture condition. Life 365 (2020) assumes 20 years for epoxy-coated steel and 6 years for any other types of rebars (i.e. Black steel and stainless steel). ACI 365.1R-17 (2017) uses 20 years for a diaphragm wall exposed to saline ground water. In this study, it is assumed that the propagation period of 20 years for epoxy-coated steel and 6 years for all other types of rebars. Then the required initiation period to reach 75 years of service life will be 55 years for epoxy-coated steel and 69 years for all other types of rebars. Chloride concentration necessary to initiate corrosion is another important parameter. The research team is using the assumptions that Life 365 (2020) uses, which is black steel = 0.05 % wt. concrete, epoxy-coated = 0.05 %, and stainless steel = 0.5 %. The depth  $t$  depends on the type of structure and its respective concrete cover. The minimum cover specified in the FDOT 2025 Structures Manual will be used in this study (Table 49). A summary of input parameters is presented in Table 48.

Table 48. Input parameters for service life model

Parameters	Recommended Values
$C_0$ , ppm	2000
$t_i$ , years	55 for epoxy-coated steel
	69 for all other types of rebars
$x$ , inches	Table 49
$C_{critical}$	black steel = 0.05 % wt. concrete
	epoxy-coated = 0.05 %
	stainless steel = 0.5 %

Table 49. Concrete cover (FDOT 2025 Structures Manual, 2025)

Component (Precast and Cast-in-Place)	Concrete Cover (inches)	
	S or M <sup>1</sup>	E <sup>1</sup>
<b>Superstructure</b>		
All internal and external surfaces (except riding surfaces) of segmental concrete boxes, and external surfaces of prestressed beams (except the top surface)	2	
Top surface of beam top flange	¾ (min.)	
Top deck surfaces: Short Bridges <sup>2</sup>	2	
Top deck surfaces: Long Bridge <sup>2</sup>	2½ <sup>3</sup>	
All components and surfaces not included above (including wall copings and traffic and pedestrian railings which are not allowed to be constructed using the slip forming method)	2	
Front and back surfaces of pedestrian railings and traffic railings, other than single-slope traffic railings, which may be constructed using the slip forming method	3	
Front and back surfaces of single-slope traffic railings which may be constructed using the slip forming method	2½	
Noise Wall Posts and Panels	2	
Precast Concrete Perimeter Wall Posts and Panels	1¾	
<b>Substructure</b>		
External surfaces cast against earth and surfaces in contact with water (excluding Drilled Shafts)	4	4½
Exterior formed surfaces, columns, and tops of footings not in contact with water and all components or surfaces not included elsewhere	3	4
Internal surfaces	3	
Beam/Girder Pedestals, Cheekwalls & MSE Wall Interface Lugs	2	
Prestressed Piling	3	
Spun Cast Cylinder Piling <sup>4</sup>	2	
Drilled Shafts	6	
Auger Cast Piles	4	
Micropiles	2	3
Retaining Walls (Excluding MSE walls <sup>5</sup> and external surfaces cast against earth)	2	3
Box and Three-sided Culverts (including wingwalls and wingwall footings)	2	3
Bulkheads	4	

Using the Fick's Second Law service life model, the target diffusion apparent diffusion coefficients were derived based on a design service life of 75 years. The target diffusion coefficients are presented in Table 50 for different structural components. Please note that only black steel is considered because FDOT does not allow the use of epoxy coated rebar, and the corrosion mechanism of stainless steel is beyond the scope of this project.

Table 50. Target apparent diffusion coefficients (extremely aggressive environment)

Structural Component	Cover, inches (mm)	Target apparent diffusion coefficients, $\times 10^{-12}$ ( $m^2/s$ )
End bent	4" (101.6 mm)	1.79
Piers in contact with water	4.5" (114.3 mm)	2.27
Piers not in contact with water	4" (101.6 mm)	1.79
Retaining walls	3" (76.2 mm)	1.01
Pier cap and intermediate bent	4" (101.6 mm)	1.79

## 5.2 Correlations between Diffusion and Resistivity

The rapid migration test (RMT) was conducted to determine the non-steady state migration coefficient ( $D_{nssm}$ ) for various concrete mixes after 28 days of curing. This coefficient is an essential parameter in assessing the chloride ion diffusion rate within concrete, which directly impacts its durability and service life in chloride-rich environments.

The relationship between the chloride migration coefficients derived from the rapid migration test (RMT) and the surface resistivity (SR) results after 28 days of curing shows valuable insights into the permeability and durability characteristics of various concrete mix designs. Figure 90 illustrates the correlation between the SR measurements at 28 days and the non-steady state migration coefficient ( $D_{nssm}$ ), emphasizing the connection between resistivity and chloride ion diffusion in concrete.

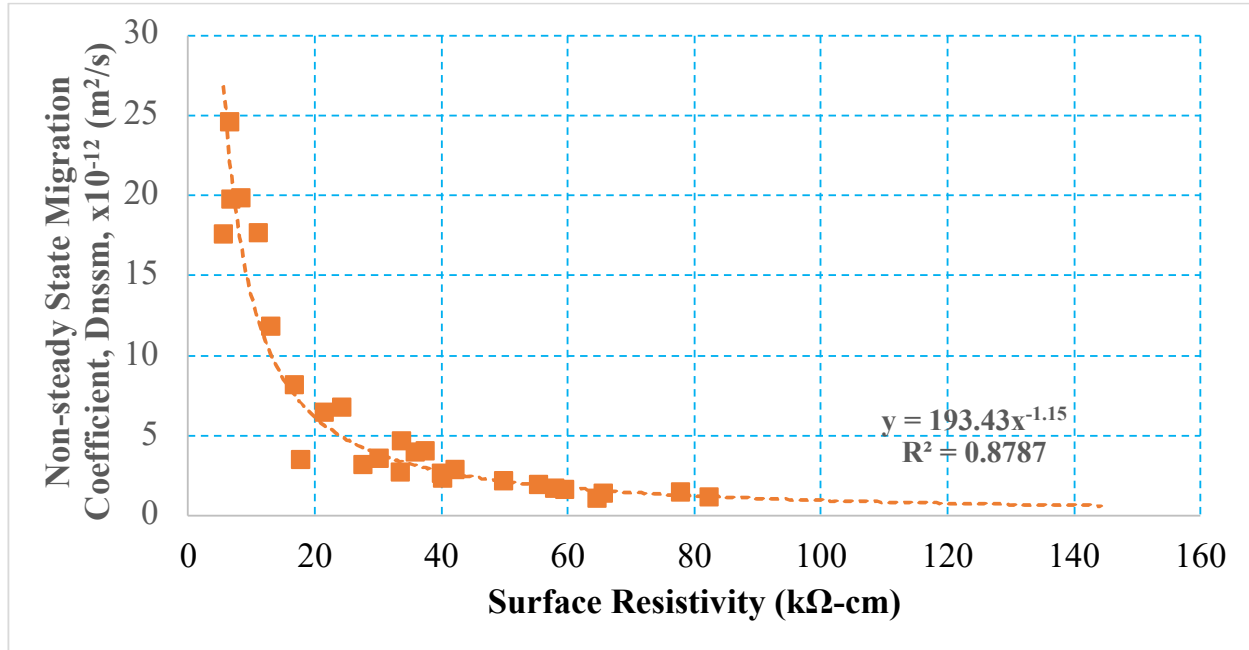


Figure 90. Correlation between surface resistivity and non-steady state migration coefficient ( $D_{nssm}$ )

In the figure, the plot demonstrates an inverse relationship between surface resistivity and the migration coefficient. As the resistivity values increase, indicating reduced permeability, the corresponding migration coefficients decrease, reflecting lower chloride ion diffusivity. This trend is consistent with the understanding that higher resistivity in concrete typically corresponds to a denser microstructure and, consequently, a greater resistance to chloride ingress.

The fitted power-law regression curve, represented by the equation  $y=193.43x^{-1.15}$ , with an  $R^2$  value of 0.8787, indicates a strong inverse correlation between SR and  $D_{nssm}$ . The high coefficient of determination ( $R$ ) suggests that approximately 93.7% of the variability in the migration coefficient can be explained by the variations in surface resistivity. This strong correlation supports the use of SR as a predictive measure for estimating the chloride diffusion rates in concrete, which is essential for assessing the long-term durability and service life of concrete structures exposed to chloride environments.

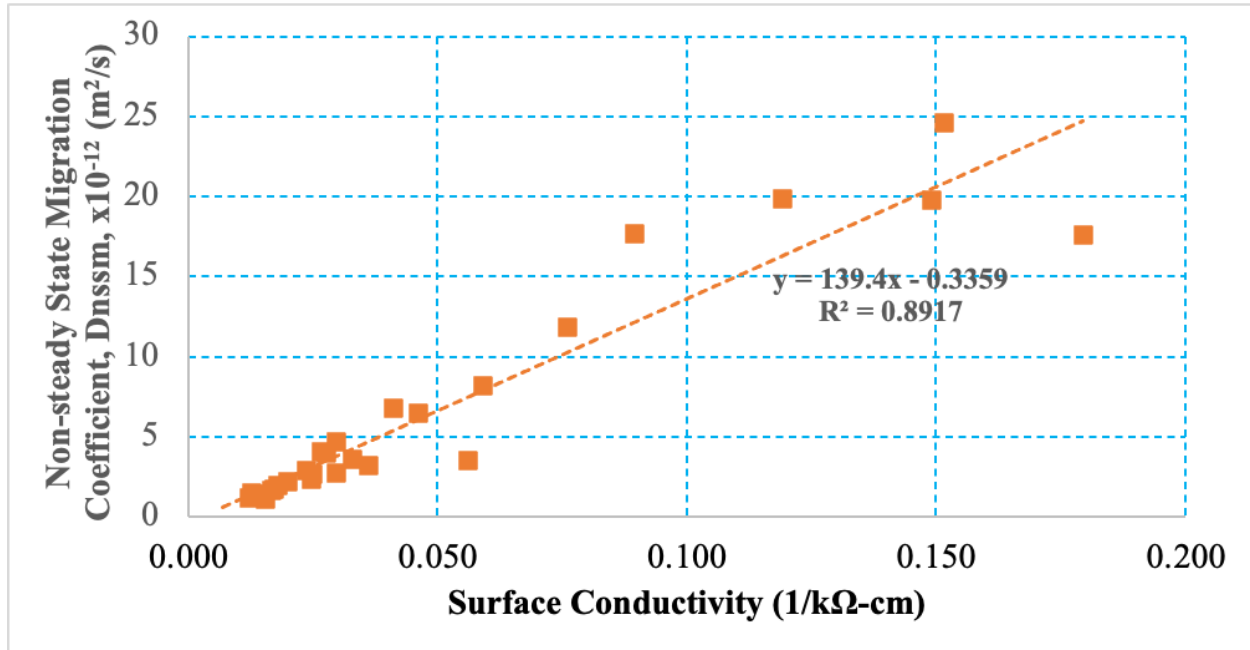


Figure 91. Correlation between surface conductivity and non-steady state migration coefficient ( $D_{nssm}$ )

Figure 91 further explores this relationship by plotting the surface conductivity, the inverse of surface resistivity, against the migration coefficient. The positive correlation observed, represented by the linear regression equation  $y=139.4x-0.3359$ , with an  $R^2$  value of 0.8917, reinforces the findings from Figure 91. Higher surface conductivity (lower resistivity) is associated with higher chloride migration coefficients, indicating greater permeability and reduced durability. The strong linear relationship, with approximately 94.7% of variability in migration coefficients explained by surface conductivity, highlights the effectiveness of resistivity measurements in evaluating chloride transport properties.

The relationship between the chloride migration coefficients derived from the rapid migration test (RMT) and the bulk resistivity (BR) results is critical for understanding the chloride ion penetration resistance of various concrete mixes. This section explores the correlations observed between BR and RMT, emphasizing their importance in predicting the long-term durability of concrete structures exposed to chloride environments.

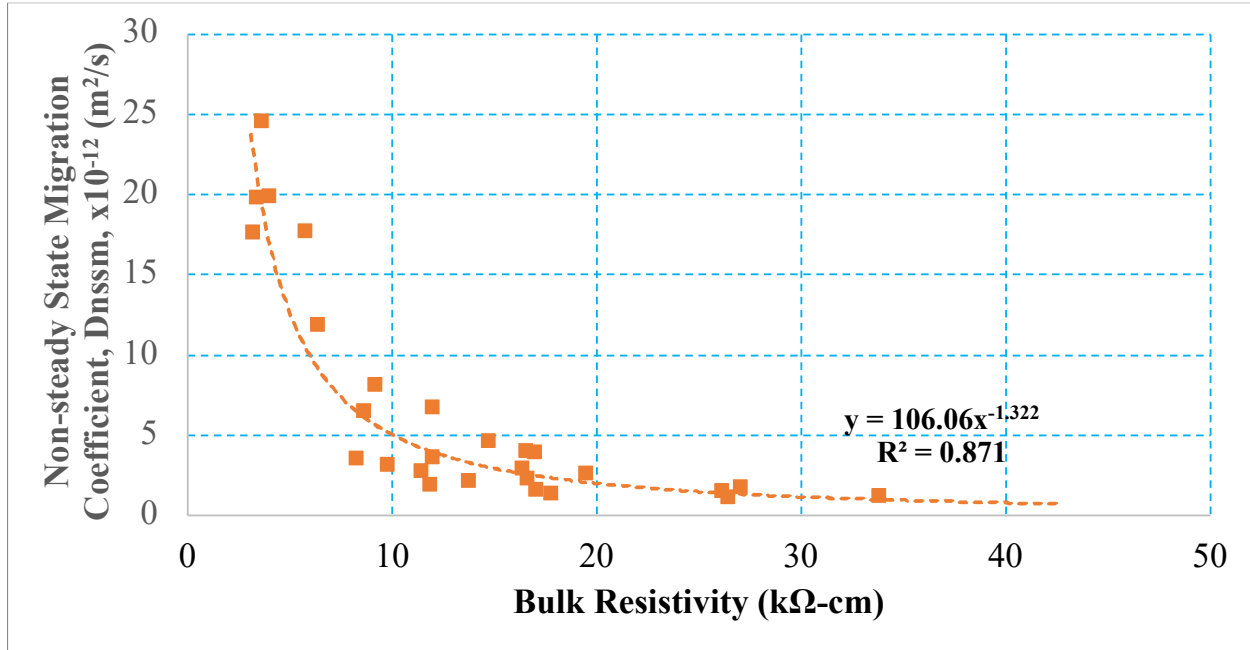


Figure 92. Correlation between bulk resistivity and non-steady state migration coefficient ( $D_{nssm}$ )

Figure 92 presents a plot demonstrating the inverse relationship between bulk resistivity (BR) measured at 28 days and the non-steady state migration coefficient ( $D_{nssm}$ ). Similar to the findings observed with surface resistivity, as BR values increase, indicating lower permeability, the migration coefficients decrease, reflecting reduced chloride ion diffusivity.

The fitted power-law regression line is represented by the equation  $y=109.06x^{-1.322}$  with an  $R^2$  value of 0.87. This high coefficient of determination suggests that approximately 94.2% of the variability in the migration coefficient can be attributed to changes in bulk resistivity. The strong inverse correlation between BR and  $D_{nssm}$  shows the potential of using resistivity measurements to estimate chloride diffusion rates and assess the permeability of concrete mixes.

Figure 93 illustrates the linear relationship between bulk conductivity values measured at 28 days and the non-steady state migration coefficient ( $D_{nssm}$ ). The positive linear trend is represented by the regression equation  $y=79.638x-2.0165$ , with an  $R^2$  value of 0.8812. This indicates that about 93.8% of the variation in the migration coefficient can be explained by bulk conductivity. The strong correlation shows that as bulk conductivity increases, signifying higher ionic mobility, the migration coefficient also increases, reflecting greater chloride diffusivity.

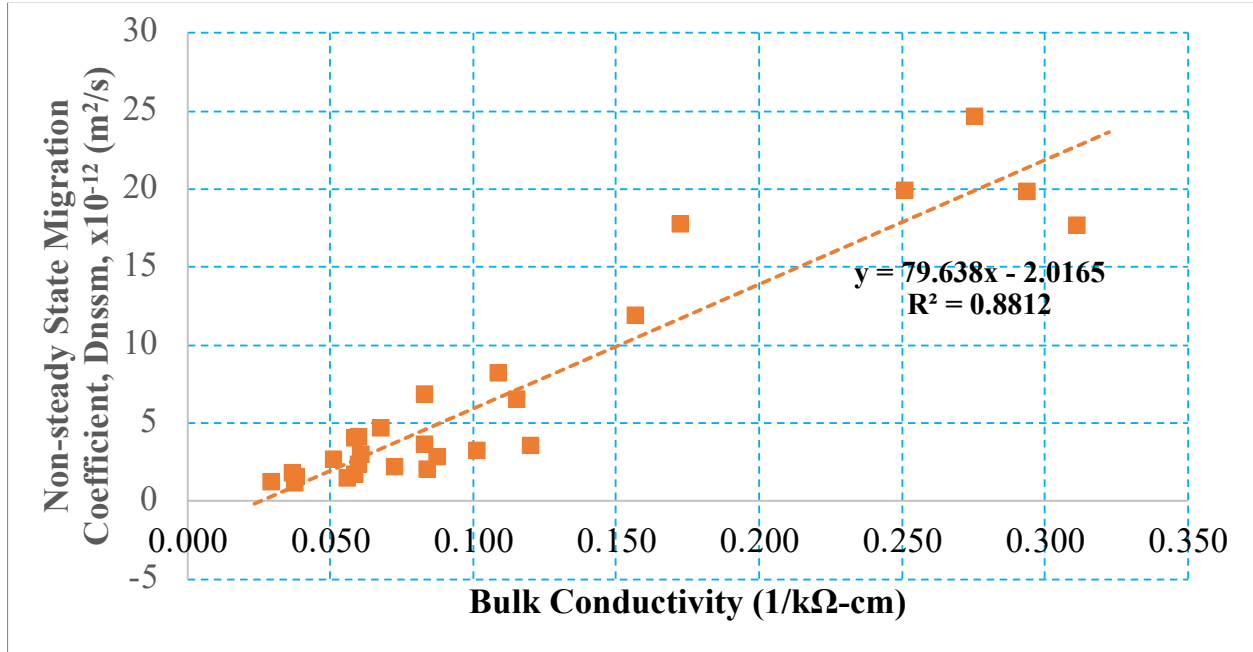


Figure 93. Correlation between bulk conductivity and non-steady state migration coefficient ( $D_{nssm}$ )

RMT results showed a good correlation with the SR and BR results and indicate that correlation can be established between chloride diffusion and concrete resistivity. However, the non-steady state migration coefficient  $D_{nssm}$  obtained from the RMT tests is a rapid migration test that doesn't reflect the characteristics of natural chloride diffusion. Thus, the bulk diffusion (BD) test results from Project BED32 "NextGEN Concrete-Chloride Diffusion" were also studied and will be used for this project.

Similar to the RMT results, the correlation between apparent diffusion coefficients from BD tests and resistivity results from SR and BR tests were investigated. Figure 94 shows the correlation between SR and apparent diffusion coefficient while Figure 95 shows the correlation between BR and apparent diffusion coefficient. Good correlations were observed between diffusion coefficients and SR/BR results. The coefficients of determination ( $R^2$ ) are 0.8576 and 0.87 for SR vs.  $D_a$  and BR vs.  $D_a$ , respectively, imply correlation coefficients of 92.6% and 93.3% which indicate good correlations between them.

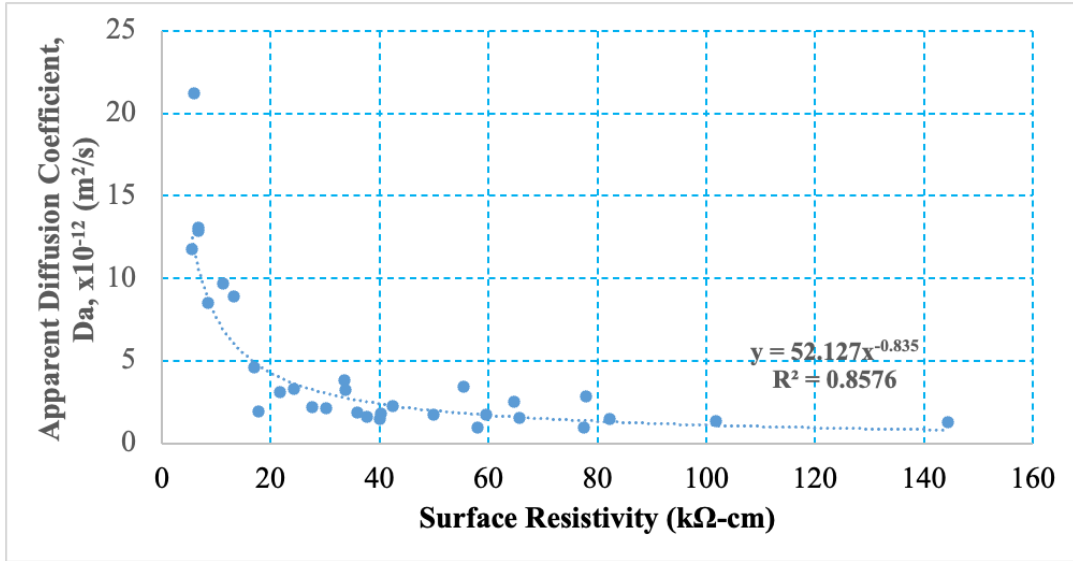


Figure 94. Correlation between surface resistivity and apparent diffusion coefficient ( $D_a$ )

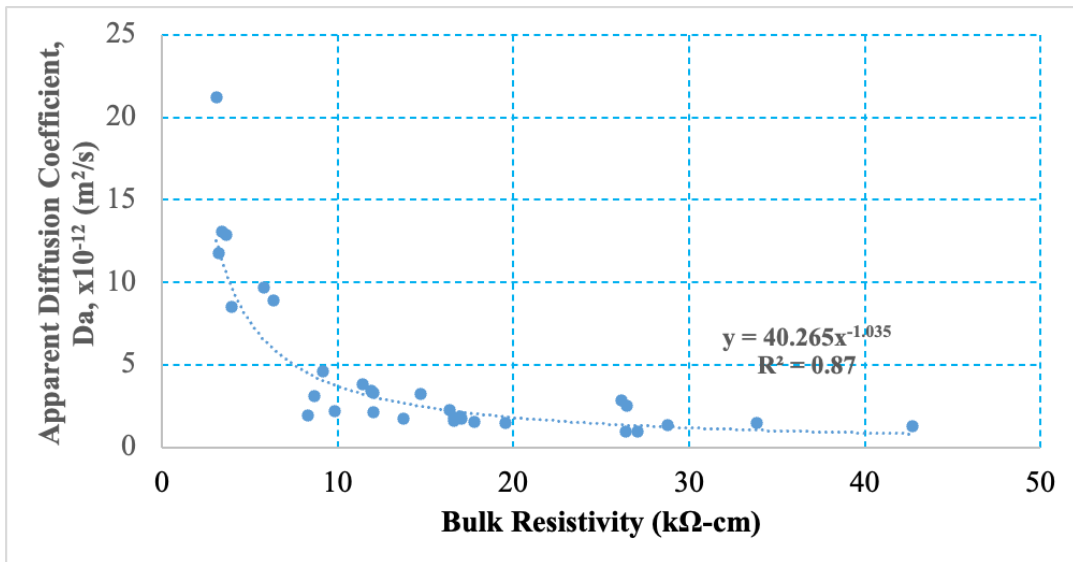


Figure 95. Correlation between bulk resistivity and apparent diffusion coefficient ( $D_a$ )

In order to depict the correlations in a more intuitive way, the inverse of resistivity, conductivity was plotted with apparent diffusion coefficients. As shown in Figure 96 and Figure 97, good correlations are observed between surface conductivity vs. apparent diffusion coefficient

as well as bulk conductivity vs. apparent diffusion coefficient. It also observed that the correlation is stronger if the conductivity and diffusion coefficient are low but weaker when conductivity and diffusion coefficient are high. In this study, the correlation equations from Figure 94 through Figure 97 will be used to correlate the diffusion coefficient and concrete resistivity.

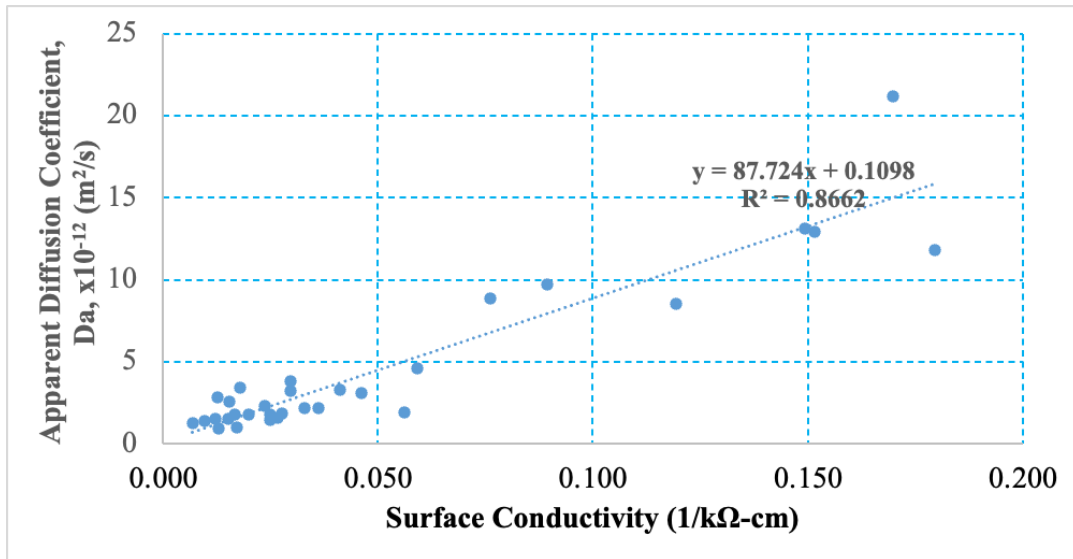


Figure 96. Correlation between surface conductivity and apparent diffusion coefficient ( $D_a$ )

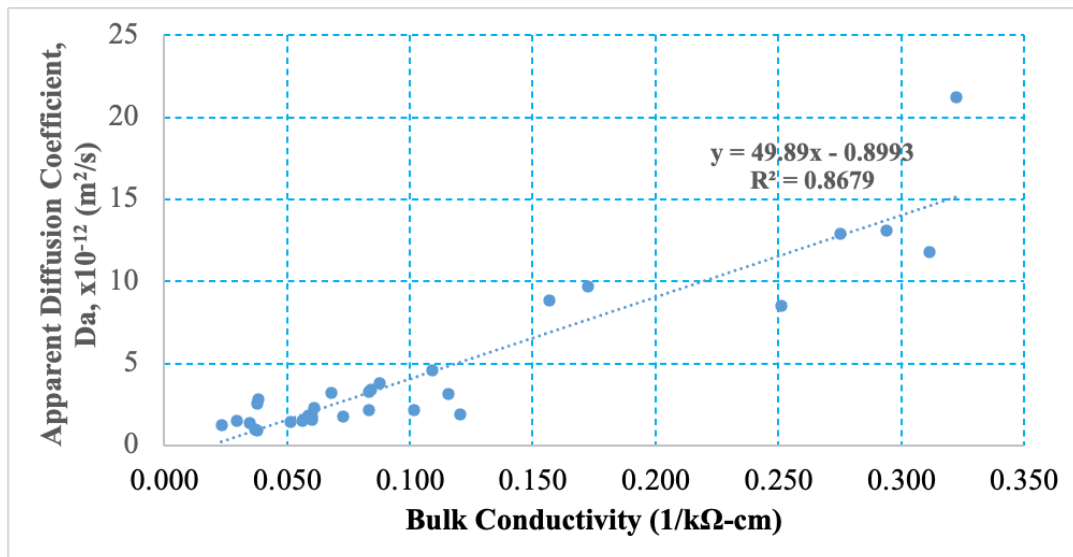


Figure 97. Correlation between bulk conductivity and apparent diffusion coefficient ( $D_a$ )

Since both conductivity and resistivity results show good correlation with apparent diffusion coefficients, the correlation between conductivity and apparent diffusion coefficients were used to predict the SR and BR thresholds for a service life of 75 years. Table 51 shows proposed SR and BR thresholds for different structural components.

Table 51. Proposed SR and BR thresholds (extremely aggressive environment)

Structural Component	Cover, inches (mm)	SR Threshold (kΩ-cm) at 28 days	BR Threshold (kΩ-cm) at 28 days
End bent	4" (101.6 mm)	52	19
Piers in contact with water	4.5" (114.3 mm)	41	16
Piers not in contact with water	4" (101.6 mm)	52	19
Retaining walls	3" (76.2 mm)	98	26
Pier cap and intermediate bent	4" (101.6 mm)	52	19

### 5.3 Comparison with Recommendation in BEE02

In BEE02 project, SR and BR thresholds were proposed based on available BD results at the conclusion of BEE02, which include data from 24 mixes. Table 52 shows the SR and BR thresholds recommended in BEE02. It can be seen that the new proposed thresholds are very similar but slightly lower to what recommended in BEE02. Because the new proposed thresholds are obtained from more mix designs that includes more types of SCM (Silica Fume and different source of slag), it is recommended to use the new proposed thresholds for future design and specification consideration.

Table 52. Proposed SR and BR thresholds for extremely aggressive environment (BEE02 Project)

Structural Component	Cover, inches (mm)	SR Threshold (k $\Omega$ -cm) at 28 days	BR Threshold (k $\Omega$ -cm) at 28 days
End bent	4" (101.6 mm)	55	21
Piers in contact with water	4.5" (114.3 mm)	42	17
Piers not in contact with water	4" (101.6 mm)	55	21
Retaining walls	3" (76.2 mm)	106	31
Pier cap and intermediate bent	4" (101.6 mm)	55	21

## 5.4 Recommendations

The main goals of this project are:

1. To investigate and determine chloride diffusion rates using bulk diffusion (ASTM C1556) and rapid migration (NT Build 492) tests for concrete mixes intended for use in an extremely aggressive chloride environment.
2. Establish a correlation between the chloride diffusion test results and the bulk (TP 119) and surface (T 358) resistivity results obtained in contract BEE02.

In order to achieve these two objectives, the research team conducted both BD and RMT tests for selected 31 mix designs. Based on the testing results and the correlations between SR&BR and BD results, the following thresholds are recommended for the structures exposed to an extremely aggressive environment:

1. For structures with 3” cover, use 98 k $\Omega$ -cm and 26 k $\Omega$ -cm for SR and BR, respectively.
2. For structures with 4” cover, use 52 k $\Omega$ -cm and 19 k $\Omega$ -cm for SR and BR, respectively.
3. For structures with 4.5” cover, use 41 k $\Omega$ -cm and 16 k $\Omega$ -cm for SR and BR, respectively.

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