JOINT TRANSPORTATION RESEARCH PROGRAM

FHWA/IN/JTRP-2010/29

Final Report

**PORTLAND CEMENT CONCRETE PAVEMENT
PERMEABILITY PERFORMANCE**

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INDOT Research

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Portland Cement Concrete Pavement Permeability Performance

Introduction

The main objective of this study was to evaluate the fluid transport properties of concrete pavements constructed in the state of Indiana.

The scope of the research included characterization of fluid transport using two primary tests that included water absorption and electrical conductivity.

Specifically this work evaluated the absorption of water and/or deicing solutions, and electrical conductivity. A series of concrete paving mixtures were tested to provide a range of values that were typical for the state of Indiana. While similar mixture proportions were used for the mixtures in Indiana differences in the

magnitude of water absorbed occurred. A series of mortars were tested to illustrate the effect of curing conditions, water to cement ratio, and paste volume.

In addition to the measurement of transport properties, the relative humidity was assessed for concrete slabs exposed to different exposure conditions. The samples considered in this investigation included a sample stored at 50% relative humidity, covered concrete, a concrete with an exposed vertical surface, a concrete on a drainable base, a concrete on a non-drainable base, and concrete that was submerged.

Findings

The following conclusions can be drawn :

- While similar mixture proportions were used for paving mixtures in Indiana differences in the magnitude of water absorbed occurred.
- Samples dried to a lower relative humidity showed a greater volume of water absorbed.
- Drying water absorption samples at 105C resulted in substantial anomalies in water absorption, as such this method is not recommended.
- Absorption samples should account for the volume of paste in the sample when this varies
- Samples were tested using deicing solutions as the absorbing fluid. In addition, samples were tested that had previously been exposed to deicing solutions. In both cases the presence of salts altered the viscosity and surface tension resulting in differences in the water absorption.
- The influence water addition to a concrete mixture was able to be determined using electrical conductivity.
- Pore solution conductivity was observed to be approximately linearly related to the degree of hydration.
- A correction must be applied to electrical conductivity or resistivity samples tested at different temperatures.
- Practical field samples the relative humidity in the concrete was always above 80% for the fall winter and spring. The samples that were exposed to precipitation events demonstrated higher relative humidities.

Implementation

The results of this investigation indicate that fluid transport can vary significantly even when the similar mixture proportions are used. This work will be combined with results of a national pooled fund project that can promote the development of a new testing procedure that can rapidly assess the fluid transport properties of concrete. This work has laid the foundation for using water absorption and electrical conductivity.

The results demonstrate the importance of the fluid being absorbed as well as the sample conditioning in obtaining meaningful results. It was demonstrated that even when samples are previously exposed to deicing salts which can

alter the rate of fluid absorption. This needs to be considered in the evaluation of field samples.

Tests for rapid conditioning of absorption samples are being evaluated as part of the pooled fund study and will be used as a rapid test is developed. Similarly, INDOT is currently involved in the evaluation of electrical conductivity testing for the potential development of a new standard testing procedure. The results of this work can be combined with the results of that study for the development of a new testing procedure to rapidly assess the quality of concrete.

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16. Abstract <p>The objective of this project was to evaluate the transport properties of concrete pavement in the state of Indiana using common testing procedures. Specifically this work evaluated the absorption of water, the absorption of deicing solutions, and electrical conductivity. A series of concrete paving mixtures were tested to provide a range of values that were typical for the state of Indiana. While similar mixture proportions were used for the mixtures in Indiana differences in the magnitude of water absorbed occurred. A series of mortars were tested to illustrate the effect of curing conditions, water to cement ratio, and paste volume. It was observed that a long duration of drying was needed to obtain equilibrium. Samples dried to a lower relative humidity showed a greater volume of water absorbed. It was observed that drying at 105C resulted in substantial anomalies in water absorption, as such this method is not recommended. It was observed that when samples were tested using deicing solutions or samples were tested that were previously exposed to deicing solutions the water absorption could be influenced. The electrical conductivity work was performed as a potential method to develop the understanding of rapid test techniques for quality control. The research used a modified parallel law to relate the electrical conductivity to the pore volume, pore solution conductivity and the tortuosity through the pore network. The influence water addition was able to be determined using electrical conductivity. In addition, the pore solution was observed to be approximately linearly related to the degree of hydration. It is critical that a correction be applied to samples tested at different temperatures. An activation energy of conduction was observed that was approximately 10 kJ/mol irrespective of water to cement ratio. In addition to the measurement of transport properties, the relative humidity was assessed for concrete exposed to different exposure conditions. The samples considered in this investigation included a sample stored at 50% relative humidity, covered concrete, a concrete with an exposed vertical surface, a concrete on a drainable base, a concrete on a non-drainable base, and concrete that was submerged. The samples showed that for practical field samples the relative humidity in the concrete was always above 80% for the samples tested. The samples that were exposed to precipitation events demonstrated higher relative humidities.</p>			
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CHAPTER 1: INTRODUCTION

1.1 Background

Several recent studies have indicated the importance of fluid transport on the durability of concrete pavements. For example, a recent survey by the Iowa Department of Transportation (Nantung and Byers 2006) indicated a correlation between permeability of a concrete pavement and its durability. The Iowa Department of Transportation measured the permeability of cores taken from concrete pavements that have been in service for over eighty years. The results of these scores showed that the low permeability of this concrete aides its ability to withstand environmental loading. Similar anecdotal observations have been made in Michigan on sections of I-94 outside of Detroit that are performing well after fifty years of service (Nantung 2006). Indiana has similar experience with their concrete pavements. For example Yang et al. (2005) demonstrated a relationship between rate of water absorption and the level of damage that exists in concrete pavements. Cores from around the state of Indiana demonstrated that pavements with high rate of water absorption tended to be performing more poorly in the field. It should be noted that Yang research measured the properties of the field concrete long after the concrete was placed so it is difficult to completely separate the initial material properties from the degradation that came over time due to loading and cracking.

Several recent studies have shown that a relationship exist between the transport properties of concrete and performance. The Virginia Department of Transportation, VDOT (Lane 2006) has recently performed two studies related to the permeability of concrete. The first VDOT study (Ozyildirim 1998) investigated the fabrication and testing of low permeability concrete while the second VDOT study compared different methods of assessing permeability (Lane 2006). VDOT has proposed the development of a database of permeability properties for pavements used in their state (Lane 2006). This would enable permeability properties to be measured and used in quality control procedures and service life prediction. It is anticipated that other states will follow this lead and build their own databases. Once such a database is developed for a state, the state highway engineer can become familiar with the transport properties that they can expect to obtain with their current proportioning and casting processes. Contractors and agencies can then become more familiar with how changes in mixture proportions and new materials lead to either improved or more detrimental behavior. This would enable agencies like INDOT to begin to develop concretes with lower transport properties (e.g. permeability) through mixture proportioning, admixtures, or surface coatings that are specifically aimed at improving long-term performance. This can lead to improved construction practices that lead to improved long-term performance in concrete pavements that would benefit the citizens of the state of Indiana.

In addition to providing a database of the materials being used in the state of Indiana, the measurement of permeability (transport) can be used in the development of service life prediction or end result specification models. Recent work in SPR 2941 (Barde 2006) has used an approach modeled after the one developed by Fagerlund (1999)

1 and Bentz et al. (2001) to estimate the service life of concrete pavements in Indiana as a
2 function of air content and secondary sorptivity as shown in Figure 1.1. This model is
3 based on the concept that a pavement which has a lower rate of water absorption would
4 require a longer time to saturate and would therefore be more resistant to freezing and
5 thawing damage resulting in a longer service life. This model also accounts for the role
6 of air content in extending the time required for the concrete to reach a critical saturation
7 level, thereby also extending the service life. The work performed in SPR 2941 was
8 based on literature review and modeling concepts that followed the approach of
9 Fagerlund (1999) and Bentz et al. (2001). As such, paving concrete mixtures and field
10 documented performance were not used to develop these predictions for Indiana. In
11 addition, the work from SPR 2941 used approximations for the degree of saturation that
12 can be expected in concrete using rainfall data (Bentz et al. 2001) rather than measuring
13 saturation directly from the concrete. It is expected that both additional measurement of
14 the permeability properties as well as direct measurements of the degree of saturation in
15 the field can improve these models and their predictions. This work is currently being
16 conducted for INDOT pavement materials as a part of SPR 3200.

Figure 1.1: An Example of the Relationship between the Permeability Properties of a Concrete to its Service Life when Exposed to Freezing and Thawing Cycles (Barde 2006)

While it is generally believed that the transport properties of concrete are related to its long-term durability and performance, there are no common tests or transport specifications used by Indiana for the specification, acceptance, or evaluation of concrete pavements. As such, a need exists to implement a transport test that can be used to develop a database of transport properties of the mixtures used in the state of Indiana. Further, research is needed to determine the current level of permeability (transport) that is achieved and to relate this property with long-term performance.

1.2 Problem Statement

This project evaluates the fluid transport properties of concrete pavements currently being placed in the field. Specifically, this work use transport tests for to asses several concrete pavements in Indiana. These tests measured the transport properties of

1 concrete pavements that are typical for the state of Indiana. These transport properties can
2 eventually be combined with exposure conditions and computer modeling concepts for
3 the purpose of developing performance prediction models. However, at the current time
4 INDOT was performing the study they are only beginning to develop the database of
5 transport properties.

6 1.3 Research Objective and Scope of Project

7 The following section describes the tasks that were performed in this project in
8 greater detail.

9 The second chapter of the report describes a review of literature pertaining to the
10 transport properties of concrete. The main objectives of this chapter are to:

- 11 1) Assemble a listing of test methods commonly used to measure transport
12 properties of paving concrete.
- 13 2) Assemble a listing of cases that link transport properties with field
14 construction, and
- 15 3) To review recent developments in relating transport properties and the service
16 life of concrete.

17 Chapter 3 reviews experimental results from fourteen different sites/paving
18 materials that were selected for this project. Several testing protocols were used fully

1 characterize the transport properties of concrete. Originally samples will be tested
2 according to the following procedures:

- 3 1) ASTM C-457 to assess the amount of air void in the concrete system,
- 4 2) ASTM C-642 to assess the volume of permeable voids,
- 5 3) ASTM C-1585 to assess the water absorption of the concrete,
- 6 4) Rapid electrical property measurement (Rajabipour 2006),
- 7 5) ISO 12572 to assess moisture diffusivity, and
- 8 6) Assess moisture absorption and desorption (using the procedure commonly
9 used at Purdue which will be performed on pastes and thin sections due to
10 the time required by this test).

11 However, after the literature review some of the test methods were updated.
12 ASTM C-457 was updated to an automated method developed after the work of Peterson
13 (2008). ISO 12572 was updated to a more accurate method using a vapor sorption
14 analyzer able to hold relative humidity from 98 % to 0 % at constant temperature. In
15 practical term, this updated method allows to have information for the entire desorption
16 spectra compared with the 2 points spectra provided by ISO 12572.

17 Chapter 4 reviews experimental results from mortars prepared in laboratory using
18 different w/c (0.35, 0.40, 0.45 and 0.50). These samples were included in this report
19 because allow a better control of the mixture proportion and the moisture history, which

1 allows a better assessment of the transport properties. Water absorption based on ASTM
2 C-1585 procedure was used to evaluate these samples.

3 Chapter 5 reports series of wetting and drying tests performed on concrete using
4 different aqueous solutions containing deicing salts. Samples were subjected to different
5 conditioning regimes to evaluate its effect on the test results. This information can be
6 used to better understand water absorption results on samples obtained from the field.

7 Chapter 6 describes a comparison of the ASTM C-1585 testing procedure with a
8 modified version of the ISAT (Initial Surface Absorption Test) for which the data
9 collection can be automated. A comparison of the two methods is presented and
10 statistical significance and repeatability is discussed for each test procedure. This
11 automated test provides additional information compared with the standard ASTM C-
12 1585. This additional data can provide information for further analysis of the absorption
13 behavior of the samples. This test method can also be used to decreases the influence of
14 the operator, and is less time consuming after the test started.

15 Chapter 7 describes the use of electrical properties to assess transport properties.
16 In addition to testing the field samples, a laboratory study was performed to illustrate the
17 influence of minor variations in water and air content on the measured transport
18 properties. Specifically the research illustrates the differences that can be measured in
19 transport properties with 1) production variation, 2) the addition of water as opposed to a
20 water-reducing admixture to maintain slump, and 3) the use of inaccurate daily aggregate
21 moisture contents. This information will be used to developing testing and production
22 variation statements. In addition this will be used for the development of educational
23 materials that will be developed for the concrete paving contractors.

Chapter 8 reports results from samples exposed to the environment to assess relative humidity and temperature that can be expected in the field. An exposure site was developed in West Lafayette that used embedded sensors. The sample geometry was held constant for the samples on the exposure site using a paving mixture from Indiana with a w/c of 0.42. Five different exposure conditions were considered to better understand the effects of boundary conditions on the level of saturation that develops inside the pavement. These boundary conditions will include: 1) exposed vertical surface - covered, 2) exposed vertical surface, 3) horizontal surface on a drainable base, 4) horizontal surface on a non draining base, and 5) submerged. In addition, a specimen was exposed to 50 % relative humidity.

Chapter 9 discusses the recommendations generated after this study. The information from this report provides the beginning of documentable database for transport properties in the state of Indiana. These results will be combined with results from an ongoing pool funded study to develop testing protocols that can be used in Indiana.

CHAPTER 2: LITERATURE REVIEW

2.1 Introduction

A literature review on fluid transport was performed. The theory of fluid transport in concrete will be presented first, including a description of the equations used to characterize absorption and permeability. This is followed with a description of the most common tests. The effect of samples preparation is discussed.

2.2 Review of Theory of Fluid Transport in Concrete

The ability of concrete to absorb fluid has an influence on its durability. Deterioration processes such as freezing and thawing, sulfate attack, reinforcement corrosion are influenced by the ability of concrete to resist ingress of fluid (Weiss 1999, Nokken and Hooton 2004).

Three mechanisms can be used to describe transport of fluid in cementitious systems: (1) permeability, (2) absorption and (3) diffusion. Permeability is the measure of the flow of water under a pressure gradient while diffusion is the movement of ions due to a concentration gradient. Absorption can be described as the materials ability to take in water by means of capillary suction.

Transport proprieties of concrete are controlled by the characteristics of its pore network. Total porosity, pore size, pore connectivity, and pore saturation all influence the

measured transport coefficients (Garboczi 1990; Bentz et al. 1999). The pore structure of concrete is complex, spanning six or more orders of magnitude (Bentz et al. 1995). In ordinary portland cement concrete, the largest pores are typically contained in the interfacial transition zones present between cement and aggregate particles (Winslow and Liu 1990).

At equilibrium, the saturation of a concrete is determined by the pore structure (size and volume) and the local relative humidity. Many of the pores in concrete are sufficiently small such that condensation will occur at RH values much less than 100%. The relationship between the smallest pore radius which remain water filled r_p , and RH (for RH greater than 50 %) is described by the Kelvin-Laplace equation (Equation 2.1) (Bentz et al 1995):

$$\ln\left(\frac{RH}{100}\right) = -\frac{2V_m\sigma}{r_pRT} \quad (2.1)$$

where V_m is the molar volume of water, σ is the surface tension of the liquid, R is the universal gas constant, T is the absolute temperature in degree Kelvin.

The validity of this equation for RH lower than 50 % is questionable due the fact that the water meniscus would be composed of only about ten molecules. A considerable hysteresis exists in the adsorption/desorption curves, due to ink-bottle and pore topology effects. Thus, simply knowing the RH of a concrete is insufficient for making an accurate estimate of its moisture content (Martys 1995, Rajabipour and Weiss 2006).

2.2.1 Absorption

According with Boddy et al (1999), when empty or partially empty pores come into contact with a wetting liquid phase, the liquid will invade due to the capillary forces present in each pore. The local capillary force is inversely proportional to the pore diameter, with smaller pores exerting a large capillary force (although the rate of ingress into a smaller pore will actually be less than that into a large one).

For the case of one-dimensional flow, absorption of concrete is generally defined by its sorptivity, S , determined using Equation 2.2 (Martys 1995):

$$M(t) = \rho AS\sqrt{t} \quad (2.2)$$

where $M(t)$ represent the mass of liquid gained by the specimen at time t , ρ is the density of the invading liquid, A is the surface area of the specimen exposed to the liquid, and S is the sorptivity in units of $\text{length}/\text{time}^{0.5}$. For the field concrete, the square root of time dependency may not always be followed, as the exponent in the power law function may vary between 0.2 and 0.5 (Sosoro 1998).

In practice, it is often observed that there is a rapid initial absorption so the Equation 2.2 is modified to Equation 2.3 (Parrot 1994):

$$M(t) = M_o + \rho AS\sqrt{t} \quad (2.3)$$

where M_o represents the initial mass gain.

Deviation from the square root of time behavior at longer times has been observed in numerous experiments (Sosoro 1998). It is generally attributed to interactions of water with the concrete. Hall and Yau (1987), and Martys and Ferraris (1997) have proposed modifications to Equation 2.3 to account for these long time deviations. The proposed equation of Hall and Yau (Equation 2.4), has the form:

$$M(t) = M_o + \rho A(S\sqrt{t} - C_H t) \quad (2.4)$$

The proposed equation of Martys and Ferraris (Equation 2.5) has the form:

$$M(t) = M_o + \rho A \left[S_g \sqrt{t} + C_M \left(1 - \exp\left(\frac{-S\sqrt{t}}{C_M} \right) \right) \right] \quad (2.5)$$

where C_H and C_M are constants obtained from fitting the experimental data, and S_g describes the sorptivity in the smaller pores or the effects of moisture diffusion.

2.2.2 Permeability

Permeability of concrete plays an important role in durability because it controls the movement and the rate of entry of water, which may contain aggressive chemical

1 (Bamforth 1987). Permeability is characterized by the permeability coefficient k . It can
 2 be measured as gas permeability and liquid permeability.

3 2.2.2.1 Gas Permeability

4 To measure gas permeability in the most direct manner, a pressure is applied
 5 across a concrete specimen and the flow rate at steady is measured. With this setup, the
 6 gas permeability, k_g , is given Equation 2.6 (Bamforth 1987):

$$7 \quad k_g = \frac{2Qx\eta P_f}{A(P_1^2 - P_2^2)} \quad (2.6)$$

8 where P_1 and P_2 are the upstream and downstream pressures respectively, Q is the
 9 volumetric flow rate in m^3/s , x is the specimen thickness in m , A is the area for flow in
 10 m^2 , and k_g is the permeability coefficient in units of m^2 . P_f correspond to the pressure at
 11 which the flow rate is measured, either P_1 or P_2 , depending on the experimental setup.

12 Gas permeability can be also calculated using the modified Darcy's equation
 13 (Equation 2.7), as proposed by Grube and Lawrence (Grube and Lawrence 1984):

$$14 \quad K_o = \frac{2\mu p_{\text{out}} QL}{A(p_{\text{in}}^2 - p_{\text{out}}^2)} \quad (2.7)$$

1 where Q is volume flow rate (m^3/s), L is sample thickness (m), p_{in} is pressure at inlet
 2 (bar), p_{out} is pressure at outlet (bar), and A is cross-sectional area of sample (m^2).

3 2.2.2.2 Liquid Permeability

4 There exist a wider set of proposed equation for measurement of liquid
 5 permeability. According with Bamforth (1987), the governing equation for determining
 6 intrinsic permeability is (Equation 2.8):

$$7 \quad k_l = \frac{Qx\eta}{A(P_1 - P_2)} \quad (2.8)$$

8 where all terms were previously defined.

9 According with Shafiq and Cabrera (2004), the intrinsic coefficient of water
 10 permeability can be calculates using the Equation 2.9:

$$11 \quad K_w = \frac{d^2 v}{2th} \left(\frac{\mu}{\rho g} \right) \quad (2.9)$$

12 where d is the depth of water penetration (m), t is the time of penetration (s), h is the
 13 applied pressure (m), v is the total porosity (fraction), ρ is the density of water (kg/m^3), μ
 14 is the viscosity of water (Ns/m^2), and g is the acceleration due to gravity (m/s^2).

15 Katz and Thompson (1986) proposed the relationship show in Equation 2.10:

$$K = c \cdot d_c^2 \cdot \left(\frac{\sigma}{\sigma_o} \right) \quad (2.10)$$

where K is the permeability (m^2), d_c is the critical pore diameter (m), σ is the electrical conductivity of the sample ($\Omega\text{-cm}$)⁻¹, σ_o is the conductivity of the pore fluid ($\Omega\text{-cm}$)⁻¹, and c is a constant.

To predict the permeability of a material using the Katz-Thompson equation, the critical pore diameter (d_c) and the normalized conductivity (σ/σ_o) must be measured. Katz and Thompson related the critical pore diameter to the inflection point in mercury intrusion curves. The physical interpretation of the critical diameter is that it is the smallest continuous pore size that percolates through the sample. The normalized conductivity is determined using electrical methods.

According to Shane et al. (1997), the main advantage of the Katz-Thompson relation is that the time required to make these measurements is much shorter and much less labor intensive than is required to make permeability measurements. Also, unlike with permeameters, there is not a limit of permeability that can be predicted, once set has occurred, and d_c can be measured. This allows specimens of any age, or composition, to be evaluated.

2.2.2.3 Gas Permeability vs. Liquid Permeability

When comparing gas permeability to liquid permeability, it is necessary to correct for the slippage of the gas, commonly referred as the Klinkenberg correction (Nokken

and Hooton 2004). By measuring the gas permeability under a variety of pressure gradients, the intrinsic permeability (permeability at infinite pressure) can be derived by fitting the data to an equation of the form (Equation 2.11):

$$k_l = \frac{k_g}{1 + \frac{b}{P_m}} \quad (2.11)$$

where k_l is the intrinsic permeability, P_m is the mean pressure of the inlet and outlet streams, and b is a constant for a given gas and a given concrete.

2.2.3 Diffusion

The present section provides a method to calculate diffusivity, the coefficient of moisture diffusion, from weight loss of a sample exposed to controlled conditions (i.e., relative humidity and temperature).

2.2.3.1 Moisture Desorption Technique

Moisture desorption is an established technique for evaluating the effect of moisture loss at a given humidity for a material. A TA Q5000 SA moisture sorption analyzer was used in this investigation (Figure 2.1). The system enables the desorption behavior to be evaluated under carefully controlled conditions of temperature and humidity (Castro et al 2010a).

Figure 2.1: Picture of Q5000

Figure 2.2 shows an example of the results that can be obtained from moisture desorption. As the relative humidity is changed, the sample undergoes a rapid change in mass. The mass change decreases as the sample approaches equilibrium. It can be seen that this general behavior is observed at each change in the relative humidity, however, the magnitude of the mass change is different at each relative humidity and would be consistent with the volume of pores from which water is being lost at each step.

Figure 2.2: A Typical Mass Change versus Time Plot for LWA Prewetted with 1 h Synthetic Pore Solution as Example of the Technique (Castro et al. 2010a).

2.2.3.2 Moisture Diffusivity

The moisture diffusivity of cement based materials is a function of RH, however, at each step during this experiment the moisture diffusivity can be considered constant since the change in RH value is small. The change in mass of sample, M_t , as a function of time, t , can be represented using Equation 2.12 (Crank 1980):

$$\frac{M_t}{M_\infty} = \frac{4}{\pi} \left(\frac{\pi t}{L^2} \right)^{1/2} \quad (2.12)$$

where M_∞ is the mass change at equilibrium, D is the diffusivity and L is the thickness of the sample. Equation (2.12) can be used to obtain the D at each step by calculating the slope of a curve that is obtained by plotting $\frac{M_t}{M_\infty}$ versus square root of time.

2.2.4 Effect of Mixture Proportions and Curing on Transport Properties

2.2.4.1 Water Content

The water to cement ratio is generally considered to be the governing parameter, which affects the strength and durability of concrete. When a high water to cement ratio is used in concrete it will generate a higher initial porosity. However the threshold values are very dependent of the curing regime will lead premature deterioration of concrete structures has created (Figg 1973).

2.2.4.2 Supplementary Material

a) Silica Fume

According to Bentz (2000), silica fume influences concrete diffusivity in several ways: (1) by densifying the microstructure if the interfacial transition zone, (2) by reducing the overall capillary porosity for a fixed degree of cement hydration, and (3) by production a pozzolanic C-S-H gel with a relative diffusivity about 25 times less than that of the C-S-H gel produced from conventional cement hydration. This beneficial effect is observed regardless of the temperature of the curing regime (Cabrera 1996).

b) Slag

Because of their low heat of hydration, slag cements help to prevent cracks in mass concrete structures resulting from temperatures stresses at early ages (Geiseler et al.

1995). For this reason, slag replacement offers stronger and more durable concrete in hot climates. According to Bleszynski et al. (2002), the incorporation of blast-furnace slag into silica fume concrete reduces the water demand. It can lead also to a lower initial porosity of the concrete with its advantages to reduce durability problems.

5 c) Fly Ash

6 Fly ash, a by-product of the combustion of pulverized coal in thermal power
7 plants, is commonly used in concrete as a partial replacement for Portland cement. Fly
8 ash is capable of reacting with the calcium hydroxide (CH) produced during cement
9 hydration to form calcium silicate hydrate (CSH) which fills large capillary voids and
10 disrupts their continuity. Class F fly ash reduces permeability even at relatively low
11 levels of cement substitution (10% by weight). In the case of Class C fly ash however,
12 relatively high cement substitution level (20 to 30% by weight) may be required to
13 produce any significant reduction in permeability (Alhozaimy et al. 1996).

14 2.2.4.3 Effect of Curing

15 Several researchers have studied the relationship between permeability and pore
16 size distribution of cement paste or mortar. They concluded that the flow of a fluid
17 through concrete is associated with the larger capillary pores rather than total porosity.
18 Pore volume above a certain size pore (in the region of 880 – 1500 Å) is thought to be
19 closely related to permeability (Gowripalan et al. 1990).

The rate of chloride ingress through the concrete cover will be influenced by the type and extend of curing. A lower degree of hydration of the cementing materials close to the surface will result in higher porosity and a more connected capillary pore system and, therefore, diffusion coefficients through the cover depth will be spatially dependent. As periods of moist curing are extended, the depth-dependent effect on diffusion coefficient is reduced (Hooton et al. 2002).

According to Wang et al. (2006) regardless of the application of curing compound, properties of the near-surface layer concrete, such as moisture content, permeability, degree of hydration, and sorptivity, differ from those of the internal concrete. However, application of a curing compound significantly increase degree of cement hydration and decreases sorptivity of the near-surface layer concrete, which reduces the difference in concrete properties between the near-surface layer and internal concrete.

The effectiveness of initial curing becomes more important when mineral admixtures like fly ash are used as partial substitution for cement concrete (Shafiq and Cabrera 2004).

2.2.5 Effect of Samples Preparation on Tests Results

2.2.5.1 Degree of Saturation and Relative Humidity

Both sorptivity and gas permeability are strongly influenced by the water content of the concrete being evaluated (Abbas et al. 1999). As the water content is increased,

1 more and more of the pores are filled with liquid water, which significantly alters both
2 the sorption liquid water and the transport gas. Thus, even in laboratory testing, the pre-
3 conditioning of the specimens is of paramount importance (Boddy et al. 1999). For liquid
4 permeability, the samples are normally vacuum saturated. Details of sample preparation
5 will not be discussed in this section.

6 Many different samples preparation methods had been used for sorptivity and gas
7 permeability. During the late 80's and early 90's drying at 105 °C was used as the
8 conditioning method (Dhir et al. 1989). However, it is now well recognized that this
9 method produces a moisture content that is atypical with respect to field conditions
10 (Boddy et al 1999). Additionally, the elevated temperature will lead to severe drying of
11 the cement gel that will alter the microstructure of the cementitious binder resulting in
12 elevated values for sorptivity (Parrot 1994).

13 The Cembureau method (Kollek 1986) recommended that one of the following
14 two pre-conditioning regimes be employed: 1) storage in a laboratory atmosphere at 20
15 °C and 65 % RH for 28 days or 2) drying in a ventilated oven at 105 °C for 7 days
16 followed by storage in a desiccator for 3 days at 20 °C. However, it is well recognized
17 that it may take several months to obtain moisture equilibrium in specimen only a few
18 centimeters thick

19 In 1994 Parrot (1994) conducted a series of experiments to analyze conditioning
20 procedures and assess the moisture distribution of concrete samples. The Parrot (1994)
21 work used 100 mm cubes of concrete with a cast-in cylindrical cavity. These samples
22 were used to measure the differences in relative humidity between the inside and the
23 outside of the sample. That research reported that drying for 4 days at 50 °C followed by

1 3 days of sealed storage at 50 °C caused a small relative humidity gradient in the concrete
2 cube that gradually diminished when the specimens were placed in a sealed container at
3 20 °C for 25 days.

4 The sample conditioning established by ASTM C-1585 (2004) consist of placing
5 the specimens in an environmental chamber at a temperature of 50 ± 2 °C and RH of $80 \pm$
6 3 % for 3 days. Alternatively, the specimens can be placed in a desiccator inside an oven
7 at temperature of 50 ± 2 °C for 3 days. If the desiccator is used, the RH is controlled
8 using a saturated solution of potassium bromide. After 3 days, the specimens are placed
9 inside a sealable container for at least 15 days at 23 ± 2 °C. The total drying period of 18
10 days recommended in the standard is reasonable and certainly more efficient than the
11 several month required for the equilibrium of moisture when conditioning chambers are
12 used.

13 When the standard recommendation is followed, specimens should obtain a
14 humidity between 50 % and 70 %. However, Nokken and Hooton (2002) established
15 experimentally that the rate of absorption is highly dependent of the actual degree of
16 saturation. Then, if a sorption test is performed on twin specimens conditioned at RH of
17 50 % and 70 %, the rate of absorption will be different.

18 2.2.5.2 Temperature

19 Temperature influences sorptivity through its effect on the surface tension (σ) and
20 the dynamic viscosity of the fluid (η). Hall indicates that absorption should scale as
21 $(\sigma/\eta)^{0.5}$ (Hall 1994). Using this relationship, measurements made at different temperatures
22 can be normalized to a common standard temperature for comparative purposes.

1 While this scaling relationship holds well for the absorption of organic fluids by
2 concrete, significant deviations from the theory are observed when water is used as the
3 sorbing fluid (Sosoro 1998). This could be due to the changing solubility of calcium
4 hydroxide with temperature and the complex interactions of water with the concrete
5 matrix (Boddy et al. 1999).

6 2.2.5.3 Critical Analysis of Effect of Samples Preparation

7 It appears clear that sample preparation could be an important issue when sorption
8 test are used to characterize different mixtures. Following the standard procedure, the
9 sample preparation is reported to result in a relative humidity between 50 % and 70 %,
10 however this is a wide range.

11 The main factor that will affect the final moisture content in samples after
12 standard preparation will be the initial moisture content. However, not work was found
13 about this topic. It will be a very interesting area for future research.

14 With respect to temperature not too much research had been performed on the
15 absorption at low temperatures. A consensus appears to exist to report sorptivity results at
16 23 °C. However it could be important develop a better understanding of what happens in
17 terms of absorption and desorption of fluids when temperatures are close to freezing
18 point.

2.3 Methods to Measure Gas and Water Permeability in Concrete

Much of the trouble encountered with concrete structures is due to the ingress of harmful materials into the concrete. Consequently, durability can be improved significantly by producing concrete that is highly resistant to the ingress of external harmful substances as Mehta said: “impermeability of concrete should be the first line of defense against any of the physiochemical deterioration processes” (Mehta 1994).

Therefore, the evaluation of the permeability in a concrete structure is an essential and important step for the definition of its durability, performance and service life. This report is intended to be brief description of some of the most commonly used methods and their advantages and disadvantages. One general point that must be borne in mind when discussing these tests is that few of them measure absolute values of fundamental transport properties; what they do provide is an index or relative value and this is useful for comparing one concrete with another.

2.3.1 Gas Permeability

Measuring the gas permeability of concrete is relatively quick and enables specimens conditioned at any age to be tested without any time delay. In addition, the conditions of the specimen do not change during the testing. For these reasons these tests have become quite common in order to measure the permeability of concrete. Testing concrete for gas permeability can be carried out by either keeping the head constant and measuring the flow or by monitoring the pressure decay over a specified time interval (Basheer 2001).

2.3.1.1 Schnlin Method

The method consists of placing a cell, equipped with a rubber gasket, on the concrete surface and creating vacuum. Figure 2.3 shows a schematic illustration of this method. Once a certain vacuum has been reached, the stopcock is closed and the time elapsed for the absolute pressure to rise from 50 to 300 mbar is recorded. These measurements allow the calculation of a permeability index, which is defined as the airflow into the chamber divided by the average chamber depression. The method is very fast (~3 minutes) and easy to perform and it is entirely non-destructive.

This test is considerably influenced by the moisture condition of the concrete. To eliminate this problem, Schnlin and Hilsdorf (1987) suggested that the concrete surface should be dried with hot air for 5 min before the start of a test.

Figure 2.3: Schematic View of the Schnlin Method

1 2.3.1.2 Surface Air Flow Test

2 The surface airflow test (SAF) is based on a method used by the petroleum
3 industry to determine the permeability of rock cores (Whiting and Cady 1992). A
4 vacuum plate is placed on the concrete surface. Valves A, B, and C (Figure 2.4) are
5 closed. The vacuum pump is started and allowed to stabilize at a vacuum pressure of
6 approximately -83 kPa. Valve A is then opened allowing the vacuum to be created inside
7 the vacuum plate. After the vacuum has been created, valve A is closed and valve B is
8 opened. To maintain the vacuum pressure, the air that has permeated into the vacuum
9 chamber through the concrete is drawn out through the flow meter. After a period of
10 approximately 15 seconds, to allow the system to stabilize, the flow rate of the air is
11 measured by a flowmeter. This flow rate is used as a measure of the surface air
12 permeability. Similar to the Schönlin test, if the surface layer has high moisture content,
13 the surface should be dried with hot air.

Figure 2.4: Schematic of Surface Airflow Test (Henderson et al. 2004)

2.3.1.3 Autoclam

Basheer et al. (1993) developed "Autoclam" permeability system for measuring the in-situ permeation properties of concrete by applying a pressure over the concrete surface to force air into it. The Autoclam Permeability System measures the air permeability, unsaturated water permeability and water absorption (sorptivity) of concrete, brick masonry and stone masonry, both in the laboratory and on site.

The instrument, which is currently available in four different versions, is totally portable and easy to use on site. Non-skilled personnel can carry out the tests. The tests can be carried out without leaving any mark on the test surface (Figure 2.5).

The total duration of each test is less than 30 minutes, which includes instrument setting up time.

Up to 12 tests can be carried out using the battery pack available within the instrument or run continually during the day if an external power supply is available. The

data from each test is stored in the controller of the instrument and can either be recorded manually or transferred to a PC for further analysis (Amphora NDT Limited 2009).

After the body of the apparatus is placed on the based ring, the pressure in the test area is increased to 1.5 bar, using the syringe attached the priming valve. The pressure decay is monitored for 15 minutes (or until the pressure reaches zero) with 1 minute interval. The slope of the pressure-time curve between 5th and 15th minute is used as the air permeability index. The test should be not carried out if the internal relative humidity of the covercrete is greater that 80 %. This should be considered in light of the results presented in Chapter 8.

Figure 2.5: Autoclam Apparatus for Permeability Tests (Zia et al. 1994)

2.3.1.4 Torrent Permeability Tester (Torrent 1992; Mastrad Limited 2009; Proceq 2009)

The TORRENT permeability tester is based on investigations which were carried out by the research center of “Holderbank Management and Consulting Ltd.”, Switzerland (Figure 2.6).

1 The particular features of the Torrent method are a two-chamber vacuum cell and
2 a pressure regulator. This ensures that an air flow at right angles to the surface is directed
3 towards the inner chamber. The cell is placed on the concrete surface and a vacuum is
4 created in both chambers with pump. Due to the external atmospheric pressure and the
5 rubber rings, the cell is pressed against the surface and thus both chambers are sealed.
6 After 1 minute, the inner chamber is insulated. From this moment, the pressure in the
7 inner chamber starts to increase, as air is drawn from the underlying concrete. The rate of
8 pressure raise, which is directly related to the permeability of concrete, is recorded.
9 Meanwhile, the vacuum pump continues to operate on the outer chamber to keep the
10 pressure equal in both chambers. The permeability coefficient can then be calculated
11 (Table 2.1).

Figure 2.6: Schematic of Torrent Test Setup

Table 2.1: Concrete Categories Based on Torrent Permeability Coefficient

Concrete category	Covercrete quality	Torrent permeability coefficient (kT) after 28 days ($\times 10^{-16} \text{ m}^2$)
1	excellent	$kT < 0.01$
2	good	$0.01 < kT < 0.1$
3	fair	$0.1 < kT < 1$
4	not very good	$1 < kT < 10$
5	poor	$kT > 10$

1 The measurement takes 2-12 minutes, depending on the permeability of the
2 concrete. In the case of dry concrete, the quality class of the concrete cover can be read
3 from a table using the kT value. In the case of moist concrete, kT is combined with the
4 electrical concrete resistance R (Ohm) and the quality class is determined from a
5 nomogram. The test is completely non-destructive, very easy to handle

6 2.3.1.5 TUD Method (Reinhardt-Mijnsbergen) (Torrent et al. 2007)

7 The method is consisted of drilling a small hole with diameter of 10 mm and the
8 depth of 40 mm on the surface of the concrete. A cylindrical hollow probe is introduced
9 to the hole. After sealing the hole the chamber the nitrogen gas at the pressure of 10 to
10 10.5 bar is introduced through the probe. After, shutting the gas flow, the time required
11 for a pressure decay in the chamber from 10 to 9.5 bar is recorded. Time for test
12 including setting up time is about 20 minutes. This method is slightly destructive (Figure
13 2.7).

Figure 2.7: Schematic of the TUD Method

2.3.1.6 Hong-Parrott Method (Parrot and Hong 1991, Torrent et al. 2007)

It consists in drilling a blind hole, with the depth of 35 mm and diameter of 20 mm, into the concrete surface. The hole is sealed with stainless steel and silicon rubber plug. A pressure transducer and a digital indicator are connected to the plug. The hole is pressurized with air slightly above 1 atmosphere, and the time taken for the relative pressure to drop from 50 to 35 kPa is measured. Soap solution is brushed on the concrete surface around the hole which produces fine bubbles as airflows across the surrounding concrete. If the concrete is cracked or poorly compacted, large bubbles can be seen. The measured time is converted into an apparent permeability, as function of the radius affected by the test (revealed by bubbles in the soap solution). Time for test including setting up time is about 40 minutes. This method is slightly destructive (Figure 2.8).

Figure 2.8: Schematic View of Hong-Parrott Method

2.3.1.7 Germann's Gas Permeation Test (Hanson et al. 1984; Germann Inst. Inc. 2009)

A hole is drilled into the surface at an angle of 45 degrees and a pressure sensor is installed at in the hole. CO₂ gas is pressurized to the surface and the sensor pressure recorded over time. The permeation relative to the porosity of the concrete is determined by recorded graphs. The test rig illustrated is secured to the surface by means of two adjustable, anchored clamping pliers. The rig contains a gasket being pressurized against the surface. If needed, the gasket may be glued to the face. Coring of the sensor hole takes place with a water-cooled diamond drill bit at a given distance from the gasket (15, 20 or 25 mm). The sensor is inserted in the cored hole, expanded and checked for air-tightness. The CO₂ surface pressure is 1, 2 or 3 bar and the time of testing is normally 2, 5 or 10 minutes. Alternatively, oxygen can be used (Figure 2.9).

Figure 2.9: Schematic View of the Germann Gas Permeation Test Method (Germann Instruments Inc. 2009)

2.3.1.8 Cembureau Method (Kollek 1986)

In this method, unidirectional gas flow between two parallel surfaces of the test specimen (typically 150 mm diameter by 50 mm thick) is caused by a constant absolute pressure difference of the test gas between the two surfaces. The testing should be carried out in a laboratory with $T = 20 \pm 2 \text{ }^{\circ}\text{C}$ and $\text{RH} = 65 \pm 5 \%$. It is recommended to determine the pressure of five absolute inlet pressure stages as: 1.5, 2, 2.5, 3 and 3.5 bar. At each stage, the flow rate should be allowed to stabilize, which normally achieved within 5-30 minutes. The mean specific coefficient of permeability is evaluated from the five permeability coefficient values obtained for the five pressure stages. The gas flow depends on the pressure difference, testing area, specimen thickness and open porosity, and the viscosity of the test gas (Figure 2.10).

Figure 2.10: Schematic of Cembureau Method

2.3.2 Water Permeability

Methods available for testing water permeability can generally be categorized as absorption tests and permeation tests.

Absorption tests involve the intake of a fluid due to capillary suction in the pores. Under perfect conditions, the magnitude of capillary rise follows a linear relationship with the square root of time elapsed, and the constant of proportionality is called the sorptivity and ideally, this is the property that should be measured in an absorption test (Basheer 1993). However, there are always practical limitations such as: difficulty in achieving a unidirectional penetration of water, problems of determining the water penetration depth without actually splitting the concrete specimen, and etc. Therefore, the absorption characteristics of concrete are usually measured indirectly by one of the following types of test.

Permeability is the property of concrete that describes resistance to a fluid penetration under the action of a pressure gradient (The Concrete Society 1988; Perraton

1 and Aitcin 1992). Sometimes, permeability is confused with porosity, and vice-versa.
 2 Porosity is simply a measure of the proportion of the total volume occupied by pores,
 3 usually expressed as a percentage (Neville 1995).

4 2.3.2.1 ISAT (Initial Surface Absorption Test) (Basheer 2001)

5 The ISAT was originally developed by Glanville in 1931 and modified by Levitt
 6 in 1970s (Levitte 1971). The test starts with placing an acrylic cap onto the concrete
 7 surface with a minimum water contact area of 5000 mm² and making it water-tight by
 8 clamping it (Figure 2.11). A pressure head of 200 mm (~0.02 bar) is set by mean of
 9 water reservoir. With the inlet tap opened, water flows to fill the gap and then through
 10 the outlet it climbs into the calibrated horizontal capillary tube. After 10 min, the tap is
 11 closed and the rate of water suction by the concrete is monitored by following the
 12 retraction of the meniscus in the capillary tube. The absorption values are determined in
 13 this manner at 10, 30, 60 and 120 minutes from the start of the test. The inlet tap is
 14 opened after each measurement in order to allow the head of the water to be maintained
 15 at 200 mm. ISA-value is given by Equation 2.13.

$$16 \quad \text{ISA} = 0.6 D / \delta t \quad (2.13)$$

17 where ISA = rate of water suction (ml/m²/s) and D = Number of scale the meniscus
 18 moves during time δt . The test results are evaluated according Table 2.2. A setup of the
 19 test is shown in Figure 2.11.

Table 2.2: Typical results of ISAT tests on well cured and oven dried concrete

	ISA (ml/m ² /s)			
	Time after starting the test			
	10 min	30 min	1 h	2 h
High	>0.50	>0.35	>0.20	>0.15
Average	0.25-0.50	0.17-0.35	0.10-0.20	0.07-0.15
Low	<0.25	<0.17	<0.10	<0.07

Figure 2.11: Schematic View of the ISAT Method

The main advantage of the ISAT is that it is a relatively quick (~150 min) and simple nondestructive in situ test method that can be used to measure water penetration into a concrete surface. The difficulty of ensuring a watertight seal is probably one of the greatest limitations of this test because of the problems achieving this in practice. Another limitation is that the measured property is greatly affected by the moisture condition of the concrete. The method cannot be applied to the underside of the concrete

1 2.3.2.2 Autoclam

2 As mentioned before, this method was developed by Basheer et al. (1993). In this
3 system, water goes into the test area through a priming pump with air escaping through
4 the bleed pipe and a pressure transducer measure the water pressure. When the test
5 chamber is completely filled with water the priming pump automatically switches off and
6 the micro pump pressurizes the test area to 0.5 bar above atmospheric, at the stage the test
7 starts. At this pressure the transport of water into capillary pores is considered to be due
8 to absorption rather than by pressure induced flow. As water absorbed by capillary
9 action, the pressure inside would tend to decrease, hence it is maintained constant by
10 pump and the control system. The volume of water delivered is measured and recorded
11 every minute for a test duration of 15 minutes. The quantity of water flowing into the
12 material is plotted against the square root of time. The slope of this graph (which is
13 linear between 5 and 15 minutes) is used to specify a water permeability index with unit
14 of $\text{m}^3/\text{min}^{1/2}$. Figure 2.12 shows the schematic setup of the test.

Figure 2.12: Schematic Setup of Autoclam Test for Water Permeability Measurement (Basheer et al. 1993)

2.3.2.3 Germann Water Permeability Test (GWT – 4000)

The GWT (Germann Water permeability Test) is used for on-site evaluation of: the water permeation of the skin-concrete in finished structure, the water permeation of masonry panels, the water tightness of construction joints and sealed control joints and effectiveness of water proofing membranes (Germann Instruments Inc. 2009).

In this method, a pressure chamber containing a watertight gasket is secured tightly to the surface by two anchored clamping pliers or by means of suction plate, (or glue). The chamber is filled with water and the valves closed.

The top lid of the chamber is turned until a desired water pressure is achieved. The pressure selected is maintained by means of a micrometer gauge pressing a piston into the chamber, submitting the water penetrating into the material. The travel of the

1 piston over time is used for characterizing the permeation of the surface tested. Time for
 2 test including setting up time is about 40 minutes.

3 The method is somewhat destructive (holes for fixing). Figure 2.13 shows the
 4 schematic setup of the test.

5
 6 Figure 2.13: Schematic Illustration of the GWT Setup (Germann Instruments Inc. 2009)

7 2.3.2.4 Figg-Poroscope Method

8 The development of this method was first reported by J. W. Figg (1973) in
 9 England. A schematic showing the apparatus and test setup is presented in Figure 2.14.

10 First, a hole with a diameter of 10mm and a depth of 40 mm is drilled
 11 perpendicularly to the surface of the concrete. After clearing, the hole is made airtight by
 12 inserting a special silicon rubber plug, which leaves 20 mm long cylindrical chamber. A
 13 hypodermic needle with two concentric tubes is pierced through the rubber after

1 hardening, and a water head of 100 mm is applied. The chamber and the capillary tube
 2 are filled with water by means of the syringe. After about 1 minute of water contacting
 3 the concrete, the stop-cock is closed and the rate of water suction by concrete is
 4 monitored by movement of a meniscus in a horizontal capillary. The time taken for
 5 concrete to absorb 0.01 ml of water is recorded and reported as the result of the test; and,
 6 the results are evaluated based on Table 2.3.

7 Table 2.3 Concrete Categories Based on the Figg Method

Concrete Category	0	1	2	3	4
Protective quality	Bad	Fair	Medium	Good	Very Good
Poroscope Absorption Time (s)	<20	20-50	50-100	100-500	>500

8 Time for test including setting up time is about 40 minutes. The main advantage
 9 of this method is that it is simple and easy test. On the other hand, drilling the hole might
 10 change the microstructure of the concrete.

Figure 2.14: Schematic Illustration of the Figg-Poroscope Method

2.3.2.5 Field Permeability Test (Florida Test) – FPT

This method is developed by Developed by Meletiou, Tia and Bloomquist in 1990s (Meletiou et al. 1992; Meletiou et al. 1993). It starts with drilling a hole (23 mm in diameter and 152 mm deep into the concrete. Then a cylindrical probe is introduced into the hole. Tightening the top nut causes the two neoprene packers to extend and seal a central chamber. A full vacuum is applied to the chamber for 5 to 10 minutes. The probe is then connected to the instrumentation unit. Water, pressurized by nitrogen bottle, is introduced into the chamber through the hollow probe, at the pressure within the range of 10 to 35 bar (normally 17 bar) and follows radically into the surrounding concrete. After steady-state flow is achieved, the flow rate of the water is recorded (mean value pf the 5

1 measurements) and the coefficient of water permeability is calculated according to
 2 Darcy's law. Time for the test including the setting up time is about 40 minutes and it is
 3 slightly destructive due to drilling the hole Figure 2.15 shows the schematic setup of the
 4 test.

Figure 2.15: Schematic View of the Florida Test Setup

2.3.3 Migration

Ion migration in concrete is a complex process which involves: diffusion, capillary suction, convective flow with flowing water and electrical field. Followings are some of the methods for monitoring/measuring the ion migration in concrete.

2.3.3.1 Electrical Resistivity Methods

In concrete, electrical current is carried out by ions dissolved in the pore solution. More pore solution, as well as more and larger pores with higher degree of connectivity cause lower resistivity and this can be used as the indication of concrete permeability.

a) Resistivity - 4 point (Wenner) Method

The four-probe method is one of the most widely used technique for field measurement of concrete resistivity. This method was originally developed by F. Wenner (1996) to measure the resistivity of soil. In this technique, four electrodes are equally spaced (As shown in Figure 2.16) and a small alternating current is applied between the outer electrodes while potential is measured between the inner electrodes. The resistivity is then calculated by using the Equation 2.14:

$$\rho = \frac{2\pi aV}{I} \quad (2.14)$$

where ρ is the resistivity ($\Omega\cdot\text{cm}$), a is the distance between inner electrodes (cm) and V and I are maximum values of voltage (volts) and current (amps), respectively. Factors that may affect the results of four probe technique measurement are:

1. geometrical constraints,
2. surface contact,
3. concrete non-homogeneity,
4. the presence of steel reinforcing bars,
5. surface layers having different resistivity from the bulk of the concrete, and
6. ambient temperature.

Figure 2.16: Schematic Illustration of the 4 Point (Wenner) Method for Resistivity Measurement

b) Resistivity – Disk Method

It consists in placing one metal electrode on the concrete surface and measuring the resistance between this electrode and the reinforcement or placing concrete between two metal electrodes and measuring the concrete resistance (shown in Figure 2.17). This technique was introduced in 1988 for field application (Newton and Sykes 1988). In this method, a short-time anodic current pulse is applied galvanostatically between metal electrode placed on the concrete surface and the rebar (or between two metal electrodes). The applied current is usually in the range of 10 to 100 μA and the typical pulse duration is between 5 to 30 seconds. The plot of resultant potential difference versus time can be used to determine the concrete resistance as shown in Figure 2.18.

Figure 2.17: Test Setup of the Galvanostatic Pulse Technique

Figure 2.18: Potential Response of the Galvanostatic Pulse Technique. R_{ohm} , Represents Concrete Resistance,

c) Electrical Impedance Spectroscopy (EIS)

EIS studies the system response to the application of a small amplitude alternating potential signal at different frequencies. If, at each excitation frequency, the real part is plotted on the x-axis and the imaginary part is plotted on the y-axis of a chart, a "Nyquist plot" is formed. From this plot, the resistance of the bulk material can be determined (Figure 2.19).

Figure 2.19: Nyquist Plot Obtained from and EIS Test

d) Ion Migration Methods

In these methods, additional ions (usually chloride) are introduced into the pore solution, by applying electrical potential between the source of ion and the concrete. The movement of ions, due to the action of the electrical field is called migration. By applying electrical field to the concrete through two electrodes, several processes develop:

- In both anode and cathode, gases may be generated.
- The ions move through the pore solution in the direction of the electrode of opposite charge.

e) ASTM C1202 and AASHTO T277

The test method involves obtaining a 100 mm diameter core or cylinder sample from the concrete being tested (Figure 2.20). A 50 mm specimen is cut from the sample. The side of the cylindrical specimen is coated with epoxy, and after the epoxy is dried, it is put in a vacuum chamber for 3 hours. The specimen is vacuum saturated for 1 hour and allowed to soak for 18 hours. It is then placed in the test device. The left-hand side (–) of the test cell is filled with a 3 % NaCl solution and the right-hand side (+) of the test cell is filled with 0.3N NaOH solution. The system is then connected and a 60-volt potential is applied for 6 hours. Readings are taken every 30 minutes. At the end of 6 hours the sample is removed from the cell and the amount of coulombs passed through the specimen is calculated and the results are interpreted based on Table 2.4.

Figure 2.20: Schematic of Rapid Chloride Permeability Test

1 Table 2.4: Interpretation of the ASTM C1202 Test (RCP)

Charge Passed (Coulombs)	Chloride Permeability	Typical of
>4000	High	High W/C ratio (>0.60) conventional PCC
2000-4000	Moderate	Moderate W/C ratio (0.40–0.50) conventional PCC
1000-2000	Low	Low W/C ratio (<0.40) conventional PCC
100-1000	Very low	Latex-modified concrete or internally-sealed concrete

2 There are many factors that may affect the accuracy of the test procedure. The
3 age and curing of the test specimen affects the results dramatically. In general, the older
4 the specimen, the lower the coulombs, assuming that the sample has been cured properly.
5 Research has also indicated that the presence in the concrete of admixtures containing
6 ionic salts may affect the results obtained. Cement factor, air content, water/cement ratio,
7 curing of the test sample, aggregate source or type are the other factors that can affect the
8 test results.

9 This test method does not replicate actual conditions that concrete would
10 experience in the field. There is no condition where concrete is exposed to a 60-volt
11 potential. This test method does not measure concrete permeability. What it does
12 measure is concrete resistance to electrical current. Resistance is calculated as volts
13 divided by current. It has been shown that there is a fair correlation between concrete
14 resistivity and concrete permeability.

f) AASHTO T259

The test requires three slabs at least 75 mm thick and having a surface area of 300 mm square. These slabs are moist cured for 14 days then stored in a drying room at 50 percent relative humidity for 28 days. The sides of the slabs are sealed but the bottom and top face are not (Figure 2.21). After the conditioning period, a 3 % NaCl solution is ponded on the top surface for 90 days, while the bottom face is left exposed to the drying environment. At the end of this time the slabs are removed from the drying environment and the chloride concentration of 0.5-inch thick slices is then determined.

Figure 2.21: AASHTO T259 Test Setup

Limitations of this method can be summarized as:

- Little information is being gathered about the chloride profile.
- The salt ponding test does provide a crude one-dimensional chloride ingress profile, but this profile is not just a function of chloride diffusion. Since the

1 specimens have been left to dry for 28 days, there is an initial sorption effect
2 when the slabs are first exposed to the solution.

- 3 • For some higher quality concretes, there has also been difficulty in developing
4 a sufficient chloride profile.
- 5 • Insufficient chloride may penetrate in the 90-day duration for a meaningful
6 profile to develop.

7 g) Bulk Diffusion Test (NordTest NTBuild 443)

8 A bulk diffusion test has been developed to overcome some of the deficiencies of
9 the salt ponding test (AASHTO T257) to measure diffusion. In this method, the test
10 specimen is saturated with limewater. This prevents any initial sorption effects. Also,
11 instead of coating just the sides of the sample and leaving one face exposed to air, the
12 only face left uncovered is the one exposed to a 2.8 M NaCl solution. It is left this way
13 for a minimum of 35 days before evaluation. To evaluate the sample, the chloride profile
14 of the concrete is determined at depth increments on the order of 0.5 mm. The chloride
15 content of the powder is then determined according to AASHTO T260 (Figure 2.22).

Figure 2.22: Schematic of the NordTest Test Setup

While the Nordtest is capable of modeling chloride diffusion into concrete, it is still a long-term test. For low quality concretes, the minimum exposure period is 35 days. For higher quality concretes, however, this period must be extended to 90 days or longer, just as for the salt ponding test.

h) The Rapid Migration Test (CTH Test or NORD492)

This setup (Figure 2.23) is suggested by Tang and Nilsson (1992). A migration cell is set up with a specimen 50 mm thick and 100 mm in diameter, and an applied voltage of 30 V. The experiment proceeds as usual for an electrical migration test, except that the chloride concentration of the downstream solution is not monitored. Instead, after a specified duration (~8 hrs.) the samples are removed and split, and the depth of chloride penetration is determined in one half of the specimen using a 0.1N silver nitrate solution as a colorimetric indicator. The color change border corresponds to the location of a soluble chloride concentration of 0.15 % by weight of cement (Otsuki et al. 1992).

Figure 2.23: Tang and Nilsson Migration Cell (Stanish et al. 1997)

2.3.3.2 TDR (Time-domain Reflectometry) (Cerny 2009)

The TDR method is a dielectric method, based on analysis of the behavior of dielectrics in time-varying electric field and consists in the measurement of permittivity of moist porous media. The determination of moisture content using the permittivity measurements is based on the fact the static relative permittivity of pure water is equal to ~ 80 at $20\text{ }^{\circ}\text{C}$, while for most dry building materials it ranges from 2 to 6. The principle of TDR device consists in launching of electromagnetic waves together with the time intervals between launching the waves and detecting the reflections. A TDR system for field measurement generally includes a TDR device, which consists of pulse generator and sampler, a connection cable, and a measurement probe. The pulse generator sends out pulse (which generally is a fast rising step pulse) and the sampler records the response from the system.

2.4 Summary and Conclusions

A literature review of transport properties affecting durability of concrete and a revision of the test methods available to measure permeability had been performed in this chapter. The main equations that describe the theory of fluid transport were presented. The effects of water to cement ratio, curing, and supplementary materials such as silica fume, slag, and fly ash were also described. It was shown that the effect of sample preparation is a paramount issue when absorption and gas permeability need to be measured. Samples prepared under different conditions will have different relative humidity or different moisture content. This can lead to a misleading test result, perhaps more reflecting sample preparation method than the actual porosity network.

An exhaustive revision of the available test methods to measure permeability was performed. Table 2.5 shows a summary of the techniques described in this chapter.

Table 2.5: Summary of Test Methods Described in this Literature Review

Test method	Lab method	In-situ method	Duration	Simplicity*	Basic concept	Limitations	Commercially available/price
Gas permeability							
Schonlin method	X	X	20 min	2	Vacuum the surface	Sensitive to the moisture condition	No
Surface Air Flow Test (SAF)	X	X	-	2	Vacuum the surface	Sensitive to the moisture condition	No
Autoclam	X	X	30 min	1	Surface pressure	Sensitive to the RH>80%	Yes • Amphora NDT Ltd (www.amphorandt.com)
Torrent Permeability Tester	X	X	20 min	1	Two-chamber vacuum cell	Needs periodic calibration, Sensitive to the moisture condition	Yes • Mastard (www.mastard.com) • Proceq (www.proceq.com)
TUD method (Reinhardt-Mijnsbergen)	X	X	60 min	2	Drilling a hole, introducing Nitrogen gas		No
Hong-Parrott method	X	X	60 min – 1 day	2	Drilling a hole, introducing pressurized air		No
Germann's Gas permeation Test (GGT-5000)	X	X	40 min	2	Drilling a hole, introducing CO ₂ gas	Sensitive to the moisture condition	Yes • Germann Instruments (www.germann.org)-3950 US\$
Cembureau method	X		Few hours	2	constant pressure difference of the test gas between the two surfaces		No

Test method	Lab method	In-situ method	Duration	Simplicity ⁺	Basic concept	Limitations	Commercially available/price
Water permeability							
ISAT (Initial Surface Absorption Test)	X		150 min	1	Water absorption through surface	Cannot be applied to the underside of the concrete. It is difficult to achieve a good seal between the reservoir and concrete surface. It is very sensitive to the moisture content of the surface layer.	Yes
Autoclam	X	X	60 min	1	Water pressure (absorption)		Yes <ul style="list-style-type: none"> Amphora NDT Ltd (www.amphorandt.com)
Germann Water Permeability Test (GWT – 4000)	X	X	40 min	2	Water pressure (absorption)		Yes <ul style="list-style-type: none"> Germann Instruments (www.germann.org)-4250 US\$
Figg-Poroscope Method	X	X	40 min	1	Water pressure (absorption)	It has relatively poor repeatability	Yes <ul style="list-style-type: none"> James Instruments Inc. (www.ndhames.com)-4895US\$ Qualitest (www.worldofest.com)
Field Permeability Test (Florida Test) – FPT	X	X	40 min	2	Pressurized water		No

Test method	Lab method	In-situ method	Duration	Simplicity ⁺	Basic concept	Limitations	Commercially available/price
Ion Migration							
Resistivity - 4 point (Wenner) Method	X	X	Few minutes	2	Applying a current and measuring potential	<ul style="list-style-type: none"> • Sensitive to surface contact • Sensitive to the presence of rebar • Temperature 	Yes
Resistivity – Disk method	X	X	Few minutes	2	Applying a current pulse and measuring the potential response		Yes <ul style="list-style-type: none"> • Germann Instruments (www.germann.org)
Electrical Impedance Spectroscopy (EIS)	X		Few minutes	3	Response to the application of a small amplitude alternating potential signal at different frequencies.		Yes

1

2

Test method	Lab method	In-situ method	Duration	Simplicity*	Basic concept	Limitations	Commercially available/price
ASTM C1202 and AASHTO T277	X		6 hours	2	Ionic migration due to application of 60 volt potential	Factors that may affect the results: the age and curing of the test specimen, air content, Water/Cement ratio, aggregate source or type	
AASHTO T259	X		132 days	2	Ionic diffusion		
Bulk Diffusion Test (NordTest NTBuild 443)	X		77-132 days	3	Ionic diffusion		
The Rapid Migration Test (CTH Test)	X		8 hours	2	Ionic migration		
TDR (Time-domain Reflectometry)	X						

* Easy =1, moderate = 2, difficult = 3

1 CHAPTER 3: TRANSPORT PROPERTIES OF SAMPLES OBTAINED FROM THE
2 STATE OF INDIANA

3 3.1 Introduction

4 This chapter describes the main transport characteristics of the samples collected
5 from the field. Originally four concrete mixtures were considered to be evaluated,
6 however a total of fourteen different concrete were analyzed, representing a range of
7 mixtures proportions that are typical across the state.

8 3.2 Concrete Samples

9 Fourteen different mixtures were collected from the field. Table 3.1 shows a detail
10 of the project selected by the SAC. Table 3.1 includes mixture proportions and the age of
11 the concrete at the time they were received for preparation.

Table 3.1 Characteristics of Concrete Sample Collected from the State of Indiana

Designation	Project	w/c	Cement lb/ft ³	Fly Ash lb/ft ³	S. Fume lb/ft ³	Slag lb/ft ³	Total lb/ft ³	Paste Vol %	Air Content %	Agg	Age of Samples
Concrete #1	E, LaPorte	0.40	539	-	-	-	539	29.5%	NA	Satndard	2 yr. (core)
Concrete #2	F, Seymour	0.39	540	-	-	-	540	29.2%	NA	Satndard	2 yr. (core)
Concrete #3	LaPorte	0.43	590	-	-	-	590	32.8%	NA	Slag	2.5 months
Concrete #4	C, LaPorte	0.39	590	-	-	-	590	31.3%	7.2	Slag	4 yr. (core)
Concrete #5	Rockville Road	0.42	400	110	-	-	510	29.2%	7.0	Satndard	2 mo.
Concrete #6	Airport, P-501	0.39	449	149	-	-	598	31.6%	5.5	Satndard	1.5 yr
Concrete #7	IMI Bridge deck	0.40	533	101	-	-	634	34.0%	NA	Satndard	1.5 yr
Concrete #8	SR 267	0.42	440	70	-	-	510	29.2%	5.5	Satndard	1.5 yr
Concrete #9	D, LaPorte	0.42	440	100	-	-	540	30.6%	NA	Satndard	2 yr. (core)
Concrete #10	IB	0.42	490	125	-	-	615	34.0%	NA	Slag	2.5 months
Concrete #11	A, LaPorte	0.38	480	110	-	-	590	31.2%	6.4	Slag	6 yr. (core)
Concrete #12	IR-29137	0.39	445	125	32	-	602	32.9%	6.5	Satndard	1 month (core)
Concrete #13	IB-29153	0.39	445	125	32	-	602	32.9%	6.5	Satndard	1 month (core)
Concrete #14	B, LaPorte	0.41	440	-	-	120	560	30.2%	6.2	Slag	1 yr. (core)

From Table 3.1 samples can be separated in six groups considering the binder material and the type of aggregate (standard or slag aggregate). These groups are:

- a) Concrete #1 and #2 : Plain cement, with standard aggregate
- b) Concrete #3 and #4 : Plain cement, with slag aggregate
- c) Concrete #5 to #9 : Plain cement plus fly ash, with standard aggregate
- d) Concrete #10 and #11 : Plain cement plus fly ash, with slag aggregate
- e) Concrete #12 and #13 : Plain cement plus silica fume, with standard aggregate
- f) Concrete #14 : Plain cement plus slag cement, with slag aggregate

3.3 Test Methods

Samples were tested under several different tests: volume of air voids, volume of permeable voids, water absorption, diffusivity, moisture absorption-desorption, and

- 1 electrical conductivity. Tests technique and results are shown separately for each test.
- 2 Electrical conductivity results are analyzed separately in Chapter 7.

3 3.3.1 Air Void System

4 The parameters of the air-void system of hardened concrete are related to the
5 susceptibility of the cement paste portion of the concrete to damage by freezing and
6 thawing. ASTM C457 (ASTM 2009) had been commonly used to quantify the air
7 content system. The procedure consists of the determination of the volumetric
8 composition of the concrete by observation of the frequency with which areas of a given
9 component coincide with a regular grid system of points at which stops are made to
10 enable the determinations of composition. The data gathered are the linear distance
11 between stops along the traverse (I), the total number of stops (St), the number of stops in
12 air voids (Sa), the number of stops in paste (Sp), and the number of air voids (N)
13 intersected by the line of traverse over which the component data is gathered. From these
14 data the air content and various parameters of the air-void system are calculated. If only
15 the air content is desired, only Sa and St need be determined.

16 Although ASTM C457 does not currently recognize any automated methods, the
17 field of concrete research has long recognized the fact that it is difficult to find people
18 willing to measure large numbers of air-voids, and thus several automated methods have
19 been developed. The earliest automated procedure was described by Chatterji (1976) and
20 relied on a sample preparation contrast enhancement step to make air-voids appear white
21 and to make aggregates and paste appear black. This principle is still widely used today
22 by and a more recently developed method was used in this work (Peterson 2008).

1 Sample preparation start by lapping the specimen with successively finer
2 abrasives until it is suitable for microscopical observation. After the samples are polished,
3 black and white contrast may be obtained by painting the surface of the polished sample
4 with a wide tipped black permanent marker. Normally three coats are used, changing the
5 orientation 90° between coats. After the ink is dry, the voids are filled with white zinc
6 powder (median size smaller than 2 µm). Powder is worked into the samples using the flat
7 face of a glass slide. A razor blade is used to scrape away excess powder, leaving behind
8 powder pressed into voids. Residual powder is removed by wiping with a clean and
9 lightly oiled fingertip. A fine tipped black marker can be used to darken voids in
10 aggregates (Peterson 2008). Figure 3.1 shows pictures of a sample prepared to be
11 scanned.

Figure 3.1: Prepared Samples for Air Void Count, Automated Method (Sample Dimension: 4x4 inches)

Once the sample is prepared, an image is captured using a high resolution scanner. The first step in the analysis of a digital image is to classify the pixels that represent the air-voids. Most segmentation procedures based on black and white contrast enhancement begin with a choice of threshold. Pixels in the digital image darker than the threshold level are classified as non-air, pixels brighter than the threshold level are classified as air. Many of these procedures employ additional digital processing to further refine the distinction between air and non-air pixels (Peterson 2008). For this study, the air void content will be quantified using an automated procedure.

Figure 3.2a shows the results from air void content, measured using an automated method. Due to difficulties in segmenting voids space in the slag aggregate, only samples containing standard aggregate were considered (total of nine concrete samples). The results show that concrete samples typically used in Indiana had an entrained air volume between 3 % and 5 % of the total concrete volume. It should be noted that has

1 been recommend (Hover 1977) that a minimum of 18% of the cement paste was
 2 entrained air as a method to prevent damage from the freezing and thawing. This
 3 percentage can be calculated from Figure 3.2a dividing the percentage of entrained air by
 4 the cement paste proportion (Figure 3.2b). It can be observed that none of these mixtures
 5 reach the recommended 18 % of entrained air expressed as a function of the paste
 6 volume.

Figure 3.2: Results for Entrained Air

3.3.2 Volume of Permeable Voids, ASTM C-642

ASTM C-642 is the standard method used to calculate density, percent absorption
 and total percent voids in hardened concrete. The method consists of determining the
 total volume of pores comparing the oven dry mass, the saturated mass after immersion,
 the saturated mass after boiling or vacuum, and the immersed apparent mass. These

1 parameters allow calculating the volume of the sample and then the total percentage of
 2 void on the concrete, including gel pores, capillary pores and air entrained pores.

3 Figure 3.3 shows the total air content for the fourteen mixtures. The results show
 4 that standard pavement concrete in Indiana contains a total pore volume in the range of
 5 11.0 % to 14.5 % of the total concrete volume. This plot also shows the influence of the
 6 porosity of the slag aggregate on the test. Sample containing just standard aggregate have
 7 total porosity in the range of 11 % to 12 %.

8
 9 Figure 3.3: Results from Total Volume of Voids, ASTM C-642

3.3.3 Water Absorption of the Concrete, ASTM C-1585

3.3.3.1 Water Absorption Test

The durability of concrete subjected to aggressive environments depends largely on the penetrability of the pore system (ASTM 2004, Sabir et al. 1998, Maltais et al. 2004, Hooton et al. 1993, Parrot 1992, Fagerlund 1996). Three mechanisms can be used to describe transport in cementitious systems: permeability, diffusion and absorption. Permeability is the measure of the flow of water under a pressure gradient, while diffusion is the movement of ions due to a concentration gradient. Absorption can be described as the materials ability to take in water by means of capillary suction. Water absorption is an important factor for quantifying the durability of cementitious systems (Parrot 1992, Hooton et al. 1993, Fagerlund 1996, Bentz et al. 2001, Yang 2006, Henkensiefken et al. 2009) and is the primary focus in this section.

ASTM C1585 (ASTM 2004) is commonly used to determine the absorption and rate of absorption (sorptivity) of water in unsaturated hydraulic cement concretes. This test method, based on work reviewed by Hall (1989), consists of preconditioning samples to a known water content, then exposing the bottom surface of the sample to liquid water and measuring the increase in mass resulting from water absorption. According to the standard conditioning procedure, the samples are conditioned for 18 days. The samples are first placed in a 50 °C and 80 % relative humidity (RH) environment for three days. Then, the samples are removed from the oven and placed in individually sealed containers where they remain for 15 days, to allow internal moisture to spatially equilibrate before the test begins.

1 The absorption test involves recording incremental mass change measurements
 2 during the first six hours after the sample comes in contact with water and subsequently
 3 taking one measurement every day for the next eight days. The amount of absorbed
 4 water is normalized by the cross-section area of the specimen exposed to the fluid using
 5 Equation 3.1:

$$6 \qquad I = \frac{m_t}{(a \cdot d)} \qquad (3.1)$$

7 where: I (mm^3/mm^2) is the normalized absorbed water, m_t (g) is the change in specimen
 8 mass at time t ; a (mm^2) is the area of the specimen exposed to water (i.e., that of the
 9 bottom face), and d (g/mm^3) is the density of the water (taken to be $0.001 \text{ g}/\text{mm}^3$ at 23
 10 °C), and the units provided in the ASTM C1585 standard (ASTM 2004) are employed.

11 These absorption measurements are then plotted as a function of the square root
 12 of time. The initial sorptivity is determined as the slope of the curve during the first six
 13 hours, while secondary sorptivity is determined using the slope of the same
 14 measurements between one and eight days, as outlined in ASTM C1585 (ASTM 2004).

15 The initial and secondary sorptivities can be used to evaluate the connectivity of
 16 the pore network. Additionally, the secondary sorptivity, combined with exposure
 17 conditions, has been used for performing service life predictions (Bentz 2001).

3.3.3.2 Sample Preparation for Water Absorption Test

Concrete samples were collected using cores, cylinder molds and beams from field mixtures. 100 mm diameter cylinders samples were collected when cores and cylinder molds were used. When samples were collected on beams, 100 mm cores were taken from the samples.

After cylindrical samples were obtained, $50 \text{ mm} \pm 2 \text{ mm}$ thick samples were cut from the central portion of cylinders with a wet saw. Nine 100 mm diameter and 50 mm thick samples were prepared for each concrete mixture.

After cutting, concrete and mortar samples were conditioned by placing them in environmental chambers at $23 \pm 0.5 \text{ }^{\circ}\text{C}$. Specimens were placed in environmental chambers at three different relative humidities ($50 \pm 1 \%$, $65 \pm 1 \%$ and $80 \pm 1 \%$) until they reached mass equilibrium, which happened after 10 months.

3.3.3.3 Water Absorption Results

Concrete samples were tested during 8 days according with ASTM C 1585. Results are shown on Figure 3.4 to 3.9.

Figure 3.4: Water Absorption Test, Concretes #1 and #2: Plain Cement with Standard Aggregate

Figure 3.5: Water Absorption Test, Concretes #3 and #4: Plain Cement with Slag Aggregate

Figure 3.6: Water Absorption Test, Concretes #5 to #9: Cement plus Fly Ash with Standard Aggregate

Figure 3.7: Water Absorption Test, Concretes #10 and #11 Cement plus Fly Ash with Slag Aggregate

Figure 3.8: Water Absorption Test, Concretes #12 and #13 Cement plus Fly Ash and Silica Fume with Standard Aggregate

Figure 3.9: Water Absorption Test, Concretes #14, Cement plus Slag with Slag Aggregate

3.3.3.4 Analysis of the Results

The effect of relative humidity on the test results is visible on all tested concretes. The same material conditioned at different relative humidity show different behaviors. The higher the relative humidity (moisture content) the lower the absorption. This is clear example of the importance of the sample preparation for further analysis.

Concrete samples containing supplementary materials show in general a lower absorption compared with similar systems containing plain cement.

Samples containing slag aggregate tends to absorb more water than concrete samples containing just normal aggregate. This may be explained by the high porosity of the slag aggregate. However, this higher absorption is not an indicator by itself about the overall quality of the concrete.

Concrete #1 and #2 have similar mixture proportions and are similar age. However after visual inspection, concrete #2 presents a considerable amount of pores,

evidence of lack of consolidation. This higher porosity of the paste generates a very high absorption value when it is compared with concrete #1. This is clear evidence of the importance of a good consolidation.

Concrete #3 and #4 have similar amount of paste and aggregates and both contain slag aggregate in similar proportion, but having a different w/c . Concrete #3 has a $w/c = 0.43$ and #4 has a $w/c = 0.39$. The result shows clearly the effect of a higher w/c on the water absorption at any of the different relative humidities.

Concretes #5 to #9 represent a widely used mixture proportion for the State of Indiana constituted of binder composed of cement and fly ash. The difference in w/c (in the range of 0.38 to 0.42), the range of cement paste volume (29 % to 34 %), and the different level of fly ash replacement make difficult to compare these results to other mixtures. However, in general results are very similar and comparable with water absorption of concrete containing other supplementary material analyzed in this research.

A special note is needed for Concrete # 8. Visual analysis of this samples shows a very porous structure, what be attributed mainly to a lack of consolidation. This concrete was tested knowing this condition, and the very high water absorption result is consistent with the observed high porosity.

Concretes #10 and #11 contain similar amounts of cement and fly ash, but concrete #10 has a higher w/c (0.42 vs. 0.38), a higher amount of cement paste (34.0% vs. 31.2%), and it is younger than concrete #11 (2.5 months vs. 6 years). As result, concrete #10 presents a higher water absorption then concrete #11.

Concretes #12 and #13 correspond to two different project having the same mixture proportion: $w/c = 0.39$, 445 lb/ft³ of cement, 125 lb/ft³ of fly ash and 32 lb/ft³ of

silica fume, and a paste volume of 32.9 %, and they were pavement with one day of difference. Water absorption test results show very similar results between the projects. This shows a high repeatability of the test under similar conditions.

Concrete # 14 containing slag cement and slag aggregate presents a water absorption comparable with the ones containing fly ash and slag aggregate.

3.3.4 Moisture Diffusivity

The present section provides a method to calculate diffusivity, the coefficient of moisture diffusion, from weight loss of a sample exposed to controlled conditions (i.e., relative humidity (RH) and temperature). The diffusivity is calculated for a cement paste sample with w/c of 0.5. The same method can be used to obtain the diffusivity of other cement based materials.

3.3.4.1 Desorption Measurements on Cement Paste

The desorption response was measured for a cement paste sample with 5 mm diameter and 1 mm thickness. The sample was kept seal for 24 hours after casting. After demolding the sample was immediately placed in a tared 180 mL quartz pan. The pan containing the sample was then suspended from the balance (± 0.001 mg accuracy) and placed in the relative humidity chamber to equilibrate at 23.0 ± 0.1 °C and 97.5 ± 0.1 % RH for up to 48 h or until the sample had achieved a stable mass (less than an 0.001 % mass change/15 minutes). After the mass of the sample equilibrated (the mass change was less than 0.001 % mass change/15 minutes) the relative humidity was reduced in 5 %

1 RH steps to 2.5 % RH. The samples were allowed to equilibrate at each humidity. After
 2 equilibrating at 2.5 % RH the samples were dried to 0 % RH.

3 Figure 3.10 illustrates the normalized weight loss of the cement paste sample
 4 exposed to the full range of relative humidity (RH). Each step presents the weight loss of
 5 the sample at a constant RH which is also plotted on in the graph. All the steps are at
 6 constant temperature of 23 °C. The insert in Figure 3.10 is a closer representation of one
 7 of the steps.

8
 9 Figure 3.10: Normalized Mass Loss of Cement Paste Sample at Different RH

10 The moisture diffusivity of cement based materials is a function of RH, however,
 11 at each step during this experiment the moisture diffusivity can be considered constant
 12 since the change in RH value is small. The change in mass of sample, M_t , as a function of
 13 time, t , can be represented using Equation 3.3 (Crank 1980).

$$\frac{M_t}{M_{\infty}} = \frac{4}{\pi} \left(\frac{D t}{L^2} \right)^{1/2} \quad (3.3)$$

where M_{∞} is the mass change at equilibrium, D is the diffusivity and L is the thickness of the sample. Equation (3.3) can be used to obtain the D at each step by calculating the slope of a curve that is obtained by plotting $\frac{M_t}{M_{\infty}}$ versus square root of time.

Figure 3.11 illustrates the calculated diffusivity of the cement using Equation (3.3). Diffusivity increases rapidly with RH. The obtained values for diffusivity are in agreement with the reported values in literature (Bazant and Najjar 1972, Bazant 1986, Anderberg and Wadso 2008).

Figure 3.11: Diffusivity of Cement Paste Sample as a Function of RH

3.3.5 Moisture Desorption

Three different mortar mixtures were prepared for this study. All they were mortars with a single volume fraction of fine aggregate (55 % of the total volume) and different w/c (0.30, 0.40 and 0.50).

An ASTM C150 Type I ordinary portland cement (OPC) was used in this study, with a Blaine fineness of 370 m²/kg and an estimated Bogue composition of 56 % C₃S, 16 % C₂S, 12 % C₃A, 7 % C₄AF and a Na₂O equivalent of 0.68 % by mass.

Two 100 mm × 200 mm cylinders were cast for each mortar mixture. After one day of curing, the samples were demolded and then sealed in double plastic bags for sealed curing. Bags were stored in a room at 23 ± 1 °C until samples reached an age of 28 d. After that, cylinders were removed from bags and eighteen 10 mm ± 2 mm thick samples were cut from the central portion of each cylinder with a wet saw. After cutting, mortar samples were placed in environmental chambers at 23 ± 0.5 °C and 50 ± 1 % for 18 months.

Samples were then vacuum saturated for 24 h. After that, specimens were placed in environmental chambers at six different relative humidities (93 ± 1 %, 87 ± 1 %, 75 ± 1 %, 65 ± 1 % and 50 ± 1 %). Weight was monitored at regular intervals until it reach equilibrium. At the end, all samples were oven dried to express water absorption in term of the dry mass the sample. Figure 3.12 shows the desorption behavior of these mortar samples.

Figure 3.12: Desorption Curve on 18 Months Old Mortar Samples

3.4 Summary and Conclusions

This section has described the absorption behavior of concretes conditioned at different relative humidities. A summary of the general conclusions from the data presented in this chapter are:

- A series of concrete paving mixtures were tested to provide a range of values that were typical for the state of Indiana.
- While similar mixture proportions were used for the mixtures in Indiana differences in the magnitude of water absorbed occurred.
- The standard absorption test ASTM C-1585 is considerably affected by the relative humidity of the samples before starting the test. Comparing samples

- 1 conditioned at different relative humidities can lead to a misunderstanding of
- 2 the actual absorption behavior.
- 3

CHAPTER 4: EFFECT OF SAMPLE CONDITIONING ON THE WATER ABSORPTION OF CONCRETE

4.1 Overview

ASTM C1585 is commonly used to determine the absorption and rate of absorption of water in unsaturated hydraulic cement concretes. ASTM C1585 preconditions the samples for a total of 18 days. Unfortunately however, the range of relative humidities that can exist in the samples after this relatively short conditioning period may provide a wide enough variation to considerably influence the results of the test. Three main variables were studied in this program to assess the effect of preconditioning. First, the role of water to cement ratio was investigated by testing mortar samples with 55 % aggregate by volume with four different water-to-cement ratios (w/c of 0.35, 0.40, 0.45 and 0.50). Second, the role of paste volume was investigated by considering samples with 55 %, 45 %, and 35 % aggregate by volume with a $w/c = 0.50$. Finally, the effect of conditioning was assessed by exposing all the samples in three different relative humidities (50 %, 65 % and 80 %) until they reached mass equilibrium (defined as constant mass over 15 days), taking approximately 14 months. Oven dry samples were also prepared and tested for comparison. The results confirm that water absorption testing is considerably influenced by sample preparation. Samples conditioned at 50 % relative humidity can show up to six times greater total

absorption than similar samples conditioned at 80 % relative humidity. Samples that were conditioned in the oven at 105 °C do not appear to follow a similar trend when compared with specimens conditioned in chambers for the longer duration. The absorption is also influenced by the volume of paste in the samples. The experiments show that a lack of control on moisture content or lack of consideration of the material composition can lead to a misunderstanding of the actual absorption behavior.

4.2 Introduction

The durability of concrete subjected to aggressive environments depends largely on the transport properties which are influenced by the pore system (Parrot 1992, Hooton et al. 1993, Fagerlund 1996, Sabir et al. 1998, ASTM 2004, Maltais et al. 2004). Three main mechanisms can be used to describe transport in cementitious systems: permeability, diffusion and absorption. Permeability is the measure of the flow of water under a pressure gradient, while diffusion is the movement of ions due to a concentration gradient. Absorption can be described as the materials ability to take in water by means of capillary suction. All three mechanisms are heavily influenced by the volume of pores as well as the connectivity of the pore network. A large fraction of concrete in service is only partly saturated and the initial ingress of water and dissolved salts is influenced, at least in part, by capillary absorption (Hearn et al., 1994). As such, water absorption has been used as an important factor for quantifying the durability of cementitious systems (Parrot 1992; Hooton et al. 1993; Hearn et al. 1994; Fagerlund 1996; Bentz et al. 2001; Yang 2004; Henkensiefken et al. 2009). Water absorption is the primary focus of this study since it is being increasingly used by specifiers and in forensic studies to provide a

parameter that can describe an aspect of concrete durability. It is also important that these properties be adequately described for use in service life models (Fagerlund 1996; Bentz 2001).

4.2.1 Water Absorption Test

ASTM C1585 (2004) is commonly used to determine the absorption and rate of absorption (commonly referred to as sorptivity) of water in unsaturated hydraulic cement concretes. This test method, based on work reviewed by Hall (1989), consists of preconditioning samples to a known moisture content, then exposing the bottom surface of the sample to liquid water and measuring the increase in mass resulting from water absorption. According to the standard conditioning procedure, samples are conditioned for 18 days. This conditioning period begins by first placing the sample in a 50°C and 80 % relative humidity (RH) environment for three days. The samples are then removed from this environment and placed in individually sealed containers where they remain for 15 days at 23 °C, to allow internal moisture to redistribute throughout the specimens before the test begins.

The absorption test involves recording incremental mass change measurements at relatively frequent intervals during the first six hours after the sample comes in contact with water and subsequently taking one measurement every day for the next eight days. The amount of absorbed water is normalized by the cross-section area of the specimen exposed to the fluid using Equation 4.1:

$$i = \frac{m_t}{(a \cdot \rho)} \quad (4.1)$$

where: i (mm^3/mm^2) is the normalized absorbed fluid, m_t (g) is the change in specimen mass at time t ; a (mm^2) is the area of the specimen exposed to the fluid (i.e., that of the bottom face), and ρ (g/mm^3) is the density of the absorbed fluid (taken to be $1000 \text{ kg}/\text{m}^3$ at 23°C).

These absorption measurements are then plotted as a function of the square root of time. The initial sorptivity is determined as the slope of the curve during the first six hours, while secondary sorptivity is determined using the slope of the same measurements between one and eight days, as outlined in ASTM C1585 (2004). It should be noted that these times work well for water though they may not work as well for other fluids with different surface tension and/or viscosity (Spragg et al. 2010).

The initial and secondary sorptivities can be used to evaluate the connectivity of the pore network. Additionally, the secondary sorptivity, combined with exposure conditions, has been used for performing service life predictions (Bentz et al. 2001).

4.2.2 The Role of the Relative Humidity

Water ingress in unsaturated concrete is dominated by capillary suction upon initial contact with water (Martys and Ferraris 1997, Martys 1999, Lockington et al. 1999; Hall 2007, Yang et al. 2007). Capillary absorption can be related to the volume of the pores as well as the size (i.e. radius) of the partially empty capillary pores (Figure

1 4.1). The relation between the equilibrated relative humidity and the radius of the
 2 smallest empty pore is given by the Kelvin-Laplace equation (Equation 4.2).

$$3 \quad \ln(RH) = \frac{2 \sigma V_m}{r_m R T} \quad (4.2)$$

4 where: RH is the relative humidity, σ is the surface tension of water (pore solution),
 5 $[N/m]$, V_m is the molar volume of water, $[m^3/mol]$, r_m is the average radius of curvature
 6 $[m]$, R is the general gas constant, and T is the absolute temperature, $[^{\circ}K]$.

7 Figure 4.1: Relation Between Relative Humidity and Partially Empty Pores in Cement
 8 Paste

9 It should be noted that this expression is simplified as it does not consider the
 10 effect of water that is absorbed on the walls of the pores. Largely the concrete community
 11 has considered two sizes of pores as introduced by Powers (1946). The gel pores are

1 considered to be small pores (< 10 nm diameter) that are a part of the hydration products.
2 Capillary pores are larger pores that occur due to excess water. Capillary porosity is
3 particularly of concern in transport, as is the interconnectivity of the capillary pores.

4 Figure 4.1 shows a conceptual illustration after Powers (1946) which used a
5 desorption isotherm to illustrate the volume of water located in the different size pores at
6 different relative humidities.

7 The relative humidity used to condition the sample prior to the sorption test can
8 have a significant impact on the results (ASTM 2004). Previous test results by Parrot
9 (1991, 1994) indicated that the water absorption rate was very sensitive to the moisture
10 content of the concrete, particularly at relative humidities above 60 % which were
11 common for field exposure. Water leaves the largest accessible pores first. It can be seen
12 from Figure 4.1 that capillary pores occupy the range of humidity from approximately 80
13 % to 100 % RH. As such, initially upon drying water leaves the capillary pores. The
14 lower the relative humidity, the greater the total volume of pores that are empty and
15 available to be filled with water during the sorption test. Further, the lower humidity will
16 empty smaller pores and create a higher suction force in the materials resulting in a
17 greater sorption rate and a larger overall total absorption.

18 According to ASTM C1585, the standardized test conditioning will generally
19 provide an internal relative humidity similar to relative humidities found near the surface
20 in some field concrete structures (DeSouza et al. 1997, DeSouza et al. 1998, ASTM
21 2004). This range of relative humidities can represent what is found in samples in the
22 field; however, it is wide enough to affect considerably the results of the test.

Results presented in Chapter 8 shows that the relative humidity of samples that were kept in the field under different exposure conditions was in the range of 80 % to 100 % depending on the type of exposure, which is somewhat higher than what is mentioned in ASTM C-1585.

4.2.3 Chapter Objectives

The objectives of this research are threefold. First, this research will examine the influence of conditioning relative humidity (oven dry, 50 %, 65 % and 80 % RH) on the results of sorption tests performed on mortars with different w/c , containing a fixed volume of aggregate. Second, this research will examine the influence of the volume of aggregate (or equivalently the paste content) on the results of sorption testing. Third, this research will examine the effect of the conditioning method proposed in ASTM C1585-04.

4.3 Materials

An ASTM C150 Type I ordinary portland cement (OPC) was used in this study, with a Blaine fineness of $370 \text{ m}^2/\text{kg}$ and an estimated Bogue composition of 56 % C_3S , 16 % C_2S , 12 % C_3A , 7 % C_4AF and a Na_2O equivalent of 0.68 % by mass.

A polycarboxylate-based high-range water-reducing admixture (HRWRA) was added in varying rates as indicated in Table 4.1 depending on the mixture proportions to maintain similar consistencies (i.e., workability). The sand used was natural river sand

with a fineness modulus of 2.71, an apparent specific gravity of 2.58, and a water absorption of 1.8 % by mass.

4.3.1 Mixture Proportioning

Six different mixtures were prepared in total. Four mixtures were mortars with a single volume fraction of fine aggregate (55 % of the total volume) and different w/c (0.35, 0.40, 0.45, and 0.50). These mixtures were designated as 55/0.35, 55/0.40, 55/0.45 and 55/0.50 with the number on the left representing the volume fraction of fine aggregate and the number on the right representing w/c . Additionally, two other mortars were prepared with w/c of 0.50, but with different volume fractions of fine aggregate (35 % and 45 % of the total volume). They were designated as 35/0.50, 45/0.50. A list of the mixture proportions can be found in Table 4.1.

Table 4.1: Mixture Proportions in Saturated Surface Dry (SSD) Conditions

Material	55/0.35	55/0.40	55/0.45	55/0.50	45/0.50	35/0.50
Volume fraction of aggregate	55%	55%	55%	55%	45%	35%
w/c	0.35	0.40	0.45	0.50	0.50	0.50
Cement (kg/m^3)	673	626	585	549	671	793
Water (kg/m^3)	235	250	263	275	336	397
Fine Aggregate (kg/m^3), SSD	1442	1442	1442	1442	1180	918
HRWRA (g/ 100 g cement)	0.60	0.40	0.20	0.00	0.00	0.00

4.3.2 Mixing Procedure

The mixing procedure used for the mortar was in accordance with ASTM C192-06 [2006]. The aggregate was oven dried and cooled for 24 h before mixing. The volume

of water was corrected by the absorption of the aggregate. The water and cement were conditioned for 24 h at room temperature prior to mixing.

4.4 Experimental Method

Six 100 mm × 200 mm cylinders were cast for each mixture. After one day of curing, the samples were demolded and then sealed in double plastic bags for sealed curing. Bags were stored in a room at 23 ± 1 °C until samples reached an age of 28 d. After that, cylinders were removed from bags and three 50 mm ± 2 mm thick samples were cut from the central portion of each cylinder with a wet saw.

After cutting, samples were conditioned by placing them in environmental chambers at 23 ± 0.5 °C. Specimens from mixtures 55/0.35, 55/0.40, 55/0.45 and 55/0.50 were placed in environmental chambers at three different relative humidities (50 ± 1 %, 65 ± 1 % and 80 ± 1 %). Specimens from mixtures 35/0.50 and 45/0.50 were placed in an environmental chamber at 50 ± 1 % relative humidity. Samples were kept in the environmental chamber until they reached mass equilibrium, defined as negligible mass change over a 15 day period. Mixture 55/0.35 placed at 50 ± 1 % relative humidity required the longest period of time (14 months) to reach mass equilibrium. However, all samples were maintained in the chambers for 14 months to test them all at the same age.

Additional specimens from mixtures 55/0.35, 55/0.40, 55/0.45 and 55/0.50 were placed at 50 ± 1 % RH. After the 14 months, these specimens were dried in an oven at 105 ± 2 °C until they reached mass equilibrium.

Once the samples were removed from the chambers or from the oven, the side surface (i.e. outer circumference) was sealed with epoxy and the top surface was covered

with plastic to avoid evaporation from the sample during testing. After the samples were prepared, testing occurred in accordance with ASTM C1585-04 (2004). Specimens from mixtures 55/0.35, 55/0.40, 55/0.45 and 55/0.50 were tested over a period of 90 days. Specimens from mixtures 35/0.50 and 45/0.50 were tested over a period of 8 days.

Two additional 100 mm × 200 mm cylinders were cast for each mortar mixture. After one day of curing, the samples were demolded and then sealed in double plastic bags for sealed curing. Bags were stored in a room at 23 ± 1 °C until samples reached an age of 28 d. After that, cylinders were removed from bags and 10 mm ± 2 mm thick samples were cut from the central portion of each cylinder with a wet saw. After cutting, mortar samples were vacuum saturated for 24 h. After that, specimens were placed in environmental chambers at six different relative humidities (93 ± 1 %, 87 ± 1 %, 80 ± 1 %, 75 ± 1 %, 65 ± 1 % and 50 ± 1 %) to determine their desorption isotherms.

4.5 Experimental Results and Discussion

4.5.1 Desorption Isotherms

Figure 4.2 shows the desorption isotherm curves measured using 10 mm thick samples. Mass change was monitored at regular intervals until it reached equilibrium, defined as no mass change over a 15 day period. At the end, all samples were oven dried to express water absorption in terms of the dry mass of the sample.

It can be noticed that while the values of the moisture content are similar at 50 % and lower RH, as it refers to the small gel pore system (Powers et al. 1946), the capillary pores at high RH are strongly influenced by the w/c .

Figure 4.2: Desorption Curve on 18 Months Old Mortar Samples (Typical Standard Deviation in the Average of 3 Samples is Lower Than 0.2 %)

4.5.2 Effect of Initial Conditioning on Water Absorption Tests

4.5.2.1 Effects of Relative Humidity on Sorption Test

Figure 4.3 shows the absorbed water during the 90 days of testing performed on mortars conditioned at different relative humidities (mixtures 55/0.35, 55/0.40, 55/0.45 and 55/0.50). It can be noticed that the water absorption is very sensitive to the relative humidity at which the specimens were pre-conditioned before testing. In each case, as the conditioning relative humidity increases, the absorption decreases.

Figure 4.3: Absorbed Water in Mortars as a Function of Relative Humidity: a) Mixture 55/0.35, b) Mixture 55/0.40, c) Mixture 55/0.45, d) Mixture 55/0.50. Error Bars Represent the Standard Deviation for the Average of Three Samples.

These results can be viewed in a slightly different manner if they include the initial amount of water held in the pores before the test. In order to do this, samples were oven dried at the end of the sorption test to calculate the amount of water they held before starting the test. Additional specimens that were kept at $50 \pm 1\%$ RH during the 14

1 months were oven dried and then saturated by the procedure described in ASTM C642-
2 06 to measure the total amount of interconnected porosity in the systems. Results from
3 Figure 4.3 were then normalized by the total amount of pores in the system, which can be
4 viewed as the degree of saturation of the sample as a function of time. This is presented
5 in Figure 4.4. Figure 4.5 shows the total degree of saturation for the samples after 90
6 days.

7 Figures 4.4 and 4.5 show that samples prepared at different relative humidities
8 with a low w/c (e.g. $w/c = 0.35$) do not reach values near to saturation even after 90 days
9 of being in contact with water. It may be attributed to the refined pore network of this low
10 w/c system which makes it difficult for water to move through the sample to fill all the
11 pores. This is commonly referred to as depercolation, which occurs after different
12 hydration times for different w/c (Powers et al. 1959).

13 In contrast after 90 days, samples prepared with a higher w/c (e.g. $w/c = 0.50$)
14 reach much higher levels of saturation. It can be noted from Figure 4.4 that samples
15 conditioned at 50 % RH reach values near saturation after about 40 days of testing,
16 similar to what is obtained with oven dry samples. Again this may be attributed to the
17 connectivity of the pore network and the size of these pores. In this case, a more
18 interconnected pore network will facilitate the movement of water to the interior of the
19 specimens and the diffusion of water vapor out of the sample. However, when these
20 samples were conditioned at higher relative humidities (65 and 80 % RH), the amount of
21 initially retained water is high enough to reduce the diffusion of vapor out of the sample.
22 As a result, this may explain why the level of saturation of these specimens is lower.

Figure 4.4: Degree of Saturation as a Function of Time During the Water Absorption Test: a) Mixture 55/0.35, b) Mixture 55/0.40, c) Mixture 55/0.45, d) Mixture 55/0.50. Typical Standard Deviation of the Average of Three Samples is Lower than 0.02 Points in the Degree of Saturation.

Figure 4.5: Degree of Saturation at 90 Days in Contact with Water as a Function of the w/c .

4.5.3 Effects of Relative Humidity on the Amount of Absorbed Water after 8 Days

Figure 4.6 shows the cumulative water that was absorbed after 8 days of testing performed on mortars conditioned at different relative humidities, expressed as a function of w/c (Figure 4-6a) and as a function of the relative humidity (Figure 4.6b).

Figure 4.6a shows that mixture 55/0.50 can exhibit six times higher absorption when the samples are conditioned at 50 % RH compared with similar samples conditioned at 80 % RH.

Figure 4.6: Cumulative Absorption at 8 Days for Mortars Versus: a) w/c , b) Relative Humidity. Solid Lines are Provided only to Show a General Tendency in the Data. Error Bars Represent the Standard Deviation on the Average of 3 Samples.

Figure 4.7: Cumulative Absorption at 8 Days Versus w/c and Relative Humidity: a) Normalized to Absorption of Mixture 55/0.35, b) Normalized to Absorption at 50 %RH.

Figure 4.7 shows a normalization of the data presented in Figure 4.6. In Figure 4.7a the normalization is made with respect to the absorption of samples with $w/c = 0.35$ (mixture 55/0.35). In Figure 4.7b the normalization is made with respect to the absorption

1 of samples conditioned at 50 % relative humidity. It can be seen that the values follow a
2 consistent trend in each case, except for the oven dry samples.

3 4.5.4 Effects of Relative Humidity on Initial Sorptivity

4 Figure 4.8 shows the initial sorptivity calculated as the slope of the absorption vs.
5 the square root of time during the first six hours of test (ASTM 2004).

6 Figure 4.8a shows that mixture 55/0.50 can exhibit a ten times higher initial
7 absorption when the samples are conditioned at 50 % RH compared with similar samples
8 conditioned at 80 % RH.

9 It needs to be noted that the oven dry samples show a much higher initial
10 sorptivity, due to the fact that the gel's capillary pores are empty and possibly to
11 microcracking. While the increase in sorptivity is observed to be linear for the specimens
12 conditioned at 50 %, 65 % and 80 % relative humidity, this trend appears to break down
13 for the oven dry samples which may be attributed to micro-cracking generated during the
14 sample preparation (Hwang and Young 1984; Chatterji 1976; Bisschop 2002; Samaha
15 and Hoover 1992; Yang et al. 2006).

Figure 4.8: Initial Absorption on Mortars Conditioned at Different RH Function of: a) w/c , b) Relative Humidity. Solid Lines are Provided to Show a General Tendency in the Data. Error Bars Represent the Standard Deviation on the Average of 3 Samples.

4.5.5 Effects of Relative Humidity on Secondary Sorptivity

Figure 4.9 shows the secondary sorptivity calculated as the slope of the absorption vs. the square root of time between 1 d and 8 d of testing. Trends are similar to those observed for the initial sorptivity. However, it needs to be noted that samples that were oven dry prior to the test present a considerably lower secondary absorption with respect to the samples conditioned in environmental chambers. This may be explained by the high initial absorption of the oven dry samples shown in Figure 4-8. During this initial absorption it can be noticed that since a majority of the water was already absorbed in the first hours of the test, the secondary rate of absorption will be much lower. It can also be expected that microcracking enabled a more rapid ingress of water (Yang, 2004).

Figure 4.9: Secondary Absorption on Mortars Conditioned at Different RH Function of: a) w/c , b) Relative humidity. Solid Lines are Provided to Show a General Tendency in the Data. Error Bars Represent the Standard Deviation on the Average of 3 Samples.

Figure 4.9 shows a similar trend to what was noted in the case of total absorption and initial sorptivity, namely that the secondary sorptivity of samples conditioned in chambers exhibits a consistent trend when the results are plotted against the w/c or the relative humidity at which samples were conditioned. However, samples that are conditioned by drying them in an oven at 105°C do not follow the same tendency.

4.6 Effects of Initial Moisture of Samples on ASTM C1585 Conditioning Method

At the age of 24 months, samples from each mixture conditioned at the three different relative humidities were removed from the chambers. The side surface was sealed with epoxy to be then “re-conditioned” using the 18 day procedure described in ASTM C-1585. In addition, three other samples from each mixture were saturated following the procedure described in ASTM C642 (2006), to then be “re-conditioned”

1 following the same 18 day procedure. After samples were fully prepared, testing was
2 performed in accordance with ASTM C-1585 over a period of 8 days, with results
3 provided in Figure 4.10. In addition, Figure 4.11 shows the calculated initial and
4 secondary sorptivities from these tests. Secondary sorptivity values are not reported when
5 the correlation coefficient is lower than 0.98.

6 Figure 4.10 and 4.11 show that the 3 days of controlled drying at 50 ± 2 °C and 80
7 % RH followed by the 15 days for internal moisture equilibration is not capable of
8 eliminating the effects of the “moisture history”. These results suggest that the ASTM
9 C1585 preparation method does not prepare all the samples to the same water content
10 before a water absorption test. As such this accelerated method can make a substantial
11 difference in how the data is interpreted. This may be due to moisture hysteresis effect
12 (Nilson 1980). It should be noted that this can be a concern for field samples evaluated
13 using this method, as their as-received relative humidities may easily vary between the
14 extremes examined in this study.

15 4.7 Effects of Volume of Aggregate on Sorption Test

16 Figure 4.12 shows the absorbed water during 8 days of testing performed on
17 mortars containing different volumes of aggregate (mixtures 55/0.50, 45/0.50 and
18 35/0.50) conditioned at 50 % relative humidity. In Figure 4.12a the effect of a higher
19 volume of paste is observed as the mixture containing the lower volume of aggregate has
20 the higher absorption. However, when the results are normalized by the volume of paste
21 (volume of the main absorbent material), a reversal in the order of the samples is

- 1 observed (Figure 4.12b). The samples with the higher volume of aggregates have a higher
 2 absorption.

5 Figure 4.10: Effect of Initial Moisture on the Conditioned Procedure Established in
 6 ASTM C1585-04: a) Mixture 55/0.35, b) Mixture 55/0.40, c) Mixture 55/0.45, d) Mixture
 7 55/0.50. Error Bars Represent the Standard Deviation for the Average of Three Samples.

Figure 4.11: Initial and Secondary Sorptivity on Mortars with Different Initial Moisture Content, Conditioned with the Procedure Established in ASTM C1585-04.

Figure 4.12: Water Absorption in Mortars Containing Different Volume of Aggregates: a) Normalized by Surface in Contact with Water, (b) Normalized by Volume of Paste.

Commonly water absorption is reported without considering the effect of the absorption of the aggregate in the samples. To better understand its effect, Figure 4.13 was calculated assuming five different sand absorptions (0.0 %, 0.6 %, 1.2 %, 1.8 %, and

2.4 %) to then subtract these values from the absorption in Figure 4.12b. When the sand absorption is assumed to be 0.0 %, the resulting absorption at 8 days will be the same as the absorption presented in Figure 4.12b. From Figure 4.13, it can be noticed that for the assumed 1.8 % sand absorption, the normalized water absorbed for the sample is the same after 8 days, independent of the amount of aggregate in the sample.

Figure 4.14 shows a desorption isotherm for the sand used in these mixtures. It can be noted that at 50 % RH (humidity at which the samples were conditioned), the amount of water on the sand is about 0.2%. Considering that the aggregate used in this study has a 24 h absorption of 1.8%, the difference on water absorption of samples containing different amounts of aggregate can be explained mainly by the amount of water absorbed by the aggregates.

Figure 4.13: Water Absorption at 8 Days Normalized by Volume of Paste, Corrected by Different Values of Aggregate Absorption.

Figure 4.14: Desorption Isotherm for the Sand used in this Study.

4.8 Summary and Conclusions

This paper has described the absorption behavior of mortars conditioned at different relative humidities. A summary of the general conclusions from the data presented in this paper are:

- As was shown in previous works by Hall (1989), Hooton et al. (1993) and Martys and Ferraris (1997), the water absorption test is considerably affected by the relative humidity of the samples before starting the test, which if not properly accounted for can lead to a misunderstanding of the actual absorption behavior.
- Samples conditioned at 50 % relative humidity can show a total absorption that is approximately six times greater than similar samples conditioned at 80 % relative humidity. This is consistent with what can be expected from the desorption curves.

- 1 • Initial sorptivity, secondary sorptivity and total absorption at 8 days for samples
2 conditioned in chambers show a linear trend related to the w/c and the relative
3 humidity at which samples were conditioned.
- 4 • Samples that are conditioned by drying in an oven at 105 °C do not follow the
5 same trend as samples conditioned in other approaches. This is attributed to two
6 factors: 1) emptying of a wider range of pores, and 2) the potential for
7 microcracking.
- 8 • The conditioning procedure described in ASTM C1585-04 is not able to eliminate
9 the “moisture history” of the samples, and thus can lead to a misunderstanding of
10 the water absorption test results, especially in field samples which have obtained a
11 lower relative humidity.
- 12 • Comparing samples containing different volumes of aggregate can also lead to a
13 misunderstanding of the actual absorption behavior. Samples containing higher
14 volumes of cement paste will absorb more water. When the results are normalized
15 by the volume of cement paste, the sample containing lower volumes of cement
16 paste will absorb more water. However, this difference can be mainly explained
17 by the amount of water absorbed by the aggregates in the sample.

18

1 CHAPTER 5: WETTING AND DRYING OF CONCRETE IN THE PRESENCE OF
2 DEICING SALT SOLUTIONS

3 5.1 Overview

4 A series of wetting and drying tests were performed on concrete using different
5 aqueous solutions containing deicing salts. The rate of absorption was generally lower for
6 aqueous solutions containing deicing salts. In addition, less fluid was absorbed for
7 aqueous solutions containing deicing salts. The change in the rate of aqueous fluid
8 absorption was proportional to the square root of the ratio of surface tension and viscosity
9 of the absorbed fluid. Concrete that has been exposed to solutions containing deicer salts
10 show less mass loss during drying. Measures of equilibrium relative humidity over the
11 salt solutions are used to interpret drying behavior. Experimental data indicates that
12 concretes that have previously been exposed to deicing solutions can also exhibit reduced
13 rate of absorption, even if water is used as the fluid being absorbed.

14 5.2 Introduction

15 Some jointed plain portland cement concrete pavements in freezing prone
16 climates have shown premature deterioration at the longitudinal and transverse joints.
17 While some have attributed this damage to a chemical attack, inadequate air entrainment,
18 poor mixture design, inadequate constituent materials, or poor construction practices; it is

the hypothesis of the authors of this paper that this joint deterioration may be attributed, at least in part, to preferential absorption of fluid at joints. This hypothesis was developed based on observations from the field that show these deteriorated locations frequently occurred at low spots in the pavement, where joint sealers were damaged, where water has collected, or where the joint does not appear to have opened thereby trapping water (Weiss and Nantung 2006). Preferential fluid ingress at joints could increase a variety of damage mechanisms including deleterious chemical reactions, crystallization pressure, or freeze thaw damage that may degrade the concrete. To fully evaluate fluid ingress at the joints it is essential that the wetting and drying behavior of concrete is evaluated using aqueous solutions containing deicing salts.

This work is limited in scope as it considers only the ingress of aqueous solutions over short time periods and does not explicitly consider any chemical reaction that occurs between the aqueous solution and the concrete or any long-term effects. This information is intended to provide reference for those developing tests to evaluate potential deicer-concrete interactions (Shi et al. 2010), for developing tests on fluid absorption, for evaluating fluid absorption in concrete (Hong and Hooton 1999), for input parameters in computer simulation of fluid ingress at joints (Pour-Ghaz et al. 2009), and for potential approaches to limit joint deterioration like penetrating sealers for possible use in concrete pavements (Coates et al. 2009).

5.3 Fluid Absorption in Porous Materials

Fluid absorption is a frequently used test to provide an indication of the durability of concrete systems since it is simple to perform. Several standard tests exist for

measuring water absorption including ASTM C 1585-04 (ASTM 2004), BS 1881-99 (British Standard Institute 1990), and ASTM D6489-99 (ASTM 2006). While the concept behind these tests is very similar, there are differences in how the samples are conditioned, treated, and tested. In each of these tests water is typically used as the fluid that is being absorbed. Hall (1997) discusses that water can interact with the cement matrix adding complexity to the interpretation of results. To overcome some of these limitations or to indicate how absorption can be reduced by fluid composition other solutions have been tested (Hall et al. 1997, Hall and Hoff 2002, Weiss 1999, Bentz et al. 2009, Sant et al. 2010).

MacInnis and Nathawad (1980) assessed the absorption of an aqueous solution consisting of a NaCl deicing salt and reported a decrease in absorption. Sutter et al. (2008) reported that sorptivity decreased from highest to lowest in the order of water, NaCl, CaCl_2 and MgCl_2 . Similar data has recently been observed by Janusz (2010). As a result, it can be observed that concrete exposed to deicing salt solutions absorb fluid at a slower rate than they would absorb water; however the previous work has not related this behavior to the fluid properties or described the influence of salt concentration or properties of the aqueous solution.

The results of one-dimensional fluid absorption tests (assuming negligible gravitational effects) are typically reported as the cumulative water absorbed per surface area (surface from which water is absorbed) versus the square root of wetting time. Equation 5.1 can be used to describe the water absorption (total volume of fluid absorbed) and the sorptivity (related to the rate of absorption).

$$i = S \tau^{1/2} \quad (5.1)$$

where i [mm³/mm²] is the cumulative water absorption, S [mm/s^{1/2}] is the sorptivity, and τ [s] is the elapsed time.

Hall (1997) proposed that the diffusion would scale proportionately with the ratio of surface tension (γ) and viscosity (η) of the fluid. Hall further related this to sorptivity since sorptivity is related to the square root of diffusion. Kelham (1988) derived an expression for fluid absorption (Equation 5.2) that shows the relationship between depth of penetration and the square root of the ratio of surface tension and viscosity.

$$x(\tau) = \sqrt{\frac{4 k \gamma \cos(\theta) \tau}{p \eta r}} \quad (5.2)$$

where $x(\tau)$ [mm] is the penetration depth, γ [N/mm] is the surface tension, θ [rad] is the liquid-solid contact angle, p [~] is the porosity of the medium, r [mm] is the pore radius, k [mm²] is the intrinsic permeability of the material, and η [Pa.s] is the viscosity of fluid. An expression similar to equation 3 was derived by Scherer and Wheeler (2009) for stone consolidates.

Previous research using organic fluids have been noticed to have an absorption rate that scales proportionally with the square root of the ratio of surface tension and viscosity of the fluid ($(\gamma/\eta)^{1/2}$). This work will use this approach to attempt to interpret results from absorption tests that used aqueous solutions containing deicing salts.

5.4 Wetting and Drying for Concrete with Deicing Solutions

5.4.1 Experimental Program of Wetting and Drying of Concrete with Deicing Solutions

The concrete mixture that was used for these tests was a typical INDOT class C bridge deck concrete. The mixture proportions of this concrete are shown in Table 5.1. The fresh air content was 5.7 % measured according to ASTM C231-09 (2009). The hardened air content of the concrete was 4.4 % as assessed using an automated optical scanning approach (Castro et al. 2010) based on the approach of Peterson et al. (2001).

Table 5.1: Mixture Proportions Assuming Saturated Surface Dry (SSD) Conditions

Material	Mass
Cement (kg/m ³)	316
Class C Fly Ash (kg/m ³)	60
Water (kg/m ³)	150
Fine Aggregate (kg/m ³)	736
Coarse Aggregate (kg/m ³)	1049
Air Entrained Admix. (ml/ 100 kg cem. materials)	20
High Range Water Reducer Admix. (ml/ 100 kg cem. materials)	456
Retarder Admixture (ml/ 100 kg cem. materials)	98

The concrete was produced in a central mix plant and discharged from a ready mix concrete truck before the concrete was sampled. A series of 100 mm × 200 mm cylinders were cast. After one day of curing, the cylinders were demolded and sealed in double plastic bags at 23 ± 0.5 °C until the samples reached an age of 28 d. After 28 days of curing the cylinders were removed from bags and three 50 mm ± 2 mm thick samples were cut from the central portion of each cylinder with a wet saw.

Two different series were used to condition the concrete samples. The first series evaluated the role of different conditioning regimes on the absorption of water using three samples for each curing condition. The samples in the first series were conditioned in five different ways (ASTM, oven-dry, 50 % RH, 65 % RH and 80 % RH). A portion of the samples were conditioned following the procedure in ASTM C1585-04 (after conditioning the samples for 12 months at 50 % RH they were vacuum saturated for 24 h and then exposed to ASTM C1585-04 conditions). The remainder of samples were conditioned in four different ways: 1) at 80 % RH for 12 months, 2) at 65 % for 12 months, 3) at 50 % for 12 months, and 4) oven dried. The second series of samples consists of concrete samples that were dried at 50 ± 2 % RH, 23 ± 0.5 °C for 36 months and two samples were tested for each curing condition.

To prepare the specimens for fluid absorption testing the sides of the sample were sealed with epoxy. After the epoxy had hardened the top surface was covered with plastic to avoid evaporation from the sample during testing.

The absorption test involves recording incremental mass change measurements during the first six hours after the sample comes in contact with the fluid and subsequently taking one measurement every day for the next eight days. The amount of absorbed fluid is normalized by the cross-section area of the specimen exposed to the fluid using Equation 5.3.

$$i = \frac{m_t}{(a \cdot \rho)} \quad (5.3)$$

where: i (mm^3/mm^2) is the normalized absorbed fluid, m_t (g) is the change in specimen mass at time t ; a (mm^2) is the area of the specimen exposed to the fluid (i.e., that of the bottom face), and ρ (g/mm^3) is the density of the absorbed fluid (this is provided in greater detail later in the paper). These absorption measurements are then plotted as a function of the square root of time. The sorptivity is the slope of this graph.

The second series of samples were tested using seven different fluids. Their composition primarily based upon, one of three different industrially available deicing products, either NaCl, MgCl_2 or CaCl_2 . A low concentration was used for each salt as well as a higher concentration that was selected to be near the eutectic composition for each salt. De-ionized water was also used as a reference fluid.

5.4.2 Experimental Results from Wetting with Different Conditioning Methods

Figure 5.1 shows the results from water absorption tests performed on the first series of samples that were conditioned with different environmental conditions as mentioned earlier (ASTM C1585-04 accelerated conditioning, 80 % RH, 65 % RH, 50 % RH and oven drying). It should be remembered that these samples were conditioned for 12 months while the remainder of the samples discussed in this paper were conditioned at 50 % RH for a much longer time. Sample preparation has an enormous impact on the

1 water absorption results as more severe drying enables a greater volume of water to be
 2 absorbed during the test.

Figure 5.1: Water Absorption on Samples Subjected to Different Conditioning Procedures

5.4.3 Experimental Results from Wetting and Drying with Deicing Solutions

Figure 5.2 illustrates the results of the fluid absorption test as a function of time (for concrete at 50 % RH for a longer conditioning time than the samples in Figure 5.1).

It can be seen that even though the concrete that is used for all the tests in Figure 5.2 has the same conditioning and exposure conditions, the volume of solution absorbed by each material is dependent on the deicing salt solution and the concentration of the deicing salt solution that was absorbed. The sample with the low concentration of NaCl showed a slight increase in the rate of absorption (as compared with water) as well as the amount of fluid absorbed. This is consistent with the data reported by MacInnis and Nathawad (1980). The absorption of all the other fluids was reduced when compared with water.

1 As a result it can be concluded that in general as the salt concentration increased a
 2 reduced rate of absorption and reduced total absorption was observed. Further work is
 3 needed to examine lower concentrations for NaCl to ascertain why a slight increase is
 4 typically reported.

5

6 Figure 5.2: Volume of Deicing Solutions Absorbed by Concrete as a Function of Time
 7 (Typical Standard Deviation less than $0.1 \text{ mm}^3/\text{mm}^2$)

8 After the fluid absorption test was performed for 8 days the samples were dried at
 9 $50 \pm 2 \% \text{ RH}$, $23 \pm 0.5 ^\circ\text{C}$ for seven days. The samples were kept in the same one-faced
 10 exposed condition for the drying test; however, the exposed surface that was facing down
 11 in the absorption testing was placed facing up to simulate drying from the top. During the
 12 drying test the mass of the samples was recorded at regular intervals.

13 Figure 5.3 shows the volume of water loss during the drying period. It is
 14 important to note that the drying test will result in only the water portion of the solution
 15 being evaporated from the system leaving the salt to become more concentrated in the

1 solution before it eventually precipitates out. It can be noticed that as the concentration
 2 of deicing solution was increased the mass loss during drying decreased. This was
 3 particularly evident in the high concentration solutions which showed nearly no mass loss
 4 or even a slight gain during drying.

5
 6 Figure 5.3: Drying of Concrete Prewetted with Different Salt Solutions as a Function of
 7 Time (Typical Standard Deviation less than 0.03 mm³ water/mm²)

8 5.4.4 Experimental Results from Wetting Previously Exposed to Deicing Solutions

9 The samples that were first tested for absorption of different aqueous solutions,
 10 then tested for drying, were then oven dried so that a second fluid absorption test could
 11 be performed. The second fluid absorption test however used only de-ionized water as
 12 the fluid that was being absorbed.

13 To prepare the samples for the second wetting test they were placed in an oven at
 14 105 ± 2 °C until the difference between any two 24 h apart successive mass
 15 measurements were less than 0.5 % (i.e., approximately 5 days). It is important to note

that this drying process will evaporate only the water portion of the solution pre-absorbed, leaving salt in the pores. Since these samples were oven dried, their absorption rates and absorbed water are not comparable with any previous tests.

Figure 5.4 shows the results for this second absorption test. It can be seen by comparing the results to the results in Figure 5.2 that the behavior of the samples was dependent on the deicing solutions and the concentrations of deicing solutions used in the first wetting test. These results are a clear indication that the history of the samples affects the results of fluid absorption. This suggests when sorption testing is performed on field concretes some understanding of the admixtures or salts that remain in the pore system is needed to fully interpret the results.

Figure 5.4: Volume of De-ionized Water Absorbed by Concrete as a Function of Time in the Second Fluid Absorption Test (Fluid from the Original Test is Shown in the Caption)

5.4.5 Drying of Mortars Saturated with Different Deicing Salts

Moisture desorption is an established technique for evaluating the effect of moisture loss at a given humidity for a material. A TA Q5000 SA moisture sorption analyzer was used to carefully control temperature and humidity. Mortar samples were prepared ($w/c = 0.42$ and 55 % aggregate by volume) and cast in a cylindrical mold with a 34 mm diameter and 50 mm height. At an age of 28 days the specimens were demolded and 34 mm diameter 0.8 ± 0.05 mm thick slices were taken from the middle of the samples. The samples were dried under controlled conditions (at 23 ± 0.1 °C and 50 ± 1 % RH) in a CO₂ free chamber until they reach mass equilibrium. Then, samples were submerged for a minimum of 5 days in aqueous solutions with 23 % NaCl, 32 % CaCl₂, and 30 % MgCl₂ by mass.

For the samples submerged in NaCl, CaCl₂, and MgCl₂ solution,s a 50 mg to 70 mg piece of sample was placed in a tared quartz pan after a minimum of 5 days of submersion. The pan containing the sample was then suspended from the balance (± 0.001 mg accuracy) and placed in the relative humidity chamber to equilibrate at 23.0 ± 0.1 °C and 97.5 ± 0.1 % RH for up to 96 h or until the sample had achieved a stable mass (less than an 0.001 % mass change/15 minutes).

Then, the relative humidity was reduced to reach 95 %. After the sample mass equilibrated, the relative humidity in the chamber was changed in 10 % RH steps to 55 % RH, allowing the sample to attempt to equilibrate (12 h or 0.01% change in mass over 15 minutes) at each new humidity. After equilibrating at 55 % RH the samples were dried to 0 % RH. For the sample submerged in de-ionized water the procedure was similar, but

1 the relative humidity was reduced in 5 % steps from 97.5 % to 2.5 %, and then reduced to
2 0 % RH.

3 Figure 5.5 shows the plot of mass change as a function of time for the mortar
4 saturated in de-ionized water. The sample soaked in water can be seen to lose mass with
5 the decrease of RH. For this system, when the environment is below 100 % RH, water
6 will move from the pores to outside of the sample and classical drying behavior is
7 observed. The maximum mass of the sample is 8.5 % higher than the mass of the oven
8 dry sample.

9
10 Figure 5.5: Mass Change at Decreasing RH for Samples Containing De-ionized Water

11 Figure 5.6 shows a plot of mass change for the mortar samples submerged in
12 aqueous solutions of 23 % NaCl, 32 % CaCl₂, and 30 % MgCl₂. It can be observed that
13 initially upon placement in the testing chamber at 97.5 % relative humidity the mass of
14 the sample increases for the first 96 h until the relative humidity of the chamber is
15 changed. The samples absorb water during this time of preconditioning, with values

1 much higher than the 8.5 % increase in mass of the sample with de-ionized water as
2 compared with the oven dry sample.

3 The sample loses weight as the relative humidity is decreased however it should
4 be noted that the sample mass does not decrease to below the initial mass obtained from
5 soaking the sample in the deicing solution until relative humidity was decreased below 85
6 %, 55 % and 55 % for NaCl, CaCl₂ and MgCl₂ respectively. This will be compared with
7 the equilibrium relative humidity of the salt solution later in the paper.

Figure 5.6: Mass Change for Samples Submerged in Aqueous Solutions Containing Deicing Salts: (a) NaCl 23% (b) CaCl_2 32 % and (c) MgCl_2 30%

5.5 Properties of Deicing Salt Solutions

Physical properties of pure solutions were gathered from literature and compared with measured values for the industrially available deicing solutions tested in this research, and they are provided here for convenience in one location. The properties of the deicing solutions will be used in interpreting the wetting and drying results. This section is divided into four sections. The first three sections describe the influence of the deicing solutions in terms of surface tension, viscosity, and equilibrium relative humidity over the aqueous solution. The fourth section describes the specific gravity of the solution as a function of concentration as this is used to determine the volume of solution absorbed during the absorption test.

5.5.1 Surface Tension of Deicing Salt Solutions

Figure 5.7a shows surface tension measurements at different concentrations for the three solutions used in this research: NaCl, CaCl₂, MgCl₂. The surface tension for NaCl was obtained from (Hall and Hoff, 2002), CaCl₂ from (Conde, 2004) and MgCl₂ from (Phang and Stokes, 1980). A Du Noüy Ring Tensiometer KRÜSS was used with a resolution of 0.1 mN/m for the industrial deicers tested in this study. The tensiometer was cleaned between measurements following ASTM D971-04 (ASTM, 2004). The tensiometer was first calibrated using de-ionized water, which provided a value of 71.0×10^{-6} N/mm. A series of three measurements were performed for each solution, with the average reported.

The closed points in Figure 5.7a are the values measured for the industrially available solutions. The lines represent values taken from literature for pure salt

solutions at different mass concentrations. While the general trends are consistent, differences between the solutions containing industrial deicing salts and literature values may be due to impurities or other additives however further work is needed to examine this in greater detail.

5.5.2 Viscosity of Deicing Salt Solutions

Figure 5.7b shows a comparison of the viscosities for the solutions used in this research between pure solutions taken from literature and measurements of the deicing solutions. Viscosity measurements for the industrial deicers were performed on the salt solutions using an Anton-Parr rheometer, model Physica MCR 301. The rheometer kept the solution being tested at 23.0 ± 0.02 °C and from the torque applied to the fluid that causes a shear from which the viscosity can be found. Calibration of the device was performed using a reference standard.

The dashed lines presented are viscosities at different concentrations and are taken from literature (Hall and Hoff, 2002; Conde, 2004; Phang and Stokes, 1980, Afsai et al., 1989), while the points represent measured viscosities of the industrially available solutions. Again, differences between literature values and those of the solutions measured can be explained by differences in possible additions or chemistries of the industrial deicers.

Figure 5.7: Properties of Deicing Salts at 23-25 °C: (a) Surface Tension (b) Viscosity (c) Relative Humidity (d) Specific Gravity (Rosenburgh 2010, unpublished data)

5.5.3 Relative Humidity of Deicing Salt Solutions

Relative humidity measurements were performed on the salt solutions using Rotronic HygrClip2S sensors (± 0.8 % RH at 23 ± 0.1 °C). The relative humidity probes were mounted in a 75 mm x 68 mm stainless steel cylinder that was placed over a water jacketed sample cup holder. The water jacket was connected with a water bath at a constant temperature of 23.0 ± 0.1 °C.

Figure 5.7c shows the relative humidity measured over salt solutions for a wide range of solution concentrations. As the concentration increased the relative humidity over the solution decreased. The measured relative humidities of these unsaturated salt solutions are higher than that of the saturated solution of these salts which are 75.4 % RH for NaCl (Greenspan, 1977), 33.0 % RH for MgCl_2 (Greenspan, 1977) and 22 % for CaCl_2 (Conde, 2004).

5.5.4 Specific Gravity of Deicing Salt Solutions

Figure 5.7d shows the specific gravity of different deicing solutions as a function of concentration. The specific gravity of the solution increases with concentration. The CaCl_2 and MgCl_2 increase at very similar rates with an increase in concentration, while the NaCl increases slightly less than the CaCl_2 and MgCl_2 (i.e., 25 % less increase with concentration). This may be attributed to the colligative properties of solutions (Diamond, 2010, Personal Communication).

5.6 Discussion of Results

5.6.1 Aqueous Solution Absorption Behavior as a Function of Surface Tension and Viscosity

Equation 5.3 showed that the rate of absorption was related to the square root of surface tension and viscosity. Figure 5.8 plots the square root of the ratio of surface tension and viscosity versus mass concentration of salt. Pure salt solutions are shown as lines while industrial deicing solutions are presented as solid points, and the open points represent the measured sorption response of concrete (i.e., salt sorptivity/water sorptivity) from Figure 5.2. Figure 5.8 confirms that as the solution concentration increases, the rate of fluid absorption (i.e., sorptivity) decreases. Further, while the properties of pure solutions may not exactly represent the response of industrially available deicing solutions they do provide a comparable trend. Reasonable agreement is seen between the measured sorption and square root of the ratio of surface tension and viscosity the measured properties. Additional work is currently being performed to extend these results to a wide range of temperatures.

Figure 5.8: Relative Sorptivity for Deicing Solutions

5.6.2 Drying Time Versus Wetting Time

Comparing Figure 5.2 and 5.3 indicates that wetting happens much faster than drying. When de-ionized water was used as the absorbed fluid, the amount of fluid that was evaporated from the sample after eight days was $0.8 \text{ mm}^3/\text{mm}^2$. In contrast, it took just two hours for samples to absorb the same amount of fluid. These differences are even larger when salt solutions were used as the absorbed fluid. When MgCl_2 solution was used as the absorbed fluid, the amount of fluid that was evaporated from the sample after eight days was $0.07 \text{ mm}^3/\text{mm}^2$, but it took just ten minutes for the samples to absorb the same amount of fluid.

This is important as it suggests that field concrete may be more susceptible to increasing its level of saturation over time rather than drying out. Further, it shows that laboratory tests that use equal times for drying and wetting increase the saturation level of the concrete over time. Researchers (Wang et al. 2005) observed an increase in sample

1 mass during wetting and drying cycling with deicers which was attributed to
2 microcracking; however an increase in mass would be consistent with the wetting and
3 behavior observed in this paper.

4 5.6.3 Reduced Drying with Salt Solutions – The Role of Solution Equilibrium Humidity

5 The relative humidity of different salt solutions presented in Figure 5.7b help to
6 understand the results from the drying tests. The equilibrium relative humidity for the
7 23% NaCl, 32% CaCl₂ and 30% MgCl₂ solutions are 80 %, 40 % and 50 % respectively.
8 When the samples are placed in an environment with a relative humidity that is greater
9 than or equivalent to the approximate equilibrium relative humidity over the aqueous
10 solution in the pores water will not be lost (Figure 5.3) and the sample can actually gain
11 mass (Figure 5.3 and 5.6). This can be seen by the thinner (dashed lines in Figure 5.6),
12 which show the initial mass of the sample after it has been submerged in a aqueous
13 solution for over 5 days. At relative humidity higher than the equilibrium of the aqueous
14 salt solution the samples will increase in mass. At relative humidities where the
15 environment is less than the equilibrium humidity over the salt solution, the samples will
16 be expected to decrease in mass. The drying behavior of systems containing concentrated
17 aqueous solutions of deicing salts is complex and requires additional research.

18 5.6.4 Effect of Solution on Rewetting

19 When samples of concrete that were previously exposed to deicing solutions were
20 rewet with water they had an absorption and rate of absorption that depended on the

1 history of the specimens (Figure 5.4). The absorption of water can be 30 % to 50 % less
2 in specimens that were exposed to deicing solutions at some point in their lives. This is
3 an important, yet subtle, factor to understand. This is important since absorption tests of
4 field concrete may be mistakenly interpreted by relating the reduction in sorption to pore
5 filling or delayed sorption microcracking. Both of these observations (lower sorptivity
6 and delayed sorption) are consistent with data here for samples that did not have reduced
7 porosity or differences in sample damage.

8 5.7 Summary and Conclusions

9 This paper has reported experimental results from aqueous solution absorption
10 measurements and drying measurements for concrete in the presence of deicing solutions.
11 The following observations can be made. First, the absorption of fluid in concrete
12 depends on the drying environment used to condition the samples. Samples stored at a
13 lower RH absorbed a greater volume of fluid. Second, it was observed that the deicing
14 solutions reduce the rate of fluid absorption. This reduction can be related to the square
15 root of the ratio of surface tension and viscosity (Hall and Hoff 2002). Third, the time
16 scale between drying and wetting is different and concrete is more likely to become
17 preferentially increasingly wet over time. Fourth, the drying of concrete containing
18 aqueous solutions (with deicers) differs from that of water. The equilibrium relative
19 humidity of the aqueous solution plays an important role on limiting drying. Finally, the
20 presence of deicing salts in field samples impacts the absorption when field samples are
21 tested in the lab using water. This suggests that care must be taken in analyzing field
22 concrete exposed to deicing salt solutions.

CHAPTER 6: SORPTION TESTING IN CEMENTITIOUS MATERIALS: A DISCUSSION OF AUTOMATING TEST PROCEDURES

6.1 Overview

Fluid penetration in concrete has received significant attention from scientists and engineers over the past several decades. To better understand the mechanisms of fluid transport many test configurations have been developed. This chapter will focus on the comparison of the ASTM C-1585 testing procedure with a modified version of the ISAT (Initial Surface Absorption Test) for which the data collection can be automated. A comparison of the two methods is presented.

This automated procedure yields results that are similar to the standard ASTM C-1585 test. The repeatability of the automated procedure is similar or in some cases better than the standard test. Further, the automated test provides additional information that can be used to determine the rate of absorption.

6.2 Introduction

The durability of concrete subjected to aggressive environments depends largely on the penetrability of the pore system (Sabir et al. 1998, ASTM 2004). Three mechanisms can be used to describe transport of water in cementitious systems: permeability, diffusion and absorption. Permeability is the measure of the flow of water

1 under a pressure gradient while diffusion is the movement of ions in a saturated or
2 partially saturated material. Absorption can be described as the materials ability to take in
3 water by means of capillary suction. Water absorption is an important factor for
4 quantifying the durability of cementitious systems (Sabir et al. 1998, Tremblay et al.
5 2005) and is the primary focus in this chapter.

6 The test method currently used to determine the rate of absorption (sorptivity) of
7 water in unsaturated hydraulic cement concretes is ASTM C1585 (2004). This test
8 consists of exposing one surface of a specimen (bottom surface) to water and measuring
9 the increase in mass resulting from absorption. The absorption test involves recording
10 incremental mass change measurements during the first six hours after sample is in
11 contact with water and taking one measurement every day for the next eight days. It is
12 important to note that before the test is conducted, samples are conditioned for 18 days.
13 During the first 3 days, samples are placed in a 50 °C and 80 % RH environment. After
14 three days the samples are removed from the chamber and placed in individual sealed
15 container where the samples remain for 15 days to allow internal moisture equilibrium
16 before the test begins.

17 While ASTM C1585 is simple, it can be a time consuming test to conduct. An
18 automated test method which allows for continuous data recording has been
19 implemented. The procedure based on test performed by Tremblay et al. (2005) and
20 Bégué et al. (2004), consists of measuring the decrease of weight of a continuous supply
21 of water connected with the tested specimen, instead of measuring the change in weight
22 of the specimen as in the standard procedure. It should also be noted that the automated
23 test considers both gravity and absorption effects. Previous work by Young (2007)

1 indicated that capillary effects were similar to the combined effect of gravity and
 2 capillary, when there are not cracks or large pores in the sample.

3 The focus of this research is the assessment of a procedure that used automated
 4 data collection system for sorption test. The standard procedure (ASTM C1585) and the
 5 proposed procedure will be performed on references mortar and concrete cylindrical
 6 specimens. By comparing the total amount of absorbed water, and the initial and
 7 secondary rate of absorption, the repeatability of both procedures will be evaluated.

8 6.3 Standard ASTM C-1585 Procedure

9 ASTM C-1585 consists of exposing one surface of a specimen (bottom surface) to
 10 water and measuring the increase in mass resulting from absorption of the water over
 11 time. The mass of the specimen is recorded at 1, 5, 10, 20, 30 and 60 min, then every
 12 hour up to 6 h, and then every 24 h up to 8 d. The amount of absorbed water is
 13 normalized by the cross-section area of the specimen exposed to the fluid using Equation
 14 6.1 (ASTM, 2004):

$$15 \quad I_t (\text{mm}^3/\text{mm}^2) = \frac{m_t}{(A \cdot d)} \quad (6.1)$$

16 where “ m_t ” is the change in specimen mass in grams at the time t ; “ A ” is the exposed
 17 area of one surface of the specimen, in mm^2 ; and “ d ” is the density of the water in g/mm^3
 18 ($0.001 \text{ g}/\text{mm}^3$ at 23°C).

This normalized value of absorption (I_t) is plotted against the square root of time beginning with the addition of water, in order to determine the initial and secondary rate of absorption (ASTM, 2004). The initial rate of water absorption is defined as the slope of the mass of water plotted versus the square root of time considering all the points from 1 minute to 6 hours. The secondary rate of absorption is calculated considering all points from 1 day to 8 days. These periods of time assumes that the fluid that is absorbed by the sample is water. However if other fluids are considered, the period of times considered for initial and secondary rates of absorption may change.

ASTM states that the repeatability can be expressed as a coefficient of variation that has been determined to be less than 6.0 % in preliminary measurements for the absorption as measured by this test method for a single laboratory and single operator. It should be noted that if the correlation coefficient is less than 0.98, the rate of water absorption can not be determined (ASTM 2004).

6.4 Proposed Procedure

This procedure consists of measuring the decrease of weight of a continuous supply of water connected with the top surface of the tested specimen, instead of measuring the change in mass of the specimen as in the standard procedure. The set-up and procedure proposed is based on test performed Tremblay et al. (2005) and Bégué et al. (2004). Figure 6.1a shows a photo of two samples being tested using the automated procedure.

a)

b)

Figure 6.1: Automated Test Procedure: a) Photo of an Actual Test, and
b) Representation of Specimen in Support Device

The specimen in the testing device is placed on a support base to avoid movement of the setup during the test (Figure 6.1). The edge between the rubber ring and the specimen support is sealed by contact at the surfaces.

The test starts (i.e., time = zero) when water is introduced in the system using the pipe 1 (Figure 6.2a). The air inside of the system is removed using the pipe 2. When air is completely removed, the stopcock #2 is closed (Figure 6.2b). Additional water is introduced using pipe 1. When the water level rises to be over stopcock #1, it is closed. Then more water is added using pipe 3 until it is complete filled with water. Subsequently, pipe 3 is turn down using a finger over the end of it to avoid the loss of water from the pipe. The pipe is then introduced in the water reservoir and it is placed on a digital balance connected to a computer with an automated data collection system

(Figure 6.2c). Paraffin oil is added carefully on the top of the water reservoir to minimize evaporation. Finally, the automated data collection system is activated recording mass and time.

The time elapsed between the moment that water is in contact with the specimen and the moment that the data collection system is approximately 4 to 5 minutes. The initial rate and secondary rate of absorption will be not affected if the absorption during the initial 5 minutes is not known. However the total absorbed mass will change. In order to know the total absorbed water, the specimen weight is recorded before and after the test. Comparing this change of mass with the data recorded by the automatic collection system, is possible to determine the absorption during the initial minutes.

Figure 6.2: Schematic Representation of Setup of Proposed Procedure

To calibrate the system and be sure that the test setup worked properly, two tests were performed. The first test has for objective to measure the lost of mass from the water reservoir because of evaporation. Three different water reservoirs were filled with approximately 150 grams of water and covered with 30 grams of paraffin oil. Then, they were placed in an environment similar to the one where the actual sorption tests are

performed (23 ± 2 °C and $50 \pm 3\%$ RH environment). The change of mass was monitored during 8 days. The results show that the average evaporation from the water reservoir is 0.15 grams over 8 days with a low variability (Figure 6.3).

The second test consisted of performing a regular test, but a plastic (impermeable) sample was used instead a real concrete sample with the objective to simulate a test on a low permeability material. The results of these tests, that include the effect of evaporation from the source of water, show that the setup is not completely impermeable, but it is controllable under the environment in the laboratory (Figure 6.3). Total measured evaporation was 0.4 g of water, while the total change in mass from a sorption test is approximately 25 g. This would result in an error of less than 2% generated by the loss of water from the reservoir of water covered with paraffin oil.

Figure 6.3: Total Water Loss During a Test of an Impermeable Plastic Sample and Total Evaporated Water from Reservoir of Water Covered with Paraffin Oil

Once evaporation value is known, it is necessary considering it to correct measurements on actual samples. According with the experimental results, 0.4 gram of water will be subtracted from the total amount of absorbed water in a regular test of 8 days. Assuming linear behavior of total evaporated water will simplify the procedure and it will not affect significantly the results (Figure 6.3).

6.5 Experiment Work

For this study four different mixtures were prepared in total. Three mixtures were mortars with a single volume fraction of fine aggregate (55 %) and different w/c (w/c of 0.40, 0.45 and 0.50). These mixtures were designated as M-40, M-45 and M-50, with the numbers representing the different w/c . The fourth mixture was concrete, designated as C-40 with the number on the left representing w/c . A list of the mixture proportions can be found in Table 6.1.

Table 6.1: Mixture Proportions, SSD Condition

Constituents	M-40	M-45	M-50	C-40
Cement type I (kg/m^3)	625.7	584.9	549.2	316.0
Pozzolan	0	0	0	60.0
Water (kg/m^3)	250.8	263.7	275.1	150.4
Fine aggregate (kg/m^3)	1389.6	1389.6	1389.6	736.3
Coarse aggregate (kg/m^3)	0	0	0	1049.2
w/c or w/b	0.40	0.45	0.50	0.40
Aggregate volume	55%	55%	55%	60%

Cylinders (101.6 mm or 4 inches and 203.2 mm or 8 inches diameter) were cast for each mixture. The specimens were cured for 28 days in double sealed bags at 23

1 $\pm 1^\circ\text{C}$. After curing, three 50.8 mm (2 inches) slices thick were extracted from central
2 portion of each cylinder. The remaining outer portions were discarded.

3 Slices were conditioned in three different environmental chambers at three
4 different relative humidities, $50 \pm 2\%$, $65 \pm 2\%$ and $80 \pm 2\%$ until they reached mass
5 equilibrium. The temperature in all three chambers was fixed at $23 \pm 1^\circ\text{C}$. After
6 specimens reached mass equilibrium (approximately 12 months), the mass and
7 dimensions of each sample was recorded. The outer edge of each slice was coated with a
8 thin film of epoxy to prevent the moisture exchange from the lateral surface during test.

9 Additionally, the specimens to be use in the proposed procedure were coated in
10 the bottom surface with epoxy to avoid evaporation during the test. If the bottom surface
11 is not sealed, unquantifiable amount water will be evaporated through that surface
12 affecting the measured absorption. A rubber ring is placed on the specimens to avoid
13 leaking between the specimen and the specimen's support during the test. The edge
14 between the sample and the ring is sealed using caulking silicone (Figure 6.4). When the
15 silicone is hardened, the total mass specimen with epoxy and ring is recorded.

(a)

(b)

1 Figure 6.4: Preparation of Specimen for Automates Procedure a) Representation of
 2 Sample Preparation b) Photo of the Sample Ready to be Tested

3 6.6 Results

4 The standard and the automated procedures were performed on the specimens.
 5 Four samples from each specimen were tested (two for each procedure). Figure 6.5 and
 6 6.6 shows a comparison between the average results of absorption of the standard and the
 7 proposed procedure for the four different specimens. In the plots, the dashed lines
 8 represent the results from the automated procedure.

Figure 6.5: Comparison of Absorbed Water of Samples Conditioned at 50%RH: a) vs time, b) vs Square Root of Time

Figure 6.6: Comparison of Absorbed Water of Samples Conditioned at 65%RH: a) vs. Time, b) vs. Square Root of Time

6.7 Summary and Conclusions

ASTM C1585 is currently used to determine the rate of water absorption by hydraulic cement concretes. ASTM C1585 consists of exposing one surface of a specimen to water and measuring the increase in mass due to water absorption. While this test is easy to perform, it can be time consuming and can require the timing of data collection to be altered if fluids other than water are used.

A procedure is used here, which allows for continuous and automated data acquisition. This automated method consists of measuring the decrease in mass of a continuous supply of water (or other fluid) connected with the tested specimen, instead of measuring the change in mass of the specimen directly. The supply of water is placed on a digital balance and the balance is connected to a computer with an automated data collection system. This enables data to be collected at a much more frequent rate.

Both the standard and automated procedures were performed on two different specimens. The proposed procedure yields reproducible values of rates of absorption that are similar to those of the ASTM standard test.

In addition, the proposed procedure decreases the influence of the operator, and is less time consuming after the test started. This additional data can provide information for further analysis of the absorption behavior of the samples.

CHAPTER 7: ELECTRICAL IMPEDANCE SPECTROSCOPY AND RELATED EXPERIMENTAL PROCEDURES

7.1 Overview

This chapter discusses the measurement of electrical conductivity in hydrating concrete. The electrical conductivity, or resistivity, of concrete is frequently used as a surrogate test method for assessing a concrete's fluid transport properties. This work indicates benefits of using the continuous monitoring of electrical conductivity from the time of casting and also discusses rapid conductivity measurements. Both of these methodologies could provide a valuable alternative to rapid chloride permeability (RCPT, ASTM C1202). This work highlights the need to carefully consider the effects of temperature, hydration, sample geometry, and pore solution concentration when interpreting the electrical measurements using the modified parallel model. The work describes the electrical conductivity measurements from a series of concretes that are cast with different water to cement ratios, aggregate volumes, and admixtures.

7.2 Background

Electrical impedance spectroscopy (EIS) describes a procedure that measures the electrical impedance of a concrete by applying a difference in potential across the concrete (Rajabipour 2006). The resulting current is measured, and an impedance and

1 phase base can be determined. The impedance can be broken into two parts: real and
2 imaginary. By measuring the electrical impedance of concrete over time, information can
3 be obtained about the fluid transport properties that will influence service life predictions
4 for concrete structures (McCarter and Curran 1984, Christensen et al. 1994, Ford and
5 Mason 1996).

6 The EIS described in this paper is performed by applying an alternating current
7 (AC) to the concrete. AC is preferred as compared to direct current (DC) for a few
8 reasons. First, DC causes the ions in the concrete to become “polarized” meaning certain
9 ions become preferentially attracted to one electrode depending on their charge (Bockris
10 and Reddy 1970, McCarter and Curran 1984, McCarter and Brousseau 1990, Gu and
11 Beeaudoin 1996). This crowding of ions around an electrode makes it hard for some
12 oppositely charged ions to migrate through the system, yielding spurious results. Second,
13 the voltage used for DC measurements is typically much greater than that of AC
14 measurements. This increased voltage means increased power needed to perform the test.
15 The increased power in the sample can input a large amount of energy into the system
16 causing an increase in the temperature or this energy can result in damage to the
17 microstructure of the concrete. The primary mode of conduction in concrete is through
18 the pore solution. Since the electrical properties of the liquid phase of a concrete (the
19 pore solution) is dependent on temperature, a change in the temperature of the sample can
20 give inconsistent results if this is not accounted for.

21 As stated previously, EIS measures the resistance of a material to an applied
22 current. In EIS the resistance of this current is measured at multiple frequencies. Two
23 graphs are frequently used to assess data from EIS measurements: the Nyquist plot and

1 the Bode plot. The Nyquist plot places the real impedance (commonly thought of as
 2 resistance) on the ordinate axis and the corresponding imaginary impedance (commonly
 3 thought of as capacitance) on the abscissa. The bulk resistance of the material is
 4 determined when the imaginary component is at a minimum. The advantage of the
 5 Nyquist plot is that the resistance value can be easily determined through visual
 6 recognition. The Bode plot contains the frequency of the electrical current on the
 7 ordinate and the impedance on the abscissa. The influence of frequency is more easily
 8 realized on the typical Bode plot (Figure 7.1).

Figure 7.1: Nyquist Plot and Bode Plot (after Macdonald and William 1987)

7.3 Objectives

The objective of this work was four-fold:

- First, the chapter outlines a procedure and sample geometry to collect field samples for possible Quality Control / Quality Assurance (QA/QC) practices. The EIS procedure will calculate similar properties yielded by the RCPT, but with more repeatable results, and more early age data.
- Second, the chapter reports experimental results of electrical impedance, temperature rise, degree of hydration, and pore solution conductivity as a function of time. The results indicate the effect of correcting conductivity measurements.
- Third, the chapter provides a systematic study of the effects of temperature on electrical conduction. This is essential to enable electrical measurements to be corrected for temperature.
- Fourth, a sensor will be evaluated that assesses changes in pore solution as a function of hydration.

7.4 Experimental Program

7.4.1 Program Overview

Nine different mixture designs were evaluated. After the samples were cast, they were monitored for the following week continuously. After the continuous first week, the samples were point measured once a week for the next three weeks. Finally, after the

1 first four weeks, the samples were point measured once every four weeks till an age of 90
 2 days was reached. This is explained in Table 7.1. The program itself can be broken
 3 down into four main categories: equal paste comparison (Table 7.2), equal w/c
 4 comparison (Table 7.3), increased water content comparison (Table 7.4), and admixture
 5 comparison (Table 7.5).

6 Table 7.1: Testing Schedule

Age (weeks)	Tested
0-1	Continuously
2-4	Weekly
5-12	Every 4 weeks

8 Table 7.2: Equal Paste Content Testing Breakdown

Equal Paste Content Samples		
Mixture #	w/c	Paste Content (% volume)
1	0.36	27.7
2	0.42	27.7
3	0.45	27.7
4	0.50	27.7

Table 7.3: Equal Water to Cement Ratio Testing Breakdown

Equal Paste Content Samples		
Mixture #	w/c	Paste Content (% volume)
2	0.42	27.7
5	0.42	34.0

Table 7.4: Increased Water Content Testing Breakdown

Increased Water Content				
Mixture #	Original w/c	kg of water added per m ³	Final w/c	Paste Content (% volume)
2	0.42	0	0.42	27.7
6	0.42	10.9	0.45	28.4
7	0.42	29.1	0.50	29.7

Table 7.5: Increased Admixture Testing Breakdown

Admixture Addition				
Mixture #	w/c	Used additive	g admixture per 100 g of cement	Paste Content (% volume)
2	0.42	none	-	27.7
8	0.42	air entrainer	0.05	27.7
9	0.42	water reducer	0.20	27.7

7.4.2. Sample Geometry and Age of Testing

Many Department of Transportation (DOT) projects require some type of QA/QC practice. These practices are usually related to strength of the concrete. Additional information is needed to find the properties that can be used to predict the service life of the concrete. An early age prediction of fluid transport properties would give DOT's

another criteria for acceptance of a concrete and overall better prediction of service life. The specimen geometry used in this research was developed with a possible QC/QA procedure in mind. A standard 6"x12" cylinder mold was outfitted with a pair of electrodes used to conduct the EIS experiments. The use of a 12" cylinder mold should make any future QA/QC development a simple transition and is compatible with any storage equipment designed to accommodate a standard cylinder. A detailed description can be found in section 7.4.5.

The specimens were monitored continuously for one week after casting, weekly from 2-4 weeks of age, and monthly from 2 months-90 days. The change in the conductivity of a 3 day old concrete sample is very small. Because of this, the testing regimen utilized provides a representative amount of data over the desired time scale.

7.4.3 Mixture Proportions

Four different w/c (0.36, 0.42, 0.45 and 0.50) and four different paste volume fractions (27.7%, 28.4%, 29.7 and 34.0% of the total volume) were used. These mixtures were designated as 27.7/0.42, 27.7/0.45, 27.7/0.50, 34.0/0.42, 28.4/0.42 and 29.7/0.42 with the number on the left representing the volume fraction of cement paste and the number on the right representing w/c. Mixtures 8 and 9 are have the same mixtures that mixture 2, but adding air entrainer and water reducer respectively, as shown in Table 7.5. A list of the mixture proportions can be found in Table 7.6.

Table 7.6: Mixture Proportion for Electrical Conductivity Testing, in SSD Condition

Constituent	Mixtures						
	27.7/0.36	27.7/0.42	27.7/0.45	27.7/0.50	34.0/0.42	28.4/0.45	29.7/0.50
Coarse Aggregate, IN #8 (kg/m ³)	1047.2	1047.2	1047.2	1047.2	944.6	1035.9	1017.6
Fine Aggregate, IN #23 (kg/m ³)	880.4	880.4	880.4	880.4	786.6	870.9	855.5
Cement Type 1 (kg/m ³)	396.4	364.2	350.2	328.7	464.5	360.3	353.9
Water, Public Source (kg/m ³)	142.7	152.9	157.5	164.3	195.4	162.1	176.9
Admixtures (g/100 g of cement)	0.0	0.0	0.0	0.0	0.0	0.0	0.0

7.4.4 Constituent Materials

7.4.4.1. Portland Cement

An ASTM C150 Type I ordinary portland cement (OPC) was used in this study, with a Blaine fineness of 370 m²/kg and an estimated Bogue composition of 56 % C₃S, 16 % C₂S, 12 % C₃A, 7 % C₄AF and a Na₂O equivalent of 0.68 % by mass.

7.4.4.2 Fine Aggregate

The sand used was natural river sand with a fineness modulus of 2.71, an apparent specific gravity of 2.58, and an absorption of 1.8 % by mass.

7.4.4.3 Coarse Aggregate

The coarse aggregate used was limestone with an apparent specific gravity of 2.65, and an absorption of 0.9 % by mass.

7.4.5 Specimen Geometry and Casting

The two major experiments in the research required two different sample geometries. Both the EIS experiment and the late age pore solution extraction geometries are detailed below.

7.4.5.1 EIS Concrete Samples

The mold preparation starts with a 6" diameter, 3/8" thick, high density polyethylene (HDPE) disk (Figure 7.2). The disk is cut using a hole saw with a 6" inner diameter from a larger sheet of HDPE. Then, two holes are drilled four inches, on center, away from each other. These holes are then tapped using a 3/8"-16 tap. Inside these holes, 12" threaded stainless steel rods are screwed until the tip of the rod is flush with one end of the HDPE disk.

It is at this time some type of 1" thick spacer material is placed at the bottom of a standard 6"x12" concrete cylinder mold. In the current experiment, a 4.25" square of extruded polystyrene insulation was used. Then, inside the cylinder mold, the disk with the stainless steel rods threaded to it is placed, disk side down, until the disk is flush with the spacer material (Figure 7.2). The rods should stick out of the top of the cylinder by about an inch to accommodate the EIS equipment connection. A schematic representation of the mold is shown in Figure 7.3.

Figure 7.2: HDPE Disk and Threaded Rod Apparatus

Figure 7.3: Schematic of Mold Design

The concrete is then placed in the mold with two lifts equaling about half the volume of the mold with each lift. The lifts are roded twenty five times each, paying special attention to the area around the stainless steel rods. After each lift, a vibrating table is used to vibrate the sample for 5-10 seconds. The top is finished with a trowel. After the mold is full of material, a standard 6"x12" cylinder mold lid with two 3/8" holes spaced 4 inches apart, on center, is placed on top (Figure 7.4). After the lid is secure, the sample is again vibrated for 3-5 more seconds while being tapped with an open hand around the outside. After the lid is place on top of the mold, be sure to wipe off the threads of the portion of the rods that are sticking out of the mold so that screws

1 can be threaded on to the rods and good connection can be made when the rods act as
2 terminals later.

3 Within one hour from the initial mixing, a thermocouple is placed in the cylinder
4 to monitor the temperature, and the electrodes are attached to the EIS analyzer to acquire
5 the impedance data.

6
7 Figure 7.4: Finished Specimen with Rods and Lid in Place
8

7.4.5.2 Pore solution extraction Samples

To understand how the conductivity of the pore solution changes over time, pore solution extractions were performed on samples of differing ages and water to cement ratios. Pore solution is considered the only primary phase in concrete due to the facts its conductivity is many magnitudes higher than the cement gel and the aggregate phases.

As stated before, the conductivity of the pore solution changes over time by an increasing ion concentration as the cement continues to hydrate and react with the water, reducing the amount of pore solution. To characterize this increase in conductivity, the pore solution must be extracted from the other media in order to determine its overall contribution to the parallel model discussed earlier. The pore solution is extracted through two different processes; one used for samples before set and the other for after set has occurred.

Samples with four different water to cement ratios were created: 0.36, 0.42, 0.45, and 0.50 mirroring all the different w/c tested in the EIS cylinder experiments. The early age pore solution extractions are taken when the concrete is still in its plastic state, according to the procedure by Penko (1983). Mortar samples are created and the extractions are taken at predetermined times. The same mixtures proportion from Table 7-6 are used, but the coarse aggregate is not added in these mixtures. In general, the pore solution extractions were taken at an age of 10 minutes and 1 hour. More pore solution extractions were taken close to set time usually around 3-4 hours, and every hour following until the concrete became too difficult to work with. The lower w/c mixtures become harder to work with more quickly than the higher w/c mixtures, yielding less data

1 overall. The pore solution samples are kept in air tight vials to protect against
2 carbonation until they undergo conductivity testing.

3 The later age pore solutions were extracted from pore solutions at 12 hour, 20
4 hour, 1 day, 3 day, and 8 day ages. The pore solution samples were obtained by a high
5 pressure piston and cylinder apparatus according to the procedure described by
6 Barneyback (1983). The test consists of placing the sample in a high strength steel
7 cylinder and crushing the sample with a high pressure piston, usually provided by a
8 standard compression testing machine. After the pore fluid is freed from the sample, the
9 fluid is extracted via a syringe. The samples used in this test were pastes only. Paste is
10 preferred when performing piston and cylinder extraction because of the higher yield of
11 pore solution per sample, reducing the time needed to obtain the minimum amount of
12 pore solution, and the lower chance of damaging the testing apparatus. Mortars can cause
13 scoring along the cylinder walls, leading to costly repairs and apparatus downtime.

14 7.5 Experimental Techniques

15 7.5.1 Impedance Measurements Procedures

16 The concrete sample was attached to a Solartron SI 1260 Impedance/Gain-Phase
17 Analyzer by the two stainless steel rods which act as electrodes. Copper wire is run from
18 the impedance analyzer to each terminal and secured by two stainless steel nuts where the
19 wire is place in between the two nuts and tightened to ensure good connection. The

1 samples were monitored for a week continuously, once a week thereafter for twenty eight
 2 days, and every four weeks until ninety days.

3 The data acquired by the EIS experiment is in the form of a resistance,
 4 specifically, the units of ohms. Hereafter, all EIS experimental data will be expressed in
 5 the form of conductivity, specifically in the unit of Siemens/meter. To go from resistance
 6 value to conductivity value, Equation 7.1 can be used.

$$\sigma = k / R_B \quad (7.1)$$

7 Where:

8 $\sigma =$ The conductivity (S/m)

9 $k =$ The geometry factor (1/m)

10 $R_B =$ The bulk resistance (Ω)

11 The geometry factor can be determined by using a solution of known
 12 conductivity. This solution is placed in the mold just as the concrete itself is placed in the
 13 mold during casting. The resistance is obtained through EIS. If the conductivity of the
 14 solution is known, a geometry factor can then be back calculated using Equation 7.1.

7.5.2 Automation of Measurements

EIS measurements can be used to monitor the concrete structure as it hydrates. Poursaei and Weiss (2010) developed a user interface using LabView to allow researchers to monitor multiple specimens simultaneously. By automating the process, the experiment has become more efficient in terms of man hours needed to perform it, and yields better results by automatically acquiring data when it would be highly inconvenient to do so in person. Automation is also employed in the data analysis portion of the testing. A program written in visual basic language scans the EIS data file for resistance and relays that value along with the time of the measurement to a spreadsheet file which can be sorted and further analyzed. With both the data acquisition and data analysis portions of the experiment automated, the only part of the test that is time intensive is the sample preparation.

7.5.3 Pore Solution Conductivity Testing

After the pore solution is extracted from the samples, the solution is tested for conductivity. To test the conductivity of the solution, the fluid is placed in a conductivity cell. The conductivity cell consists of two parallel plates placed on either side of a small plastic cylinder, and can be seen in Figure 7.5. The plates are kept in place using a clamp to apply pressure to both ends. The fluid is injected by a syringe into the cell using a pair of holes drilled on into the wall of the cylinder. From there the cell has a current applied to it using the EIS equipment and the resistance of the bulk material is found. The resistance is converted to a conductivity value. A geometry factor was calculated exactly the same as the EIS sample, by using a fluid of known conductivity, obtaining the

1 resistance of the fluid in the cell through EIS testing, and back calculating a geometry
2 factor using Equation 7.1. A typical plot of pore solution conductivity versus time can be
3 seen later in Figure 7.9.

4
5 Figure 7.5: Pore Solution Conductivity Cell

6 A sample of the raw conductivity data from the concrete can be seen in Figure
7 7.6. The conductivity is high initially for the fresh concrete. There is decrease in the
8 conductivity of nearly 90 % during the first few days for this specific mixture design.
9 The high level of repeatability in the measurements can be seen by examining the error
10 bars in Figure 7.6.

Figure 7.6: Geometry Corrected EIS Conductivity Data

7.6 Review of the Modified Parallel Law

Concrete has been modeled by using the modified parallel law (Dullien 1979, McLachlan et al. 1990, Gu et al. 1992, Ford et al. 1995, Torquato 2002). The model states that the conductivity of concrete can be attributed to three components: the conductivity of the pore solution as it is the primary conductive phase in concrete, the volume fraction of pore solution, and the connectivity of the system which reflects how well an ion can pass from one place to another within the system. The model is expressed in equation form in Equation 7.2.

$$\sigma_t = \sigma_o * \phi * \beta \quad (7.2)$$

1 Where,

2 σ_t = The conductivity of the bulk cement paste (S/m),

3 σ_o = The conductivity of the pore solution (S/m),

4 ϕ = The volume fraction of the liquid that can be found inside the pores (NA),

5 β = The connectivity of the pore system (bounds are from 0 to 1)

6 The parallel model can be used to describe how the conductivity changes during
7 the hydration process. As the hydration of cement occurs, chemical changes occur in the
8 pore solution as it becomes more concentrated. Further, as hydration progresses the pore
9 fluid volume fraction decreases, ϕ . While the pore fluid volume is decreasing, it is also
10 become more concentrated as the ions in solution have less fluid to occupy making the
11 conductivity of the pore fluid, σ_o , increase. Lastly, the connectivity factor, β , of the
12 parallel model will change over the hydration process. When the concrete is in the plastic
13 state, ion mobility will be very easy through the sample due to the fact that there are very
14 little obstructions. However, as the hydration products start to form a pore structure, ion
15 transport paths will become much more tortuous and disconnected, making it much more
16 difficult for ions to move within the system, decreasing the connectivity factor. A
17 qualitative understanding of the components of the parallel model is understood,

1 however, one of the main goals of this research is to quantify how the change each of
2 these components affects the overall system.

3 7.6.1 Powers Model for Computation for Hydration

4 To determine the components of the modified parallel law it is essential that we
5 consider how these components evolve during hydration. Initially it may be tempting to
6 simply think of these parameters in terms of their change as a function of time; however
7 it is likely more fundamentally appropriate to consider them as a function of the degree of
8 hydration. Conceptually the degree of hydration can be thought of as the change in the
9 volume of water or unreacted cement. A degree of hydration of 0 means that none of the
10 cement has reacted while a degree of hydration of 1 implies that all of the cement has
11 reacted.

12 7.6.1.1 Degree of Hydration

13 Isothermal conduction calorimetry was used to estimate the degree of hydration
14 for the mixtures described in this report. Isothermal calorimetry experiments were
15 conducted on 2 samples of each of the four pastes that reflect the w/c of each of the EIS
16 samples: 0.36, 0.42, 0.45, and 0.50.

17 An isothermal conduction calorimeter is a device that measures how much energy
18 is released by a chemical process; in this case, the hydration of cement. The amount of
19 energy released during the cement hydration can be related to the degree of hydration of
20 the cement if the ultimate heat that is liberated by complete hydration of the cement is

known. While this may be difficult to calculate it can be estimated as 475 J/g for the cement used in this study (Castro et al. 2010b).

The key assumption that is being made when correlating heat released at a certain time and degree of hydration is that all the constituents of the cement are hydrating to provide an average rate of hydration. The calorimeter returns a value of cumulative energy over time as shown in Figure 7.7. For this energy to be converted to a degree of hydration, the theoretical maximum energy of hydration must be calculated. This can be done for a specific cement using a copy of the mill certification. The mill certification will give a value of how much of each constituent is in the cement. From there, the maximum amount of energy each constituent could produce during the reaction is calculated from thermodynamic properties. The value of total energy released at any time over the total maximum possible energy released is the degree of hydration value.

Figure 7.7: Total Energy Evolved from Calorimeter

The calorimeter was used to calculate the DOH for the first week of hydration of the cement paste as shown in Figure 7.8.

Figure 7.8: Degree of Hydration for 0.45 Paste

The degree of hydration of a 14 days, 28 days, 56 days, and 90 days cement pastes were calculated using the nonevaporable water method. The nonevaporable water method consisted of crushing the cement paste into a fine powder and immersing this dust in acetone to extract any pore fluid from within the pores to stop the hydration process. The sample is then let sit in the acetone for 24 hours and then placed in a dish for 6 hours to let the acetone evaporate off. The then dry sample is further dried in a 105 °C oven for 24 hours. Then a known mass of sample is placed in a crucible of known mass. The sample is placed in a muffle furnace at 1050 °C for three hours and then weighed. The difference in the sample weight before and after the muffle furnace

1 ignition is the loss of non-evaporable water, which, through Powers model can be related
2 to degree of hydration.

3 7.6.2 Pore Solution Conductivity vs. DOH

4 The pore solution extractions were performed on samples that were small enough
5 to be considered isothermal, therefore there is no need to apply and equivalent time
6 maturity correction to the pore solution conductivity data. Using the degree of hydration
7 to measure a process can also give a better idea of what changes are occurring in the
8 structure itself. To get a graph of pore solution conductivity vs. degree of hydration, the
9 respective values at equal ages from Figure 7.8 and Figure 7.9 are compared and plotted
10 in Figure 7.10. It is seen from this plot that the pore solution conductivity of a cement
11 paste is linearly related to the degree of hydration of the sample. This is a key trend
12 when trying to quantify the relationships of all the different components of the parallel
13 model.

Figure 7.9: 0.45 w/c Pore Solution Conductivity vs. Time

Figure 7.10: Pore Solution Conductivity vs. DOH for a Paste with a w/c of 0.45

7.6.3 Pore Solution Conductivity Sensor

Pore solution extractions yield satisfactory results for pastes and mortars at early ages. However, pore solution extraction is a labor intensive procedure. As it stands, to make the EIS procedure a possible QA/QC practice, pore solution extractions would need to be made on every different mix tested; an accomplishable task, but a deterrent for any simple QC/QA use. Ideally, one would be able to measure the change in conductivity of the pore solution over the hydration process, in real time. Rajabipour (2006) has made a huge step towards this goal with the development of the Pore Solution Conductivity Sensor.

The sensor is made from a specific aggregate called Mississinewa Shale. After research was performed on different types of porous stone, the Mississinewa stone was chosen for its pore size and pore distribution. Its unique pore structure allows surrounding pore solution to diffuse into the system, making the monitoring of real-time changes in the pore solution possible.

The concept of the sensor follows the same logic as the EIS, where a saturated material has a potential applied to it and a resistance is calculated. A small, prismatic, sample of the stone, 8 mm x 8 mm x 2 mm, has electrodes attached to each of the 8mm faces of the sample. These electrodes are covered with epoxy to eliminate the short circuit that would develop (Figure 7.11). From there, the sensor is dried to 1 % RH, then vacuum saturated in a 0.4 molar potassium hydroxide solution. After these steps are carried out, the sensors are ready to be placed in a fresh sample.

Figure 7.11: Front and Side View of the Rajabipour Developed Pore Solution Sensor

When EIS is applied to a concrete sample immediately after mixing, the system is undergoing radical changes as the cement is undergoing hydration; the fluid volume is decreasing, the pore solution is becoming more concentrated, and the pore network is developing. Conversely, as long as the pore solution sensor stays saturated, the pore fluid volume fraction and pore network are unchanging. The only variable in the pore solution sensor system is the pore solution conductivity. A typical plot of the sensor data can be seen in Figure 7.12. It is displayed as a function of the pore solution conductivity that was acquired in the research discussed in 7.6.2. The acquisition of the data collected by the sensor began within an hour of casting of the sample and ran at half hour intervals for one week. The sample the sensor was imbedded in was a 0.45 w/c 45% paste by volume, mortar.

Figure 7.12: Sensor Conductivity vs. Pore Solution Conductivity

The data acquired from the pore solution sensor differed from the experiment performed by Rajabipour. However, the slopes of the linear fit lines from the experiment were similar in value with this experiments slope as 0.017 and Rajabipour's as 0.015. Further research is needed to determine the repeatability of the pore sensor as well as its validity over a range of different mix designs.

7.6.4 Pore Fluid Volume Fraction

The EIS concrete samples are a sealed system. The degree of hydration data can be used to calculate the pore fluid volume fraction.

The pore fluid volume fraction itself can be defined as any fluid in the system that can be used to transport ions in the system. This includes both fluid in the capillary pores and the fluid within the gel pores.

Using the equations developed by Jensen, a pore fluid fraction vs. degree of hydration graph was developed and can be seen in Figure 7.13. This pore fluid fraction is for the paste component only. However, concrete is composed of two other components; air and aggregate. The pore fluid volume fraction of a concrete can be changed greatly depending on the volume of aggregate found in your mixture design. Figure 7.14 shows the volume fraction of the conductive phase in concrete.

Figure 7.13: Pore Fluid Fraction vs. Degree of Hydration for 0.42 w/c Paste

Figure 7.14: Pore Fluid Volume Fraction vs. DOH for a 65.7% Agg. Fraction 0.42 w/c Concrete

7.6.5 Effect of Temperature on Pore Solution and Concrete Conductivity

A change in the temperature of a pore solution has a profound effect on the conductivity of the pore solution. Due to the heat of hydration experienced when a cement grain undergoes hydration, the system always undergoes a temperature change in ambient conditions. However this change in conductivity due to temperature can be monitored and expressed through mathematical equations, primarily a modified version of the Arrhenius equation developed by Sant et al., seen in Equation 7.3 (Sant, 2008). Since the temperature of the EIS samples are monitored throughout the experiment, all of the values are known in Equation 7.3 except for E_{ac} , the activation energy of conduction. This value can be obtained by performing temperature experiments on the pore solutions samples themselves.

$$\sigma(T_{REF}) = \frac{\sigma(T)}{\exp\left[\frac{-E_{ac}}{R} \cdot \left(\frac{1}{T} - \frac{1}{T_{REF}}\right)\right]} \quad (7.3)$$

1 where $\sigma(T_{REF})$ = The Conductivity of the Material (S/m), at the Chosen Reference
 2 Temperature T_{REF} ($^{\circ}\text{K}$), $\sigma(T)$ = The Conductivity of the Material at Temperature (S/m),
 3 at the temperature T ($^{\circ}\text{K}$), E_{ac} = The Activation Energy of Conduction (J/mole), and R
 4 = The Universal Gas Constant (J/(K * mole)).

5 To obtain the E_{ac} for a pore solution, the resistance of the pore solution must be
 6 measured at three chosen separate temperatures. The resistances are converted to
 7 conductivities through Equation 7.1. Then, because of the relationships of the Arrhenius
 8 Equation, the natural log of the conductivities is plotted vs. in inverse of temperature. A
 9 line is fit through these points, and the slope of this line gives the value of E_{ac}/R . A
 10 typical graph created from this procedure can be seen in Figure 7.15. This procedure is
 11 repeated for all the different age pore solutions to obtain an E_{ac} at the different ages. An
 12 interesting trend is found when comparing activation energies of conduction at different
 13 times within the same mixture. There seems to be no trend in the change of E_{ac} over
 14 time. Furthermore, the values of E_{ac} are all within a small standard deviation of each
 15 other. For this reason, an average of the activation energies of conduction was
 16 calculated, and this value was used as a constant over time for a specific mixture. The
 17 range of values can be seen over the different w/c samples tested in Table 7.7. It can be

- 1 seen that there is no trend across the different w/c which leads one to believe that the
- 2 activation energy of conduction is a constant for a specific cement.

3

4 Figure 7.15: Natural Log of Pore Solution Conductivity vs. the Inverse of the

5 Temperature

6 Table 7.7: Activation Energy of Conduction for Different W/C Mixtures

w/c	Eac (KJ/mole)
0.36	9.39
0.42	10.19
0.45	10.06
0.50	9.69
Average	9.83
SD	0.36

1 Now that the activation energy of conduction is known, the temperature
2 correction for conductivity can be applied to the EIS data. The process starts with the
3 raw data that is acquired by the EIS experiment that can be seen in Figure 7.16. Because
4 of the increase in temperature of the samples due to heat of hydration, an equivalent time
5 must be calculated before the temperature correction can be employed. After the
6 equivalent time calculation is complete, the temperature correction can be applied to the
7 EIS data.

8 By looking at Figure 7.17, the end of the dormant stage of hydration can see by a
9 sharp increase in the conductivity around five hours, and characterized over time as a
10 “bump” in the data. Again, this is due to the increase in temperature during the
11 acceleration period. When looking at Figure 7.18, which has been corrected for
12 equivalent time and temperature effects, the bump has been almost completely removed.
13 This bump has been claimed to be an effect of many different chemical processes,
14 however, through this research, it seems to be only a temperature effect.

Figure 7.16: 0.45 w/c 27.7% Raw EIS Data

Figure 7.17: 0.45 w/c Concrete (27.7% Paste Volume) – Early Age Data

Figure 7.18: 0.45 w/c (27.7% Paste Volume) - Time and Temperature Corrected Conductivity Data – Early Age

7.6.6 Formation Factor

With three out of four elements of the parallel model equation known, σ , σ_o , and ϕ , the connectivity factor, β , can be calculated at any point in the data, giving a better understanding of how the pore structure develops over time. But, another important factor that comes from normalizing the concrete conductivity with the pore solution conductivity is called the formation factor. The pore solution volume fraction and conductivity factor are both unitless values that are indications of how the structure of the concrete is changing. As the cement is hydrating and decreasing the fluid volume, the pore system is becoming much more complex. This is causing both the variables to drop simultaneously. This trend is expressed in Figure 7.19. By normalizing the graph in

- 1 Figure 7.19 with the pore fluid volume fraction data, a graph of the connectivity
 2 component of the parallel model can be viewed in Figure 7.20.

3
 4 Figure 7.19: Formation Factor vs. Degree of Hydration for a 0.45 w/c 27.7% Paste
 5 Volume Concrete

6 The connectivity or “beta” factor as it is called, due to the Greek letter used to
 7 describe it in the parallel model equation, is a measure of the connectivity of the pore
 8 system in a cementitious material. It is a unitless value describing the connectivity of the
 9 pores. It ranges in value from 1 to 0; 1 meaning the system is totally connected, without
 10 any obstructions, and 0 meaning the substance stops all ion transport.

11 As would be expected, the more difficult it is for an ion has to travel across a
 12 material, the lower the conductivity of that material will be. An ion’s travel can be made
 13 difficult by both the length and size of the path traveled by the ion. As the pore system of

a cementitious system develops, the pores become more tortuous, and less connected to one another, making the path of the ion more difficult over time. Another phenomena of the hydrating process is the pores become more constricted with increasing hydration. As the pore constricts at a point, it creates a “bottleneck” where ions start to crowd allowing a smaller amount of ions to pass through. This decreased flow of ions decreases the conductivity of the system over time.

Figure 7.20: Beta Factor vs. Degree of Hydration for a 0.45 w/c 27.7% Paste Volume Concrete

7.7 The Effect of Mixture Design on the Conductivity of Concrete

If the EIS procedure explained above is ever to be implemented as a QA/QC practice, a better understanding on how variations of mixture designs affect the results

1 must be known. The following section is by no means a complete breakdown on the
2 most common changes made to mix design, however it is a good start to see how a
3 variable paste volume, water to cement ratio, basic admixtures, and water content affect
4 the electrical impedance of a concrete system. Certain components frequently added to
5 concrete, such as silica fume, have a profound effect on the EIS results. Further research
6 will need to be performed to test enough of the possible components of concrete to make
7 the EIS a viable choice for QA/QC testing.

8 7.7.1 Mix Designs

9 The mixture proportions selected for the initial testing of the EIS procedure
10 display how four variables influence the measured electrical response. The four variables
11 are: water to cement ratio, paste volume, water content, and admixture addition.

12 7.7.2 Equal Paste Comparison

13 The first series of tests maintained a constant paste volume but varied the water to
14 cement ratio. Initially, after mixing the difference in the conductivity between the three
15 mixtures is not very significant. The 0.50 w/c mixture has a greater proportion of water
16 and as a result it has the highest conductivity immediately after mixing. This is followed
17 by the mixture with a w/c of 0.45 and 0.42 respectively. It is approximately around the
18 time of set when the values of conductivity between the three mixtures begin to deviate.
19 At nearly one week, the mixture with a w/c of 0.5 has a 50 % higher conductivity

- 1 compared to the mixture with a w/c of 0.42. This trend can be seen in Figure 7.21 and
2 Figure 7.22.

3
4 Figure 7.21: Temperature Corrected Conductivity vs. Equivalent Time – Early Age

1

2 Figure 7.22: Temperature Corrected Conductivity vs. Equivalent Time – Late Age

3 Figure 7.23 plots the normalized conductivity of the parallel model as a function
 4 of degree of hydration. It can be seen that the system with a higher w/c has a higher
 5 conductivity. This is due to higher porosity (ϕ) and a higher connectivity factor (β).
 6 Powers model can be used to estimate the porosity enabling the β factor to be estimated
 7 directly as shown in Figure 7.24.

Figure 7.23: Equal Paste Comparison – Normalized Conductivity

Figure 7.24: Equal Paste Comparison – Beta Factor

7.7.3 Differing Paste Volumes

The second series of experiments were performed to assess the influence of the volume of paste in the concrete. The standard mixture with a w/c of 0.42 has a paste volume of 27.7 %, which is a relatively low amount of paste. A second mixture was prepared with a higher paste volume, a w/c of 0.42 paste by volume of 34 %. By maintaining the w/c constant between the two mixtures in the experiment, the pore solution conductivity will be the same throughout the hydration process, meaning the other two parameters in the parallel model, ϕ and β , will be the only factors affecting the results of the experiments.

The increased water fraction of the mixture with an increased paste volume results in a higher initial conductivity since the pore solution conductivity is equal. This difference is most pronounced at early ages (Figure 7.25) with the increased paste mixture having around a 40 % higher conductivity value compared to the standard mixture. However, as the individual mixtures pass set, the difference in the values has a sharp decrease, then a more steady decrease until a DOH value of 0.5 is achieved as shown in Figure 7.26 when the decrease starts to increase again, presumably due to pore refinement.

Figure 7.25: Conductivity vs. Degree of Hydration for Equal W/C Concretes with Differing Paste Volumes

Figure 7.26: Percent Greater Conductivity of the Increased Paste Mixture Compared to the Standard Mixture

Figure 7.27: Conductivity of the System / Pore Solution Conductivity vs. Degree of Hydration for Equal W/C Concretes with Differing Paste Volumes

One very important conclusion can be drawn regarding the connectivity as there appears to be no correlation between an increased paste volume and connectivity. As can be seen in Figure 7.28, the connectivity factor of the 27.7 % paste samples and the 34.0 % paste samples are virtually identical throughout the process. An increase in the amount of aggregate does little to increase the tortuosity of the pore system.

Figure 7.28: Connectivity Factor vs. Degree of Hydration Comparison of Equal w/c Concretes with Different Paste Volumes

7.7.4 Increased Water Content

The comparison of water content was designed to simulate a situation that could be encountered in the field. If the workability of an already delivered concrete batch needed to be increased, how would adding water to the truck change the transport properties of the mixture compared to the original concrete delivered. The control mixture (0.42 w/c - 27.7% paste) was used. However, additional water was added to the mixture to increase the water to cement ratio to 0.45 and again to 0.50. This does change the water to cement ratio as well as the paste volume. As one would expect, the increased water in the mixture increases the overall conductivity (Figures 7.29, 7.30, 7.31).

Figure 7.29: Temperature Corrected Conductivity vs. Degree of Hydration Over an Increase in Water Content

Figure 7.30: $1 / \text{Formation Factor}$ vs. Degree of Hydration Over an Increase in Water Content

Figure 7.31: Connectivity Factor vs. Degree of Hydration over an Increase in Water Content

An alternative method was investigated to increase workability without the addition of water, namely the use of a water reducing admixture. This is discussed in the following section.

7.7.5 Comparison of Admixtures

The mixture designs compared in the previous section did not have any admixtures within their mixture design. This was to limit the number of variables that could affect the results of the tests. However, nearly all commercial “Ready-Mix” batches contain some sort of admixture. As a preliminary test, an air entrainer, as well as a high-range water reducer were respectively added to the 0.42 w/c 27.7% paste standard mixture. The results can be seen in Figures 7.32, 7.33 and 7.34.

Figure 7.32: Comparison of Conductivity of Samples Containing Admixtures

Figure 7.33: Conductivity of the System Normalized to the Pore Solution Conductivity of the Admixture Comparison Samples

Figure 7.34: Connectivity of the System vs. DOH for the Admixture Comparison Samples

It can be seen from all three graphs that neither air entrained nor the high-range water reducer samples deviate from the standard mix by a significant amount. It can be argued that air filled porosity will not transport electrical signal. The behavior is the same for all three comparisons, highlighting the fact that in small amount, both air entrainer and high-range water reducer do not change the conductivity or transport properties of a concrete system.

7.8 Summary and Conclusions

This chapter has reviewed the use of electrical measurement as a method to assess fluid transport properties. The influence of temperature, paste volume and pore solution

- 1 conductivity are discussed. This work will be a valuable addition to the background that
- 2 forms a new standardized method for newer electrical properties.
- 3

1 CHAPTER 8: MONITORING INTERNAL RELATIVE HUMIDITY IN EXPOSED
2 CONCRETE SLABS WITH VARIOUS EXPOSURE CONDITIONS

3 8.1 Introduction and Overview

4 To better understand the relative humidity that can be expected to develop in
5 concrete in the field, a series of instrumented slabs were tested with several different
6 exposure conditions. These tests considered the effects of boundary conditions on the
7 wetting and drying of concrete. Six exposure conditions were evaluated including:

- 8 1) a covered surface,
9 2) an exposed vertical surface,
10 3) a horizontal surface on a drainable base,
11 4) a horizontal surface on a non drainable base,
12 5) a completely submerged sample and
13 6) a slab stored at 23 °C and 50 % relative humidity.

14 The slabs were prepared using a mixture that was similar to a typical INDOT
15 paving mixture; however hollow cylindrical shafts were placed in the sample to enable
16 sensors to be placed in the sample. By placing sensors at different depths in the exposed

1 samples, the internal relative humidity (RH) and temperature (T) profile that can be
2 expected in field concrete in Indiana is obtained.

3 The relative humidity of concrete in the field is important for evaluating the long-
4 term durability of this concrete. It is known that if the internal RH of a concrete drops
5 below approximately 80% RH, the hydration of cement particles slows substantially.
6 Improper curing can lead to problems such as excessive shrinkage and cracking or poor
7 strength development. Another reason to monitor the relative humidity in a sample
8 which is due to its influence on the moisture transport properties of concretes. If external
9 moisture application to a sample is known, an accompanying change in internal RH can
10 be correlated to the transportation properties of the sample itself (Castro, 2010).

11 8.2 Boundary Conditions

12 Six different boundary conditions were proposed for this experiment. The slabs
13 prepared to evaluate the first five conditions mentioned in section 7.1 were prepared and
14 placed in an outdoor exposure site in West Lafayette, Indiana at the INDOT research
15 facility. The samples were exposed to the environment from November 8, 2009 to May
16 1, 2010. During this time the slabs were exposed to fall, winter, and spring conditions.
17 The weather conditions were monitored by wireless weather station positioned near the
18 samples. The samples were placed outside after approximately 48 days of curing at $23 \pm$
19 1°C and $98 \pm 2\%$ relative humidity. The sixth sample was placed in a constant
20 temperature ($23 \pm 0.2^\circ\text{C}$) and relative humidity ($50 \pm 2\%$) environmental chamber for an
21 equivalent exposure time as a control sample. This is shown in Figure 8.1.

Figure 8.1: Exposure Samples at the INDOT Research Test Facility

Several attempts were made to obtain an agreement from the weather department of the television station located near the INDOT research office. While it was initially thought that this would be able to be done, the television stations unresponsiveness caused the research team to install a weather station. Figure 8.2 shows the weather station that was used in this study that was alongside the slabs being tested.

The first sample (a covered surface) was covered at the surface from direct contact with surface moisture a tent like structure made out of two pieces of plywood, and can be seen in Figure 8.3. Wood planks were used to raise the slab off the ground so

- 1 any pooled water would not be in contact with the specimen. This boundary condition
2 would represent a concrete that is protected from the direct precipitation.

3
4 Figure 8.2: Weather Station at the INDOT Research Test Facility
5

Figure 8.3: Covered Sample at the INDOT Research Test Facility

The second sample tested was the exposed vertical surface. To create this boundary condition, a slab was placed on its end so the exposed surface was perpendicular to the ground as shown in Figure 8.4. The slab was placed on a wooden pallet so any pooled water would not be in contact with the specimen. This boundary condition represents a concrete capable of shedding water like an exposed concrete bridge column that may or may not always be directly contacted by precipitation depending on the prevailing winds.

Figure 8.4: Exposed Vertical Surface Sample at the INDOT Research Test Facility

The third and fourth boundary conditions were designed to evaluate the conditions of the base the concrete is placed on different subgrades (Figure 8.5 and Figure 8.6). The third sample was placed on a drainable base while the fourth sample was placed on a non-drainable based. These base conditions were created using a plastic tub with open graded aggregate lining the bottom. The only difference between the third and fourth samples is that the drainable base (sample three) had holes drilled in the bottom of the tub to facilitate the water loss. The non-drainable condition started with a small amount of water in the bottom of a fully intact tub. The non-drainable boundary condition had a

- 1 varying water level in the tub according to the precipitation and evaporation. This was
2 designed to represent concrete pavements that may be on different subgrades.

3
4 Figure 8.5: A Horizontal Surface on a Drainable Base at the INDOT Research Test
5 Facility

Figure 8.6: A Horizontal Surface on a Non-Drainable Base at the INDOT Research Test Facility

The fifth boundary condition consisted of placing a sample was fully submerged from the start of the experiment. This was done by placing the concrete in a plastic bin under water as shown in Figure 8.7. This boundary condition represents a concrete that is in the worst case scenario with respect to developing high internal relative humidity or saturation.

Figure 8.7: A Submerged Concrete Sample at the INDOT Research Test Facility

The sixth boundary condition was simulated by using a controlled environment with a constant temperature (23 ± 1 °C), RH (50 ± 2 %), or precipitation. This sample is shown in Figure 8.8 and represents a sample that may exhibit a high level of drying.

Figure 8.8: A Sample Exposed to a Constant Drying Environment

8.3 Evaluation of the Relative Humidity Sensors

To test the capabilities of the RH sensors, the sensors were placed above saturated salt solutions according to ASTM E-104. The salt solutions were used to determine whether the relative humidity (RH) sensors could return a stable measurement for higher relative humidities. The salts used to evaluate the sensors were potassium sulfate (K_2SO_4), potassium chloride (KCl), sodium chloride (NaCl), and potassium iodide (KI). Tables 8.1 and 8.2 provide an illustration for typical salts and relative humidities measured in this program. The humidity created by these salts is temperature dependant; therefore all the salt solutions were maintained at 23 ± 1 °C.

1 Table 8.1: Salt Solution Calibration of Humidity Sensor #28

Humidity (%)		
Salt	Sensor measurement	Actual RH over saturated salt solution
K ₂ SO ₄	97.3	97.4
KCl	84.7	84.7
NaCl	75.4	75.4
KI	70.0	69.3

2 Table 8.2: Salt Solution Calibration of Humidity Sensor #21

Humidity (%)		
Salt	Sensor measurement	Actual RH over saturated salt solution
K ₂ SO ₄	96.7	97.4
KCl	84.4	84.7
NaCl	75.0	75.4
KI	70.1	69.3

3

4 The salt solutions themselves have a variance associated with them, the greatest at
5 23 °C is potassium sulfate with plus/minus 0.6 % RH and the lowest being sodium
6 chloride with plus/minus 0.2 % RH.

8.4 Selection of a Relative Humidity Sensor

There are many commercially available relative humidity (RH) sensors. Testing was needed to assess which sample would be the most appropriate for this study considering durability, cost, precision, and accuracy, of the sensors. All sensors have a recommended operating range for both temperature and relative humidity that must be considered. Since the experiment was to be conducted for over a year, outdoors, and in north-central Indiana, a sensor that could function in both freezing and classically warm temperatures was needed. Also, when dealing with a sealed concrete specimen, one must expect the humidity to be relatively high and in an alkaline environment in the sample when the experiment first begins. Typically, as a sensor is subjected to a RH above 80 %, the variance and error in the measurement become greater compared to a lower RH range. The following section describes the sensors that were evaluated as a part of this study.

8.4.1 Honeywell Sensors

These relative humidity sensors cost approximately \$20 - \$25. These sensors (HIH-3610, HIH 4000) needed to be coupled with some type of temperature probe running simultaneously with the RH sensor. The sensors were a capacitive type of sensor. The RH sensor works by measuring a voltage, which, when coupled with the accompanying temperature, can be used to ascertain a relative humidity measurement. The sensors were soldered to standard copper wires and need to be continually connected to data logging equipment. Since the concrete slabs had to be placed outside, there would need to be shelter nearby so the cords run outside the building with the computers

1 safely inside. When testing the environmental chamber type RH sensor over the
2 saturated salt solutions, the sensor read a maximum value of approximately 80 % RH,
3 even though the sensor should have returned a reading over 97 %. This pointed out that
4 substantial corrections would be needed to make these sensors work in the range if this
5 was ever possible. Since the internal relative humidity for a sealed concrete system would
6 be expected to be between 80 % and 100 % and the sensors would require separate data
7 acquisition this sensor was not a good candidate for this research program.

8 8.4.2 I button Sensors

9 A commercial sensor called the I-button® produced by Maxim Products was
10 evaluated as a potential candidate for this research. The I-button is a single device that
11 measures both relative humidity and temperature. The device is a stand alone device
12 which means that the device will log data without being attached to any data logging
13 device. Because of the I-buttons internal battery and onboard memory, the device can
14 hold over 4000 data points (the data collection rate can be adjusted during the sensor set-
15 up) and that can be downloaded at any time. Because of the I-button's stand alone
16 capability, this introduced the possibility of easily placing the slabs outdoors to get real
17 weather exposure conditions.

18 The size of the I-button made it capable of being placed in a sample, though an
19 even smaller sensor would be optimal. Although, one specific limitation of the I-button's
20 set up needed to be overcome. As explained before, the I-button is a stand alone device,
21 meaning it will acquire data without being attached to a data acquisition system.

1 The primary method for using the I-button was directly connecting an I-button to
2 a reader provided by the manufacturer. The reader consists of a molded plastic head with
3 electrical contacts inside to connect with the I-button. The other end of the cable is a
4 USB connection that enables the data to be downloaded to a computer. The I-button is
5 placed inside this head and can data can be obtained at any point during the acquisition.

6 The need to monitor the progress of the experiment without removing the sensors
7 from the slab was desired. Toward this end the direct connection to the I-button was
8 needed so that the sensor could be read while the experiment was in progress. As can be
9 seen in Figure 8.9, the size of the I-button by itself is approximately the size of a quarter
10 in diameter. If the I-button were connected with the reader, it would roughly double the
11 size of the I-button, negating any advantage gained by the small size of the I-button. To
12 get around using the oversized reader for direct connection, a procedure was created to
13 manufacture a custom “reader”. A phone wire was connected to contacts located on the
14 top and bottom face of the I-button, respectively to enable the phone wire to protrude
15 from the specimen.

Figure 8.9: Maxim I-Button ®

The I-buttons responded well over the saturated salt solutions, easily reaching the 97 % RH mark with an acceptable amount of variance. It should however be noted that when the sensors were placed in fresh concrete there appeared to be a reaction with the alkaline pore solution which caused several of the sensors to malfunction. One problem with simply placing a sensor directly into concrete is the aggressive nature of fresh concrete. With its highly basic chemistry, fresh paste can damage sensitive electronics that come in contact with it. To overcome this limitation the sensor was not placed in contact with the fresh concrete, but instead was placed in a cavity inside the concrete after it hardened.. The cost of the sensor was between 60 and 100 dollars depending on the quantity ordered.

8.5 Design of the Slab Specimens

Due to the problems that were observed with placing the sensors in direct contact with the pore solution, a decision was made to place cylindrical cavities in the sample to monitor relative humidity (Parrot 1982, Grasley 2006). To obtain a relative humidity profile throughout the depth of the sample several sensor locations were used. It was decided early on in the research that the sample geometry should be a slab with one- or two sided exposure to the environment. This was for ease of construction, handling, and analyzing of the obtained data.

Many different depth configurations and designs were considered for this slab. It was originally envisioned that an object would be cast in the concrete which could be removed after set thereby creating a cavity in the concrete. The samples would then be sealed and cured. When the experiment was set to begin, the sensors would be placed in the cavity left in the sample and the ends of the cavity could be filled with some type of material to prevent moisture flow through the ends of the cavity.

The type of material for which to fill the cavity was also a major point of discussion. In order to get a good relative humidity measurement from the sensors, the cavity in which the sensors rest must come to into equilibrium with the surrounding concrete system. The original hypothesis was, the faster the equilibrium process, the more reflective the measurement would be of the actual sample's state. It was also logically assumed that a smaller cavity would come into equilibrium faster with the surrounding system faster than a larger one.

Using this logic direction of thoughts, it was suggested to place some type of cementitious material inside the sample cavity. A cementitious material would mimic the

1 thermal and diffusive properties of the concrete from which the sample itself is
2 composed. However, there was a desired to keep the sensor away from the paste while it
3 was in the fresh state. Another problem with using a cementitious material to fill the
4 cavity was that it would add water to the sample. An alternate solution needed to be
5 explored. An advantage to not using cementitious filler is there is no permanent block on
6 the cavity, restricting access to the sensor if it somehow malfunctions during the test. A
7 sensor can easily be changed out without having to destroy the test specimen.

8 The wetting and drying occurs all in the vertical direction. As such the cavities
9 should not be placed in line with each other along the vertical direction. This means that
10 all sensors must have a horizontal offset from one another to avoid possible influence
11 from the other cavities. The sensors also need to be placed a sufficient distance from the
12 sample ends to avoid edge effects. As a result, the first sensor was placed at least half the
13 depth of the sample away from any boundary of the sample.

14 The creation of a cavity, or “tunnel” that runs completely through the sample
15 made the construction of the slab easier. It was also easier to ensure that the cavity was
16 placed at the correct location.

17 The final slab dimensions can be seen in Figure 8.10. The form was made using
18 2”x8” (nominal dimensions) lumber as the sides of the mold, and plywood as the bottom
19 of the mold. The center of the cavity that is used for measuring relative humidity was as
20 follows: 0.5”, 1”, 2”, and 3.625” from the top surface. The samples were cast upside
21 down to have a consistent surface finish.

Figure 8.10: Test Slab Dimensions

The mold was prepared with the sides of the mold being covered with plastic sheeting to ease the demolding process. In ASTM tests where concrete surface integrity is needed it is preferred to not use form release agents to stay consistent with these practices. The rectangular steel bars were not covered in form oil. The sample was cast and let sit overnight to set, covered in a plastic tarp. The rectangular steel bar was not able to be removed from the slab without cracking the slabs. Another approach was attempted by first wrapping the steel bars in plastic wrap to help the release of the bars upon demolding. This approach appeared to create a wedging effect that made it more difficult to remove the bars. A similar experience was observed after coating the bar with Teflon tape.

It was determined from these trials that the rectangular steel bar were poorly suited for this application. A circular cross section would allow a torque to be applied to break the initial static bond. This twisting action could be continued while a longitudinal

1 force was applied in conjunction, to remove the piece from the tunnel. A PVC pipe with
2 a ½ inch inner diameter fit these characteristics well.

3 Slabs were cast with the PVC pipe. Ample vibration was applied to make certain
4 material makes it between the PVC pipe and the plywood below. A picture of the casting
5 can be seen in Figure 8.11.

6
7 Figure 8.11: Casting of the Slabs

8 The PVC pipe was removed from the concrete using mechanical means of
9 removal using a tensile force of a Universal Testing Machine (UTM). The slab was

1 secured to the base of the UTM and clamping the head of the UTM was secured to the
2 PVC pipe as seen in Figure 8.12. The UTM was used to apply a tensile load to the pipes
3 thereby removing the PVC pipes from the concrete.

4
5 Figure 8.12: Removing PVC Pipes from Slab Using UTM

6 The samples were sealed and cured for approximately 60 days. Since there were
7 five exposure conditions being tested, a minimum of five slabs needed to be cast;
8 however only two molds used. To help minimize the effect of different ages, the slabs
9 were let cure for a long period of time to let the pore structures reach a point where a few

1 days maturity would have little effect on the results (i.e., 59 days is similar to 60 days
2 which is similar to 61 days).

3 8.6 Design of the Relative Humidity Sensor Connection

4 The I-buttons were attached to phone wires so that they could be placed in the
5 slab. The phone line had a phone line on one end that could be placed in an extension
6 cable that consisted of a phone jack and USB plug for attachment to the computer. The I-
7 button was wired to the free end of the telephone wire as seen in Figure 8.13.

Figure 8.13: I-button Telephone Line Setup

Since telephone wire itself is very thin, a short length of thicker gauge wire was soldered to the ends of the telephone wire. To prevent a short circuit from occurring, each splice was adequately insulated with electrical tape shrink tube was placed over the whole connection to help create a more continuous unit for easier handling.

8.7 Placement of the Relative Humidity Sensor in the Cylindrical Hole

The sensor was designed to be placed into the slab with a minimal potential for leakage from the ends. The cylinder was prevented from being directly exposed to the open atmosphere. A rubber stopper was used to seal the holes. A small hole was drilled in a #3 rubber stopper to enable the electrical wires to be fed through the hole. After the

1 wire was passed through the hole it was injected with pure silicon sealer. After the
2 rubber stopper was placed in the tube end the outside of the rubber stopper was sealed to
3 the concrete interface to create a watertight seal. Since latex sealers release moisture as
4 they cure and silicon sealer hardens by sucking ambient moisture out of the air to cure;
5 both of these sealers could influence the moisture content. The silicon sealer was used on
6 the outside of the specimen since the sealers will cure in the open environment having
7 little effect on the sample results.

8 The electrical wire would typically be soldered to the I-button, however this was
9 not an option with the I-buttons since the housing of the I-button is made of stainless
10 steel. This difficulty arises since soldering stainless steel is difficult because of the
11 chemical composition; the stainless steel must become much hotter compared to mild
12 steel to have solder adhere to it. Further, the I-buttons internal electronics encased inside
13 the small unit, the danger of destroying the I-button with the intense heat was too great.
14 As a result a conductive adhesive was invested as a method to attach the wires. The
15 stainless steel cover displayed poor adhesion properties with the conductive adhesive.
16 The surface can be roughened to increase adhesion, but still does not meet the adhesion
17 of normal steel. To improve the adhesion and electrical properties of solder, a
18 combination of super glue and conductive paint was used. Most adhesives insulate
19 against electrical conduction, making the application of the glue to the wire and I-button
20 difficult. Special care was taken to ensure the super glue was applied to a portion of the
21 wire that still had the insulation attached (Rajabipour 2006). The superglue was left to
22 dry overnight to develop the full strength of the glue. After the glue had cured, the
23 electrical connection was installed from the wire to the I-button. This electrical

1 connection was made using a silver conductive paint (Pour Ghaz et al. 2010). Before the
2 wires were attached to the I-buttons using super glue, a small piece of wire,
3 approximately one quarter of an inch long, was stripped from the end, exposing the
4 copper strands. With the super glue keeping the insulated part of the wire attached to the
5 I-button housing, the conductive paint could be applied to the area where the copper
6 strands and I-button met, completing the electrical connection and the I-button setup
7 itself.

8 An adaptable and customizable setup is another huge advantage when using the I-
9 button. The main reason a custom setup can be achieved is the I-buttons internal
10 memory. With many relative humidity sensors, a capacitor is used to monitor the
11 ambient humidity in the air. A current is applied across the capacitor and the voltage is
12 monitored by the attached system. This voltage can be converted into a relative
13 humidity. The drawback with this type of sensor is the setup itself must be taken into
14 account when the voltage is output. If too much resistance is intrinsically built into the
15 system, the data output can be incorrect. This is not the case when dealing with the I-
16 button. Since the I-button stores it's data on internal memory and, in turn, relays this
17 memory from the I-button to the user, all that is needed for data transfer is a sufficient
18 electrical connection. No attention needs to be paid to how long the wire is running from
19 the I-button, or how conductive the paint used to complete the connection is; the data is
20 passed by code not voltage output.

8.8 The Field Slab Concrete Mixture Design

A concrete that was similar to a typical Indiana Department of Transportation (INDOT) paving mixture was used (Table 8.3). The mixture contained entrained air in the range of 5-8% by volume. As it can be seen from Table 8.4, the amount of entrained air present in the slab is on the high side of this range at 8 %. The mixture had a water to cement ratio of 0.42 with approximately 61 % aggregate by volume. This relatively high paste content made the concrete have a consistent 6" slump using the standard ASTM C-143.

Table 8.3: Field Slab Mixture Proportions by Mass in SSD Condition

Constituent	Mass (kg/m ³)
Coarse Aggregate (IN #8)	944.6
Fine Aggregate (IN #23)	786.6
Cement (Type 1)	464.5
Water (Public Source)	195.4
Air Entrainment (Micro-Air)	0.3

Table 8.4: Field Slab Mixture Proportions by Volume

Constituent	Volume (%)
Coarse Aggregate (IN #8)	33.1
Fine Aggregate (IN #23)	27.7
Cement (Type 1)	13.7
Water (Public Source)	18.1
Air	8.0

8.9 Field Conditions

Figures 8.14 to 8.17 provide an illustration of the temperature, rainfall, relative humidity, and wind speed measured at the slab locations. It can be seen that during the time of testing the temperature of the air underwent a fluctuation from 30 °C to -20 °C. It can be noticed that the temperature swing of approximately 20 °C is not uncommon. It can be seen that during the time of monitoring there were approximately 30 weather events that resulted in precipitation. While the average relative humidity in the state of Indiana is 65 % the humidity can change rapidly and swings over a wide range. This implies that one can expect substantial drying and wetting hysteresis. It was also observed that a reasonable maximum wind speed for design would be 20 km/hr however specific days may exist when the winds gust higher (e.g., during storms). This data will be used to further examine the potential correlations with measured slab behavior.

Figure 8.14: Temperature Response from the Weather Station

Figure 8.15: Rainfall Response from the Weather Station

Figure 8.16: Relative Humidity Response from the Weather Station

Figure 8.17: Wind Speed from the Weather Station

The following section describes the relative humidity that was measured in the slabs. Two items should be noted however. First, many of the sensors malfunctioned. This points to serious questions with relying on the use of these sensors to provide reliable results when they are placed in a slab for a long time exposure. It is believed that the main reason for the malfunction was related to the potential for water condensation in a high pH environment around the sensors. In addition, the sensors demonstrated drift over time and needed to be recalibrated. This was done by removing the sensors from the slab and recalibrating them and then reinserting them in the slab. The I-buttons were removed from their cavities inside the slabs and recalibrated over the same saturated salts as referenced earlier. An example of the recalibration can be seen in Table 8.5. A post-experiment calibration correction was applied to all of the data collected in the entire experiment.

Table 8.5: Recalibration of an I-button after the Test was Finished (i.e, Post Experiment Calibration Correction)

Salt	Theoretical	Measured
KCl	84.6	91.4
NaBr	58.2	68
NaCl	75.4	84.2

The following section provides results from the humidity sensor measurements taken from the slabs (Figure 8.18 to 8.23). It can be observed that even in the slab that was exposed to a constant drying of 50 % relative humidity that the humidity in the core

1 of the slab remains relatively high dropping to only 75 % RH 12 mm from the surface
2 while the core remains above 90 % RH. The covered slab shows similar behavior with
3 the sensor near the surface remaining at a slightly higher relative humidity (i.e., 80 %)
4 which is consistent with the fact that the average relative humidity in Indiana (65 %) is
5 higher than the humidity in the chamber (50 %). Similar behavior was observed with the
6 exposed vertical surface however the relative humidity appeared to increase at the end of
7 the test possibly due to rewetting. The remaining slabs consisted of horizontal surfaces
8 placed on a drainable or undrainable base or a slab that was submerged. In each of those
9 cases the relative humidity was higher (> 90 %, 95 % and 100 % RH) than the constant
10 drying, non-exposed vertical surface and exposed vertical surface. As expected the
11 horizontal surfaces are more likely to collect water than the vertical surface causing the
12 RH to be higher. The sensors in the submerged sample all failed or read slightly higher
13 than 100 % relative humidity suggesting that the concrete was on its way to saturation. It
14 was slightly surprising to see the drainable and undrainable bases providing similar
15 results.

1

2

Figure 8.18: Relative Humidity Measured in the Concrete in the 50 % Environment

3

4

Figure 8.19: Relative Humidity Measured in the Concrete in the Covered Slab

Figure 8.20: Relative Humidity Measured in the Concrete in the Exposed Vertical Slab

Figure 8.21: Relative Humidity in the Concrete in the Slab on a Drainable Base

1

2

Figure 8.22: Relative Humidity in the Concrete in the Slab on an Undrainable Base

3

4

Figure 8.23: Relative Humidity Measured in the Concrete in the Submerged Slab

8.10 Summary and Conclusions

This chapter has described the development and testing of concrete samples that were used to monitor field concrete that may be exposed to a variety of environmental conditions. As a part of this study five slabs were placed outdoors and one slab was maintained in an environmental chamber at constant conditions. The slabs tested in this program were used to examine different exposure conditions including: a covered surface, an exposed vertical surface, a horizontal surface on a drainable base, a horizontal surface on a non drainable base, a completely submerged sample and a slab stored at 23°C and 50% relative humidity. The design of the samples is discussed as well as the evaluation and use of relative humidity sensors. Not surprisingly, the sensors nearest the surface showed the greatest variations in temperature and relative humidity. The main conclusions of this research are that for slabs with a surface protected from direct moisture experienced a relative humidity between 75 % and 100 % while samples that were in direct contact with moisture had a much higher relative humidity. This has implication on the water absorption and diffusion that may be expected in the field.

CHAPTER 9: SUMMARY, CONCLUSIONS AND RECOMMENDATIONS

9.1 Introduction

This research addressed several issues raised concerning the evaluation of transport properties on concrete pavement in the state of Indiana. This chapter provides a summary of the research study including the results of an extensive literature review and analysis of the experimental results. The results of this study will be used along with results of the Pooled Fund Study that finishes in 2012 to develop and establish new testing procedures.

9.2 Summary and Conclusions from Experimental Studies

This report addresses several issues raised concerning the appropriate evaluation of transport properties on concrete pavement in the state of Indiana. This chapter provides an overall summary of the study including the results of an extensive literature review and analysis of the experimental results.

9.2.1 Transport Properties on Concrete Pavement from the State of Indiana

The properties of currently used Indiana concrete mixtures were evaluated in terms of water absorption, porosity, and electrical conductivity. A series of concrete paving mixtures were tested to provide a range of values that were typical for the state of

1 Indiana. While similar mixture proportions were used for the mixtures in Indiana
2 differences in the magnitude of water absorbed occurred.

3 9.2.2 Water absorption on Mortar Samples Cast on Laboratory

4 A series of mortars were tested to illustrate the effect of sample conditioning,
5 water to cement ratio, and paste volume on the water absorption test. It was observed
6 that the water absorption test is considerably affected by the conditioning of the samples
7 before starting the test, which if not properly accounted for can lead to a
8 misunderstanding of the actual absorption behavior.

9 The conditioning procedure described in ASTM C1585-04 is not able to eliminate
10 the “moisture history” of the samples, and thus can lead to a misunderstanding of the
11 water absorption test results, especially in field samples which have obtained a lower
12 relative humidity. It is recommended that field samples which have dried be saturated
13 prior to being exposed to the conditioning regimen of ASTM C1585.

14 As an alternative to the conditioning procedure described in ASTM C1585-04, in
15 this study samples were also allowed to conditioning for over 14 months at three different
16 relative humidities: 0 % (oven dry at 105 °C), 50 %, 65 % and 80 %. Results show that
17 samples conditioned at a 50 % relative humidity can show a total absorption that is
18 approximately six times greater than similar samples conditioned at 80 % relative
19 humidity. Initial sorptivity, secondary sorptivity and total absorption at 8 days for
20 samples conditioned between 50 % and 80 % relative humidity show a linear trend
21 related to the w/c and the relative humidity at which samples were conditioned. However
22 samples that are conditioned by drying in an oven at 105 °C do not follow the same trend

1 as samples conditioned in other approaches. This is attributed to two factors: emptying of
2 a wider range of pores, and the potential for microcracking. Therefore, oven drying
3 samples is not recommended as a proper conditioning procedure for this test. Currently
4 work is examining the use of ovens at 50 °C and different relative humidity and the
5 results will appear in the Pooled Fund Study.

6 Comparing samples containing different volumes of aggregate can also lead to a
7 misunderstanding of the actual absorption behavior. Samples containing higher volumes
8 of cement paste will absorb more water. When the results are normalized by the volume
9 of cement paste, the sample containing lower volumes of cement paste will absorb more
10 water. However, for the materials examined in this study, this difference can be mainly
11 explained by the amount of water absorbed by the aggregates in the sample.

12 9.2.3 Fluid Absorption on Concrete Samples Using Deicing Solutions

13 This portion of the report describes experimental results from aqueous solution
14 absorption measurements and drying measurements for concrete in the presence of
15 deicing solutions. It was observed that the deicing solutions reduce the rate of fluid
16 absorption. This reduction can be related to the square root of the ratio of surface tension
17 and viscosity of the absorbed solutions. It was also noted that the drying of concrete
18 containing aqueous solutions (with deicers) differs from that of water. The equilibrium
19 relative humidity of the aqueous solution used as absorbent fluid plays an important role
20 on limiting drying.

21 According with these observations, the presence of deicing salts in field samples
22 may considerably impact the fluid absorption when field samples are tested in the lab

1 using water. This suggests that care must be taken in analyzing field concrete exposed to
2 deicing salt solutions.

3 9.2.4 Automated Water Absorption Test

4 ASTM C1585-04 is currently used to determine the rate of water absorption of
5 water by hydraulic cement concretes. While this test is easy to perform, it can be time
6 consuming and can require the timing of data collection to be altered if fluids other than
7 water are used.

8 This study described a procedure which allows for continuous and automated data
9 acquisition. This enables data to be collected at a much more frequent rate and eliminate
10 the influence of the operator. This additional data can provide information for further
11 analysis of the absorption behavior of the samples.

12 Both the standard and automated procedures were performed on different
13 specimens. The proposed procedure yield reproducible values of rates of absorption that
14 are similar to those of the ASTM standard test.

15 9.2.5 Electrical Conductivity on Concrete

16 Electrical conductivity tests were performed as a potential method to develop the
17 understanding of rapid test techniques for quality control. This method is directly
18 applicable to concrete in three ways: 1) wenner probe measurements, 2) direct current
19 measurements, and 3) embedded sensors. This study used a modified parallel law to
20 relate the electrical conductivity to the pore volume, pore solution conductivity and the

1 tortuosity through the pore network. The influence water addition was able to be
2 determined using electrical conductivity. The results also indicated sensitivity to the
3 volume of paste. In addition, the pore solution was observed to be approximately linearly
4 related to the degree of hydration. It was also shown that it is critical that a correction be
5 applied to samples tested at different temperatures. An activation energy of conduction
6 was observed that was approximately 10 kJ/mol irrespective of water to cement ratio from
7 the cement tested.

8 9.2.6 Effect of Exposure Conditions

9 In addition to the measurement of transport properties, the relative humidity and
10 temperature were assessed for concrete exposed to different field conditions. The
11 samples considered in this investigation included a sample stored at 50 % relative
12 humidity, covered concrete, a concrete with an exposed vertical surface, a concrete on a
13 drainable base, a concrete on a non-drainable base, and concrete that was submerged in
14 water. The samples showed that for practical field samples the relative humidity in the
15 concrete was always above 80% for the samples tested. The samples that were exposed
16 to precipitation events demonstrated higher relative humidities and approached 100 %.

17 9.3 Recommendations and Future Work

18 1) If ASTM C1585 is used by INDOT for mixtures that may have dried, it is
19 suggests that the samples be saturated prior to test.

- 1 2) If concrete is tested from the field that contains salts, the influence of salts on
2 fluids transport should be considered.
- 3 3) Electrical methods show promise as a rapid QC/QA technique. They are sensitive
4 to w/c and paste content.
- 5 4) The results of this study will be used along with results of the Pooled Fund Study
6 that finishes in 2012 to develop and establish new testing procedures.

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