

EVALUATION AND ENHANCEMENT OF CURING EFFICIENCY OF JOINTS IN CONCRETE PAVEMENTS

by

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ABSTRACT

The concrete hardening process should take place in a manner that provides optimum hydration development, which can be achieved through proper curing practices. Curing of concrete is essential at controlling its mechanical and durability performance during service, however, premature deterioration at joints presents a critical durability issue of concrete pavements associated with considerable repair costs. Durability of concrete exposed to aggressive environments depends mainly on the penetrability of its pore structure. Absorption has been used as an important indicator for quantifying the durability of bulk concrete, while the reliability of current absorption test methods with respect to curing efficiency and geometry of joints in concrete pavements is still unexplored. Curing efficiency of joints at early-age may be compromised due to uncontrolled evaporation resulting from saw-cutting processes. Therefore, providing an optimum curing and monitoring its efficiency by a real-time continuous measure is appealing. Also, a quantitative model of unsaturated flow ingress with respect to curing applications may provide a holistic understanding to predict the durability of concrete pavements. Therefore, this thesis aimed at assessing the effect of different curing compound applications on concrete pavements and overfilling joints with curing compound immediately after saw-cutting on improving the quality of concrete microstructure. Also, an effort was made to develop a customized test protocol for determining the absorption capacity of joints in concrete pavements. In addition, this thesis explored the correlation between the dielectric response of real-time sensor embedded in concrete with hydration development of paste as well as setting time. Moreover, this thesis investigated and developed an analytical model based on Katz-Thompson relationship to determine the absorption capacity of joints in

concrete pavements according to an absorption test customized to the joint geometry of pavements.

This thesis program involved experiments on cores extracted from constructed field pavement sections and laboratory slabs, and specimens produced from similar concrete under laboratory conditions. Absorption, rapid chloride penetrability, maturity, thermogravimetry, mercury intrusion porosimetry, and scanning electron microscopy tests were conducted on cores/specimens. Also, the absorption trends were modeled based on the unsaturated flow theory with 3D finite element software. The results indicated that applying a thorough coat of curing compound and overfilling the joints with curing compound immediately after late saw-cutting significantly improved the microstructure and durability of joint zones in concrete pavements. The results also revealed that the proposed absorption protocol was efficient, robust and reliable in reflecting the physical features of the microstructure of field pavement sections, including joint locations. In addition, the results showed that the dielectric response of concrete is strongly correlated to the hardening threshold and strength/hydration development of concrete, and thus it may be potentially used as a field indicator for these parameters. Finally, the results indicated that the unsaturated flow model reliably simulated fluid transport at joint locations in concrete with accurate predictions relative to experimental results.

CO-AUTHORSHIP

This thesis has been prepared in accordance with the regulation of integrated-article format stipulated by the Faculty of Graduate Studies at the University of Manitoba. Substantial parts of this thesis were either published in or submitted for publication to peer-reviewed technical journals and international conferences as well as technical and research reports as shown below:

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NOMENCLATURE

The following symbols were used in this thesis:

A	cross-sectional area of flow (m^2);
a	cross sectional area of the exposed surfaces (mm^2);
AAE	average absolute error;
A_t	percentage of water absorption at a certain point of time;
c_i	calculated absorption values (mm^2);
D	non-steady-state migration coefficient ($\text{m}^2/\text{s} \times 10^{-12}$);
d_c	critical pore diameter (m);
d_{max}^e	$0.34d_c$;
dL	length (m);
dP	pressure loss over a flow path of length (Pa);
g	$9.81 \text{ (m/s}^2\text{)}$;
I	empirical parameter for the interconnectivity and tortuosity of pores;
I_a	absorption depth of concrete at a certain point of time (mm);
k	intrinsic permeability (m^2);
K_s	saturated permeability (mm/h);
$K(\theta)$	unsaturated capillary conductivity (mm/h);
L	thickness of the specimen (mm);
M_{DRY}	mass of dry specimen before immersion (g);
M_{WET}	mass of wet, but surface-dry, specimen (g);
$M(t)$	temperature-time factor at age t ($^{\circ}\text{C}\cdot\text{h}$);
m_i	measured absorption value (mm);
m_t	difference between wet mass and dry mass at a specific point of time (g);

n	curve fitting parameter;
n_D	number of data points;
$S(d_{max}^c)$	fraction volume of pores larger than d_{max}^e ;
T	average value of initial and final temperatures in the anolyte solution (°C);
t	elapsed time (s);
T_a	average concrete temperature during the time interval Δt (°C);
T_o	datum temperature (°C);
Q	discharge (m ³ /s);
r	pore radius (m);
V	applied voltage (V);
WA	percentage of water absorption at one minute;
x_d	average value of penetration depth (mm);
α	curve fitting parameter (mm ⁻¹);
β	contact angle (deg);
γ	surface tension (N/m);
Δt	time interval of curing (h);
η	dynamic viscosity of fluid (Pa.s);
θ	normalized moisture content;
θ	moisture content per unit volume of dry medium (mm ³ /mm ³);
θ_r	residual moisture content (mm ³ /mm ³);
θ_s	saturated moisture content (mm ³ /mm ³);
ρ	density of the fluid (kg/m ³);
σ	bulk conductivity (S/m);
σ_0	pore solution conductivity (S/m);

φ	total porosity;
χ	absorption depth (m); and
$\psi(\theta)$	capillary potential (mm).

CHAPTER 1: INTRODUCTION

1.1 Deterioration of Joints in Concrete Pavements

During early-age, tensile stresses may develop in concrete pavements due to multiple factors including thermal stresses and restrained shrinkage, resulting in random cracking. This phenomenon can be controlled by saw-cutting about one-third of pavement depth to create a plane of weakness and force the concrete pavement to crack at desired locations. However, premature deterioration near to and at the joints still presents a critical durability issue of concrete pavements associated with considerable repair costs (Sutter et al. 2006; Leech et al. 2008). The damage at the joints can be problematic because it compromises the performance (e.g. ride quality) and potentially service life of otherwise sound pavement sections. The United States Federal Highway Administration (FHWA) estimates that \$85 billion would be needed in annual capital investment up to 2028 to be directed towards improving the physical condition of existing road assets in order to achieve the Department of Transportation's state of good repair benchmark for a better ride quality (ASCE 2013). Joint deterioration in concrete pavements is not only attributed to design factors such as thickness, dowels or joint spacing but also is considered as one of the most serious materials-related distresses which triggers key durability issues for concrete. For example, joint deterioration may initiate due to environmental effects such as freeze-thaw damage, especially with the application of de-icing salts in cold regions and corrosion of steel reinforcement/dowels (Litvan 1976; Şahin et al. 2010). The distress is usually observed as shadowing when micro-cracking near the joints (e.g. due to saw-cutting) traps water. Later, the development of cracking parallel to the joints tends to cause disintegration and spalling of concrete. Joints with their unique geometry of width and depth are vulnerable locations

to a higher degree of infiltration. These locations facilitate two-directional absorption, from vertical joint wall and top surfaces near the joint, at each side of the joint, which can result in a high degree of saturation with salt solutions (especially in cold regions where de-icing salts are frequently applied to pavements in winter), and in turn aggravate premature degradation of concrete pavement at joint locations.

Concrete pavements are particularly susceptible to moisture loss via evaporation during the hardening process due to their high surface-to-volume ratio. Moreover, drying of the concrete surface and thermal gradient at early-age may lead to a network of shrinkage and thermal cracks. Therefore, curing compounds are typically applied to the surface of concrete pavements immediately after placement and texturing when bleeding is complete [roughly at initial setting] (Taylor et al. 2006; CW3310–R17 2015). On the other hand, any deficiency in curing of concrete pavements may cause insufficient hydration of the cementitious matrix, which may lead to forming a continuous pore structure within the surface, facilitating the penetrability of moisture carrying aggressive agents (e.g. de-icing salts), and thus adversely affecting the long-term performance of concrete (Bentz and Jensen 2004). The behaviour of concrete exposed to aggressive environments (e.g. de-icing salts and freezing-thawing cycles) depends on the mode of transport of moisture and ionic species through the pore system, which indicates the ease of saturation of concrete. Absorption is one of the key transport properties, as it implicates the porosity of concrete surface as well as the general characteristics of pore structure and it has been used as an indicator for assessing the durability of concrete (Bentz et al. 2001; Neithalath 2006). Standard test methods for absorption (ASTM C1585 2013; ASTM C642 2013; NBN B15-215 1989; BS 1881-122 2011) can give a quantitative indication of transport properties of concrete; however, such tests have been criticized for being highly influenced by the

sampling location and their sample pre-conditioning procedures that may cause insufficient or excessive level of dryness (Castro et al. 2011) which may affect their reliability to differentiate among concretes with variable qualities.

Concrete at newly formed joints is susceptible to uncontrolled evaporation since, in current practice, no remedial action is pursued curing of these surfaces. Therefore, it is most likely that the concrete pavement's curing efficiency is compromised at the joint locations. Saw-cutting in concrete pavements is generally applied through two methods; conventional and early-entry cuts. In conventional sawing operation, the joints are usually cut with water-cooled blades. However, in the early-entry cut, as the device is much lighter than the conventional saw equipment, the saw cutting process can be applied to concrete at an earlier age when the concrete is not too hard and thus the blade does not require water for cooling. This would be done as soon as the concrete has sufficiently hardened to avoid raveling, whereas for the conventional method it may extend up to 24 hours after casting. Hence, joint walls can be exposed significantly sooner in early-entry cut than the conventional method (about 20 hours), which makes concrete of joint walls at much higher risk of evaporation.

1.2 Need for Research

Despite the current knowledge, standards and specifications for concrete, numerous cases of premature failure and costly rehabilitation of joints in concrete pavements serving especially in cold climatic regions have been reported (Sutter et al. 2006; Leech et al. 2008). It seems that there is a gap between practical research outcomes on the topic and the actual practices in the field. In addition, current knowledge on evaluating and enhancing joints curing efficiency at early-age of concrete pavements appear to be inadequately addressed in the scientific literature. For example, there is still limited field data on the effect of

applying different rates or multiple coats of curing compounds on the quality of concrete, especially with respect to concrete pavements and joints. Hence, the degree of curing efficiency of applying multiple coats of curing compounds on concrete pavements, and whether this practice can significantly enhance their durability properties remain uncertain. This is a critical question to a number of transportation agencies including Public Works Department, City of Winnipeg linked to changing current construction specifications and associated with a considerable increase of initial construction costs.

In current practice, vertical joint walls due to saw-cutting are exposed to ambient conditions at early-age, as curing precedes saw-cutting (Taylor et al. 2006). Creating and exposing these surfaces at early-age in the absence of curing, make them susceptible to uncontrolled evaporation, especially in hot, arid and windy conditions. Later on during service, these critical surfaces facilitate absorption, which may result in higher degree of saturation with salt solutions (especially in cold regions where de-icing salts are frequently applied to pavements in winter), and in turn aggravate degradation of concrete in these locations. Currently, there are no test procedures available to assess the fluid absorption capacity of concrete with respect to the unique geometry of joints in concrete pavements. Neither the one-dimensional sorptivity test nor the full immersion tests replicate the configuration of joints in concrete pavements; moreover, the fluid specified in these absorption tests is pure water, which does not correlate to field conditions in cold regions. This warrants focused investigation to develop an absorption test method customized to the geometry of joints in concrete pavements. In addition, joints can be exposed significantly sooner in early-entry cut than the conventional method which makes the concrete at the joint walls at much higher risk of evaporation. Yet, limited studies have been focused on investigating the effect of saw-cutting on concrete pavements at joint

locations (Vepakomma et al. 2002; Krstulovich et al. 2011). Such studies are needed to understand the effect of the saw-cutting method on the long-term durability of concrete pavements at the joint locations.

In the field practice, concrete at the fresh state may experience thermal gradients and heat exchange due to ambient conditions resulting in different levels of maturity. Since continuous techniques such as electrical methods can be used to study the cement hydration in-situ, several attempts have been reported to address dielectric properties of fresh or hardened cement paste or concrete (Camp and Bilotta 1989; Gu and Beaudoin 1996; Zang et al. 1996; Korhonen et al. 1997; van Beek et al. 1997; Sun 2008; Lee and Zollinger 2012). It was reported that the dielectric constant changes during the hydration process reflecting the changes of the bonding state of cement paste and hence it can be used as a measure of the change of hydration products (McCarter and Afshar 1988; Camp and Bilotta 1989). In most of the aforementioned studies, laboratory specimens were sealed to avoid any loss of water during the curing period. However, regardless of the curing method, in-situ concrete might suffer from some evaporation through the surface, even if the rate of evaporation is below the critical limit [0.50-0.75 kg/m²/h] (ACI 308R 2016). In addition, to the best of the author's knowledge, there have been no studies directed towards relating the dielectric properties of cement paste during hydration with the corresponding degree of hydration.

Finally, numerical simulations have been established to understand the effect of fundamental properties of medium and fluid on understanding flow through complex geometries (Therrien and Sudicky 1996; Wallach and Parlange 1998; Dershowitz and Fidelibus 1999). The concept of sorptivity has been developed from the theory of unsaturated flow originally for water transport in soil physics (Richards 1931), and it was used to describe the process of capillary water absorption in many porous building

materials (Hall et al. 1983; Hall et al. 1984; Carpenter et al. 1993). Since capillary conductivity is strongly dependent on water content (Hall and Hoff 2012) and it is usually nonlinear for most porous materials (Song et al. 2011; Hall and Hoff 2012), there is no analytical solution for the unsaturated flow equation (Richards 1931). Nevertheless, there is a numerical solution proposed originally for soil physics (Muallem 1976; van Genuchten 1980; van Genuchten and Nielson 1985) tailored for porous building materials. For this numerical solution, determining water retention curve (WRC) and permeability of medium are necessary. It was reported the method of water sorption isotherms used to determine WRC for concrete (Baroghel-Bouny et al. 1999) is unreliable at high humidity levels (Gregg and Sing 1982). In addition, it is a time-consuming procedure (about 2 years) for concrete samples to reach equilibrium moisture content (Leech et al. 2006) which makes it impractical for evaluating the transport characteristics of concrete at early-age. Also to determine permeability through permeability cell method, there are several parameters that must be addressed such as drying cycle that may increase the concrete permeability as much as two times that of the undried sample due to micro-cracking originating from the drying procedure (Powers et al. 1959). Moreover, for concrete mixture designs with low water-to-binder ratios (w/b), it is challenging to make the specimen saturated prior to the test. In addition, applying adequate confinement pressure is necessary to prevent leaking that may result in loss of exerted pressure (Scherer et al. 2007). Furthermore, water permeability tests are laborious to perform in terms of time and required equipment, which makes other testing techniques (e.g. absorption) appealing.

1.3 Objectives

To respond to the aforementioned research needs, the fundamental theme of this thesis was to improve the current level of knowledge on the efficiency of curing of joints in concrete

pavements to minimize the risk of premature deterioration. The specific objectives of this thesis are to:

- Investigate the effect of different rates of curing compound applications on the penetrability of field concrete pavements, especially at joint locations, to project their long-term durability.
- Develop a customized test protocol for determining the absorption capacity of joints in concrete pavements, to help transportation agencies reliably evaluating and projecting the longevity of these critical locations in concrete pavements.
- Assess and evaluate the effect of different saw-cutting methods and remedial curing procedures on the long-term durability of concrete pavements at the joint locations in the field.
- Explore the concept of dielectric response of concrete and establish its correlation to setting and hydration of concrete, in order to use it as a non-destructive means for reflecting the efficiency of curing in concrete pavements.
- Develop an analytical model based on the unsaturated flow theory to determine the absorption capacity of joints in concrete pavements.

1.4 Scope of Research

To achieve the aforementioned objectives, the scope of the work involves field work, laboratory work, and modeling as described below:

- Investigating the effect of three different curing compound applications (no curing compound, one coat of curing compound, and two coats of curing compound) on the behaviour of concrete pavements in terms of their resistance to infiltration of fluids and microstructural evolution to project their long-term durability in trial field sections constructed in Winnipeg, Canada.

- Develop a customized test protocol for determining the absorption capacity of joints in concrete pavements by defining and evaluating parameters such as conditioning, type of fluid, and surface area through different procedures.
- Evaluating the effect of two different saw-cutting methods, conventional and early-entry cuts, on the long-term durability of concrete pavements at the joint locations in trial field sections constructed in Winnipeg, Canada.
- Studying the effect of overfilling joints with curing compound (remedial procedure) immediately after saw-cutting (early- and late-cuts) on improving the quality of concrete microstructure at joint locations in trial field sections constructed in Winnipeg, Canada.
- Studying the dielectric response of developed real-time sensor embedded in concrete during hydration to assess the hydration development of paste in concrete as well as correlate it to setting time of concrete to use it as a benchmark for determining the saw-cutting time in the field.
- Develop an analytical model based on the unsaturated flow theory and Katz-Thompson relationship to determine the absorption capacity of joints in concrete pavements according to an absorption test customized to the joint geometry of pavements.

1.5 Structure of the Thesis

This thesis consists of eight chapters as described below:

- Chapter 1 provides a brief description of the problem statement and the significance of the proposed research.

- Chapter 2 provides a comprehensive literature review on different aspects of concrete pavements including curing, saw-cutting of joints, absorption tests, dielectric response of concrete and absorption modeling.
- Chapter 3 involves experiments on cores extracted from recently constructed pavement sections in comparison to corresponding specimens produced from similar concrete under laboratory conditions.
- Chapter 4 focuses on developing a customized test protocol for determining the absorption capacity of joints in concrete pavements which involves three phases and thorough statistical analyses of results.
- Chapter 5 evaluates the effect of overfilling joints with curing compound immediately after saw-cutting (early- and late-cuts) on improving the quality of concrete microstructure at joint locations in laboratory slabs and trial field sections.
- Chapter 6 investigates the optimum frequency needed to determine the dielectric response of concrete by gauging its sensitivity to different curing conditions of concrete and correlates the dielectric response of a real-time sensor to setting time and strength/hydration development of concrete mixtures with a range of reactivity.
- Chapter 7 focuses on developing an analytical model based on Katz-Thompson relationship to determine the absorption capacity of joints in concrete pavements according to the absorption test customized to the joint geometry of pavements developed in Chapter 4.
- Chapter 8 contains a summary of the major findings and significant conclusions of this thesis as well as recommendations for future research.

CHAPTER 2: LITERATURE REVIEW

This chapter provides a focused literature review on topics related to the subject matter of this thesis and its scope of work.

2.1 Curing of Concrete Pavements

The aspect of curing involves keeping concrete saturated as much as possible, till water-filled space in fresh concrete has been occupied by cement hydration products to the desired extent (Neville 2011). This can be done by maintaining a satisfactory level of moisture content (RH above 90%) and temperature in concrete for a period of time (preferably seven days or more) immediately following placing and finishing procedures to attain the desired properties. For example, properly cured concrete should achieve target strength, abrasion resistance, volumetric stability, water tightness and resistance to freeze-thaw cycles. The maximum accessible degree of hydration in concrete is heavily dependent on water to cement ratio (w/c) and water availability (Mills 1966). The consumed water during the hydration of portland cement is considered as the sum of incorporated physically adsorbed water onto the gel surfaces and chemically bound water into the hydration products (Neville 2011; Mindess and Young 2002; Taylor 1997). Curing of concrete should be applied as soon as the concrete surface starts to dry when the rate of evaporation is higher than that of bleeding water rises to the surface of concrete (Kosmatka 1994; AL-Fadhala and Hover 2001). Therefore, the time of curing application depends on bleeding characteristics of concrete as well as environmental conditions and rate of evaporation. The rate of evaporation depends on relative humidity, wind speed, concrete temperature, and also energy radiation through sunlight (ACI 308R-16 2016). Bleeding rate and duration are influenced by concrete mixture design, concrete thickness and method of consolidation

(Kosmatka 1994; Suprenant and Malisch 1998). As concrete reaches its initial setting, the rate of bleeding diminishes and the concrete surface eventually will be dried even under low evaporation rates (AL-Fadhala and Hover 2001). If the concrete surface dries out before the initial setting and finishing operation (due to low bleeding or high evaporation rates or both), it is necessary to decrease moisture loss through initial curing methods such as fogging or using sunshades (ACI 308R 2016).

Water curing of concrete is applied by different methods such as ponding or immersion, fogging, wet coverings, and membrane (e.g. with curing compounds). The ponding method is considered as water curing which consists of immersion of concrete after final setting in water. This method can be used for slabs, culverts, floors, bridge decks, and pavements. However, this method is seldom used as concrete surface may get weak due to dilution of paste (Van Aardt 1953). Fogging or sprinkling by sprays or lawn sprinklers is an effective curing method which is applied when concrete reaches its final setting. Wet covering techniques (absorbent materials, cotton mats, and burlap) can keep water even on the vertical surfaces of concrete. Some absorbent materials consist of vapor barriers which prevent moisture loss and provide enough absorbent resulting in effective curing (Hover 2002). However, membrane (e.g. with curing compounds) is considered as the most efficient and practical procedure for fast construction of concrete elements such as floors, pavements, and sidewalks, that provides significant economic benefits for especially in water-scarce regions (Mehta and Monteiro 2014). Also, it was reported that applying curing compound on concrete may lead to less plastic and drying shrinkage compared to wet covering techniques (Al-Gahtani 2010). Concrete pavements are typically cured by membrane-forming curing compounds (Figure 2.1) that usually consist of a resin or wax dissolved in a solvent or emulsified in water. They are applied to pavement surface,

and by evaporation of the solvent, the wax or resin forms an impermeable membrane to help retaining moisture in concrete. For an efficient coverage, the curing compound application is required to take place immediately after final finishing when the bleeding water evaporates (ACI 308R-16 2016). Delayed application of curing compound increases the chance of absorption into the surface and failure to form a membrane. In North American standards for concrete (CSA A23.1/A23.2 2014; ACI 308R-16 2016), curing compounds are specified by AASHTO M148 (2005) and ASTM C309 (2011). Applying an adequate rate of curing compounds and providing a uniform coverage onto concrete are required to ensure efficiency of the curing process (Vandenbossche 1999).



Figure 2.1- Applying curing compound on concrete pavement.

Curing-affected zone is defined as the concrete portion which is most influenced by the curing process (Cather 1992). The depth of this zone at the age of 28 days is varying between about 10-13 mm depending on concrete mixtures, ambient conditions, and curing

duration (Hover 1984; Dhir et al. 1991; Cather 1992). Curing efficiency highly affects concrete properties in the curing affected zone. While the ultimate goal of applying curing is considered as developing concrete properties, investigating the effectiveness of curing is necessary to assure whether this goal is achieved (Wainwright et al. 1990). Improper curing of concrete causes insufficient hydration of the cementitious matrix, which is associated with performance risks during service; for example, inadequate curing of concrete pavements may lead to forming a continuous pore structure within the surface, which facilitates the penetrability of moisture carrying aggressive agents (e.g. de-icing salts), and thus provoking durability issues such as surface scaling and frost damage (Marchand et al. 1994; Bouzoubaâ et al. 2011). It was reported without curing, the oxygen permeability of concrete in the affected zone at the depth of 6 mm is about eightfold higher than that in depth of 44 mm, Figure 2.2, (Gowriplan et al. 1990).

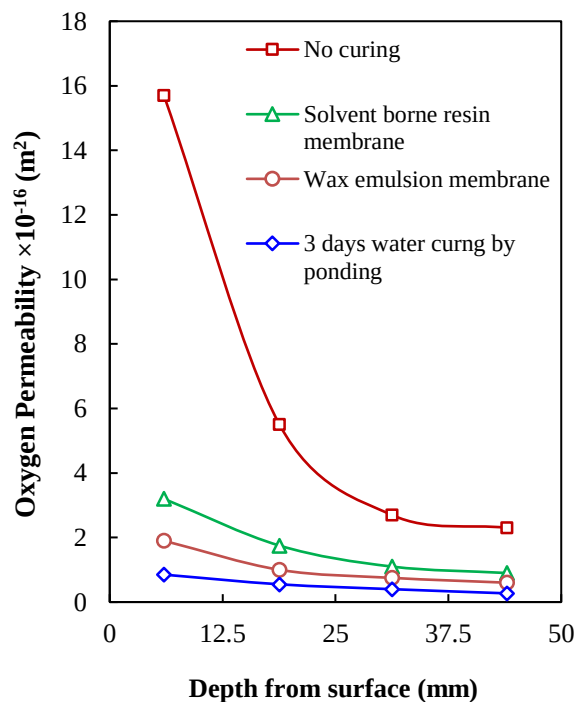


Figure 2.2- Influence of different types of curing on the concrete oxygen permeability
(adapted from Gowriplan et al. 1990).

Moreover, drying of the concrete surface at early-age may lead to a network of plastic and drying shrinkage cracks, which adversely affects the long-term performance of concrete (Bentz and Jensen 2004). In addition to the mixture design of concrete, the prevailing conditions in the field including the relative humidity, temperature, and wind velocity affect the evaporation rate of moisture from the concrete. It was reported that reduction of the relative humidity during the curing process from 100 to 94% led to a marked increase of the water absorption capacity of concrete (Ho et al. 1989), implicating the alteration of the microstructure. If the external relative humidity falls below 80% during the initial curing process, the proportion of macro-pores (larger than 0.1 μm) in the hydrated paste may significantly increase and cause higher penetrability of concrete (Mehta and Monteiro 2014).

Numerous studies reported the effect of different curing methods (e.g. water curing, covering with plastic sheets or curing compound) on hardened properties of concrete. (Marchand et al. 1994; Nassif et al. 2005; Ibrahim et al. 2013). Curing compounds can efficiently maintain the moisture content, increase the degree of cement hydration and decrease sorptivity of the near-surface layer of concrete (Ibrahim et al. 2013). It has been reported that premature deterioration of concrete pavements, especially at joint locations are strongly linked to the penetrability of concrete (MDOT 1996; Chen et al. 2011; Liu et al. 2014), which is highly affected by the efficacy of curing practices. In North America, there are recommendations to apply curing compounds at a rate of 1.5 to 2 times the normal rate specified by the manufacturer in repair applications of concrete pavements (GPDR-COW 2015) with the perception that this practice substantially improves the curing efficiency of repair concrete, and thus it may be applied to concrete pavements during the construction process. Also, it is recommended that the specified rate of application can be

achieved by two coats (each one applied at half the specified rate) to attain a uniform application of curing compound on concrete (TxDOT 2004). Yet, there are scarce field data on the effect of applying different rates or multiple coats of curing compounds on the quality of concrete, especially with respect to concrete pavements. Hence, the degree of curing efficiency of applying multiple coats of curing compounds on concrete pavements, and whether this practice can significantly enhance their durability properties remain uncertain. This is a critical question to a number of transportation agencies including Public Works Department, Winnipeg (Manitoba, Canada) linked to changing current construction specifications and associated with a considerable increase of initial construction costs.

2.2 Absorption of Joints in Concrete Pavements

Failure of concrete pavements, especially joint deterioration, is not usually attributed to design factors such as thickness, dowels or joint spacing, but it is often linked to materials distress, for example, due to environmental effects such as freeze-thaw damage, especially with the application of de-icing salts (Litvan 1976; Şahin et al. 2010). In North America, premature deterioration near to and at the joints presents a critical durability issue of concrete pavements associated with considerable repair costs (Sutter et al. 1979; Leech et al. 2008). The United States Federal Highway Administration (FHWA) estimates that \$85 billion would be needed in annual capital investment up to 2028 to be directed towards improving the physical condition of existing road assets in order to achieve the Department of Transportation's State of Good repair benchmark for a better ride quality (ASCE 2013). The damage at the joints can be problematic because it compromises the performance (e.g. ride quality) and potentially service life of otherwise sound pavement sections. This damage might be correlated to the quality of concrete in terms of its fluid transport

properties, which depend on the mixture design parameters of concrete, curing efficiency and the consequent evolution of microstructure (Taylor et al. 2006). Due to their unique geometry (width and depth), joints in concrete pavements are vulnerable locations to a higher degree of saturation, which triggers key durability issues for concrete in cold regions such as frost damage and corrosion of steel reinforcement/dowels. Thus, assessing the transport properties (e.g. absorption) of concrete pavements at joint locations may project their long-term performance.

Concrete durability depends fundamentally on transport properties, which are influenced by the pore structure (ASTM 1585 2015; Maltais et al. 2004; Sabir et al. 1998; Hearn et al. 1994; Hooton et al. 1993; Parrot 1992). Absorption is a primary mechanism of chloride ingress in the concrete, also it can be considered as an index which is well related to other durability parameters (Basheer and Cleland 2011). Water absorption has been used as an indicator for assessing the durability of concrete (Bentz et al. 2001; Neithalath 2006). It can be mathematically described with Eq. 2.1 (Kelham 1988):

$$\chi(t) = \sqrt{\frac{4k\gamma \cos(\beta)t}{\phi\eta r}} \quad \text{Eq. 2.1}$$

where χ is absorption depth (m), k is intrinsic permeability (m^2), γ is surface tension (N/m), β is contact angle (deg.), ϕ is porosity (%), η is dynamic viscosity (N.s/m²), r is pore radius (m), and t is time (s). Experimentally, it can be determined by one-dimensional absorption of an exposed surface following ASTM C1585 (2013), based on the procedure introduced by Hall (1989). In this test, a concrete specimen is conditioned (air-vacuumed for 3 hours followed by 1 hour vacuum saturation in water, dried at 50°C and 80% relative humidity (RH) for three days, and sealed for 15 days), and then the bottom surface of specimen is exposed to water to determine total absorption and sorptivity (rate of absorption). The

absorption test includes measurements of incremental mass change after the specimen comes in contact with water at relatively frequent intervals for the first six hours and successively taking one recording each day for the next eight days.

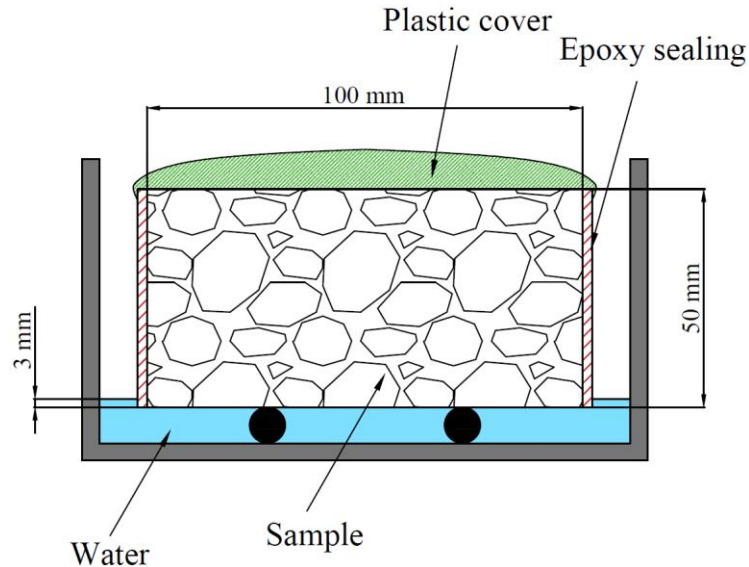


Figure 2.3- Schematic of the absorption test procedure (adapted from ASTM C1585 2013).

The amount of water absorption is normalized by the cross-section area of the sample exposed to water. The normalized values of absorbed water are then plotted as a function of square root of time. The initial and secondary sorptivity are defined as the slopes of the fitted lines during the first six hours, and between one and eight days, respectively (ASTM C1585 2013). However, the conditioning procedure may give an insufficient level of dryness and lower absorption of concrete (Castro et al. 2011), which may affect its reliability to differentiate among concretes with variable qualities. It was reported the relative humidity (RH) of concrete and its temperature are the two parameters that may highly affect the results of water absorption test (Basheer and Nolan 2001; Nokken and Hooton 2002). Variations in these parameters (RH, and temperature) during

testing may result in misrepresentative durability assessments; however, considering proper corrections to these testing conditions, water absorption can provide reliable results regarding concrete durability. Also, the one-dimensional absorption value is highly influenced by the sampling location (i.e. exposed surface to absorption) of concrete specimens/cores, and thus the sampling location should be clearly identified with absorption results. The microstructure of concrete surface may not develop to a similar extent to that of the inner core, for example, due to effects of surface temperature and moisture loss. Internal concrete generally has a higher capacity for moisture retention, which facilitates cement hydration and microstructural evolution. Therefore, absorption of the surface of the concrete is generally higher than its inner core (Senbetta and Scholar 1981; Khatib 2014).

Water absorption of concrete can also be assessed by full immersion of specimens as described in ASTM C642 (2013), NBN B15-215 (1989) and BS 1881-122 (2011). In ASTM C642 (2013), specimens with a volume of 350 cm³ are dried at 105°C until a mass change in a 24 h period is less than 0.5%; subsequently, the specimens are submerged in water until a mass change in a 24 h period is less than 0.5%. In NBN B15-215 (1989), specimens with a volume-to-surface ratio of 12 to 20 mm are submerged in water until the change in mass is less than 0.1% in 24 h period. The specimens are then dried at 105°C till the change in mass is less than 0.1 % in 24 h period. In BS 1881-122 (2011), cylindrical discs of 75 mm diameter and thickness are dried at 105°C for 72 h, followed by submersion in water for 30 min. These test methods are listed in Table 2.1. In all these immersion tests, the absorption of concrete is determined as the water uptake of concrete specimens relative to their dry mass. However, it was reported that drying at 105°C can cause severe drying shrinkage of the hydrated cement paste and decomposition of some hydration products,

which alter the microstructure of the cementitious matrix and misleadingly give high absorption values (Senbetta and Scholar 1981; McCarter et al. 1992; Parrot 1994; Bai et al. 2002).

Table 2.1: Typical concrete absorption test methods

Test	Conditioning for dry state	Duration	Disadvantage
ASTM C1585 (2013)	3h dry vacuuming, 1h water saturation vacuuming, 3d drying at (50°C, 80% RH), 15d sealed in 23°C	8d	Insufficient level of dryness, Unidirectional flow
ASTM C642 (2013)	Drying at 105°C	Minimum 1d	Severe drying may alter hydrated cement paste
NBN B15-215 (1989)			
BS 1881-122 (2011)		30 min	

Concrete pavements are vulnerable to moisture loss via evaporation due to their high surface-to-volume ratio. Therefore, curing compounds are applied to the surface of concrete pavements immediately after placement and texturing when bleeding is complete (roughly at initial setting) (Taylor et al. 2006; CW3310–R17 2015). However, tensile stresses may develop in pavements during early-age due to multiple factors including thermal stresses and restrained shrinkage, resulting in random cracking. This phenomenon can be controlled by saw-cutting to create a plane of weakness and force the concrete pavement to crack at desired locations (joints). Determining the appropriate saw-cutting depth is difficult; applying deeper cuts are effective in controlling cracks; however, shallow cuts are usually considered as less expensive and faster. The saw-cutting intervals for creating transverse contraction joints in plain jointed concrete pavements is between 4.5 and 6.1 m, with a depth of one-quarter to one-third of the pavement thickness and width of 3 to 8 mm (Taylor et al. 2006). Relative to the direction of saw-cut, the joints' geometry in

concrete pavements consist of two symmetrical planes to the right and left of the centerline of each joint: the vertical joint wall and top/horizontal surface next to the joint. In current practice, these vertical joint walls are exposed to ambient conditions at early-age, as curing precedes saw-cutting (Taylor et al. 2006). Creating and exposing these surfaces at early-age (final setting) in the absence of curing, make them susceptible to uncontrolled evaporation, especially in hot, arid and windy conditions. Later on during service, these critical surfaces facilitate absorption, which may result in higher degree of saturation with salt solutions (especially in cold regions where de-icing salts are frequently applied to pavements in winter), and in turn aggravate degradation of concrete in these locations. Currently, there are no test procedures available to assess the fluid absorption capacity of concrete with respect to the unique geometry of joints in concrete pavements. Neither the one-dimensional sorptivity test nor the full immersion tests replicate the configuration of joints in concrete pavements; moreover, the fluid specified in these absorption tests is pure water, which does not correlate to field conditions in cold regions.

2.3 Curing Efficiency of Joints in Concrete Pavements

Joint deterioration in concrete pavements is considered as one of the most serious materials-related distresses which may be observed as shadowing at first, when micro-cracking near the joints traps water. Later, development of cracking parallel to the joint tends to cause disintegration at joints and loss of materials (Figure 2.4). It has been reported premature joint deterioration of concrete pavements is strongly linked to the penetrability of concrete (MDOT 1996; Chen et al. 2011; Liu et al. 2014), which is highly affected by the efficacy of curing, saw-cutting operations and the consequent evolution of microstructure even with a well-accepted concrete mixture design. It has been reported that

applying a thorough coat of curing compound at a rate of 0.23 l/m^2 is essential to ensure moisture retention in concrete pavements (elements with high surface-to-volume ratio) for efficient hydration reactions and microstructural development (Tiznobaik et al. 2015).



Figure 2.4- A sample of premature deterioration of joint in concrete pavement showing trapped water in developed microcracks parallel to the joint.

Saw-cutting in concrete pavements is generally applied by two methods; conventional and early-entry cuts. Conventional saw-cutting is usually applied as the concrete reaches final set, which can be influenced by a number of factors; concrete mixture design, including the amount and type of portland cement, supplementary cementitious materials (SCMs), and chemical admixtures, as well as external factors like ambient conditions, curing applications, and restraint conditions (Okamoto et al. 1999). In conventional sawing operation, the joints are usually cut with a water-cooled blade.

However, in the early-entry cut, as the device is much lighter than the conventional saw equipment, the saw-cutting process can be applied at an earlier age to reduce the potential risk of early-age cracking when the concrete is not too hard and thus the blade does not require water for cooling. It is recommended by ACPA (2002) that the appropriate saw-cutting depth for longitudinal joints to be one-third of pavement depth ($D/3$), and for the transverse joint to be $D/4$ in concrete pavements with dense granular subbase, while for concrete pavements on a stabilized and open-graded subbase the depth is considered as $D/3$. Early-cut should begin as soon as the concrete has hardened sufficiently to avoid raveling, which is considered the beginning of saw-cutting window (Figure 2.5). It is suggested that early-entry saws without significant raveling can be applied at about the halfway point between the initial and final setting times of concrete (Their 2005). Also, it was reported that raveling was visually acceptable when the concrete compressive strength varied from 2.1 to 7.0 MPa based on the type of aggregate in the mixture (Okamoto et al. 1994).

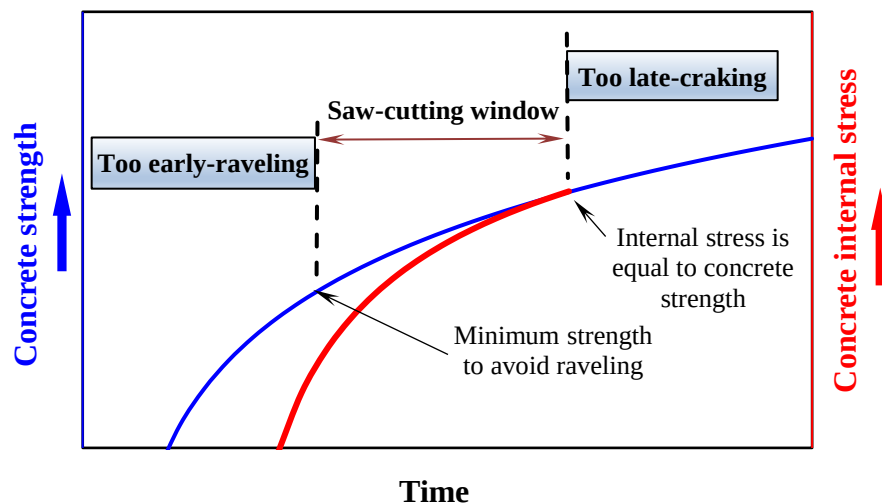


Figure 2.5- Saw-cutting window (adapted from ACPA 1997).

In practice, the time of saw-cutting is commonly determined by a professional saw operator's judgment with the aid of a simple and subjective test which involves scratching the concrete surface to see if it can maintain the surface texture. Conventional sawing windows generally begin at about the final setting time (Krstulovich et al. 2011) and may extend up to 24 h after casting (Zollinger 2001). Therefore, in practice, joint walls resulting from early-entry cuts may be exposed to uncontrolled evaporation significantly earlier than the ones from conventional sawing method. Most of the reported studies (Vepakomma et al. 2002; Krstulovich et al. 2011) focused on whether the saw-cutting methods (conventional or early-cut) were successful to avoid raveling at the joints and control random cracking in the pavement. However, limited field studies have been conducted to explore the effect of saw-cutting on concrete pavements at the joint locations with respect to curing efficiency and microstructural evolution, which warrants further investigation. Hence, the aim of this thesis was to assess whether the effect of overfilling joints with curing compound immediately after saw-cutting (early- and late-cuts) can significantly improve the quality of concrete microstructure at joint regions in laboratory slabs and trial field sections constructed in Winnipeg, Canada.

2.4 Using Dielectric Response to Evaluate Concrete Hydration Process

Isothermal conduction calorimetry has been customarily used in the laboratory to assess the hydration process by measuring the heat flux liberated from small cement paste samples. However, since it only captures the processes involving significant changes in heat, it may give limited information during the induction period, which involves the dissolution of ions from cement grains, nucleation, and growth of hydration products and initial setting. After a few days of hydration, limited heat emission at the steady-state stage

(ASTM C1679 2014) does not provide information about the development of hydration products. Moreover, the small size samples of cement paste, in the absence of fine and large aggregates which contribute to heat exchange processes, do not fully represent typical scenarios of concrete (McCarter and Afshar 1988). In the field, concrete at the fresh state (especially elements with a high surface-to-volume ratio) may experience thermal gradients and heat exchange due to ambient conditions resulting in different levels of maturity at the surface relative to the inner core. Indeed, this is different from isothermal conditions of conduction calorimetry.

Continuous techniques such as electrical methods may be used to study the hydration of cement. Dielectric response measurement is one of the methods in characterizing materials as it is easier to apply than chemical analysis methods. For example, several attempts have been reported in the literature to study dielectric properties of fresh or hardened cement paste/concrete in the laboratory (McCarter and Afshar 1988; Camp and Bilotta 1989; Gu and Beaudoin 1996; Zang et al. 1996; Korhonen et al. 1997; van Beek et al. 1997; Sun 2008; Lee and Zollinger 2012). For example, McCarter and Afshar (1988) showed that the temporal change in dielectric response of cement paste during the first day of hydration can be used to monitor the hydration progress. Gu and Beaudoin (1996) used dielectric measurements of one-year-old hardened cement paste to provide a rapid estimation of the water-to-cement ratio (w/c). Zhang et al. (1996) reported the increase of dielectric constant of cement paste with w/c, within the first 30 hours of hydration. Also, Lee and Zollinger (2012) used dielectric properties of fresh concrete to estimate the volume fraction of the different phases in hardened concrete.

Thermogravimetric analysis (TGA) is commonly used to evaluate the degree of hydration of cement paste by determining the chemically bound water which corresponds

to the mass loss of a sample typically heated up between 105°C and 1050°C (Halamickova et al. 1995; Feng et al. 2004; Gómez-Zamorano and Escalante-García 2010). The mass loss occurs through dehydration of chemically bound water in calcium silicate hydrate (C-S-H), calcium hydroxide (CH) and other hydration products (e.g. ettringite), as well as decarbonation of calcite (CaCO_3). Different references have considered the endothermic range of up to 105°C for evaporable water (Wang et al. 2004, Cassagnabère et al. 2009). There are several aspects which affect the accuracy of the obtained degree of hydration from TGA. For example, it was reported that drying samples at 105°C to eliminate evaporable water may cause severe drying shrinkage of the hydrated cement paste and decomposition of some hydration products [e.g. ettringite] (Bai et al. 2002; Snoeck et al. 2014). In addition, although the average bound water values of different types of fully hydrated portland cement pastes are in the narrow range of 0.23 to 0.25 g/g of ignited sample (Pane and Hansen 2005; Neville 2011), the hydration process in blended cement pastes comprising supplementary cementitious materials (SCMs) is more complex as hydration and pozzolanic reactions are involved in bounding water exchange. In general, thermogravimetric analysis can be used as a reasonable indication of hydration development of concrete prepared from portland and/or blended cement (Scrivener et al. 2015).

From an electrical perspective, dielectric materials can sustain a charge from an applied electric field and then transmit that energy (Halliday et al. 2013). The dielectric constant (or relative permittivity) is a physical property of materials and defined as the ratio of a material's electrical permittivity to that of free space (vacuum). Dielectric constant magnitude is identified by electrical dipole moment per unit volume of material (Halliday et al. 2013). As the dielectric property of water is very high, the dielectric properties of

building materials in a wet-state are mainly governed by the amount of water preserved (free, adsorbed or bound) in the system (van Beek et al. 1997). It is reported that re-saturation of concrete produces similar values of dielectric constant to those obtained during drying which illustrates the significant effect of absorbed water in dielectric response of a dry porous media (Camp and Bilotta 1989). The dielectric constant of water at 0°C changes from about 77 in a liquid state to 3.4 in a solid state (ice). Table 2.2 shows the dielectric constant of different materials.

Table 2.2: Dielectric constant of different materials (adapted from Lee and Zollinger 2012)

Material	Dielectric constant	Material	Dielectric constant
Air	1	HCP*	4-5
Clay	2-6	Limestone	4-8
Cement	3-4	Silt	4-8
Sand	3-6	Water	79-81

*Note: HCP is hydrated cement paste

This constant will be different in the states intermediate between water and ice, such as gels, hydrates, and interfacial water (Gu and Beaudoin 1996). Water molecule with its large dipole moment has the ability to orient in an applied electric field. During cement hydration, the water molecules change from free to bound water in several states of hydration or crystallization. As the water molecule passes from one bonding state to another, the intensity of this ability will change and affect the dielectric relaxation of cement paste (Zhang et al. 1996).

Setting/hardening of cement paste is a consequence of chemical reactions and phase transition on both grain surface and the aqueous phase. Continual monitoring of these processes may give an insight of hydration and microstructural development of concrete. Dielectric response of cement paste/concrete during hydration may reflect the changes of

bonding state of water; yet, in the previous laboratory studies in this area (McCarter and Afshar 1988; Camp and Bilotta 1989) sealed paste specimens were used to avoid loss of water during the curing period. However, in-situ concrete at fresh state is vulnerable to adiabatic conditions and perhaps uncontrolled evaporation. For example, field concrete may suffer from some evaporation through the exposed surface, even if the rate of evaporation is below a critical threshold [0.50-0.75 kg/m²/h] (ACI-308R 2016). In addition, to effectively study the dielectric response of cement paste/concrete during the hydration process, understanding its correlation with key indicators such as setting time, strength, the degree of hydration and incorporating supplementary cementitious materials (SCMs) is necessary.

Previous studies showed SCMs, such as fly ash (Class F, low calcium oxide) and nanosilica improve durability properties of concrete; however, there are some technical concerns regarding using SCMs. Fly ash retards the rate of hydration and microstructural developments at early-age resulting in low strength gain rate (Malhotra et al. 2000; Huang et al. 2013). Also, concrete pavements incorporating fly ash are vulnerable to surface scaling. These are the reasons that limit using fly ash to less than 15 to 25% replacement of portland cement in concrete pavements. For instance, Public Work Department, City of Winnipeg (CW3310–R17 2015) limits the use of fly ash in concrete pavements, sidewalks, and curbs to be optional or at a maximum replacement level of 15%. Nonetheless, these limitations can be alleviated by using nanosilica in concrete incorporating with fly ash. It has been suggested that even agglomerates of nanosilica particles (silica aggregates) can speed up the kinetics of cement hydration by nucleation and creation of additional sites for early precipitation of hydration products, resulting in shortening of setting time (Kong et al. 2013) and refining concrete pore structure (Sanchez and Sobolev 2010). For example,

incorporating nanosilica in the systems containing up to 30% fly ash (Class F) improves microstructure development and reduces the amount of calcium hydroxide (CH) phase within 28 days (Said et al. 2012).

2.5 Modeling of Fluid Ingress into Joints of Concrete Pavements

The study of capillary water absorption is considered fundamental to understanding the durability performance of porous building materials (Bentz et al. 2001; Neithalath 2006). The water sorptivity in porous building materials are typically evaluated by gravimetric measurements through determining the sample mass at progressive time intervals using unidirectional flow (ASTM 1585 2013) or immersion (ASTM C642 2013) tests. Also, advanced methods have been applied to demonstrate fluid transport experimentally in porous building materials such as X-ray attenuation and Nuclear Magnetic Resonance (NMR) (Leech et al. 2008). In addition, numerical simulations have been introduced to understand the effect of fundamental properties of medium and fluid on unsaturated flow through complex geometries (Therrien and Sudicky 1996; Wallach and Parlange 1998; Dershowitz and Fidelibus 1999). In many of the these studies, the concept of sorptivity developed from the theory of unsaturated flow originally for water transport in soil physics (Richards 1931) has been used to describe the process of capillary water absorption in porous building materials (Hall et al. 1983; Hall et al. 1984; Carpenter et al. 1993). Richard (1931) showed that the unsaturated flow equation of water can be derived from the continuity equation as follows:

$$\nabla \cdot [K(\theta)\nabla\psi(\theta)] = \frac{\partial\theta}{\partial t} \quad \text{Eq. 2.2}$$

where, θ is moisture content per unit volume of dry medium (mm^3/mm^3), $K(\theta)$ is unsaturated capillary conductivity (mm/h), $\psi(\theta)$ is capillary potential (mm), and t is elapsed time (h). Since $K(\theta)$ is strongly dependent on water content (Hall and Hoff 2012) and it is usually nonlinear for most porous materials (Song et al. 2011; Hall and Hoff 2012), Eq. 2.2, has no analytical solution. Mualem (1976) proposed an analytical model to predict the unsaturated conductivity curve by using the moisture content-capillary potential curve and saturated hydraulic conductivity:

$$K(\theta) = K_s \theta^I \left(\int_0^\theta \frac{d\theta}{\psi(\theta)} / \int_0^1 \frac{d\theta}{\psi(\theta)} \right)^2 \quad \text{Eq. 2.3}$$

$$\theta = \frac{\theta - \theta_r}{\theta_s - \theta_r} \quad \text{Eq. 2.4}$$

where, K_s is saturated permeability (mm/h), θ is normalized moisture content, θ_s is saturated moisture content (mm^3/mm^3), θ_r is residual moisture content (mm^3/mm^3), and I is empirical parameter accounting for the interconnectivity and tortuosity of pores. To ease concrete modeling the residual moisture content is reported to be considered as zero ($\theta_r=0$), which does not represent a significant modeling effect (Pour-Ghaz et al. 2009; Smyl et al. 2016). While Mualem (1976) proposed the value of I for most of the soil types to be as 0.5, it is reported I does not represent a material characteristics hence it should be considered as a fitting parameter (Kosugi 1999). Smyl et al. (2016) calculated the value of I through a model training by using maximum likelihood approach as -7.0 and -9.0 for concrete and mortar, respectively. This value is reported to be in a range of -7 to -5 and -9.0 to -8.0 for damaged concrete and mortar, respectively (Smyl et al. 2016). In order to calculate the integral in Eq. 2.3, different models were proposed for relating the moisture content to the capillary potential (Brooks and Corey 1964; van Genuchten 1980; van Genuchten and

Nielson 1985; Kosugi 1994) mainly for soil physics, as for example shown below (van Genuchten 1980; van Genuchten and Nielson 1985):

$$\theta = \left(\frac{1}{1 + (\alpha\psi)^n} \right)^{\left(1 - \frac{1}{n}\right)} \quad \text{Eq. 2.5}$$

where, both α (mm^{-1}) and n (dimensionless) are fitting parameters. This numerical model was recently adopted to study capillary suction in mortar and concrete as unsaturated porous media (Pour-Ghaz et al. 2009; Smyl et al. 2016; Smyl et al. 2017).

2.5.1 Water retention curve (WRC)

Concrete durability in an unsaturated state essentially depends on its moisture transport characteristics (ASTM 1585 2015; Sabir et al. 1998; Maltais et al. 2004). The WRC for concrete shows its capability to retain moisture at different levels of humidity which is essential in modeling a complex unsaturated medium. However, determination of complete wetting WRC is a difficult procedure. It is reported that water sorption isotherms method that is used to determine WRC for concrete (Baroghel-Bouny et al. 1999; Savage and Janssen 1997) is unreliable at high humidity levels (Gregg and Sing 1982). In addition, the water sorption isotherms method is a time-consuming procedure (about 2 years) for concrete samples to reach equilibrium in moisture content (Leech et al. 2006) which makes it impractical to evaluate the concrete transport characteristics at early-age. However, it is possible to determine the complete WRC based on Van Genuchten parameters, α and n (van Genuchten 1980) from mercury intrusion porosimetry (MIP) test (Leech et al. 2006).

2.5.2 Water permeability

Permeability is defined as the ability of a saturated porous material with a fluid medium to allow the flow of fluid passing through, under the action of a pressure differential. This

steady-state rate of flow in the porous material is directly related to the hydraulic gradient according to Darcy's law:

$$Q/A = \frac{k}{\mu} \left(\frac{dP}{dL} \right) \quad \text{Eq. 2.6}$$

where, Q is flow rate (m^3/s), A is cross-sectional area of flow (m^2), k is intrinsic permeability (m^2), η is dynamic viscosity of fluid ($\text{Pa}\cdot\text{s}$), dP is pressure loss (Pa) over a flow path of length, dL (m). Intrinsic permeability is the concept of permeability that depends purely on the porous medium characteristics and is independent of fluid characteristics that govern the flow, such as viscosity. It was reported (Powers et al. 1954) that the permeability of cement paste in the fresh state is in the order of 10^{-9} mm/hour. As the hydration proceeds, the capillary pores reduce, and the permeability declines to the order of 10^{-13} mm/hour in 24 days (Table 2.3).

Table 2.3: Decrease in permeability of cement paste (w/c 0.7) with hydration progress (adapted from Powers et al. 1954)

Age (day)	Saturated hydraulic permeability $\times 10^{-13}$ (mm/hour)
Fresh	5555555.0
5	1111.0
6	278.0
8	111.1
13	13.9
24	2.8
Ultimate	1.7

Permeability can be measured by different methods such as permeability cell, triaxial method, pressure relaxation technique, and beam bending. Among these, permeability cell is the most common method for measuring permeability. In this method, permeability is determined by establishing a pressure gradient passing through a specimen to measure the flux by using a device such as a permeameter (Figure 2.6). The specimen is required to be sealed to prevent the leak of the fluid around the sides. This process can be done by fixing the specimen in place with an adhesive material (resin) which requires the specimen's side surface to be dried. This drying step affects the specimen and alters the microstructure (Powers et al. 1959).

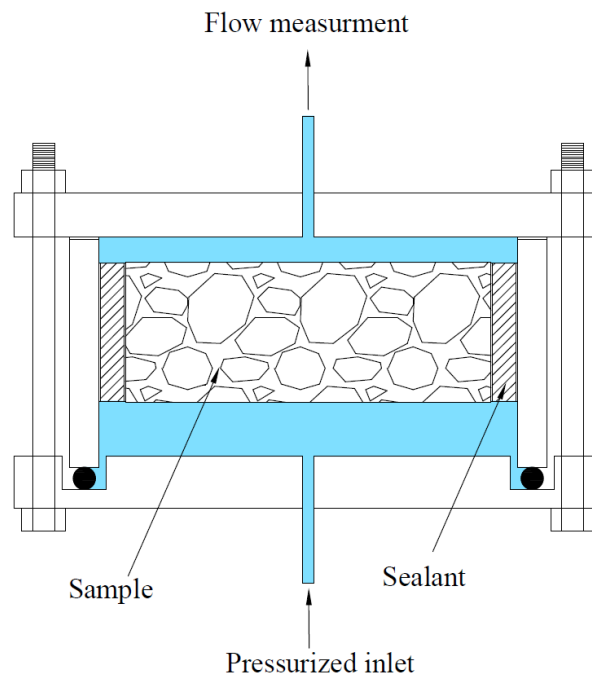


Figure 2.6- Schematic of a conventional permeameter.

There are several parameters that must be addressed while using this method. First, whether the specimen has been dried before or it is an undried sample. This is critical as a single drying cycle may increase the concrete permeability as much as two times that of the

undried sample due to micro-cracking from the drying process (Powers et al. 1959). Micro-cracking caused by mechanical loading is also reported to be responsible for raising the permeability when the stress level exceeds more than a third of compressive strength (Banthia et al. 2005). Second, for mixture designs with low water to cement ratios it is challenging to make the specimen saturated prior to the test. In addition, to prevent pore blocking of the sample by air bubbles, the water needs to be de-aired. Third, applying adequate confinement pressure is necessary to prevent leaking that may result in dropping of exerted pressure (Scherer et al. 2006). Forth, the capillary pressure at the outlet side of the sample that is exposed to atmospheric pressure may distort the permeability results if the exerted pressure is not high enough (Scherer et al. 2006). Even by addressing all aforementioned limitations, the variation in permeability results is reported to be in the magnitude of $\pm 50\%$ (Scherer et al. 2006). Also, water permeability tests are laborious to perform in terms of time and equipment which makes other tests (e.g. absorption) more appealing.

2.5.3 Katz-Thompson theory

Percolation theory was applied by Katz and Thompson (1986) on sedimentary rocks to develop a relationship between critical pore diameter and air permeability. They suggested a correlation between the permeability of saturated porous media to critical pore diameter and the ratio of bulk to pore solution conductivity based on the following equation:

$$k = \frac{1}{226} d_c^2 \frac{\sigma}{\sigma_0} \quad \text{Eq. 2.7}$$

where, d_c is critical pore diameter (m), σ is bulk conductivity (S/m), and σ_0 is pore solution conductivity (S/m). To evaluate the validation of Katz-Thompson equation (Eq. 2.7) that is based on air permeability, studies have been conducted using water permeability. For

instance, Christensen et al. (1996) supported the proposed relationship; however, El-Dib and Hooton (1994) and Tumidajski and Lin (1998) disagreed. Also, Halamickova et al. (1995) supported the relationship only for paste with w/c of 0.50. The published water permeability and pore diameter data for cement paste and concrete were reanalysed by El-Dieb and Hooton (1994). Instead of measuring the formation factor ($\frac{\sigma_0}{\sigma}$) directly, they calculated the ratio from pore parameters of the equation proposed by Katz and Thompson (1987);

$$\frac{\sigma}{\sigma_0} = \frac{d_{max}^e}{d_c} \eta S(d_{max}^c) \quad \text{Eq. 2.8}$$

where, $d_{max}^e = 0.34d_c$, φ is total porosity (%), and $S(d_{max}^c)$ is fraction volume of pores larger than d_{max}^e . Nokken and Hooton (2008) tested concretes with a wide range of w/c ratios (0.30-0.90) and reported a high correlation between measured water permeability and Katz-Thompson method (Eq. 2.6 and 2.7). It is believed that the previous published data that reported poor correlations suffered from a deficiency in measuring permeability (Nokken and Hooton 2008). Hence, in the current thesis, an effort was made to develop an analytical model based on Katz-Thompson relationship to determine the absorption capacity of joints in concrete pavements according to the customized absorption test protocol developed in this thesis.

CHAPTER 3: APPLICATION OF CURING COMPOUNDS ON CONCRETE PAVEMENTS

This chapter evaluates the effect of different curing applications through experiments on cores extracted from recently constructed pavement sections in comparison to corresponding specimens produced from similar concrete under laboratory conditions.

3.1 Introduction

The aspect of curing involves keeping concrete saturated as much as possible, till water-filled space in fresh concrete has been occupied by cement hydration products to the desired extent (Neville 2011). This can be done by maintaining a satisfactory level of moisture content (RH above 90%) and temperature in concrete for a period of time (preferably seven days or more) immediately following placing and finishing procedures to attain the desired properties. For example, properly cured concrete should achieve target strength, abrasion resistance, volumetric stability, water tightness and resistance to freeze-thaw cycles. Water curing of concrete is applied by different methods such as ponding, immersion, fogging and wet coverings; however, membrane (e.g. with curing compounds) is considered as the most efficient and practical procedure for fast construction of concrete elements such as floors, pavements and sidewalks (Mehta and Monteiro 2014). Concrete pavements are typically cured by membrane-forming curing compounds that usually consist of a resin or wax dissolved in a solvent or emulsified in water. They are applied to pavement surface, and by evaporation of the solvent, the wax or resin forms an impermeable membrane to help retaining moisture in concrete. In North American standards for concrete (CSA A23.1/A23.2 2014; ACI 308R-16 2016), curing compounds are specified by AASHTO M148 (2005) and ASTM C309 (2011). Applying an adequate rate of curing compounds

and providing a uniform coverage onto concrete are required to ensure efficiency of the curing process (Vandenbossche 1999).

Concrete pavements are particularly vulnerable to moisture loss via evaporation during the hardening process due to their high surface-to-volume ratio. Improper curing of concrete causes insufficient hydration of the cementitious matrix, which is associated with performance risks during service; for example, inadequate curing of concrete pavements may lead to forming a continuous pore structure within the surface, which facilitates the penetrability of moisture carrying aggressive agents (e.g. deicing salts), and thus provoking durability issues such as surface scaling and frost damage (Marchand et al. 1994; Bouzoubaâ et al. 2011). Moreover, drying of concrete surface at early-age may lead to a network of plastic and drying shrinkage cracks, which adversely affect the long-term performance of concrete (Bentz and Jensen 2004). In addition to the mixture design of concrete, the prevailing conditions in the field including the relative humidity, temperature and wind velocity affect the evaporation rate of moisture from concrete. It was reported that reduction of the relative humidity during the curing process from 100 to 94% led to a marked increase of the water absorption capacity of concrete (Ho et al. 1989), implicating the alteration of microstructure. If the external relative humidity falls below 80% during the initial curing process, the proportion of macro-pores (larger than 0.1 μm) in the hydrated paste may significantly increase and cause higher penetrability of concrete (Mehta and Monteiro 2014).

Numerous studies reported the effect of different curing methods (water curing, covering with plastic sheets or curing compound) on hardened properties of concrete. (Marchand et al. 1994; Nassif et al. 2005; Ibrahim et al. 2013). Curing compounds can efficiently maintain the moisture content, increase the degree of cement hydration and

decrease sorptivity of the near-surface layer of concrete (Ibrahim et al. 2013). Also, curing compounds may lead to less plastic and drying shrinkage of concrete (Al-Gahtani 2010). It has been reported that premature deterioration of concrete pavements, especially at joint locations are strongly linked to the penetrability of concrete (MDOT 1996; Chen et al. 2011; Liu et al. 2014), which is highly affected by the efficacy of curing practices. In North America, there are recommendations to apply curing compounds at a rate of 1.5 to 2 times the normal rate specified by the manufacturer in repair applications of concrete pavements (GPDR-COW 2015) with the perception that this practice substantially improves the curing efficiency of repair concrete, and thus it may be applied to concrete pavements during the construction process. Also, it is recommended that the specified rate of application can be achieved by two coats (each one applied at half the specified rate) to attain a uniform application of curing compound on concrete (TxDOT 2004). Yet, there are scarce field data on the effect of applying different rates or multiple coats of curing compounds on the quality of concrete, especially with respect to concrete pavements. Hence, the degree of curing efficiency of applying multiple coats of curing compounds on concrete pavements, and whether this practice can significantly enhance their durability properties remain uncertain. This is a critical question to a number of transportation agencies including Public Works Department, Winnipeg (Manitoba, Canada) linked to changing current construction specifications and associated with considerable increase of initial construction costs. Hence, the motive for this research is to investigate the effect of three different curing compound applications (no curing compound, one coat of curing compound, and two coats of curing compound) on the behaviour of concrete pavements in terms of their resistance to infiltration of fluids and microstructural evolution to project their long-term durability. The project consisted of macro- and micro-scale experiments on cores extracted from

recently constructed pavement sections in Winnipeg, Manitoba, Canada in comparison to corresponding specimens cast from similar concrete under laboratory conditions. This collaborative research study between the City of Winnipeg (COW) and University of Manitoba (U of M) was conducted in order to informatively guide the curing practices incorporated in the COW construction specifications for concrete pavements.

3.2 Experimental Program

3.2.1 Materials and mixtures

For the field pavement sections, the COW specified a ready mix concrete with target performance of 35 MPa and a class of exposure C-2 (plain concrete subjected to chlorides and freezing-thawing) according to CSA A23.1 (2014). Also, the same type of concrete was delivered to the U of M in order to prepare the laboratory specimens. The binder comprised General Use (GU) portland cement and fly ash (Class F), as a supplementary cementitious material (SCM), meeting the current requirements of CSA A3001 (2013), and the water-to-cementitious materials ratio (w/cm) was in the range of 0.36 to 0.38. The COW construction specifications for jointed plain concrete pavements (CW3310–R17 2015) permit replacement of GU cement with up to 15% Class F fly ash. A chemical air-entraining admixture complying with ASTM C260 (2010) was used in concrete to achieve a fresh air content of 5 to 8%. Also, a high-range water reducing agent complying with ASTM C494/C494M (2013) was used to obtain a target slump of 30 to 70 mm for slip forming. Due to a confidentiality agreement between the U of M and the concrete supplier, the specific proportions of the concrete mixture design cannot be disclosed. The curing compound applied was white in color and made of high-grade hydrocarbon resins in a water-based emulsion conforming to ASTM C309 (2011) Types 1, 2 and 1-D, Class B.

3.2.2 Field pavement sections and laboratory specimens

Three pavement sections were cast in an urban arterial road at the central area of Winnipeg, Manitoba. The pavement sections consisted of a 150 mm thick jointed plain concrete pavement with a slab width of 3.70 m on a granular base (Figure 3.1). The test sections comprised a two-way undivided road with one traffic lane and one parking lane in each direction. The speed limit in this area is 50 km/h and the average week-day daily traffic is 12,800 vehicles over the two lanes of traffic with 2% trucks and buses. The pavement in the test location is expected to be subjected to large temperature changes, as the maximum and the minimum air temperatures during the last 10 years were +36°C and –36°C, respectively.



Figure 3.1- Casting, finishing and curing procedures of field pavement sections.

The placement, consolidation, finishing and curing procedures of the pavement sections complied with the COW construction specification (CW3310–R17 2015). Placement of concrete was done via the discharge chute of ready-mix concrete trucks for continuous supply to the slipform paver, which consolidated fresh concrete using a series

of internal vibrators. This was immediately followed by finishing with bull floats and wooden trowels for fine tuning and final surfacing. Three pavement sections received different types of curing: no curing, one coat of curing compound, and two coats of curing compound, referred to hereafter as NC, 1CC and 2CC, respectively. Shortly (30 to 45 min) after the finishing operations, the first coat of curing compound was applied by labor, using portable sprayers on the designated 1CC and 2CC sections. Each coat of curing compound was applied at the rate of 0.23 l/m^2 according to the manufacturer's specifications. In addition, visual inspection was mandated by the COW specifications to cover any left out spots "holidays", where the surface of the pavement sections was uniformly white in color. The pavement slabs were saw-cut eight hours after casting to establish the contraction joints with the dimensions of 3 mm wide and 40 mm deep. For the 2CC section, the second coat of curing compound was applied immediately after saw-cutting. The second cut for joint sealing took place three weeks after the initial saw-cut with the dimensions of 10 mm wide and 30 mm deep. The joints were subsequently filled with a backer rod and sealed. At 28 days, forty eight cores 100 mm in diameter and twenty four cores 75 mm in diameter were extracted from the pavement slabs. Eight cores were used for each test according to Table 3.1; four cores were obtained from the center of the slab (mid-slab).

Table 3.1: Testing scheme for the field cores

Test	No. and (diameter) of Field Cores		
	NC	1CC	2CC
Rate of Absorption (ASTM C1585)	8 (100 mm)	8 (100 mm)	8 (100 mm)
COW Test (COW 2013-01)	8 (75 mm)	8 (75 mm)	8 (75 mm)
RCPT (ASTM C1202)	8 (100 mm)	8 (100 mm)	8 (100 mm)
Total No. per section	16 (100 mm)	16 (100 mm)	16 (100 mm)
	8 (75 mm)	8 (75 mm)	8 (75 mm)
Total	48 (100 mm)		
	24 (75 mm)		

For ASTM C1585 (2013) and C1202 (2012) tests, the other four cores were extracted from areas immediately right (two cores) and left (two cores) of transverse joints to obtain full cores. Comparatively, for the COW absorption test (COW-2013-01 2013), the other four cores were obtained directly at transverse joints (split cores). Figure 3.2 illustrates the locations of the cores extracted from the field sections.

Laboratory specimens were prepared from the same ready mix concrete delivered by the same supplier. Thus, the effect of standard (laboratory) casting relative to actual field practice on the transport properties of concrete (i.e. ‘controlled’ vs. ‘uncontrolled’) was taken into account. This is to ensure that any variation in the trends and microstructure of concrete would be originating from the curing method rather than the field practice or ambient conditions (e.g. effect of evaporation rate in the field).

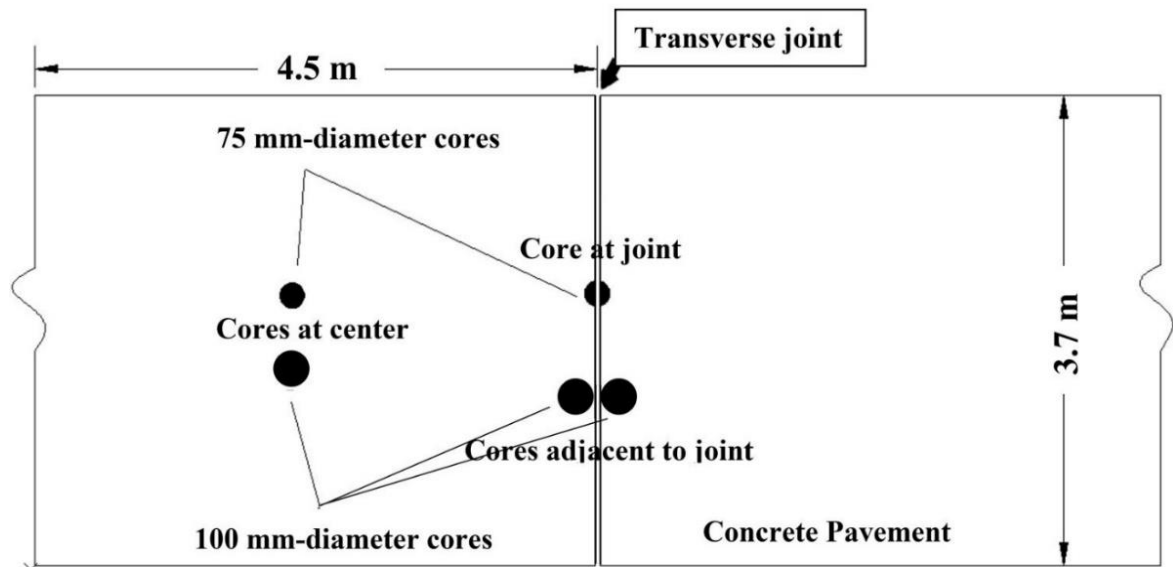


Figure 3.2- A schematic diagram showing the location of cores extracted for tests.

Thirty six 100 mm × 200 mm concrete cylinders and nine 75 mm × 150 mm concrete cylinders were cast in PVC moulds. The cylinders were filled in two layers, where each layer was compacted 25 times by a standard tamping rod according to ASTM C192/C192M

(2013). The top surface of specimens were cured in a similar way to the field pavement sections (i.e. NC, 1CC and 2CC), and they were kept in moulds for 28 days (to simulate center of slab cores) under laboratory conditions ($23\pm 2^{\circ}\text{C}$ and $55\pm 5\%$ RH). The testing scheme was similar to that of the field cores (Table 3.1), except that three replicates were used for each test and curing application, as the cylinders were prepared in a standard way.

3.2.3 Tests

3.2.3.1 Strength and absorption tests

The compressive strength of both field and laboratory concretes were tested according to ASTM C39/C39M (2014), the results were in the range of 41 to 43 MPa meeting the COW requirements for strength. The rate and total absorption were determined for concrete specimens according to ASTM C1585 (2013). At 28 days, the top skin (~ 3 mm, including the curing compound layer) of the cylindrical specimens was removed and the adjacent 50 mm thick discs were cut as the test specimens. Also, COW absorption test (absorption by immersion; COW-2013-01 2013) was conducted on both laboratory specimens and field cores. After 28 days, the surface of concrete was gently cleaned with a brass brush to remove the curing compound layer, and 50 mm thick slices were cut from cylindrical specimens and cores. For the laboratory specimens and cores extracted from the center of slab, top and bottom 50 mm (baseline) slices were obtained, whereas only the top 50 mm part was cut from the cores obtained at the joints, as required by the test procedures. The concrete discs were placed in a sealed desiccator under vacuum pressure (~ 85 kPa) for six hours. Subsequently, the specimens were removed from the desiccator and each one dried out with compressed air. Afterwards, the initial mass of the dried specimens to the nearest 0.01 g was recorded. Each specimen was immersed in deionized water for 60 ± 3 s, and then excess water was blotted off and the wet mass of the specimen was recorded within 25 s to

the nearest 0.01 g. The amount of water absorbed into concrete was determined by subtracting the dry mass from the wet mass and converted to a percent of the wet mass of the specimen. The percentage of water absorption is important because the actual volume of specimens may vary due to coring operations, and it was calculated by:

$$WA = \frac{M_{WET} - M_{DRY}}{M_{WET}} \times 100 \quad \text{Eq. 3.1}$$

where WA is the percentage of water absorption of concrete, where, M_{WET} is mass of wet, but surface-dry, specimen (g), and M_{DRY} is mass of dry specimen before immersion (g). To evaluate the efficiency of curing of concrete, the absorption of top part for each location was compared to the absorption of the bottom slices of the center of slab. The bottom slices of the center of slab are considered as the baseline for curing efficiency in the field according to COW-2013-01 (2013).

3.2.3.2 Rapid chloride penetrability test (RCPT)

The interconnectivity of pore system in concrete specimens/cores was assessed by the rapid chloride penetrability test (ASTM C1202 2012). At 28 days, the surface skin (~3 mm) from the top and bottom of cylindrical specimens was removed to reach a clear surface, and the top 50 mm thick discs were cut as the test specimens. Since physical observation provides better representation of the penetration of chloride ions into concrete, the physical penetration depth of chloride ions was measured immediately after the completion of the test by spraying 0.1 M silver nitrate solution on the surface of equally split discs (Bassuoni *et al.*, 2006). The discs were allowed to set under fluorescent light for about 15 minutes to obtain the whitish precipitate of silver chloride. Five penetration depths (every 20 mm) were measured on each half disc (i.e. 10 readings per specimen) to calculate the average penetration depth for each specimen. To account for the possible heat effect of RCPT and

physical features of the pore structure, the migration coefficient of concrete was calculated by (NT BUILD 492 1999):

$$D = \frac{0.0239(273+T)L}{(V-2)t} (x_d - 0.0238\sqrt{\frac{(273+T)Lx_d}{V-2}}) \quad \text{Eq. 3.2}$$

where D is the non-steady-state migration coefficient ($\text{m}^2/\text{s} \times 10^{-12}$), V is the applied voltage (V), T is the average value of initial and final temperatures in the anolyte solution ($^{\circ}\text{C}$), L is the thickness of the specimen (mm), x_d is the average value of penetration depth (mm), and t is time (h).

3.2.3.3 Thermogravimetry (TG), mercury intrusion porosimetry (MIP) and microscopy tests

The effect of different curing compound applications on the development of microstructure in concrete was assessed by thermogravimetry (TG), mercury intrusion porosimetry (MIP) and backscattered scanning electron microscopy (BSEM). Samples for TG tests were prepared by crushing chunks around 4–7 mm in size taken from the top 15 mm of the laboratory concrete cylinders (prepared with GU and a maximum of 15% fly ash) and grinding them to fine powder passing sieve #200 ($75\mu\text{m}$). The TG tests were conducted using a heating rate of $10^{\circ}\text{C}/\text{min}$ at 1, 3, 7 and 28 days. The content of portlandite (calcium hydroxide) was calculated by determining the drop in the percentage mass of TG curves at a temperature range of 400 to 450°C and multiplying it by 4.11 (ratio of the molecular mass of portlandite to that of water). To capture the characteristics of pore structure, the MIP technique was conducted on field concrete samples at 28 days. Small pea-sized chunks were taken from at least two concrete cylinders and were used as test samples for MIP (Kumar and Bhattacharjee 2003; Said et al. 2012). These chunks were around 4–7 mm in size, and carefully selected so that large aggregates were not included. These chunks were

taken from the top 15 mm of the concrete cores (75 mm cores) extracted at the center of slabs and joints (split cores). The samples were oven dried for 72 h at a temperature of 55 ± 2 °C; they were then kept in a desiccator containing calcium sulphate for 24 h. This method of drying – using a lower temperature for a longer period – was adopted to avoid decomposition of calcium silicate hydrate (C-S-H) and formation of micro-cracks, which may occur at high temperatures. The contact angle and the surface tension of mercury were taken as 130° and 485 dynes/cm, respectively (Shi and Winslow 1985; Said et al. 2012). To complement the trends observed in the various tests, BSEM was conducted on polished thin sections from the field cores extracted at the center of slabs after 180 days. Slices were cut from the top part (25 mm) of cores; they were then dried and impregnated with a low-viscosity epoxy resin under vacuum pressure and polished by successive diamond surface-grinding to a thickness of about 50 μm . The sections were coated then with carbon to enhance the conductivity for the BSEM imaging.

3.2.4 Statistical analysis

The results of absorption and RCPT were statistically evaluated by the Analysis of Variance (ANOVA) method at a significance level (α) of 0.05. ANOVA is a statistical model used to analyze the differences between group means and their variation among and between groups. The variance of a specific parameter is separated into components attributed to different sources of variation. ANOVA tests the hypothesis that the means of several groups are comparable via a *t*-test, and it can generalize the test to more than two groups. According to ANOVA, exceeding the F_{cr} for an *F*-distribution density function indicates that the variable tested has a statistically significant effect on the average results (Montgomery 2012). The ANOVA results were further statistically evaluated by Tukey's post hoc test (Tukey's test), when applicable, to declare pairwise comparisons between the

means of each two groups. Tukey's post hoc test declares two means significantly different if the absolute value of their sample differences exceeds a critical range, q_{cr} (Montgomery 2012).

3.3 Results and Discussion

3.3.1 Environmental conditions

During casting the field sections, the average ambient air temperature and relative humidity were 20°C and 78%, respectively, with a wind velocity of less than 31 km/h (Figure 3.3).

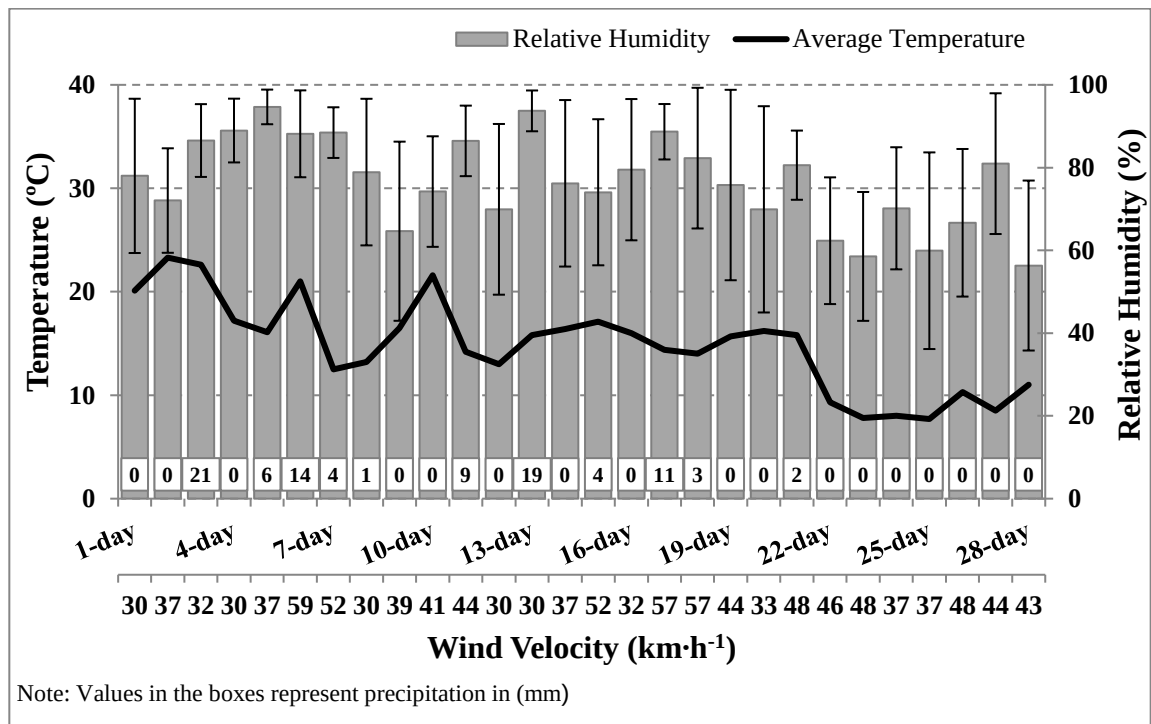


Figure 3.3- Average daily temperature, relative humidity, precipitation and wind velocity data in Winnipeg from the date of casting field pavement (GCC 2015).

The measured temperature of concrete pavement was about 19°C. Under these weather conditions, the moisture evaporation rate at the concrete surface was estimated at 0.25 kg/m²/h according to the chart of the Canadian standard CSA A23.1/A23.2 (2014). This value was well below the critical evaporation rate (0.50-0.75 kg/m²/h) stipulated by ACI

308R-16 (2016) mainly to minimize the risk of surface drying and plastic shrinkage cracking. Also, four rainy days were recorded during the first week of casting (Figure 3.3). Laboratory specimens were cast under standard room conditions (21°C and 50%RH) with a negligible wind velocity, and the concrete temperature measured was about 18°C. These conditions led to a surface moisture evaporation rate of 0.20 kg/m²/h. Hence, it can be deduced that the field concrete sections originally planned to receive no treatment in terms of curing compound application (NC) were beneficially affected by the prevailing climatic conditions (low rates of evaporation, high relative humidity and rainfall, [Figure 3.3]).

3.3.2 Absorption tests

The behaviour of concrete exposed to aggressive environments (e.g. deicing salts and freezing-thawing cycles) depends on the mode of transport of moisture and ionic species through the pore system, which indicates the ease of saturation of concrete. Hence, assessing the transport properties of concrete is critical to concrete pavements. Absorption, which is defined as the fluid uptake into the surface of concrete through capillary action/suction (Mehta and Monteiro 2014) is one of the key transport properties, as it implicates the porosity of concrete surface as well as the general characteristics of pore structure. This transport mechanism is a function of the surface tension of the penetrating fluid with capillary walls and moisture state in the pore system (dry or partially dry). The ASTM C1585 absorption results are shown in Table 3.2 and Figure 3.4. The initial and secondary rates of absorption for the laboratory and field specimens were in the narrow ranges of 1.0×10^{-3} to 1.1×10^{-3} mm/ $\sqrt{s}$ and 0.3×10^{-3} to 0.4×10^{-3} mm/ $\sqrt{s}$, respectively, without a distinctive effect of the scheme of curing. However, the general trend of the absorption curves shown in Figure 3.4 indicates reduction (up to 15%) of the total absorption of concrete with the application of the curing compound (1CC and 2CC) relative

to NC (Figure 3.5). This behaviour can be ascribed to the improvement in the hydration process near the top surface of concrete with the application of the curing compound, especially with 2CC, which led to densified microstructure in the hydrated paste and thus reduced the absorption capacity of concrete. However, the ANOVA results for the total absorption results at 8 days for both laboratory and field specimens (summarized in Table 3.3) showed the effect of the type of curing on the total absorption of concrete to be statistically insignificant. This insignificance is attributed to the overlapping range of results for replicate specimens among the groups tested which can be graphically noted in the range of error bars (standard deviation) depicted in Figure 3.5.

Table 3.2: Average results of the absorption test (ASTM C1585) for laboratory and field specimens

Source	Curing Scheme	Initial		Secondary	
		Rate of absorption $\times 10^{-3}$ (mm/ $\sqrt{s}$)	Coefficient of determination (R^2)	Rate of absorption $\times 10^{-3}$ (mm/ $\sqrt{s}$)	Coefficient of determination (R^2)
Laboratory Specimens	NC	1.0	0.99	0.3	0.99
	1CC	1.1	0.99	0.4	0.99
	2CC	1.0	0.99	0.4	0.99
Cores at Center	NC	1.1	0.98	0.4	0.99
	1CC	1.1	0.99	0.4	0.99
	2CC	1.0	0.98	0.3	0.99
Cores adjacent to Joint	NC	1.0	0.99	0.3	0.99
	1CC	1.0	0.99	0.3	0.99
	2CC	1.0	0.99	0.3	0.99

Also, it can be noted that field cores, especially specimens with NC had higher variability of results, which contributed to this insignificance. Overall, the general trend of results highlights the importance of curing in improving the microstructure of concrete, and consequently reducing its absorption capacity.

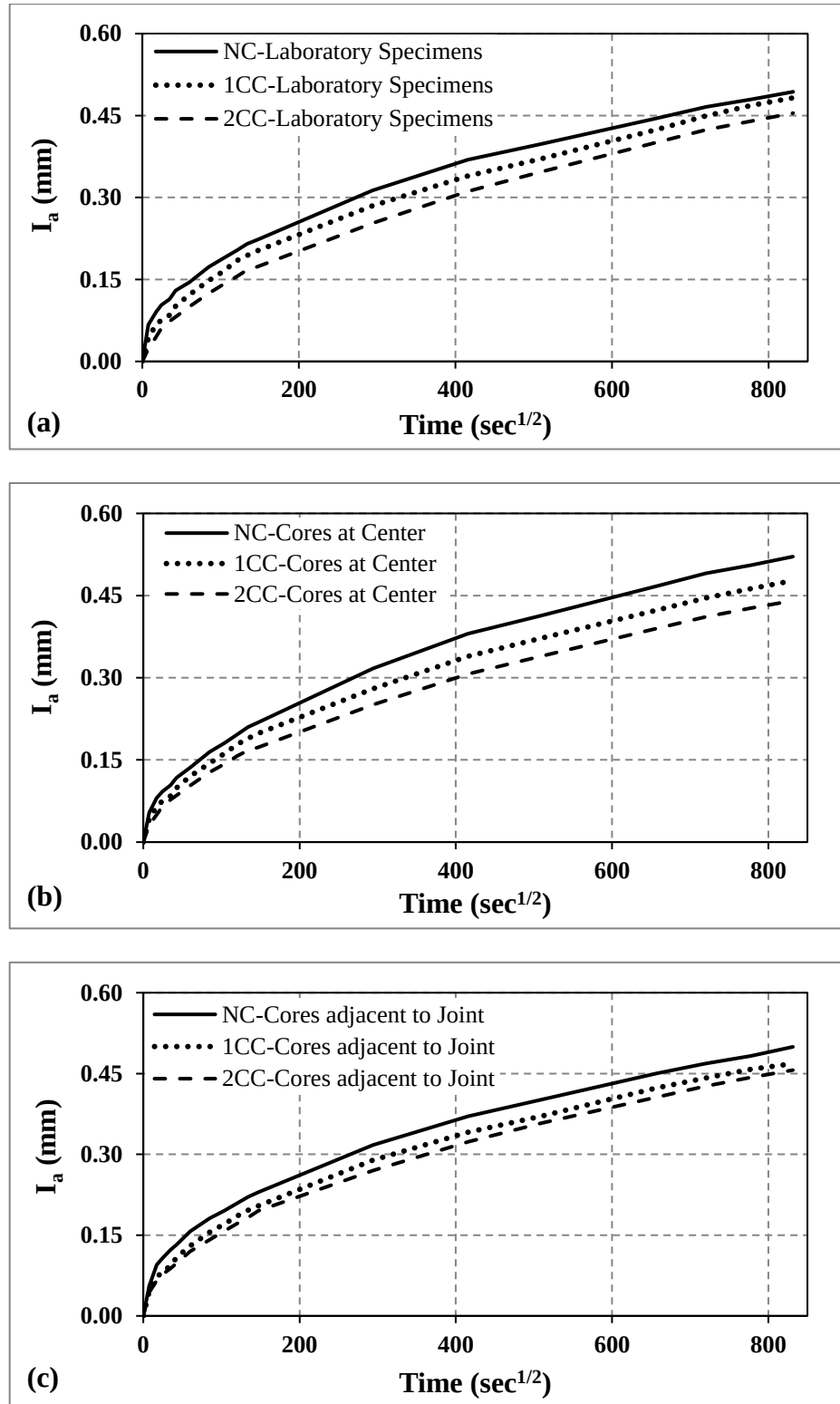


Figure 3.4- Rate of water absorption of concrete (ASTM C1585): (a) laboratory specimens, (b) cores at center, and (c) cores adjacent to joint.

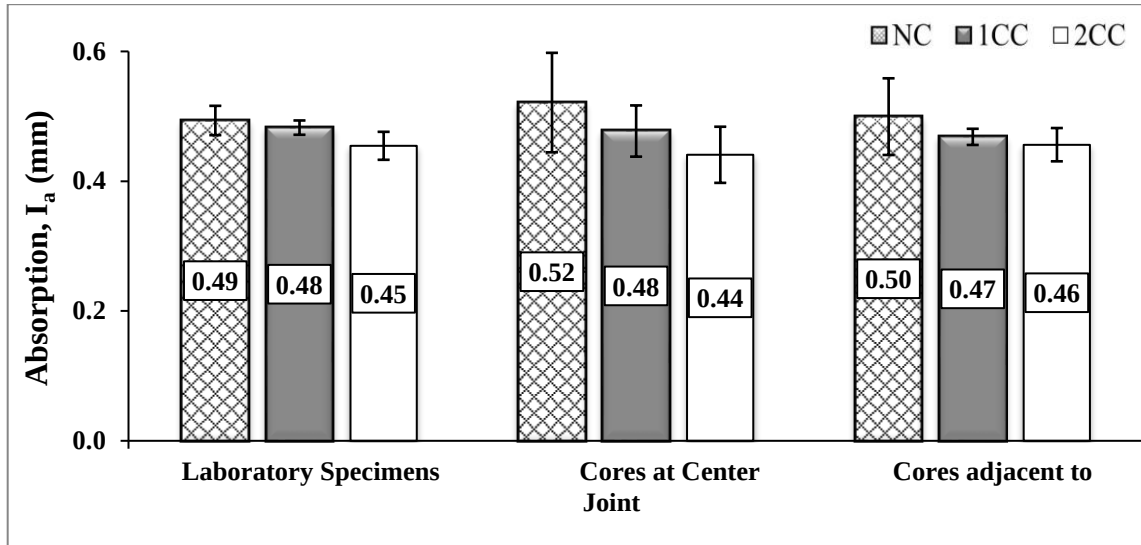


Figure 3.5- Total water absorption (ASTM C1585) of laboratory and field specimens.

Table 3.3: ANOVA for the results of ASTM C1585, COW and ASTM C1202 tests

Test	Source	Parameter Tested	F	P-Value	F _{cr}
Rate of Absorption (ASTM C1585)	Laboratory Specimens	Total Absorption	3.32	0.1068	5.14
	Cores at Center		2.09	0.1792	4.26
	Cores adjacent to Joint		1.39	0.2984	4.26
COW Test (COW 2013-01)	Laboratory Specimens	Absorption Ratio	30.32*	0.0007	5.14
	Cores at Center		25.76*	0.0002	4.26
	Cores at Joint		0.68	0.5300	4.26
RCPT (ASTM C1202)	Laboratory Specimens	Migration Coefficient	5.12	0.0504	5.14
	Cores at Center		0.51	0.6148	4.26
	Cores adjacent to Joint		3.61	0.0704	4.26

* Indicates statistical significance.

The results of the COW absorption (by full immersion) test are given in Table 3.4, and the absorption ratios (top-to-bottom) are shown in Figure 3.6. It can be noted that the average absorption ratios for the cores from the center of slab were 0.90 and 0.77 for specimens with 1CC and 2CC, respectively. This might be attributed to the difference in the level of consolidation between the top and bottom layers at the center of slab; in addition, inadequate moistening or excessive drying of this area prior to placement of fresh

concrete might have led to some moisture loss from the bottom portion of concrete, and thus influenced the hydration process and in turn the characteristics of pore structure. Irrespective of the method of curing, it can be observed that the absorption values and ratios for the cores extracted at joints are significantly higher than that of corresponding cores extracted from the center of slab or specimens prepared in the laboratory. The higher absorption values of the cores at the joints can be attributed to increasing the exposed surface area in contact with water due to the existence of a groove (vertical walls) in the split cores. The saw cutting process at early- and later- ages could have generated weak planes with micro-cracks in concrete at the joint locations. In addition, creating and exposing these surfaces at early-age (final setting) in the absence of curing, may make them susceptible to uncontrolled evaporation. These critical surfaces facilitate absorption, which can result in higher degree of saturation with salt solutions (especially in cold regions where de-icing salts are frequently applied to pavements in winter), and in turn aggravate degradation of concrete in these locations.

For laboratory concrete, the absorption ratios of specimens with NC, 1CC and 2CC were 1.58, 1.20, 1.05, respectively (Figure 3.6). ANOVA for the absorption ratios (Table 3.3) indicates that this trend is statistically significant for specimens prepared in the laboratory and cores extracted from the center of slab. For example, for laboratory specimens, changing the scheme of curing had an F value of 30.32 compared to a critical value F_{cr} of 5.14. These results were further evaluated by Tukey's test to investigate the difference in each possible pair of absorption ratios. Tukey's test declared a significant difference between the curing schemes. According to Table 3.5, the pairwise comparisons of the absorption ratios were 0.39, 0.15 and 0.53 for curing method pairs of NC versus 1CC, 1CC versus 2CC, and NC versus 2CC, respectively, compared to q_{cr} of 0.22. This

trend is consistent with the results of field concrete obtained from cores at the center of slab, which signifies the importance of curing in reducing the total absorption of concrete. Complying with the general trend of the ASTM C1585 test, the reduction of absorption capacity of concrete with 1CC and 2CC is due to the retention of moisture within the surface layer of concrete, which led to efficient hydration and microstructural development and consequently less water absorption relative to concrete with NC. This suggests that either applying 1CC or 2CC significantly improves the hydration process of concrete.

Table 3.4: Average water absorption (COW test) of laboratory and field specimens

Curing Scheme	Laboratory Specimens		Cores at Center		Cores at Joint
	Absorption	Absorption	Absorption	Absorption	Absorption
	Top (%)	Bottom (%)	Top (%)	Bottom (%)	Top (%)
NC	0.16	0.10	0.16	0.13	0.24
1CC	0.12	0.10	0.12	0.13	0.23
2CC	0.11	0.10	0.11	0.15	0.24

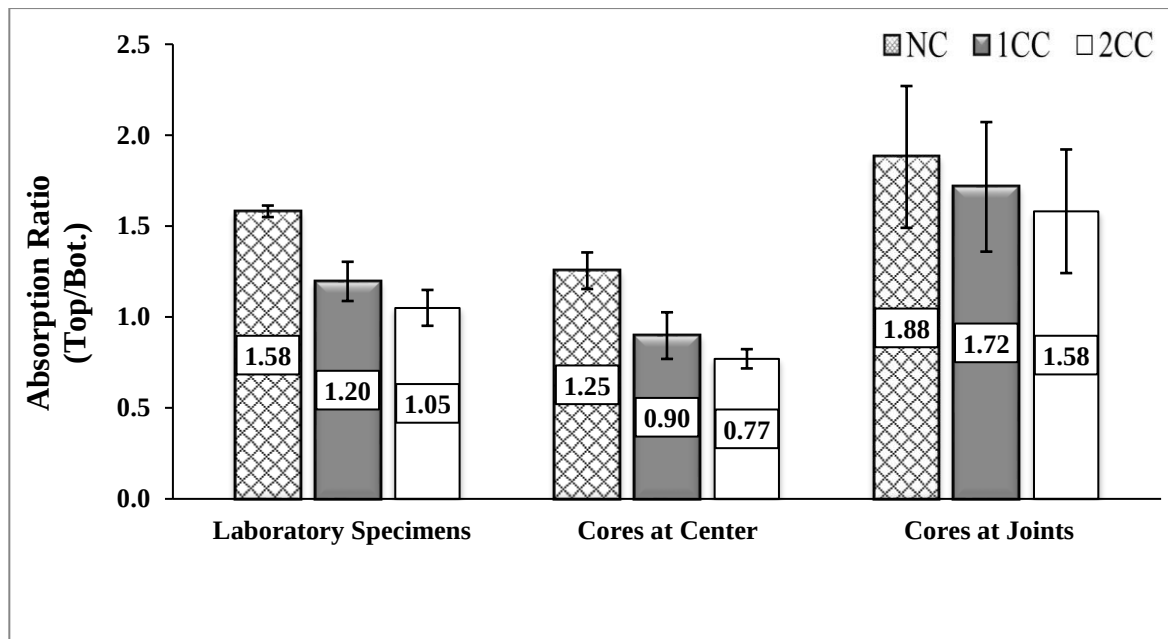


Figure 3.6- Absorption ratio (COW test) of laboratory and field specimens.

Table 3.5: Absorption ratios and Tukey's test results for the COW test

Curing Scheme	Laboratory Specimens			Cores at Center		
	Absorption Ratio	Difference	q_{cr}	Absorption Ratio	Difference	q_{cr}
NC	1.58	0.39*		1.25	0.36*	
1CC	1.20	0.53*	0.22	0.90	0.48*	0.20
2CC	1.05	0.15		0.77	0.13	

* Indicates statistical significance.

The absorption ratios for the cores extracted at the joints with NC, 1CC and 2CC were 1.88, 1.72 and 1.58, respectively (Figure 3.6). Unlike the case of laboratory specimens and field cores at the center of slab, ANOVA of these results yielded an insignificant effect of the curing scheme on the absorption results at the joints (Table 3.3). The error bars (standard deviation) of the absorption ratios for the three curing methods at the joints had higher variability, which contributed to this statistical insignificance. Again, this suggested that the COW test had been highly affected by the vertical faces within the groove at joint areas, which might have led to this high variability among results. This trend is corroborated by the microstructural results from MIP as discussed later in Section 3.4.

3.3.3 RCPT

The penetrability classification according to ASTM C1202 (2012), physical penetration depth (Bassuoni et al. 2006) and migration coefficient (NT BUILD 492 1999) of concrete were determined after operating the RCPT for 6 hours. The whitish color showing the penetration depth of chloride ions was broadly visible, as for example shown in Figure 3.7. The passing charges, penetration depths and migration coefficients of laboratory and field concrete are given in Table 3.6. The classification of chloride ions penetrability of concrete was consistent with the penetration depth results. The passing charges in all concrete specimens were in the range of 1000 to 2000 Coulombs (Table 3.6); hence, all the

specimens were classified as “Low Penetrability” according to ASTM C1202. This conforms to small penetration depths (below 13 mm), irrespective of the curing scheme.

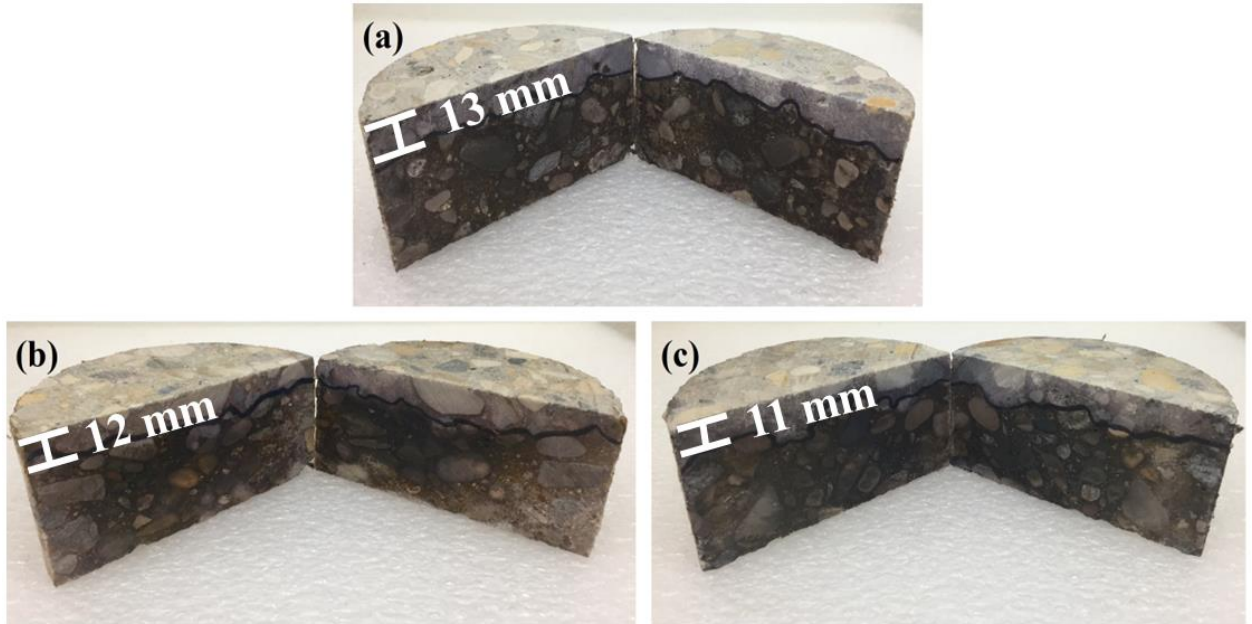


Figure 3.7- Whitish color showing the chloride front in the top part of laboratory specimens: (a) NC, (b) 1CC, and (c) 2CC.

Table 3.6: Average passing charges (ASTM C1202), penetration depth, and migration coefficient for the top part of laboratory and field specimens

Source	Curing Scheme	Charges		Penetration		Migration Coefficient ($\times 10^{-12}$ m ² /s)
		Passing (Coulombs)	Standard Deviation	Depth (mm)	Standard Deviation	
Laboratory Specimens	NC	1765	119	13	0.4	12.0
	1CC	1665	29	12	0.8	11.0
	2CC	1658	114	11	0.5	10.0
Cores at Center	NC	1755	85	12	1.1	11.0
	1CC	1548	54	12	1.5	11.0
	2CC	1806	82	12	1.1	11.0
Cores adjacent to Joint	NC	1864	56	12	0.9	11.0
	1CC	1798	28	11	0.3	10.0
	2CC	1813	114	10	0.5	9.0

Note: all specimens had a ‘low penetrability’ class according to ASTM C1202.

The RCPT trends were consistent with that of the absorption tests in the sense that there was generally a slight reduction in the migration coefficient of concrete with 1CC and 2CC relative to the corresponding specimens without curing (NC). For example, specimens taken from cores adjacent to joints with NC, 1CC and 2CC had migration coefficient of 11×10^{-12} , 10×10^{-12} and 9×10^{-12} m²/s, respectively. ANOVA (Table 3.3) for the results of migration coefficients showed that the change in curing method had an insignificant effect on the results. For instance, for laboratory specimens, changing the curing scheme had an F value of 5.12 compared to a critical value F_{cr} of 5.14. This trend may be attributed to the high quality of this concrete with low w/b (0.36 to 0.38) which led to a dense and disconnected microstructure that discounted ingress of chloride ions. It should be mentioned that specimens from the pavement section without curing compound (NC) were exposed to periods with high relative humidity and intermittent rainfall (natural curing, Figure 3.3), which improved the efficiency of hydration reactions in a comparable manner to corresponding specimens from sections with 1CC and 2CC. Correspondingly, laboratory specimens without curing (NC) were kept in the moulds for 28 days and had a limited evaporative surface, which did not affect the evolution of hydration reactions within 50 mm depth from the surface. It should be noted that the groove effect on the results was better captured in the COW absorption test relative to the ASTM C1585 and RCPT due to the locations of the cores extracted (split vs. full cores, Figure 3.2).

3.3.4 Development of microstructure

The TG results for portlandite (CH) content at different ages are shown in Figure 3.8. Irrespective of the scheme of curing, the laboratory concrete specimens had a similar pattern characterized by a general increase in the portlandite content followed by a marginal decrease up to 28 days. The former is related to the hydration of cement, while

the latter is due to an incipient stage of pozzolanic activity. In this concrete, GU cement was replaced with a low dosage of Class F fly ash (up to 15%), and thus significant consumption of portlandite in pozzolanic reactions is unlikely, especially within 28 days (Mehta and Monteiro 2014). The relatively higher portlandite content of specimens with 2CC at 3, 7 and 28 days allude to better hydration activity; which is consistent with the previous tests.

The results from mercury intrusion porosimetry (MIP) for the field cores obtained from the center of slabs and joints are shown in Table 3.7, which includes the apparent total porosities, total cumulative intrusions, threshold pore diameters and proportion of micro-pores (less than 0.1 μm). The MIP tests were done on at least four small chunks extracted from two replicate cores for each section with different curing applications, which were put in the same test compartment (porosimeter). Thus, the results shown in Table 3.7 can be reasonably considered as the averages of representative populations (Said et al. 2012). The trends of MIP at 28 days conformed to the durability properties (absorption and RCPT) in the sense that the cores at the joint area had a relatively coarser pore structure than that in the corresponding cores at the center of slab; in addition, there was reduction in the total porosity of field cores with 1CC and 2CC relative to the corresponding cores without curing (NC) [Table 3.7]. The threshold pore diameters for cores at center and joint areas with 1CC and 2CC were markedly less than those of the corresponding cores with NC. For instance, the threshold pore diameter of the cores obtained at the center of slab with 1CC and 2CC were 50% and 22%, respectively of the threshold pore diameter of cores with NC (3.6 μm). Correspondingly, the proportion of micro-pores in these samples was more than 70% of the total pore volume (Table 3.7). This directly showed the pore structure refinement achieved by the application of curing compound (with marked difference between 1CC and

2CC) to the pavement, at the joint area and center of slabs, resulting in efficient hydration reactions and better microstructural development. These trends allude to some insensitivity of the COW test (bulk absorption of concrete) in accurately evaluating the quality of microstructure of concrete at joint locations and distinctively differentiating between concretes with 1CC and 2CC at the center of slab.

Table 3.7: Mercury intrusion porosimetry (MIP) test results for field cores at 28 days

Source	Curing Scheme	Total Cumulative Intrusion $\times 10^{-2}$ (ml/g)	Apparent Total Porosity (%)	Threshold Pore Diameter (μm)	Proportion of Micro-Pores ($<0.1 \mu\text{m}$) (%)
Cores at Center	NC	6.0	13.5	3.6	67.7
	1CC	5.6	12.2	1.8	70.8
	2CC	4.8	11.3	0.8	74.1
Cores at Joint	NC	6.6	14.2	4.9	62.3
	1CC	6.1	13.1	2.0	69.6
	2CC	5.7	12.1	0.9	72.0

In general, the BSEM observations at 180 days conformed to the results of the other tests at 28 days. For instance, as shown in Figure 3.9(a), the interfacial transition zone (ITZ) in field cores with NC shows scattered porous areas and some micro-cracks, which can be attributed to an insufficient level of hydration in this weak zone. Comparatively, notable densification and homogeneity are observed in the matrix at the ITZ of specimens from corresponding field cores with 1CC and 2CC [Figures 3.9(b) and 3.9(c)], due to the low w/b (0.36-0.38), incorporation of fly ash and application of curing compound at early-age. This trend is confirmatory to the MIP pore size distribution results, as corresponding specimens with NC had the highest total porosity and threshold pore diameter and the lowest percentage of micro-pores at 28 days (Table 3.7). The BSEM images signify the importance of early-age curing for efficient microstructural development of the cementitious matrix, and hence improved long-term performance of concrete pavements.

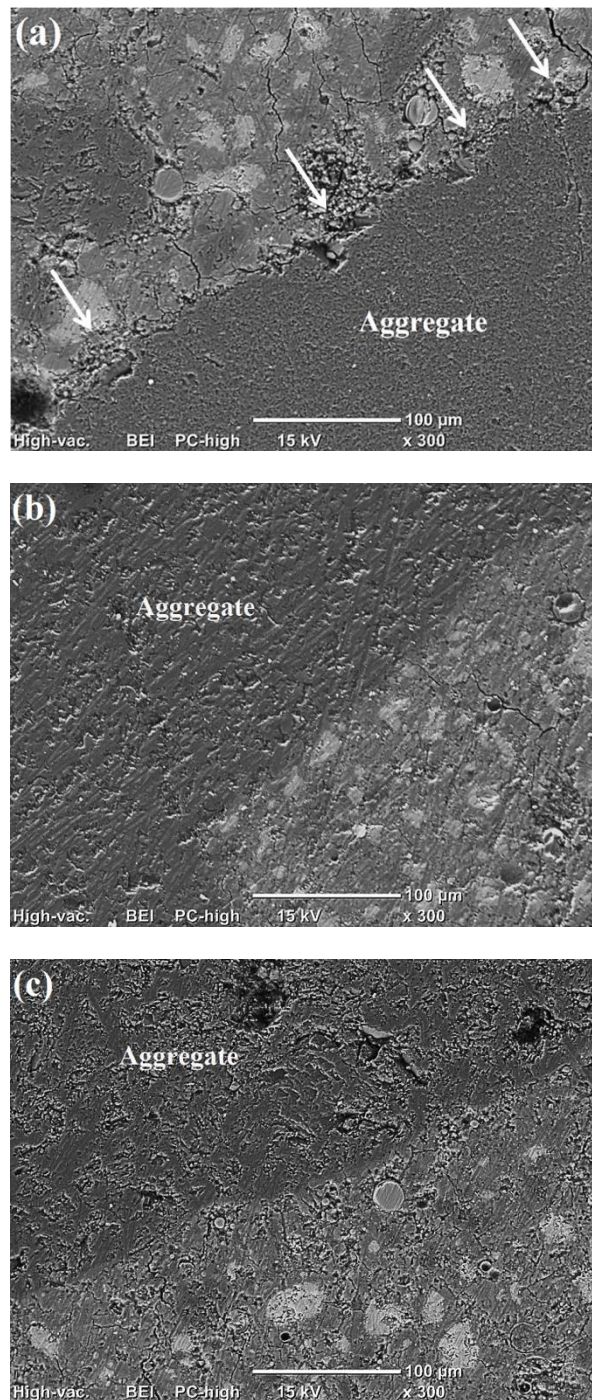


Figure 3.8- BSEM micrographs for thin sections from top part of field cores at mid slab at 180 days: (a) NC, (b) 1CC, and (c) 2CC.

CHAPTER 4: A TEST PROTOCOL FOR EVALUATING ABSORPTION OF JOINTS IN CONCRETE PAVEMENTS

This chapter focuses on developing a customized test protocol for determining the absorption capacity of joints in concrete pavements which involves three phases and thorough statistical analyses of results.

4.1 Introduction

Failure of concrete pavements, especially joint deterioration, is not usually attributed to design factors such as thickness, dowels or joint spacing, but it is often linked to materials distress, for example due to environmental effects such as freeze-thaw damage, especially with the application of de-icing salts (Litvan 1976; Şahin et al. 2010) . In North America, premature deterioration near to and at the joints still presents a critical durability issue of concrete pavements associated with considerable repair costs (Sutter et al. 2010; Leech et al. 2008). The United States Federal Highway Administration (FHWA) estimates that \$85 billion would be needed in annual capital investment up to 2028 to be directed towards improving the physical condition of existing road assets in order to achieve the Department of Transportation's State of Good repair benchmark for a better ride quality (ASCE 2013). The damage at the joints can be problematic because it compromises the performance (e.g. ride quality) and potentially service life of otherwise sound pavement sections. This damage might be correlated to the quality of concrete in terms of its fluid transport properties, which depend on the mixture design parameters of concrete, curing efficiency and the consequent evolution of microstructure (Taylor et al. 2006). Due to their unique geometry (width and depth), joints in concrete pavements are vulnerable locations

to higher degree of saturation, which triggers key durability issues for concrete in cold regions such as frost damage and corrosion of steel reinforcement/dowels. Thus, assessing the transport properties (e.g. absorption) of concrete pavements at joint locations may project their long-term performance.

Water absorption has been used as an indicator for assessing the durability of concrete (Bentz et al. 2001; Neithalath 2006). It can be mathematically described with Eq. 4.1 (Kelham 1988):

$$\chi_{(t)} = \sqrt{\frac{4k\gamma \cos(\beta)t}{\phi\eta r}} \quad \text{Eq. 4.1}$$

Where, χ is absorption depth (m), k is the intrinsic permeability (m^2), γ is the surface tension (N/m), β is the contact angle (deg), ϕ is the porosity (%), η is the dynamic viscosity (N.s/m^2), r is the pore radius (m), and t is the time (s). Experimentally, it can be determined by one-dimensional absorption of an exposed surface following ASTM C1585-13 (2013), based on the procedure introduced by Hall (1989). In this test, a concrete specimen is conditioned (air-vacuumed for 3 hours followed by 1 hour vacuum saturation in water, dried at 50°C and 80 % relative humidity (RH) for three days, and sealed for 15 days), and then the bottom surface of specimen is exposed to water to determine total absorption and sorptivity (rate of absorption) for up to eight days. However, this conditioning procedure may give insufficient level of dryness and lower absorption of concrete (Castro et al. 2011), which may affect its reliability to differentiate concretes with variable qualities. Also, the one-dimensional absorption value is highly influenced by the sampling location (i.e. exposed surface to absorption) of concrete specimens/cores, and thus the sampling location should be clearly identified with absorption results. The microstructure of concrete surface may not develop to a similar extent to that of the inner core, for example due effects of

surface temperature and moisture loss. Internal concrete generally has higher capacity of moisture retention, which facilitates cement hydration and microstructural evolution. Therefore, absorption of the surface of concrete is generally higher than its inner core (Senbetta and Scholar 1981; Parrot 1994; Wang et al. 2002; Katib 2014). Water absorption of concrete can also be assessed by full immersion of specimens as described in ASTM C642-13 (2013), NBN B15-215 (1989) and BS 1881-122 (2011). In ASTM C642-13 (2013), specimens with a volume of 350 cm³ are dried at 105°C until mass change in a 24 h period is less than 0.5%; subsequently, the specimens are submerged in water until mass change in a 24 h period is less than 0.5%. In NBN B15-215 (1989), specimens with volume-to-surface ratio of 12 to 20 mm are submerged in water till the change in mass is less than 0.1% in 24 h period. The specimens are then dried at 105°C till the change in mass is less than 0.1 % in 24 h period. In BS 1881-122 (2011), cylindrical discs of 75 mm diameter and thickness are dried at 105°C for 72 h, followed by submersion in water for 30 min. In all these immersion tests, the absorption of concrete is determined as the water uptake of concrete specimens relative to their dry mass. However, it was reported that drying at 105°C can cause severe drying shrinkage of the hydrated cement paste and decomposition of some hydration products, which alter the microstructure of the cementitious matrix and misleadingly give high absorption values (Senbetta and Scholar 1981; McCarter et al. 1992; Parrot 1994; Bai et al. 2002).

Concrete pavements are vulnerable to moisture loss via evaporation due to their high surface-to-volume ratio. Therefore, curing compounds are applied to the surface of concrete pavements immediately after placement and texturing when bleeding is complete (roughly at initial setting) (Taylor et al. 2006; CW 3310-R15 2015). However, tensile stresses may develop in pavements during early-age due to multiple factors including

thermal stresses and restrained shrinkage, resulting in random cracking. This phenomenon can be controlled by saw-cutting to create a plane of weakness and force the concrete pavement to crack at desired locations (joints). The time of saw-cutting for early-age saws is slightly before final setting, and for conventional saws is just after final setting. The saw-cutting intervals for creating transverse contraction joints in plain jointed concrete pavements is between 4.5 and 6.1 m, with a depth of one-quarter to one-third of the pavement thickness and width of 3 to 8 mm (Taylor et al. 2006). Relative to the direction of saw-cut, the joints' geometry in concrete pavements consist of two symmetrical planes to the right and left of the centerline of each joint: the vertical joint wall and top/horizontal surface next to the joint. In current practice, these vertical joint walls are exposed to ambient conditions at early-age, as curing precedes saw-cutting (Taylor et al. 2006). Creating and exposing these surfaces at early-age (final setting) in the absence of curing, make them susceptible to uncontrolled evaporation, especially in hot, arid and windy conditions. Later on during service, these critical surfaces facilitate absorption, which may result in higher degree of saturation with salt solutions (especially in cold regions where de-icing salts are frequently applied to pavements in winter), and in turn aggravate degradation of concrete in these locations. Currently, there are no test procedures available to assess the fluid absorption capacity of concrete with respect to the unique geometry of joints in concrete pavements. Neither the one-dimensional sorptivity test nor the full immersion tests replicate the configuration of joints in concrete pavements; moreover, the fluid specified in these absorption tests is pure water, which does not correlate to field conditions in cold regions. Hence, in the current study an effort was made to develop a customized test protocol for determining the absorption capacity of joints in concrete

pavements, which should help transportation agencies reliably evaluating and projecting the durability/longevity of these critical locations in concrete pavements.

4.2 Methodology

4.2.1 Materials and mixtures

General Use (GU) cement and fly ash (Class F), as a supplementary cementitious material (SCM), meeting the current requirements of CAN/CSA-A3001 (2013) standard were used in all concrete mixtures. Table 4.1 shows the chemical composition and physical properties of GU cement and fly ash. An air-entraining admixture complying with ASTM C260-10 (2010) was used in concrete mixtures to achieve a fresh air content of 5 to 8%. Also, a high-range water reducing admixture (HRWRA) complying with ASTM C494-12, Type F (2012) was used to obtain a target slump of 30 to 70 mm. Locally available natural gravel was used as coarse aggregate with specific gravity and absorption of 2.65 and 2%, respectively. The fine aggregate was well graded river sand with a specific gravity, absorption, and fineness modulus of 2.53, 1.5% and 2.9, respectively. The proportions of the concrete mixtures are given in Table 4.2.

Table 4.1: Chemical composition and physical properties of GU cement and fly ash

	GU	Fly Ash
<u>Chemical Composition</u>		
SiO ₂ (%)	19.8	56.0
Al ₂ O ₃ (%)	5.0	23.1
Fe ₂ O ₃ (%)	2.4	3.6
CaO (%)	63.2	10.8
MgO (%)	3.3	1.1
SO ₃ (%)	3.0	0.2
Na ₂ O (%)	0.1	3.2
CaCO ₃ (%)	--	--
MgCO ₃ (%)	--	--
<u>Physical Properties</u>		
Blaine (m ² /Kg)	410	290
Specific Gravity	3.17	2.12

Table 4.2: Proportions of concrete mixtures per cubic meter

Constituent	Unit	Mix 1	Mix 2	Mix 3	Mix F1 ^a	Mix F2 ^a
20 mm Stone	kg	0	0	0	1170	1213
10 mm Int. Stone	kg	1226	1109	1050	0	0
Sand	kg	646	584	553	655	679
Air entrainment	l	0.22	0.22	0.22	0.25	0.21
Fly Ash (Class F)	kg	51	51	51	57	48
Cement (GU)	kg	289	289	289	323	272
Water	kg	102	170	204	133	128
Water Reducer	l	1.19	1.19	1.19	1.33	1.12
w/b		0.30	0.50	0.60	0.35	0.40
Air Content				5-8%		

^aMix F1 and F2 represent typical concrete pavement mixtures in North America.

4.2.2 Procedures

This three-phase study involved various absorption test procedures to reflect the mass transport of fluids into concrete pavements, especially at joint locations. In Phase I, different absorption procedures were applied on laboratory specimens prepared with water-to-binder ratios (w/b) of 0.3, 0.5 and 0.6 (Mix 1, 2 and 3, respectively) to determine the most effective procedure in terms of sensitivity to this key mixture design variable (w/b). In Phase II, the procedure selected from Phase I was applied to laboratory specimens prepared with a close range of w/b (0.35 and 0.40) to further explore the sensitivity of this method; these mixtures (Mix F1 and F2) represent typical concrete pavement mixtures in the province of Manitoba, Canada. Phase III involved further validation of this procedure using field cores extracted from concrete pavements with the same mixture designs of F1 and F2, with respect to the characteristics of pore structure of concrete as determined by mercury intrusion porosimetry (MIP).

4.2.2.1 Phase I

In this phase, three different concrete mixture designs (Mix 1, Mix 2 and Mix 3) were prepared. For each mixture, twelve 75×150 mm (diameter and height, respectively)

cylindrical specimens were cast in plastic moulds. As curing in the field may range from absence of curing to continuous moist-curing, selection of the curing condition in this phase was driven by adopting the worst-case scenario (i.e. no curing on the top face of specimens). This intensified the effect of moisture evaporation from the surface of concrete. To simulate core samples extracted at joint locations in concrete pavements, the specimens were de-moulded and saw-cut (dry-cut), using a diamond saw, at a depth of 50 ± 3 mm and width of 3 to 4 mm after eight hours of casting. Subsequently, each specimen was sealed with a plastic wrap around the side surface and at the bottom. After 28 days under laboratory conditions ($23\pm 2^{\circ}\text{C}$ and $55\pm 5\%$ RH), the specimens were unwrapped and 50 ± 2 mm thick slices [as per ASTM C1585-13 (2013)] were cut from the top and bottom of cylinders using a water-cooled diamond saw. At this stage, four different absorption procedures were applied to each set of three specimens (three top and three bottom discs). It should be noted that joint (top) samples consisted of split discs, whereas bottom samples consisted of non-split discs.

4.2.2.1.1. Procedure I, City of Winnipeg (COW) Absorption Test

The City of Winnipeg (COW) absorption test, CW3310–R17 (2015), was used since it is adopted in the province of Manitoba, Canada, as a quality control indicator for concrete pavements. In principle, this test method is similar to ASTM C1151-98 (1998), withdrawn in 2000, which is considered as a useful indicator for evaluating the curing efficiency of concrete at different locations of the pavement (CW3310–R17 2015). In this method, full-depth 75 mm cores are extracted from concrete pavement slabs and cut into 50 ± 2 mm slices parallel to the surface of concrete. Absorption of the top slice for each location is compared to that of the bottom slice of the center slab, which is considered the baseline for curing efficiency, presumably representing the optimal location in terms of moisture retention

(Wang et al. 2002). In this study, procedure I consisted of the COW absorption test for the discs cut from concrete cylinders prepared in the laboratory. The discs were placed in a sealed desiccator under a vacuum pressure of 80-85 KPa for 6 h. The specimens were then removed from the desiccator and each one dried out with compressed air. Afterwards, the initial masses of the dried specimens to the nearest 0.01 g were recorded. Each specimen was freely immersed in deionized water for 60 ± 3 s, and then excess water was blotted off and the wet mass of the specimen was recorded within 25 s to the nearest 0.01 g. The percentage of water absorption (WA) is calculated by (CW3310–R17 2015):

$$WA = \frac{M_{WET} - M_{DRY}}{M_{WET}} \times 100 \quad \text{Eq. 4.2}$$

where, M_{WET} is mass of wet, but surface-dry, specimen (g), and M_{DRY} is mass of dry specimen before immersion (g). The absorption ratio of each cylinder was determined by dividing the absorption of the top part by that of the bottom part.

4.2.2.1.2. Procedure II, Modified COW Absorption Test

For efficient drying, the 50 ± 2 mm concrete discs were placed in an environmental chamber at $50 \pm 2^\circ\text{C}$ and 40% RH until reaching a constant mass, and then placed in a sealed desiccator under vacuum pressure (~ 85 KPa) for 6 h. Afterwards, the specimens were removed from the desiccator and the initial dry mass to the nearest 0.01 g was recorded. Each specimen was freely immersed in deionized water for up to 360 min, and the amounts of absorption after 1, 5, 10, 20, 40, 80, 160 and 360 minutes were recorded to the nearest 0.01 g. It is well-documented that the rate of absorption of fluids into concrete diminishes with time due to the progressive saturation of specimens (ASTM C1585-13 2013); therefore, recording the absorption value within a relatively short time interval up to 360 min may be a reasonable measure to differentiate between concretes with variable qualities

based on capillary suction. This is similar to the time interval for initial absorption stipulated by ASTM C1585-13 (2013). The percentage of water absorption at a specific point of time (A_t) was calculated by:

$$A_t = \frac{m_t}{M_{DRY}} \times 100 \quad \text{Eq. 4.3}$$

Where, m_t is the difference between M_{WET} and M_{DRY} at a specific point of time, and M_{DRY} is mass of dry specimen before immersion (g).

4.2.2.1.3. *Procedure III, Protocol for an Absorption Test for Joints*

This protocol consisted of assessing the water absorption of the vertical joint walls plus the top surfaces of the specimen. The 50 ± 2 mm concrete discs were placed in an environmental chamber at $50 \pm 2^\circ\text{C}$ and 40% RH until reaching a constant mass (difference in mass in two successive days is less than 0.1%). All surfaces except the joint walls and top surfaces were sealed by a rapid setting epoxy (Figure 4.1). Afterwards, the specimens were placed in a sealed desiccator under vacuum pressure (~ 85 KPa) for 6 h and their initial masses to the nearest 0.01 g were recorded. The specimens were then submerged in deionized water for up to 360 min, and the amount of absorption of specimens after 1, 5, 10, 20, 40, 80, 160 and 360 minutes were calculated similar to procedure II (Eq. 4.3).

4.2.2.1.4. *Procedure IV, Modified Protocol for an Absorption Test for Joints*

The proposed protocol (Procedure III) was repeated, except that 4% calcium chloride (CaCl_2) solution was used instead of deionized water. This simulates ingress of de-icing salts in concrete pavements. This type and concentration of salt is adopted by ASTM C672/C672M-12 (2012). Valenza and Scherer (2007) postulated that this concentration of salt is deleterious to concrete, when combined with freezing-thawing cycles.

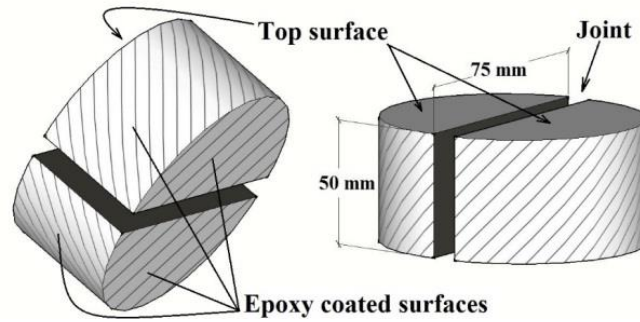


Figure 4.1- Typical top part of sealed specimens mimicking cores extracted at joints in concrete pavement for procedures III, and IV. (Note: This is the same geometry for unsealed specimens used in procedure I and II)

4.2.2.2 Phase II

Based on the results of Phase I, the most efficient test procedure was selected to focus on concrete mixtures F1 and F2 with a close range of w/b (0.35 and 0.40) to test the sensitivity of the method selected, especially that these w/b are common for concrete pavements in North America. Also, the vacuuming step (no vacuum, or 6 h vacuuming) and solution concentration (4%, or 20% CaCl_2) were varied to check their effect on the absorption values.

4.2.2.3 Phase III

Based on the results of Phases I and II, an absorption test protocol was selected and applied to field cores to further validate its reliability for evaluating concrete pavement. Two pavement sections were cast with Mix F1 and F2 according to the COW construction specifications (CW 3310-R15 2014), in an urban arterial road at the central area of Winnipeg, Manitoba. The COW used an approved ready mix supplier to provide the desired mixture designs, and the supplier made multiple batches to ensure compliance with the proportions of mixture designs. Also, the water content of fresh concrete was measured

by the microwave method (CSA-A23.2-18C 2014) to verify the water contents. After subtraction of water absorption of aggregate, the w/b came to be in the range of 0.345-0.358 and 0.391-0.423 corresponding to the target (nominal) w/b of 0.35 and 0.40, respectively. The pavement sections consisted of a 150 mm thick jointed plain concrete pavement with a slab width of 3.70 m on a granular base. Shortly (30 to 45 min) after the finishing operations, one coat of curing compound conforming to ASTM C309-11 (2011) Types 1, 2 and 1-D, Class B was applied to the top surface of pavement. Applying a curing compound should provide better hydration development for the surface of concrete pavement relative to the laboratory specimens (no curing) prepared in Phase II, which would capture the reliability of the absorption procedure with respect to the variation in the curing method. The pavements slabs were saw-cut (dry-cut) eight hours after casting to establish the contraction joints with the dimensions of 3 to 4 mm wide and 50 ± 3 mm deep. For each w/b, six cores were obtained directly at transverse joints (split cores) and another six cores were obtained from the center of slab (mid-slab) at 28 days for the absorption and MIP tests. The curing compound residuals were removed with a steel wire brush, and 50 ± 2 mm thick slices were cut from the top part of cores at joints. Also, 10 mm of the bottom part of the mid-slab cores were removed followed by cutting 50 ± 2 mm discs [baseline for optimal curing (Wang et al. 2002)]. The absorption procedure selected from the previous phases was performed on these discs to compare field concrete with close w/b (0.35 and 0.40) and identify the difference in quality of the cores at the top part of joint relative to the bottom of mid-slab. These results were presented as the absorption (I) of concrete specimens by:

$$I_a = \frac{m_t}{a \cdot \rho} \quad \text{Eq. 4.4}$$

Where, I_a is absorption depth of concrete at a certain point of time (mm), m_t is the difference between M_{WET} and M_{DRY} in g at a specific point of time, a is the cross sectional area of the exposed surface (mm^2), and ρ is density of fluid (g/mm^3).

To complement the trends observed in the absorption test and correlate them to the characteristics of the pore structure, MIP was conducted on the field concrete cores at 28 days. Small pea-sized chunks taken from at least two concrete cores were used as test samples for MIP (Kumar and Bhattacharjee 2003; Said et al. 2012). To prepare samples for MIP, bigger chunks (8-12 mm) were first extracted from the cores, visually inspected and an effort was made to remove large aggregate. This process produced the best possible 16-22 chunks with the size of 4-7 mm, approximately representing the concrete micro-structure of the two cores at specific locations in the pavement. These locations were the joint walls of the concrete cores (split cores) and bottom 25 mm of the cores extracted at mid-slab. The samples were oven dried for 72 h at a temperature of 45 ± 2 °C; they were then kept in a desiccator containing calcium sulfate for 24 h. This method of drying (lower temperature for a longer period) was adopted to avoid decomposition of hydrated paste and formation of micro-cracks, which may occur at high temperatures. The contact angle and the surface tension of mercury were taken as 130° and 485 dynes/cm, respectively (Shi and Winslow 1985; Said et al. 2012).

4.2.3 Statistical Analysis

All the results of absorption tests were statistically evaluated by the analysis of variance (ANOVA) method, Tukey's post hoc test, unequal-variance two-tail t -test and F -test, at a significance level (α) of 0.05. ANOVA is a statistical model often used to analyze the

differences between groups' means and their variations among groups. The variance of a specific parameter is separated into components attributed to different sources of variations. ANOVA tests the hypothesis that the means of several groups are comparable via a generalized t -test for more than two groups. According to ANOVA, exceeding the F -critical (F_{cr}) for F -distribution density function indicates that the variable tested has a statistically significant effect on the average results (Montgomery 2012). If applicable, the ANOVA results were further statistically evaluated by Tukey's post hoc test (Tukey's test) to declare pairwise comparisons between the means of each two groups. Tukey's post hoc test declares two means significantly different if the absolute value of their sample differences exceeds a critical range, q_{cr} (Montgomery 2012). In the two-tail t -test, exceeding the t -critical (t_{cr}) for t -statistic indicates that the variable tested has a statistically significant effect on the groups' means. For groups with a similar sample size, the t -statistic is calculated as the ratio of the difference between the means divided by square root of the summation of variances over the sample size. In the F -test, the variances of two groups are classified statistically unequal (heterogeneous) if the ratio of the larger variance to the smaller one (F -statistic) exceeds the F_{cr} , then the p -value from the F distribution has to be doubled to assign a confidence interval for a two-tail test (Montgomery 2012). When there is a desire to compare the central tendency of two populations based on samples from unrelated data, the unequal-variance t -test (Welch's t -test) should be used in preference to the equal-variance t -test (Moser et al. 1989; Ruxton 2006). Also, it has been reported that using a preliminary test to compare variances (e.g. F -test) as a measure to decide which t -test (equal or unequal variances) to use, increases the risk of type I error (rejecting the null hypothesis when it is true) (Moser and Stevens 1992; Hayes and Cai 2007). The general method in hypothesis testing is to specify a value of α (e.g. 0.05) as the probability of type

I error (Montgomery 2012). Therefore, in this study Welch's t -test was used to compare the means of two groups from unrelated data (due to applying different absorption procedures), while the F -test was performed to check the homogeneity of their variances.

4.3 Results and Discussion

4.3.1 Phase I

The ANOVA for all absorption results from the four procedures is shown in Table 4.3 to generally determine the statistical significance of each protocol. While all the F values in Table 4.3 are larger than the F_{cr} of 5.14, indicating the success of all tests in capturing the difference among the three w/b ratios (0.3, 0.5 and 0.6), further statistical analysis by other methods is needed to validate this finding. The absorption and Tukey's test results for procedure I (COW-2013-01 2013) are given in Table 4.4. As expected, higher w/b led to higher absorption values for the top and bottom parts of the specimens, which can be ascribed to the higher porosity and interconnectivity of the pore structure. For example, changing the w/b (0.3, 0.5 and 0.6) in the top part of specimens had F value of 53.04 compared to F_{cr} of 5.14 (Table 4.3), and the pairwise comparison of the absorption percentage were 0.34, 0.21 and 0.55 for w/b pairs of 0.3 and 0.5, 0.5 and 0.6, and 0.3 and 0.6, respectively, which were larger than the corresponding q_{cr} of 0.16 (Table 4.4). These absorption trends indicate that procedure I was capable of providing distinctive trends among the different w/b.

The results of absorption ratios at each w/b show the effect of higher absorption in the top (evaporative zone) relative to the bottom part (not subjected to moisture loss during the hydration process) of specimens (Table 4.4), which confirms the vulnerability of this part of the joint to less hydration development as discussed in the introduction section. However, unlike the absorption percentage trends, the results of absorption ratios did not

show a distinctive trend with respect to the variation in w/b; the absorption ratios for mixtures with w/b of 0.3, 0.5 and 0.6 were 3.68, 5.02 and 3.35, respectively. In addition to ANOVA, these results were further evaluated by Tukey's test to investigate differences in each possible pair of absorption ratios. If all possible pairwise comparisons declare significant differences, it would be valid to accept the test procedure's sensitivity in capturing the difference among all w/b.

Table 4.3: ANOVA for the absorption results from procedures I, II, III, and IV

Procedure	<i>F</i>								<i>F_{cr}</i>
	1	5	10	20	40	80	160	360	
I	53.04 ^a (7.17 ^a)	-	-	-	-	-	-	-	5.14
II	222.15 ^a	140.43 ^a	59.65 ^a	46.86 ^a	41.90 ^a	38.95 ^a	35.88 ^a	32.83 ^a	
III	15.46 ^a	14.42 ^a	13.54 ^a	13.21 ^a	13.39 ^a	12.85 ^a	10.44 ^a	8.54 ^a	
IV	49.99 ^a	117.23 ^a	203.40 ^a	326.53 ^a	661.69 ^a	1057.45 ^a	353.75 ^a	313.76 ^a	

Note: The value in bracket is for the absorption ratios; ^aIndicates statistical significance.

Table 4.4: The results of absorption, absorption ratios, and Tukey's test for procedure I

w/b	Top				Bottom			Top/Bottom		
	Absorption [%]	Difference	<i>q_{cr}</i>		Absorption [%]	Difference	<i>q_{cr}</i>	Absorption ratio	Difference	<i>q_{cr}</i>
0.3	0.24	0.34 ^a			0.07	0.05 ^a		3.68	1.34	
0.5	0.58	0.55 ^a	0.16		0.12	0.17 ^a	0.02	5.02	0.33	1.44
0.6	0.79	0.21 ^a			0.24	0.12 ^a		3.35	1.67 ^a	

^aIndicates statistical significance.

Although the ANOVA results (Table 4.3) revealed a significant difference among the absorption ratios, pairwise comparisons by Tukey's test (Table 4.4) declared a significant difference only between w/b of 0.5 and 0.6. While changing the w/b (0.3, 0.5 and 0.6) had *F* value of 7.17 compared to *F_{cr}* of 5.14, the pairwise comparisons of the absorption ratios were 1.34, 1.67 and 0.33 for w/b pairs of 0.3 and 0.5, 0.5 and 0.6, and 0.3 and 0.6, respectively, compared to *q_{cr}* of 1.44 (Table 4.4). These trends suggest that procedure I with its criterion (absorption ratio) is not sensitive to the change in w/b. In addition, as will

be shown later in the text, the one minute test duration for absorption may not be enough to reflect the available open pores in the surface of concrete which are susceptible to the process of capillary suction during service. Therefore, it may be unreliable to estimate the quality of concrete microstructure on the basis of procedure I.

The results of absorption and Tukey's test for procedures II, III and IV are given in Figure 4.2. The specimens used in these tests were the top part of specimens due to its critical nature as discussed earlier. For procedure II, ANOVA results indicated significant differences among the absorption values of concrete mixtures with different w/b starting from 1 min up to 360 min (Table 4.3). For instance, for absorption of concrete at 1 min, changing the w/b (0.3, 0.5 and 0.6) had F value of 222.15 compared to a critical value (F_{cr}) of 5.14. For one minute absorption of concrete, procedure II yielded about three times higher absorption of concrete at the same w/b than that of procedure I. This signified the effect of the oven drying step implemented in procedure II, which is needed to condition specimens to a drier state prior to the absorption test, and thus better reflecting the process of capillary suction in the surface layer of concrete. However, pairwise comparisons declared significant differences in absorption only up to 5 min, whereas the absorption values after 5 min did not show statistical difference between the w/b pairs of 0.5 and 0.6 (Figure 4.2(a)). It appears that the sensitivity of procedure II diminished after 5 min of absorption; thus, similar to procedure I, the reliability of procedure II to assess the quality of microstructure of concrete in the top joint location is questionable. Similar to procedure II, the ANOVA for procedure III results showed significant differences among the absorption values starting from 1 min up to 360 min with varying the w/b; however, pairwise comparisons by Tukey's test declared no statistical significance between the

absorption of w/b pairs of 0.5 and 0.6 (Figure 4.2(b)). Again, this indicates that procedure III did not have adequate sensitivity to the change of microstructure of concrete.

It can be noted that the results from procedures II and III for the top part of the specimens with similar w/b (0.3, 0.5 or 0.6) showed no significant differences in the total water absorption after 360 min (Figure 4.2(a) and Figure 4.2(b), and Table 4.5). For example, specimens with a w/b of 0.3 had a total absorption of 3.0 and 2.7% according to procedures II and III, respectively. This was statistically supported by Welch's t -test, as for a w/b of 0.3 the absorption results at 360 min had a t value of 1.42 which is smaller than the critical value (t_{cr}) of 2.31 (Table 4.5). This indicates that sealing the side and bottom surfaces of the specimens in procedure III did not significantly minimize the total amount of water absorption in comparison to the unsealed specimens (procedure II) despite that the exposed surface area of each specimen (116.63 cm^2) in procedure III is about 43% of the one (272.76 cm^2) used in procedure II. Since the specimens in procedure III simulate the critical surfaces at joint locations in concrete pavements, it can be substantiated that the joint geometry selected herein (vertical joint walls and top surfaces) facilitates a high degree of infiltration, which may be linked to the vulnerability of these locations to deterioration in the field.

Although the absorption results showed no significant differences between procedure II and III, a marked increase was captured for the variances of the two methods at a w/b of 0.6. This was statistically shown by the F -test for the total absorption results (360 min) of procedures II and III at a w/b of 0.6, as the F value was 18.48 which is higher than the critical value (F_{cr}) of 5.05, with a p -value of 0.006 (Table 4.5). In procedure II (similar to procedure 1), a bulk volume of well cured concrete is included in the test specimens, which may mask the true characteristics of the pore structure of the critical

faces. This signifies the effect of epoxy coating to isolate the critical faces and capture their absorption trends due to heterogeneous microstructure, especially at higher w/b, originating from moisture evaporation from these surfaces during curing.

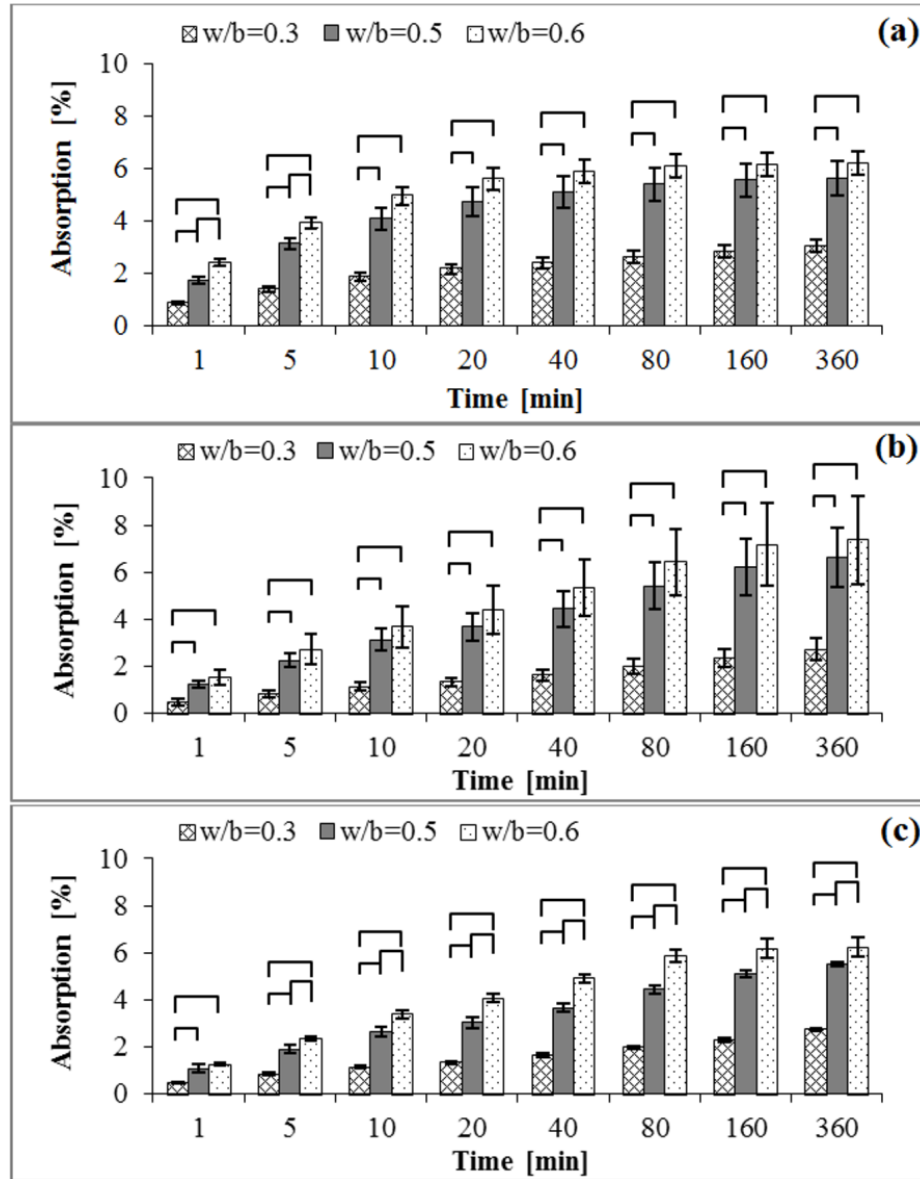


Figure 4.2- Absorption of the top part of concrete specimens according to: (a) procedure II, (b) procedure III, and (c) procedure IV. (Notes: The error bars represent standard deviation; pairwise statistically significant differences based on Tukey's test are designated by "□")

Similar to the previous procedures, the ANOVA for the results of procedure IV indicated significant differences among the absorption results with changing the w/b starting from 1 min up to 360 min (Table 4.3). Unlike procedures II and III, pairwise comparisons by Tukey's test showed significant differences between each pair of w/b starting at 5 min up to 360 min (Figure 4.2(c)). The trends from procedure IV suggest that the capillary suction process needs to be established within a period of time longer than 5 min because of a marked increase in the volume of fluid uptake within the pore space, which leads to a clear distinction among concrete with different microstructural features. For instance, at a w/b of 0.30, the volume of fluid absorbed by concrete at 1 min and 360 min was 2.4 and 13.7 cc, respectively. Hence, evaluating the quality of concrete by a very short time (e.g. 1 min in procedure I) absorption test is not reliable.

The sensitivity of procedure IV to differentiate among the concrete specimens with different w/b was tested with further statistical analysis (Table 4.5). The results indicated that the total absorption (at 360 min) of the top part of the specimens with the same w/b had no significant differences when the fluid changed from deionized water (procedure III) to 4% CaCl₂ solution (procedure IV). For example, specimens with a w/b of 0.5 had a total absorption of 6.6 and 5.6% according to procedures III and IV, respectively. This was statistically supported by Welch's *t*-test (Table 4.5) as the *t*-test of the total absorption results (360 min) at a w/b of 0.5 from procedures III and IV yielded *t* value of 2.24, which was smaller than the critical value (*t_{cr}*) of 2.57. Absorption through pore structure of concrete is proportional to the square root of surface tension (γ) to fluid viscosity (η) [Eq. 4.1], with other factors being almost unchanged (fluid-solid contact angle and pore structures) (Kelham 1988). Therefore, comparable absorption results in both fluids may be

obtained as the ratio of $\sqrt{\frac{\gamma}{\eta}}$ for 4% CaCl_2 solution and deionized water is almost 1.0 (Conde 2004). However, the results of the F -test revealed significant reduction in variances, when the absorbed fluid changed from deionized water to 4% CaCl_2 solution. For example, at a w/b of 0.5, the analysis of results showed F value of 30.02, which was greater than the critical value (F_{cr}) of 5.05, with a p -value of 0.002 (Table 4.5). This significant reduction in variances started to appear just after one minute absorption up to 360 min for w/b of 0.3 and 0.6, and after ten minutes for w/b of 0.5, which can be noted by comparing the error bars depicted in Figure 4.2(b) and Figure 4.2(c). It appears that the effect of ionic traction from absorbing a low concentration (4%) salt solution within the concrete surface led to homogeneous volume of solution uptake among the surface of replicates of the same mixture, which discounted the effect of pore size (r) variability, resulting in improved repeatability of results. Therefore, the overall outcome of Phase I suggested that procedure IV was the most effective procedure in terms of sensitivity to the key mixture design variable (w/b) tested in this study. Further validation for this procedure was carried out in Phases II and III to confirm robustness of results.

Table 4.5: Welch's t -test results for total absorption at 360 min of the top part of specimens and F -test results for the associated variances

Procedures	w/b	Welch's t -test			F -test		
		t	t_{cr}	p -value	F	F_{cr}	p -value
II & III	0.3	1.42	2.31	0.196	3.65		0.182
	0.5	1.80	2.31	0.112	3.71	5.05	0.176
	0.6	1.48	2.45	0.192	18.48		0.006 ^a
III & IV	0.3	0.03	2.57	0.976	30.02		0.002 ^a
	0.5	2.24	2.57	0.074	166.30	5.05	0.000 ^a
	0.6	1.43	2.57	0.207	21.98		0.004 ^a

^aIndicates statistical significance.

4.3.2 Phase II

To further evaluate the sensitivity of the test method selected in Phase I (procedure IV), two other concrete mixtures (F1 and F2) with a close range of w/b (0.35 and 0.40), which are typical of concrete pavement mixtures in North America, were tested in Phase II. Three absorption procedures were applied in Phase II: procedure IV (identified from Phase I), procedure IV without vacuum drying (instead of 6 h vacuum drying) and procedure IV with 20% CaCl₂ solution (instead of 4% CaCl₂ solution). The results and analysis of Phase II are shown in Figure 4.3 and Table 4.6. The pairwise comparison of data indicated significantly higher absorption due to changing the w/b from 0.35 to 0.40 for the data sets starting from 5 min up to 360 min [Figure 4.3(a)]. This substantiated the efficacy of procedure IV and further indicated the sensitivity of this protocol in providing a clear-cut, even among concretes of almost similar quality (narrow range of w/b). In addition, for procedure IV, the *F*-test results showed no significant difference between the variances of absorption data for mixtures with w/b of 0.35 and 0.40 starting from 1 min up to 360 min (Table 4.6), which suggests the consistency and repeatability of this test protocol.

Figure 4.3(b) shows the results of procedure IV without vacuum drying prior to the absorption test. According to Welch's *t*-test, this omission reduced the procedure's sensitivity to an extent that only two time points (5 and 20 min) gave significant distinction among specimens with w/b of 0.35 and 0.40. Correspondingly, the *F*-test showed that differences between the variances of data sets were insignificant up to 20 min; however, significant differences in variances were obtained from 40 min up to 360 min (Table 4.6). This may indicate that eliminating the vacuum step, after drying in the environmental chamber, led to considerable moisture differences within the outer skin of specimens with different w/b, as suggested by the absence of uniform variances among the absorption data

up to 360 min. This is augmented by the analysis shown in Table 4.7, which shows the pairwise comparison between 6 h vs. no vacuum drying for the total absorption results at the same w/b. The results indicated the vacuum drying step had a significant effect to cause a systematically higher absorption with time at each w/b. For example, concrete specimens with w/b of 0.35 that received 6 h vacuuming had an increase of the total absorption (360 min) by about 22% relative to the corresponding specimens without vacuuming. This led to t value of 8.22 which was greater than t_{cr} of 4.30; this can be attributed to the effect of vacuuming at conditioning the specimens initially to a drier state prior to the absorption test.

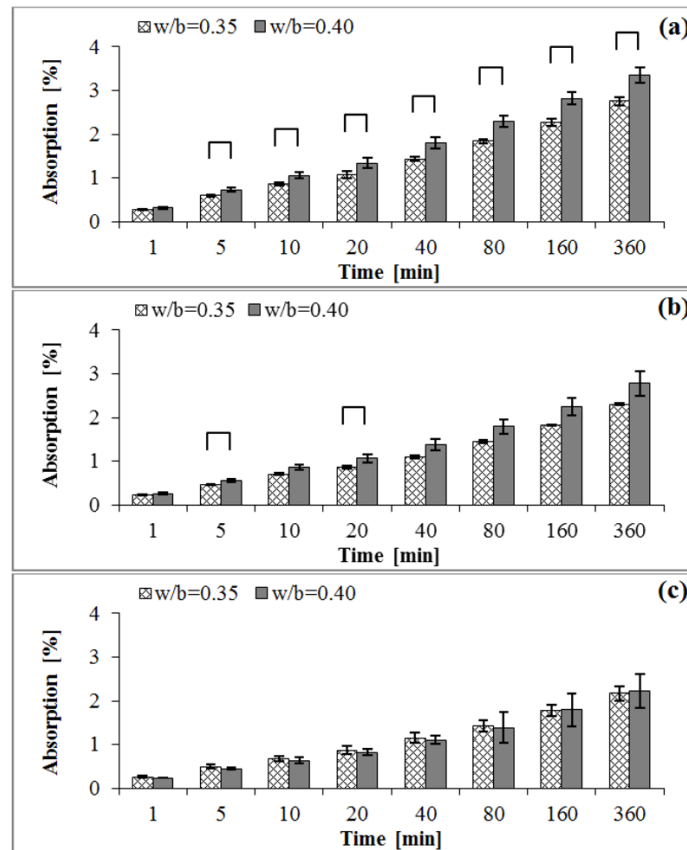


Figure 4.3- Absorption of top part of concrete specimens using procedure IV: (a) 6 h vacuuming, 4% CaCl_2 , (b) no vacuuming, 4% CaCl_2 , and (c) 6 h vacuuming, 20% CaCl_2 . (Notes: Statistically significant differences based on Welch's test are designated by "□")

Table 4.6: *F*-test results for the variances of absorption data of the top part of Phase II specimens (w/b of 0.35 and 0.40)

Time [min]	Proc. IV: 6 h vacuum, 4% CaCl ₂		Proc. IV: no vacuum, 4% CaCl ₂		Proc. IV: 6 h vacuum, 20% CaCl ₂	
	<i>F</i>	<i>p</i> -value	<i>F</i>	<i>p</i> -value	<i>F</i>	<i>p</i> -value
1	9.99	0.182	5.74	0.296	2.78	0.517
5	2.70	0.540	7.76	0.228	2.20	0.625
10	5.12	0.326	7.90	0.224	1.15	0.930
20	1.58	0.776	4.74	0.348	1.77	0.721
40	8.90	0.202	19.24	0.098 ^a	1.50	0.801
80	5.49	0.308	36.63	0.054 ^a	7.23	0.243
160	3.60	0.434	219.17	0.010 ^a	7.44	0.237
360	3.45	0.450	116.78	0.016 ^a	5.20	0.322

Note: $F_{cr}=19.00$; ^aIndicates statistical significance.

Table 4.7: Welch's *t*-test for absorption of the top part of Phase II specimens considering modifications to procedure IV

Time [min]	6 h vacuuming vs. no vacuuming						4% CaCl ₂ vs. 20% CaCl ₂					
	w/b=0.35			0.40			0.35			0.40		
	<i>t</i>	<i>t_{cr}</i>	<i>p</i> -value	<i>t</i>	<i>t_{cr}</i>	<i>p</i> -value	<i>t</i>	<i>t_{cr}</i>	<i>p</i> -value	<i>t</i>	<i>t_{cr}</i>	<i>p</i> -value
1	7.25	2.78	0.002 ^a	3.02	2.78	0.041 ^a	0.61	3.18	0.589	5.10	3.18	0.018 ^a
5	7.37	3.18	0.006 ^a	4.80	2.78	0.009 ^a	3.60	2.78	0.028 ^a	9.25	3.18	0.002 ^a
10	6.69	3.18	0.004 ^a	3.60	2.78	0.024 ^a	4.42	3.18	0.020 ^a	7.38	2.78	0.002 ^a
20	4.13	3.18	0.035 ^a	1.69	2.78	0.167	2.94	2.78	0.042 ^a	10.82	2.78	0.000 ^a
40	11.64	2.78	0.001 ^a	4.27	2.78	0.013 ^a	4.04	4.30	0.056	7.96	2.78	0.002 ^a
80	11.00	3.18	0.002 ^a	4.01	2.78	0.019 ^a	5.03	3.18	0.020 ^a	4.10	3.18	0.036 ^a
160	10.31	4.30	0.007 ^a	4.05	2.78	0.019 ^a	5.46	3.18	0.011 ^a	4.43	3.18	0.030 ^a
360	8.22	4.30	0.009 ^a	3.30	3.18	0.048 ^a	5.30	3.18	0.012 ^a	4.63	3.18	0.023 ^a

^aIndicates statistical significance.

Figure 4.3(c) shows the results of procedure IV using 20% CaCl₂ solution. According to Welch's *t*-test in this figure, the high salt concentration eliminated the procedure's sensitivity to distinguish between the absorption of concrete with w/b of 0.35 and 0.40 at all points of time, without differences between the higher variances of the two datasets starting from 1 to 360 min (*F*-test, Table 4.6). In addition, considering the pairwise comparison between the total (360 min) absorption of 4% vs. 20% CaCl₂ at the same w/b (Table 4.7), the trends indicated that the higher concentration of the salt solution had a significant effect on decreasing the absorption of concrete at each w/b. For example, specimens with w/b of 0.40 had total absorption values of 2.2% and 3.4% (35% reduction),

respectively when exposed to 20% and 4% CaCl_2 solutions; this reduction is statistically significant according to Welch's t -test, as the total absorption results at a w/b of 0.40 had t value of 4.63, which was greater than t_{cr} of 3.18 (Table 4.7). This trend is in agreement with earlier studies (MacInnis and Nathawad 1980; Spragg et al. 2011) which reported that higher salt concentrations ($>4\%$) led to lower absorption of concrete. Since the ratio of $\sqrt{\frac{\gamma}{\eta}}$ of the 20% CaCl_2 solution to that of deionized water, and 4% CaCl_2 solution, is about 82% (Conde 2004), the total absorption of concrete was notably reduced to the extent that a clear distinction between concrete with w/b of 0.35 and 0.40 could not be obtained (Figure 4.3(c)). Therefore, the overall outcomes of Phase II substantiated that procedure IV (without modifications) is still the most sensitive procedure to the key mixture design variable (w/b) tested in the present study.

4.3.3 Phase III

Based on the findings of Phases I and II, procedure IV was applied to cores extracted from field sections made from mixtures F1 and F2 (similar mixture designs to the ones tested in Phase II) for further validation of the test protocol. The absorption and the statistical analysis results of Phase III are shown in Figure 4.4. Again, Welch's t -test results indicated significantly higher absorption due to changing the w/b from 0.35 to 0.40, starting from 20 min up to 360 min with uniform variances (Figure 4.4). In conformance to the trends of Phase II, this indicated that procedure IV was sensitive to providing a clear-cut for field concrete prepared with a narrow range of w/b (0.35 and 0.40). It was noted that the total absorption of field cores (Figure 4.4) had 46% reduction in comparison to the corresponding laboratory specimens tested in Phase II [Figure 4.3(a)]. This can be ascribed to the effect of better hydration of the cementitious matrix due to the application of a curing

compound and more efficient consolidation of field concrete by a series of internal vibrators of the slipform paving machine. Hence, procedure IV was also able to capture the effect of the curing method on the quality of concrete at the same w/b.

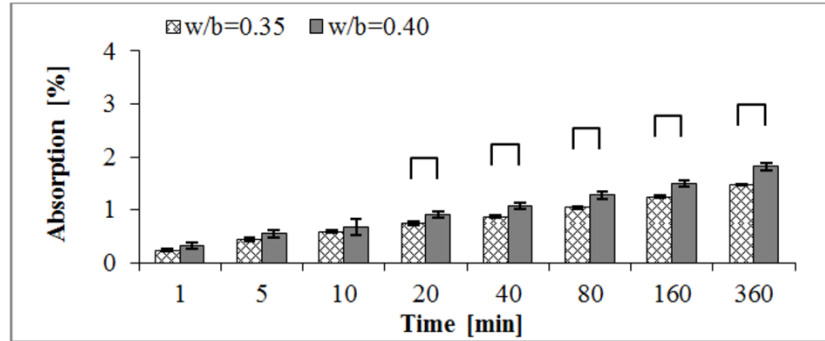


Figure 4.4- Absorption (procedure IV) of the top part of field cores extracted at transverse joints after 28 days. (Note: statistically significant differences based on Welch's *t*-test are designated by "□")

In addition to the previous data, the absorption of concrete can be expressed in terms of depth of penetration considering the density of fluid and exposed surface area (Eq. 4.4), as shown in Figure 4.5. These results represent absorption of the top part of the cores at joint locations and the bottom part of the cores at mid-slab (optimal location in terms of moisture retention and hydration development). The effect of higher absorption in the top part at joints relative to the bottom part at mid-slab started after 20 min up to 360 min at both w/b, which was statistically supported by Welch's *t*-test. Similar to the trends observed in Phase I, procedure IV in Phase III substantiated the notion that joints are more vulnerable locations to ingress of fluids, and consequently saturation and damage during service. It is important to re-emphasize that the exposed surface of sealed specimens in procedure IV is 43% of the total area of specimens, but this represents the vulnerable/critical surfaces at joints. In addition, the general trends (Figure 4.4, and Figure

4.5) from Phase III substantiate the argument that an absorption test of concrete (even for a high sensitivity protocol such as procedure IV) should be run for more than 40 min to distinctively establish cumulative absorption of fluid within the surface skin of concrete over time, and thus provide robust trends and reliable conclusions on the quality of concrete tested. Therefore, absorption tests such as procedure I that run for very short periods of time (e.g. 1 or 5 min) might give misleading conclusions about the quality of concrete microstructure.

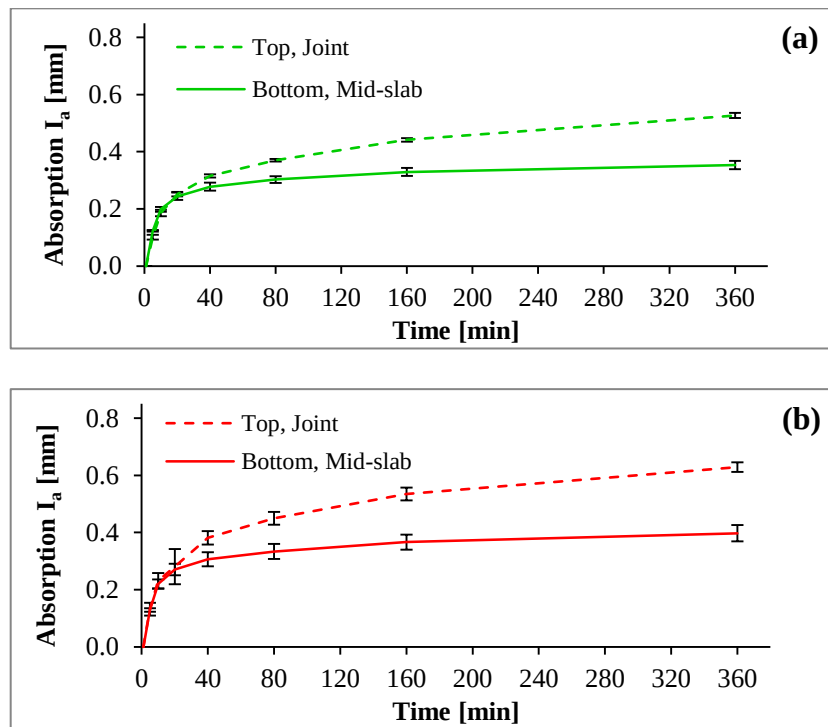


Figure 4.5- Absorption of field cores (procedure IV): (a) $w/b = 0.35$ and (b) $w/b = 0.40$

The results of mercury intrusion porosimetry (MIP) for the field cores obtained from the joints and mid-slabs are shown in Figure 4.6 and Table 4.8. The results shown in Table 4.8 can be considered the averages of representative populations (Kumar and Bhattacharjee 2003) and approximate depiction of the physical features of microstructure for the concrete cores tested in this phase. The effect of increasing the w/b was captured

by MIP in terms of higher proportion of macro-pores, threshold pore diameter and total porosity (Table 4.8), which conforms to the well-documented effect of w/b on the microstructure of concrete (Mehta and Monteiro 2014). This complied with the absorption results of procedure IV for cores at joints in the sense that there was 20% higher total absorption (at 360 min) due to increasing the w/b from 0.35 to 0.40 (Figure 4.4). For both w/b, the results of MIP showed an increase in the total volume of intrusion and total porosity in cores at the joints relative to the ones at mid-slabs (Table 4.8). Correspondingly, the proportion of macro-pores ($>0.1 \mu\text{m}$) was higher (13 to 15%) for the cores extracted at the joints.

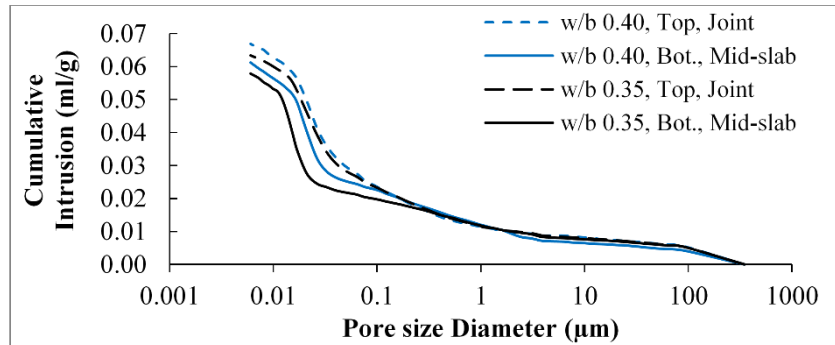


Figure 4.6- Pore size distribution of field cores at 28 days.

Table 4.8: Mercury intrusion porosimetry (MIP) test results for field cores at 28 days

w/b	location	Intrusion (ml/g)			Threshold Pore Diameter (μm)	Apparent Total Porosity (%)
		Total	Micro-Pores ($<0.1 \mu\text{m}$)	Macro-Pores ($>0.1 \mu\text{m}$)		
0.35	Top, Joint	6.3×10^{-2}	4.0×10^{-2}	2.3×10^{-2}	2.10	13.5
	Bottom, Mid slab	5.8	3.8	2.0	0.03	12.3
0.40	Top, Joint	6.7	4.1	2.6	3.10	14.1
	Bottom, Mid slab	6.1	3.9	2.3	0.05	13.0

The threshold pore diameters for the cores at the joints were 2.10 and 3.10 μm at w/b of 0.35 and 0.40, respectively, which were one order of magnitude higher than the lowest threshold of macro-pores (0.1 μm). This is ascribed to the ease of percolation in the macro-

pores range (Figure 4.6), which facilitated the absorption process in cores extracted at the joints. The threshold pore diameters for cores at the joints were markedly greater than those of the corresponding cores at the bottom of mid-slabs at both w/b. For instance, at a w/b of 0.35, the threshold pore diameters were 2.10 (macro-pores range) and 0.03 (micro-pores range) μm for the cores at the joints and bottom of mid-slab, respectively. Indeed, all the trends from MIP are in good agreement with the significantly higher absorption of cores at the joints relative to the corresponding ones at mid-slabs for both w/b (Figure 4.5), which further validates the efficacy and reliability of procedure IV in reflecting the physical features of microstructure of field pavement.

CHAPTER 5: INVESTIGATION INTO ENHANCING AND EVALUATING CURING EFFICIENCY OF JOINTS IN CONCRETE PAVEMENTS

This chapter evaluates the effect of overfilling joints with curing compound immediately after saw-cutting (early- and late-cuts) on improving the quality of concrete microstructure at joint locations in laboratory slabs and trial field sections.

5.1 Introduction

Premature deterioration near to and at the joints still present a critical durability issue of concrete pavements associated with considerable repair costs (Sutter et al. 2006; Leech et al. 2008). The United States Federal Highway Administration (FHWA) estimates that \$85 billion would be needed in annual capital investment up to 2028 to be directed towards improving the physical conditions of existing road assets in order to achieve the Department of Transportation's State of Good repair benchmark for a better ride quality (ASCE 2013). Joint deterioration in concrete pavements is considered as one of the most serious materials-related distresses, for example due to environmental effects such as freeze-thaw damage, especially with the application of de-icing salts in cold regions (Litvan 1976; Şahin et al. 2010). The distress may be observed as shadowing at first, when microcracking near the joints traps water; the development of cracking later parallel to the joint tends to cause disintegration at joints and loss of materials. Joints with their unique geometry (width and depth), are vulnerable locations to higher degree of infiltration since these locations facilitate two-dimensional absorption (from vertical joint wall and top surface near the joint) at each side of the joint (Tiznobaik and Bassuoni 2017c). During service, these critical surfaces facilitate a high degree of saturation with salt solutions, and

in turn aggravate degradation of concrete pavement in joint locations. It has been reported premature joint deterioration of concrete pavements are strongly linked to the penetrability of concrete (MDOT 1996; Chen et al. 2011; Liu et al. 2014), which is highly affected by the efficacy of curing, saw-cutting operations and the consequent evolution of microstructure even with a well-accepted concrete mixture design.

Curing is maintaining a satisfactory level of moisture content (RH above 90%) and temperature in concrete for a period of time (preferably seven days or more) immediately following placing and finishing procedures to attain the desired properties. It has been reported applying a thorough coat of curing compound at a rate of 0.23 l/m^2 is essential to ensure moisture retention in concrete pavements (elements with high surface-to-volume ratio) for efficient hydration reactions and microstructural development (Tiznobaik et al. 2015). However, severe tensile stresses may develop during the first 72 hours after placement due to multiple factors including thermal stresses and restrained shrinkage, resulting in uncontrolled and random cracking (FHWA 2007). This phenomenon can be controlled by saw-cutting to create planes of weakness and force the concrete pavement to crack at those desired locations (transverse joints). However, saw-cutting creates two joint walls at early-age, and since in current practice no remedial action in terms of curing is typically applied after saw-cutting, joint walls will be susceptible to uncontrolled evaporation. This can lead to coarse microstructure at joint locations with respect to the rest of the pavement sections, and consequently potential for joint distress during service.

Saw-cutting in concrete pavements is generally applied by two methods; conventional and early-entry cuts. In the conventional sawing operation, the joints are usually cut with a water-cooled blade. However, in the early-entry cut, as the device is much lighter than the conventional saw equipment, the saw-cutting process can be applied

at earlier age when the concrete is not too hard and thus the blade does not require water for cooling. Early-cut should begin as soon as the concrete has hardened sufficiently to avoid raveling, which is considered the beginning of saw-cutting window. It is suggested that early-entry saws without significant raveling can be applied at about the halfway point between the initial and final setting times of concrete (Their 2005). Also, it was reported that raveling was visually acceptable when the concrete compressive strength varied from 2.1 to 7.0 MPa based on type of aggregate in the mixture (Okamoto et al. 1994). In practice, the time of saw-cutting is commonly determined by a professional saw operator's judgment with the aid of a simple and subjective test which involves scratching the concrete surface to see if it can maintain the surface texture. Conventional sawing windows generally begin at about the final setting time (Krstulovich et al. 2011) and may extend up to 24 h after casting (Zollinger 2001). Therefore, in practice, joint walls resulting from early-entry cuts may be exposed to uncontrolled evaporation significantly earlier than the ones from conventional sawing method. Most of the reported studies (Vepakomma et al. 2002; Krstulovich et al. 2011) focused on whether the saw-cutting methods (conventional or early-cut) were successful to avoid raveling at the joints and control random cracking in pavement. However, limited field studies have been conducted to explore the effect of saw-cutting on concrete pavements at the joint locations with respect to curing efficiency and microstructural evolution, which warrants further investigation. Hence, the aim of this study was to assess whether the effect of overfilling joints with curing compound immediately after saw-cutting (early- and late-cuts) can significantly improve the quality of concrete microstructure at joint regions in laboratory slabs and trial field sections constructed in Winnipeg, Canada. This is a critical question (remedial curing) to a number of transportation agencies in North America (e.g. Public Works, Manitoba) linked to

upgrading current construction specifications and associated with increase of initial construction costs.

5.2 Methodology

5.2.1 Materials and mixtures

The City of Winnipeg (COW) specifies ready mix for concrete pavement with target performance of 32 MPa and a class of exposure C-2 (concrete subjected to chlorides and freezing-thawing) according to Canadian Standards Association (CSA) A23.1 (2014). This mixture was provided by an approved ready mix supplier for the field sections. The binder comprised General Use (GU) portland cement and Class F fly ash, as a supplementary cementitious material (SCM), meeting the current requirements of CSA A3001 (2013). The COW construction specifications for concrete pavements (CW3310–R17 2015) permit replacement of GU cement with up to 15% Class F fly ash, and thus the amounts of GU cement and fly ash in the binder were 272 kg/m³ and 48 kg/m³, respectively. The water-to-cementitious materials ratio (w/cm) was 0.40 and an air-entraining admixture complying with ASTM C260 (2010) was used in concrete to achieve a fresh air content of 5 to 8%. Also, a high-range water reducing agent (HRWRA) complying with ASTM C494/C494M (2013) was used to obtain a target slump of 30 to 70 mm for slip forming. Locally available natural gravel was used as coarse aggregate with specific gravity and absorption of 2.65 and 2%, respectively. The fine aggregate was well-graded river sand with a specific gravity, absorption, and fineness modulus of 2.53, 1.5% and 2.9, respectively. The coarse and fine aggregates contents were 1213 and 679 kg/m³, respectively. The curing compound applied was white in color and made of high-grade hydrocarbon resins in a water-based emulsion conforming to ASTM C309 (2011) Types 1, 2 and 1-D, Class B.

5.2.2 Establishing the maturity-strength relationship

To establish the maturity-strength relationship for concrete before casting the field pavement sections, a laboratory investigation was performed on the concrete mixture used according to the procedure described in ASTM C1074 (2011). The datum temperature was determined using mortar specimens cured in water baths maintained at three different temperatures (7, 21 and 35 °C, representing the expected minimum, average and maximum temperatures during casting). The Nurse-Saul function was used to compute the temperature-time factor (maturity index) as:

$$M(t) = \sum (T_a - T_o) \cdot \Delta t \quad \text{Eq. 5.1}$$

where, $M(t)$ is the temperature-time factor at age t (°C-h), Δt is the time interval of curing (h), T_a is the average concrete temperature during this time interval (°C), and T_o is the datum temperature [estimated minimum temperature for hydration of cement] (°C).

In addition, eighteen cylindrical (100×200 mm) specimens were prepared from the same concrete mixture design, according to ASTM C192/C192M (2014). Two temperature sensors were embedded in the centers of two specimens and connected to data-loggers. The specimens were cured in a standard moist curing room (maintained at 20±2°C, RH ≥ 95%) until testing. The time interval between temperature measurements was 4 min starting from casting up to 14 days. Since the maturity test results were needed to project strength development at low values of maturity index, the compressive strength test (ASTM C39/C39M 2014) was performed as soon as practicable at the ages of 17 and 22 h followed by 3, 7, 10 and 28 days (ASTM 1074 2011). At each testing age, the corresponding maturity index for both instrumented specimens was determined. The results of

compressive strength were then plotted versus the maturity index and a best-fit curve through the data was drawn to establish the strength-maturity relationship to estimate the strength of concrete under field conditions.

5.2.3 Field pavement sections and laboratory slabs

Two pavement sections each with the length of 50 m were cast in an urban arterial road at the central area of Winnipeg, Manitoba, Canada. The pavement sections consisted of a 150 mm thick jointed reinforced concrete pavement with a slab width of 3.70 m on a granular base. The pavement in the test location is expected to be subjected to large temperature changes, as the maximum and the minimum air temperatures during the last 10 years were +36°C and -36°C, respectively. To determine the internal RH and maturity of concrete, eight RH/Temperature wireless sensors were installed in an arrangement parallel to the transverse joints prior to casting as shown in Figure 5.1. Each group of two sensors was used to record the RH and temperature of one transverse joint approximately at the middle length of each test section.

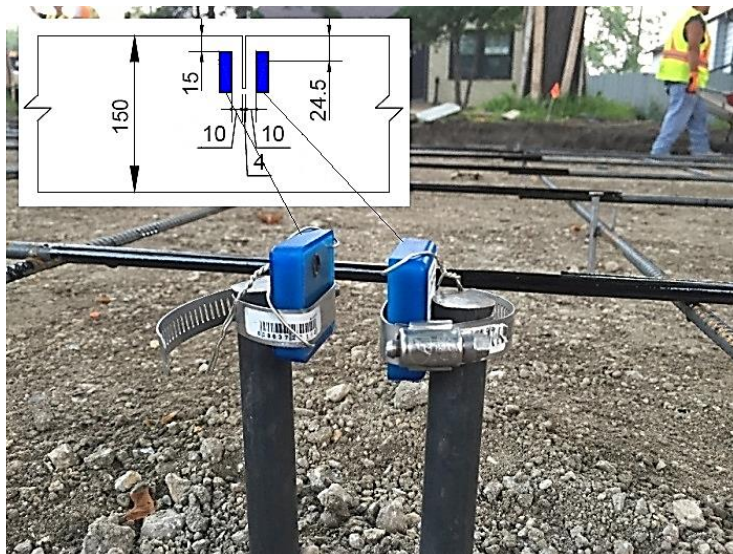


Figure 5.1- RH/Temperature sensors configuration. (Note: all dimensions are in mm)

The placement, consolidation, finishing and curing procedures of the pavement sections complied with the COW construction specifications (CW3310–R17 2015). The two pavement sections received two different types of curing: one coat of curing compound as per the COW specifications, and one coat of curing compound plus filling the joint immediately after saw-cutting with the curing compound, referred to hereafter as 1CC and 1CC+J, respectively. After excess moisture due to bleeding had evaporated (30 to 45 min after the finishing operations) the curing compound was applied by labor, using portable sprayers on the designated 1CC and 1CC+J sections. The coat of curing compound was applied at the rate of 0.23 l/m^2 according to the manufacturer's specifications. In addition, visual inspection was mandated by the COW specifications to cover any left out spots “holidays”, where the surface of the pavement sections was uniformly white in color. The pavement slabs were saw-cut in two different scenarios to establish the contraction joints with the dimensions of 3 to 4 mm wide and 50 ± 3 mm deep. According to the COW specifications, the time of saw-cutting is typically left to the discretion of the contractor in such a way that excessive raveling and/or random cracking are prevented. The time of early-entry dry cut (early-cut) was chosen around the final setting time of this mixture (7h, 25 min) which corresponded to 4.6 MPa according to the in-situ concrete strength derived from the strength-maturity relationship. This value complies with the compressive strength range reported by Okamoto et al. (1994). The conventional wet-cut (late-cut) was chosen to be roughly triple the time (20 h, 30 min) of the early-cut, which corresponded to a strength of 12.3 MPa, when wet cutting activities were still feasible without much risk of random cracking (Zollinger 2001). At 28 days, twenty four cores 75 mm in diameter were extracted from the pavement slabs to perform the tests. Figure 5.2 illustrates the locations of the cores extracted from the field sections. For the absorption/microstructure tests, four

cores were obtained directly at each transverse joint (split cores) and another four cores were obtained from the center of slab (mid-slab).

Laboratory slabs were prepared from the same ready mix concrete delivered by the same supplier. Thus, the effect of standard (laboratory) casting relative to actual field practice on the transport properties of concrete was taken into account. This is to verify that any variation in the trends and microstructure of concrete would be originating from the curing method rather than the field practice or ambient conditions (e.g. effect of evaporation rate in the field). Two 780 mm×520 mm×150 mm concrete slabs were cast in wooden formwork.

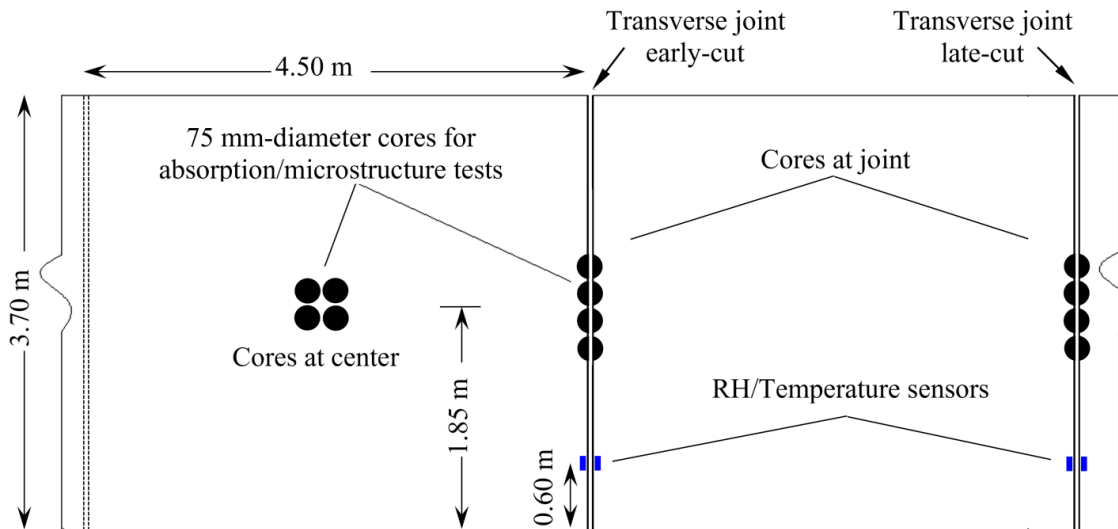


Figure 5.2- A schematic diagram showing the location of the cores extracted from each pavement section (Note: the clear distance between cores was 0.05 m).

Four RH/Temperature wireless sensors were installed in an arrangement parallel to the joints prior to casting in a similar manner to the field sections. The curing regimes on the surface of slabs were applied similar to that of the field pavement sections (i.e. 1CC and 1CC+J). The formwork was removed after 7 h and the curing compound was applied to the

side surfaces of the slabs. Early- and late-cuts were applied on each slab at 8h, 10 min and 22 h, 20 min after casting, respectively. These saw-cutting times corresponded to the same concrete maturities of the field sections. The slabs were kept under laboratory conditions ($23\pm 2^{\circ}\text{C}$ and $55\pm 5\%$ RH) for 28 days; the location of cores and testing scheme were similar to that of the field sections [Figure 5.3(a)].

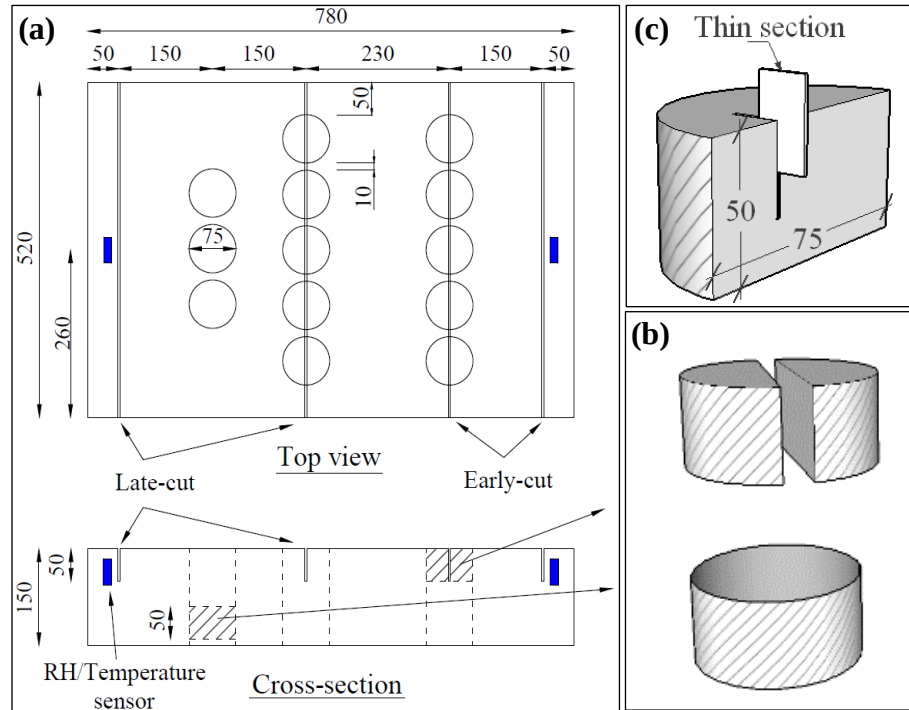


Figure 5.3- A schematic diagram of the laboratory slab showing; (a) the location and number of the cores extracted, (b) top joint and bottom mid-slab specimens, and (c) thin section. (Note: all dimensions are in mm)

5.2.4 Tests

5.2.4.1 Absorption test

A customized absorption test was performed on the cores extracted from both field sections and laboratory slabs, as it was found to characterize concrete with variable microstructural quality with high repeatability (Tiznobaik and Bassuoni 2017c). In this method, full-depth

75 mm cores are extracted from concrete pavement slabs and cut into 50 ± 2 mm slices parallel to the surface of concrete. The curing compound residue was gently cleaned with a brass brush, and 50 ± 2 mm thick slices were cut from the top part of cores at joints [Figure 5.3(b)]. Also, 10 mm of the bottom part of the mid-slab cores were removed followed by cutting 50 ± 2 mm discs representing the optimal location in terms of moisture retention [Figure 5.3(b)]. The concrete discs were placed in an environmental chamber at $50 \pm 2^\circ\text{C}$ and 40% RH until reaching a constant mass (difference in mass in two successive days is less than 0.1%). All surfaces except the joint walls and top surfaces were sealed by a rapid setting epoxy. The specimens were then placed in a sealed desiccator under vacuum pressure (~ 85 KPa) for 6 hours.

Afterwards, the specimens were removed from the desiccator and the initial dry mass to the nearest 0.01 g was recorded. Each specimen was freely immersed in 4% calcium chloride (CaCl_2) solution for up to 360 min, and the amount of absorption after 1, 5, 10, 20, 40, 80, 160 and 360 minutes were recorded to the nearest 0.01 g. These results were presented as the absorption (I) of concrete specimens by:

$$I_a = \frac{m_t}{a \cdot \rho} \quad \text{Eq. 5.2}$$

where, I_a is absorption depth of concrete at a certain point of time (mm) m_t is difference between wet mass and dry mass at a specific point of time (g), a is cross sectional area of the exposed surfaces (mm^2), and ρ is density of the fluid [i.e. 4% CaCl_2 solution at 23°C] (g/mm^3).

5.2.4.2 Mercury intrusion porosimetry (MIP) and microscopy tests

The effect of curing compound application on the development of microstructure in concrete was assessed by mercury intrusion porosimetry (MIP) and backscattered scanning

electron microscopy (BSEM). To capture the characteristics of pore structure, the MIP technique was conducted on field and laboratory concrete samples at 28 days. To prepare samples for MIP, bigger chunks (8-12 mm) were extracted from the cores, visually inspected and an effort was made to find and remove large aggregate. The result of this process was 16-22 chunks with the size of 4-7mm, representing the concrete microstructure at specific locations. These chunks were extracted immediately at the joint walls of the concrete cores (split cores) and bottom 25 mm of the cores extracted at mid-slab. The samples were oven dried for 72 h at a temperature of 55 ± 2 °C; they were then kept in a desiccator containing calcium sulphate for 24 h. This method of drying was adopted to avoid decomposition of calcium silicate hydrate (C-S-H) and formation of micro-cracks, which may occur at high temperatures. The contact angle and the surface tension of mercury were taken as 130° and 485 dynes/cm, respectively (Shi and Winslow 1985, Said et al. 2012). To complement the trends observed in the various tests, BSEM was conducted on polished thin sections from the field and laboratory cores extracted at the joint locations at 28 days. Slices were cut from the top part (25 mm) of cores which were vertically perpendicular to the joint walls [Figure 5.3(c)]; they were then dried and impregnated with a low-viscosity epoxy resin under vacuum pressure and polished by successive diamond surface-grinding to a thickness of about 50 μm . The sections were coated with carbon to enhance the conductivity for the BSEM imaging.

5.2.5 Statistical analysis

The results of absorption test were statistically evaluated by the Analysis of Variance (ANOVA) method at a significance level (α) of 0.05. ANOVA is a statistical model used to analyze the differences between group means and their variation among and between groups. The variance of a specific parameter is separated into components attributed to

different sources of variation. ANOVA tests the hypothesis that the means of several groups are comparable via a t -test, and it can generalize the test to more than two groups. According to ANOVA, exceeding the F_{cr} for an F -distribution density function indicates that the variable tested has a statistically significant effect on the average results (Montgomery 2012). The ANOVA results were further statistically evaluated by the Least Significant Difference (LSD) post hoc test (Fisher's test), when applicable, to declare pairwise comparisons between the means of each two groups. LSD post hoc test declares two means significantly different if the absolute value of their sample differences exceeds the least significant difference, LSD (Montgomery 2012).

5.3 Results and Discussion

5.3.1 Environmental conditions

During casting the field sections, the average ambient air temperature and relative humidity were 21°C and 91%, respectively, with a wind velocity of about 17 km/h (Figure 5.4) [GCC, 2016]. The measured temperature of concrete pavement was about 19°C. Under these weather conditions, the moisture evaporation rate at the concrete surface was estimated at 0.25 kg/m²/h according to the chart of CSA A23.1/A23.2 (2014). This value was well below the critical evaporation rate (0.50-0.75 kg/m²/h) stipulated by ACI 308R-16 (2016) mainly to minimize the risk of surface drying and plastic shrinkage cracking. Also, a day with heavy rain (49 mm) was recorded seven days after casting (Figure 5.4). During the first 28 days after casting, RH values recorded by the sensors in the field concrete were above 95% in all sections, as shown in Figure 5.5. Therefore, it can be deduced that even the vertical joint walls originally expected to receive no treatment in terms of curing compound application (i.e. 1CC section) were beneficially affected by the

prevailing climatic conditions (low rates of evaporation, high relative humidity, and rainfall).

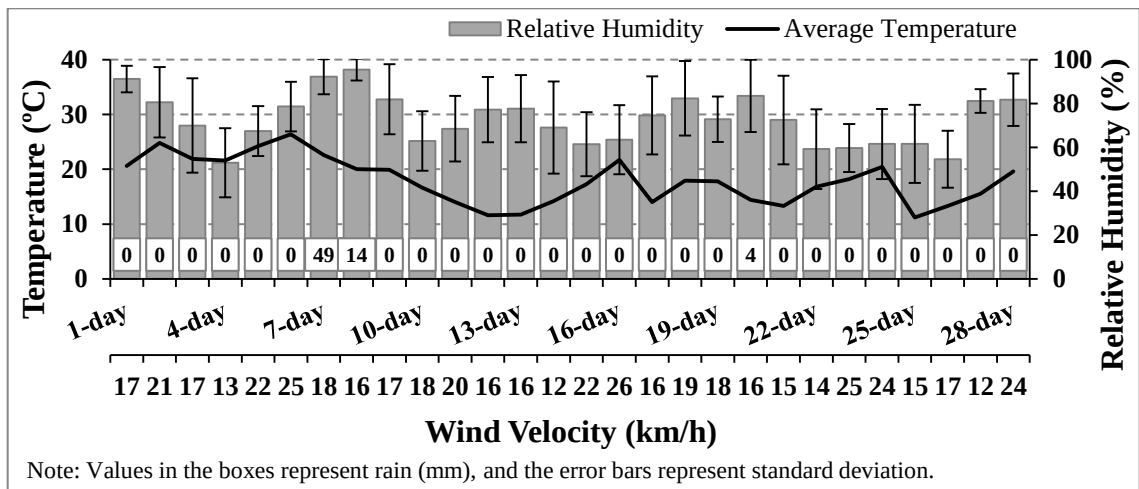


Figure 5.4- Average daily temperature, relative humidity, precipitation, and wind velocity data in Winnipeg for the period of 28 days from the day of casting.

In the 1CC laboratory slabs with early-cut [Figure 5.6(a)], the RH remained at 100% up to the second day after casting, but afterwards, there was a sharp drop to 66% up to the 17th day at an average rate of 1.5% per day. However, it was observed that the late-cut in the 1CC laboratory kept RH values above 95% up to the fourth day, followed by a gradual drop to 70% at the 18th day [Figure 5.6(b)]. The results indicate that irrespective of the saw-cutting time, overfilling the joints with a curing compound provided better moisture retention at these locations. For instance, by comparing Figures 5.6(a) and 5.6(c) for the early-cut, the effect of applying the curing compound applied at the joint extended moisture retention at 100% RH by five days (from the second to seventh day). Correspondingly, the drop in RH to 80% happened on the 18th day in 1CC+J compared to the 7th day in 1CC. In Figures 5.6(c) and 5.6(d) for the early-cut and late-cut slabs treated by the 1CC+J, the RH values remained 100% up to the 7th and 9th days, respectively,

which means that the efficiency of curing period at early-age has been extended by about 30%. Afterwards, the early-cut with 1CC+J showed a gradual drop to 80% up to the 18th day with an average rate of 1.8% per day, while the late-cut with 1CC+J showed a gradual decrease to 81% up to the 28th day with an average rate of 0.9% per day. Hydration of cement slows down if the RH inside concrete drops below 80% (Neville 2011); therefore, it can be deduced the former (early-cut with 1CC+J) had less efficiency of curing than the latter (late-cut with 1CC+J).

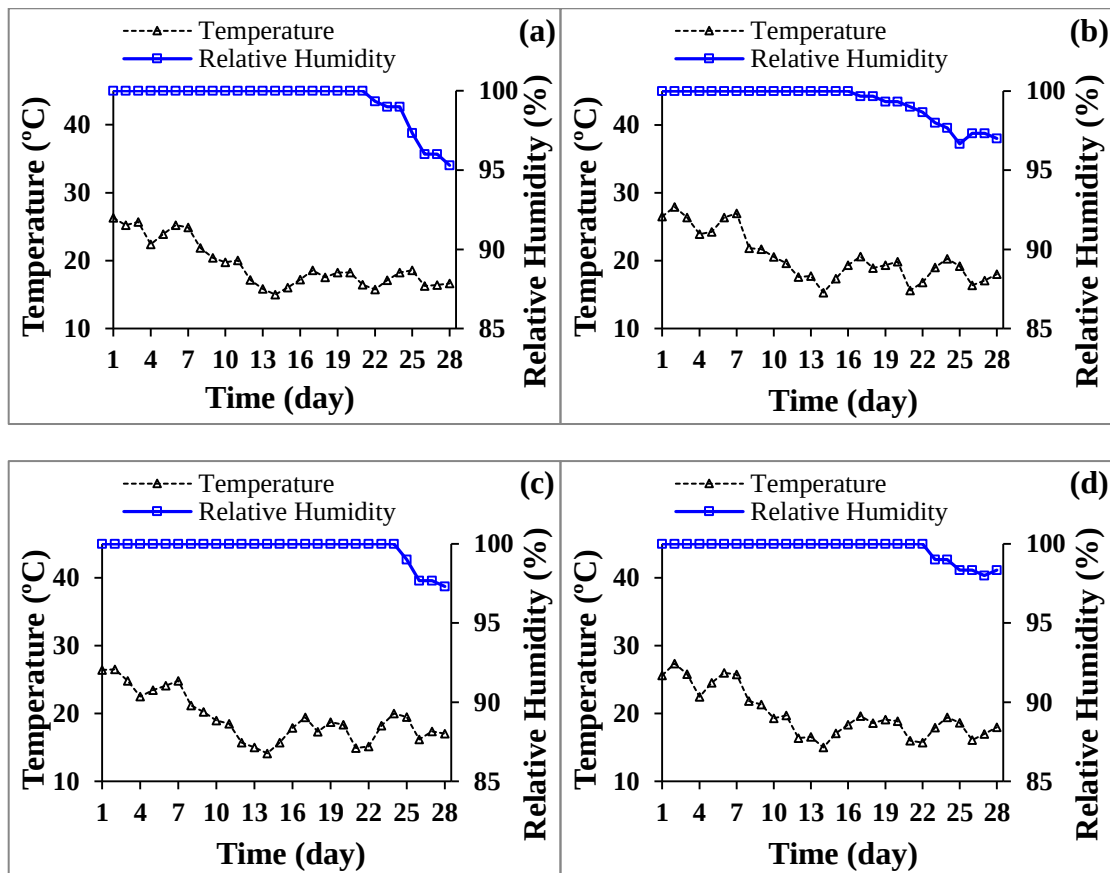


Figure 5.5- Average temperature and relative humidity of field concrete: (a) early-cut (1CC), (b) late-cut (1CC), (c) early-cut (1CC+J), and (d) late-cut (1CC+J).

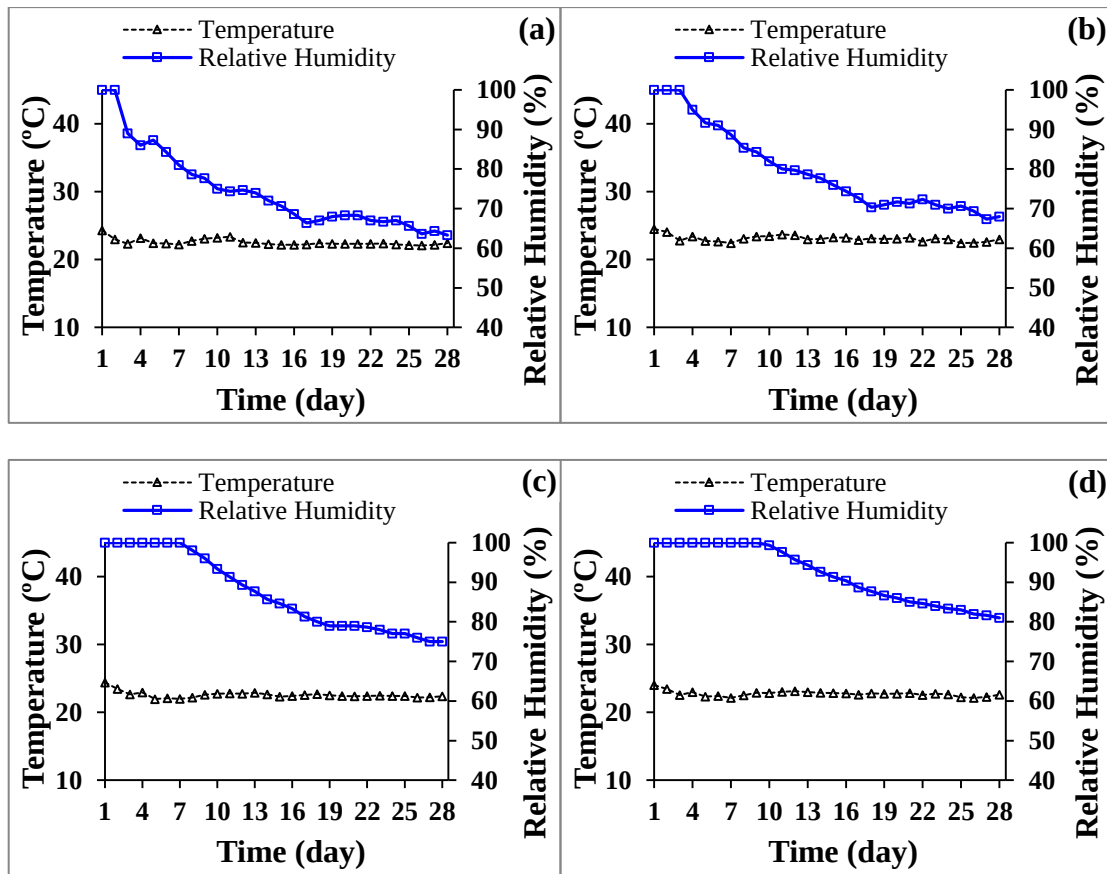


Figure 5.6- Average temperature and relative humidity of laboratory slabs: (a) early-cut (1CC), (b) late-cut (1CC), (c) early-cut (1CC+J), and (d) late-cut (1CC+J).

5.3.2 Maturity of concrete

The maturity of concrete depends on its time-temperature history provided that moisture is available to sustain hydration reactions. Laboratory results for this mixture design used in this study indicated that the datum temperature was 2.6°C (ASTM C1074 2011). The compressive strength results of concrete cylinders were plotted as a function of the maturity index, and a best-fit line through the data was drawn to establish the strength-maturity relationship as depicted in Figure 5.7 to predict the strength evolution of field concrete and laboratory slabs. The average temperatures, recorded at a depth of about 25 mm below the surface of concrete (Figure 5.1), of the pavement sections were recorded as 25.7 and 25.6°C

for the first 7 h, 25 min (early saw-cut) and 20 h, 30 min (late saw-cut) after casting. Thus, the corresponding compressive strength from the maturity function was calculated as 4.6 and 12.3 MPa for early- and late-cuts, respectively, for both field sections and laboratory slabs. The corresponding compressive strength at early-cut (4.6 MPa) was in a good agreement with the results reported by Okamoto et al. (1994) where raveling was visually insignificant when the compressive strengths were varied from 2.1 to 7.0 MPa based on type of aggregate in the mixture. In addition, at 28 days the maturity index of the pavements sections ranged between 10752 and 11424 °C-h, which corresponded to a compressive strength about 36 MPa, meeting the COW target strength for concrete pavements. Also, maturity index for the laboratory slabs were about 15120 and 15523 °C-h at 28 days, corresponding to a compressive strength of about 38 MPa.

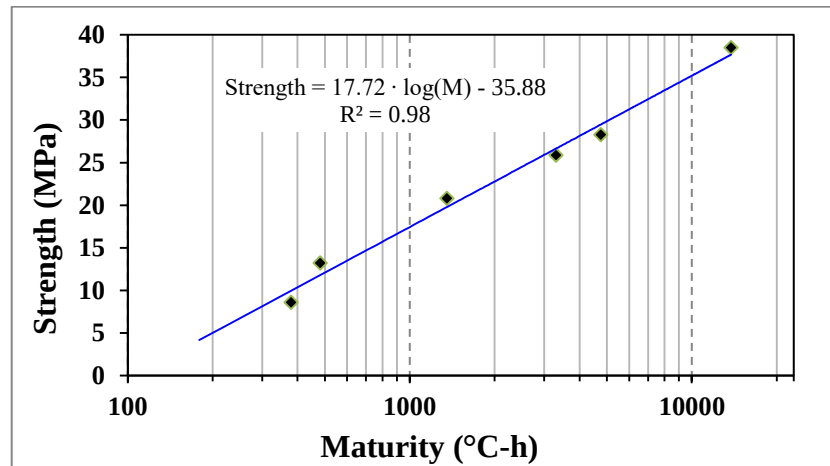


Figure 5.7- Relationship between strength and maturity of the concrete mixture used in the field sections and laboratory slabs.

5.3.3 Absorption

Absorption indicates the ease of saturation of concrete pavement exposed to aggressive environments (e.g. deicing salts and freezing-thawing cycles); therefore, it is a useful

metric to reflect the durability of concrete pavement. In this study, absorption of concrete up to 360 min was determined according to the procedures described in the Tests section. Concrete absorption (Figure 5.8) was expressed in terms of depth of penetration considering the density of fluid (4% CaCl_2) and exposed surface area (Eq. 5.2).

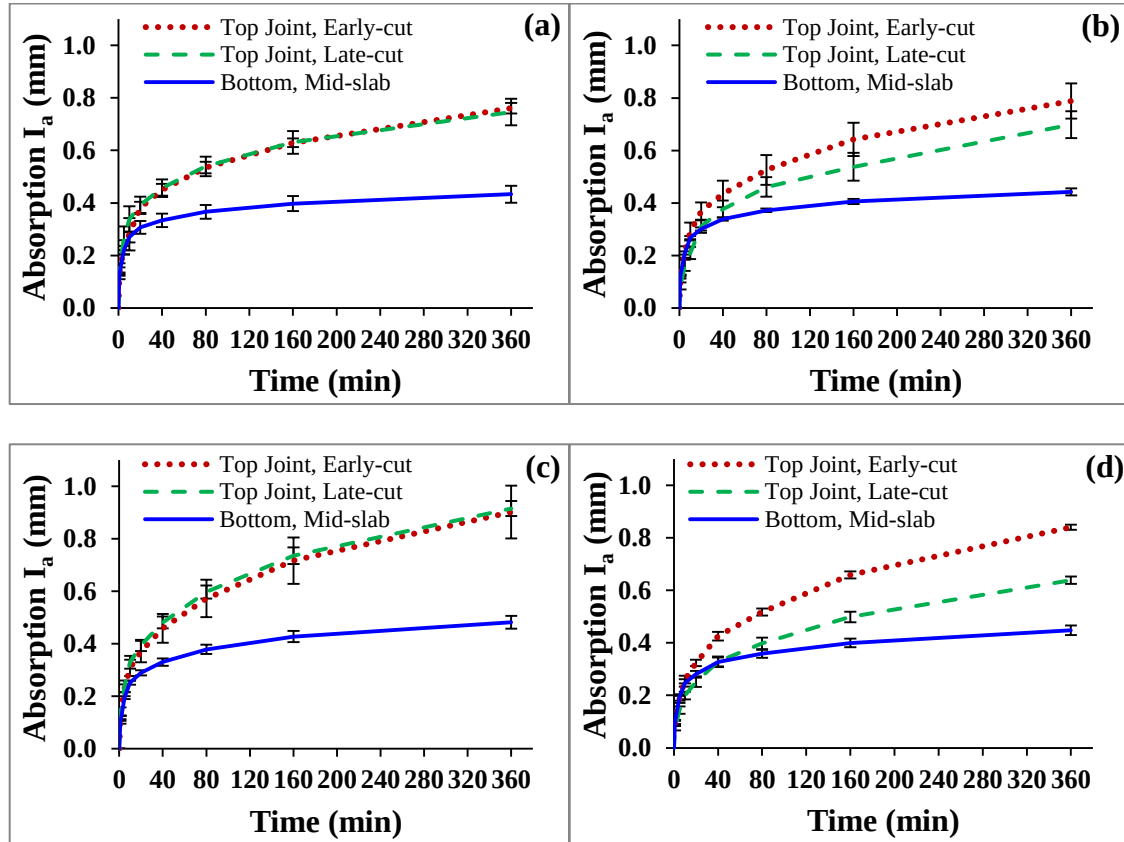


Figure 5.8- Absorption of cores: (a) field 1CC, (b) field 1CC+J, (c) laboratory 1CC, and (d) laboratory 1CC+J.

Each series of data shown refers to the three samples tested from each location. These results represent absorption of the top part of the cores at joint locations and the bottom part of the cores at mid-slab (optimal location in terms of moisture retention and hydration development as discussed earlier). Higher cumulative absorption in the top part of joints (early- and late-cuts) relative to the bottom part of mid-slab was pronounced from 80 min

up to 360 min for both field sections and laboratory slabs (Figure 5.8). Since the top joint specimens simulate the critical areas at joint locations in concrete pavements, it can be substantiated that the joint geometry (vertical joint walls and top surfaces) facilitates a higher degree of infiltration, and consequently saturation which may be linked to the vulnerability of joints to deterioration in concrete pavements.

Table 5.1 shows ANOVA results for absorption comparison which generally determines the statistical significance among four groups of top joints specimens (early-cut and late-cuts in the 1CC and 1CC+J sections) for both field sections and laboratory slabs.

Table 5.1: ANOVA results for absorption of field and laboratory cores

Comparison Groups	Parameter Tested	Source	<i>F</i>								<i>F_{cr}</i>
			1(min)	5	10	20	40	80	160	360	
E, L, E+J and L+J	Absorption	Field	8.01*	6.01*	4.22*	4.66*	2.98	2.48	3.15	1.68	4.07
		Lab.	13.93*	20.80*	19.93*	17.49*	13.03*	14.95*	14.90*	17.37*	

*Indicates statistical significance. E: Early-cut, L: Late-cut, J: Joint filled with curing compound.

For the field sections, up to 20 min, all the *F* values (e.g. 4.66 at 20 min) were greater than the *F_{cr}* of 4.07, indicating significant differences among the data groups. However, for the laboratory slabs, these significant differences (e.g. *F* values of 17.37 at 360 min) were maintained up to 360 min. Since the top surface of concrete received one coat of curing compound in both sections (1CC and 1CC+J), it may be deduced the main reason for the main differences in the absorption values originated from the absorption through joint walls. However, it should be considered that uncured joint walls in the field sections (early- and late-cuts in 1CC) were beneficially affected by the prevailing climatic conditions conforming to the recorded high RH values (above 98%) at a distance of 10 mm parallel to the joint walls (Figure 5.5) which might lead to limited drying in the curing period, and

consequently better microstructure and reduced long-term absorption. Conversely, uncured joint walls in the laboratory slabs (early- and late-cuts in 1CC) were exposed to controlled conditions ($23\pm 2^{\circ}\text{C}$ and $55\pm 5\%$ RH) which made vertical joints undergo drying in comparison to the 1CC joints in the field sections.

The ANOVA results were further statistically evaluated by Fisher's test to declare pairwise comparisons between the means of each two groups. The absorption and Fisher's test results are given in Table 5.2 for field sections and laboratory slabs, respectively.

Table 5.2: Fisher's test for absorption depth of field and laboratory cores

Source	Comparison Aspect	Time (min)	Absorption (mm)	Difference (mm)			LSD** (mm)
				L	E+J	L+J	
Field	E	20	0.381	0.011	0.013	0.070*	0.053
	L		0.392	-	0.024	0.081*	
	E+J		0.368	-	-	0.057*	
	L+J		0.311	-	-	-	
Laboratory	E	360	0.901	0.013	0.061	0.263*	0.098
	L		0.915	-	0.075	0.276*	
	E+J		0.840	-	-	0.202*	
	L+J		0.638	-	-	-	

*Indicates statistical significance. E: Early-cut, L: Late-cut, J: Joint filled with curing compound.

**LSD is calculated as; $LSD = t_{(\alpha/2, n)}(2 \cdot MSE/n)^{0.5}$, t is the critical value from t -distribution, MSE is the mean of all groups' variances, n is number of data in each group.

The results indicate no significant differences between the absorption values of early- and late-cuts with 1CC starting from 1 min up to 360 min for both field sections and laboratory slabs. Although the early-cuts were applied earlier than the late-cuts, both types of joints were subjected to surrounding environment for more than 27 days without curing, resulting in comparable absorption values. Also, it can be noted that there are no significant differences between the absorption values of early- or late-cuts with 1CC and early-cut with 1CC+J starting from 1 min up to 360 min for both field sections and laboratory slabs. This suggests that the curing compound applied at the early-cut joints did not significantly

improve the microstructure of concrete. It was observed that the curing compound residual on the joint walls created by late-cut was much thicker than early-cut. This is attributable to the presence of dust powder from early-cut (dry process) which prevented the curing compound to adhere efficiently to the joint walls. Perhaps, removing this dust, for example by an air compressor could have improved the performance of early-cut with 1CC+J; however, this procedure was not explored in the current study. The field results indicated that filling the joint with curing compound immediately after late saw-cutting (late-cut with 1CC+J) significantly reduced (e.g. 20% at 20 min) the absorption at joint locations with respect to the late-cut joints without curing compound (late-cut with 1CC). These results were supported by Fisher's test at 20 min as filling the joint with curing compound caused an absorption reduction of 0.081 mm which was greater than *LSD* of 0.053 mm (Table 5.2). This is ascribed to applying curing compound in the joints immediately after wet (late) saw-cutting which prevented joint walls from uncontrolled evaporation resulting in better microstructural development in the 1CC+J section. It should be mentioned the absorption values for both late-cut sections (1CC and 1CC+J) at 20 min were approximately half the ones at 360 min, which signifies the 20 min absorption values as a threshold in differentiating among different qualities of concrete in this case.

Correspondingly, the results of laboratory slabs indicated that applying curing compound at the late-cut joint locations (late-cut with 1CC+J) significantly reduced (e.g. 33% at 360 min) the absorption with respect to late-cut joints with 1CC starting from 1 min up to 360 min. These were supported by Fisher's test at 360 min as filling the joint with curing compound caused an absorption reduction of 0.276 mm which was greater than *LSD* of 0.098 mm (Table 5.2). Whereas the absorption results of field sections show significant reduction between these scenarios only up to 20 min, the absorption results at 360 min of

late-cut with 1CC+J for both field and laboratory concretes were in a close range. As mentioned previously, the field joints with 1CC were left uncured, but they were positively affected by the prevailing climatic conditions (high RH and rainfall), which improved the quality of concrete resulting in reduction of absorption. However, in the laboratory slabs, uncured (1CC) joints were affected by drier laboratory conditions ($23\pm 2^{\circ}\text{C}$ and $55\pm 5\%$ RH) that caused significant differences between 1CC and 1CC+J up to 360 min, especially in the late-cut case. An image analysis was conducted over specimens' cross sections (split cores at joints), and it was observed that an average of 53% of the joint wall area was composed of hardened cement paste (e.g. Figure 5.9).

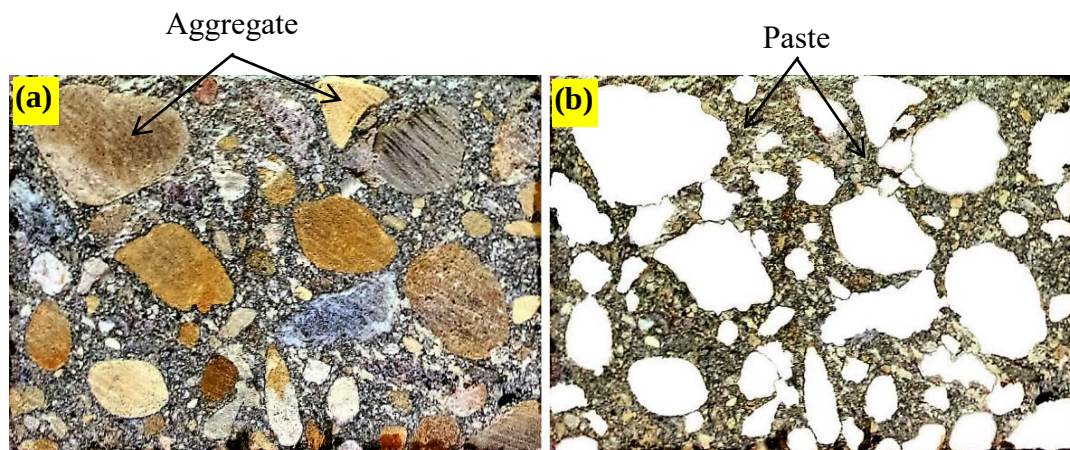


Figure 5.9- A sample of aggregate area isolation on a joint wall used in image analysis:

(a) before, and (b) after aggregate isolation.

Since the penetrability of concrete is generally controlled by the bulk properties of hardened cement paste (Neville 2011), applying curing compound on the exposed paste area (more than 50% of the cross section) would essentially improve the microstructure of concrete. Hence, the overall results of both field and laboratory tests emphasize the

significant effect of applying curing compound after saw-cutting on reducing absorption of fluids at and in the vicinity of joint walls.

5.3.4 Microstructure

The results from mercury intrusion porosimetry (MIP) for the field and laboratory cores obtained from the joints are presented in Figure 5.10 and Table 5.3. The MIP tests were done on 16-22 small chunks extracted at the joint walls from two replicate cores for each section, which were put in the same test compartment (porosimeter). Thus, the results shown in Table 5.3 can be reasonably considered as the averages of representative populations. The trends of MIP at 28 days generally conformed to the absorption results where for both field sections and laboratory slabs, joint walls had a relatively coarser pore structure than that of specimens extracted from the bottom of mid-slabs [Figures 5.10(a), 5.10(b) and Table 5.3]. Complying with the absorption results, the MIP results for field cores at joints were influenced by the beneficial effect of natural curing (high RH), since the pore size distribution curves were comparable [Figure 5.10(a)].

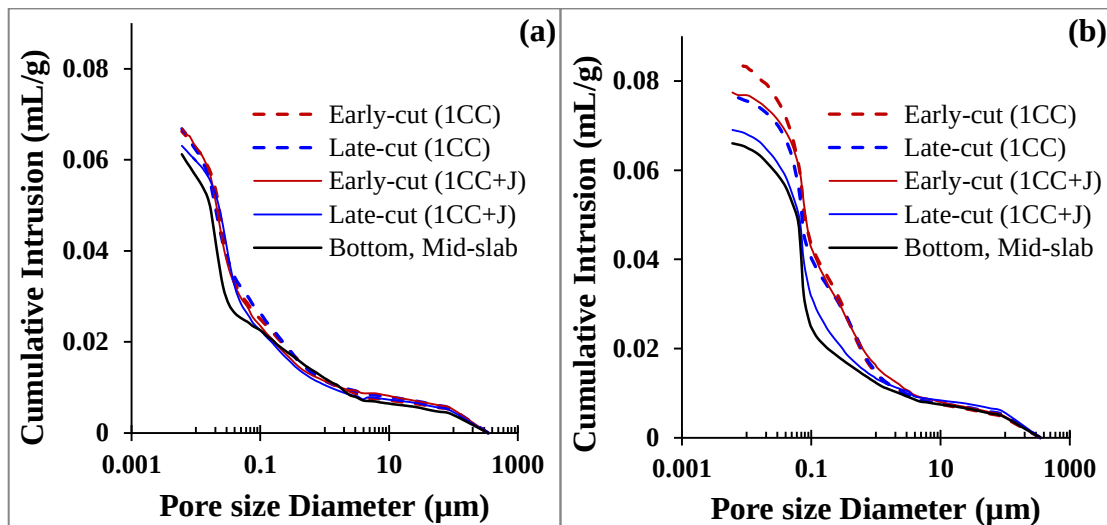


Figure 5.10- Pore size distribution of concrete at 28 days: (a) field, and (b) laboratory cores.

Table 5.3: Mercury intrusion porosimetry (MIP) test results for field and laboratory cores at 28 days

Source	Scheme	Total Intrusion $\times 10^{-2}$ (ml/g)	Threshold Pore Diameter (μm)	Apparent Total Porosity (%)
Field	1CC-E	6.6	1.45	14.2
	1CC-L	6.7	0.68	14.1
	1CC+J-E	6.7	0.98	14.1
	1CC+J-L	6.3	0.10	13.5
	Bottom-Mid slab	6.1	0.01	13.0
Laboratory	1CC-E	8.4	1.62	17.7
	1CC-L	7.7	1.23	16.2
	1CC+J-E	7.7	1.33	17.0
	1CC+J-L	6.9	0.18	13.8
	Bottom-Mid slab	6.6	0.10	13.5

E: Early-cut, L: Late-cut, J: Joint filled with curing compound.

However, the trends were more distinctive in the case of laboratory slabs due eliminating this effect of natural curing. For example, the cumulative intrusion for laboratory cores showed higher values in early-cut (8.4×10^{-2} ml/g) than late-cut (7.7×10^{-2} ml/g) with 1CC, which reflects a more porous microstructure at the joint walls. The laboratory results also indicated the early-cut with 1CC+J had similar trends to the late-cut with 1CC, as the cumulative intrusion for both were 7.7×10^{-2} ml/g and the threshold pore diameters were 1.23 μm and 1.33 μm , respectively. Hence, it may be concluded that applying curing compound after early-cut will provide a similar microstructural development at the vicinity of joint walls as late-cut without filling the joint with a curing compound (i.e. 1CC). In addition, the MIP results showed that applying the curing compound at the late-cut joint location (i.e. late-cut with 1CC+J) for both field sections and laboratory slabs decreased the cumulative intrusion and significantly reduced the threshold pore diameter with respect to late-cut with 1CC. For instance, the threshold pore diameter reduced by 90%, and 86% for field sections and laboratory slabs, respectively. This is in a good agreement with the absorption results of field sections and laboratory slabs.

It was observed by microscopy that saw-cutting caused occasional micro-cracking at the joint walls in aggregates with a depth up to 200 μm , irrespective of saw-cutting method (e.g. Figure 5.11). This might be attributed to the higher stiffness of aggregate which leads to more damages compared to surrounding paste at early-age. Presence of these micro-cracks at joint walls may contribute to the occurrence of localized defects affecting long-term durability of joints. While this micro-cracking of aggregates is inevitable, it signifies the need for at least improving the microstructural evolution of surrounding paste, which represents more than 50% of the area at joint walls.

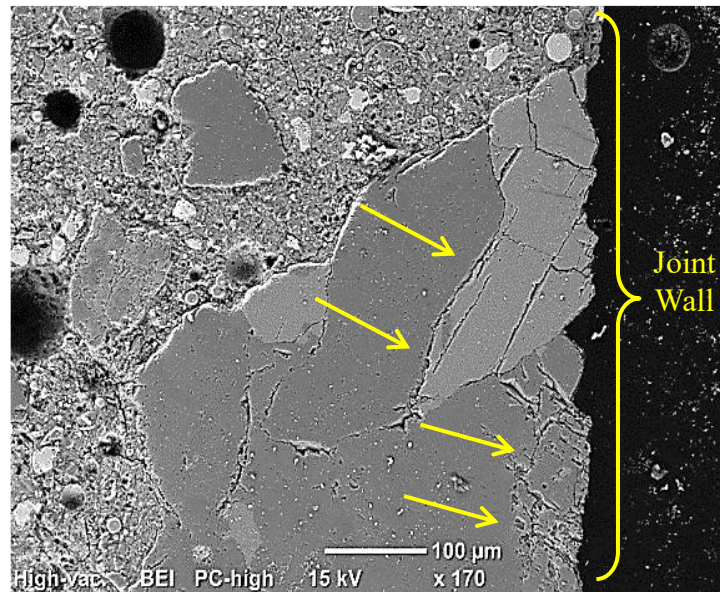


Figure 5.11- BSEM micrographs for thin sections at joint walls of the field cores, early-cut with 1CC.

In general, the BSEM observations in the 1CC field cores revealed systematic gaps between 2 to 5 μm in the vicinity of joint walls at the interfacial transition zones (ITZ) with aggregates, Figures 5.12(a) and 5.12(b). It was observed that these gaps were occupied by more hydration products and became narrower further away from joint walls. The presence,

size, and intensity of these gaps may contribute to the effect of uncontrolled evaporation and consequently higher infiltration through the joint walls. In contrast, in 1CC+J thin sections even in the vicinity of the joint walls, these gaps were significantly densified and filled with hydration products, Figures 5.12(c) and 5.12(d), which can be attributed to the effect of applying the curing compound in preventing uncontrolled evaporation. Thus, the most improved microstructure of concrete at joint walls was obtained by late saw-cutting with 1CC+J.

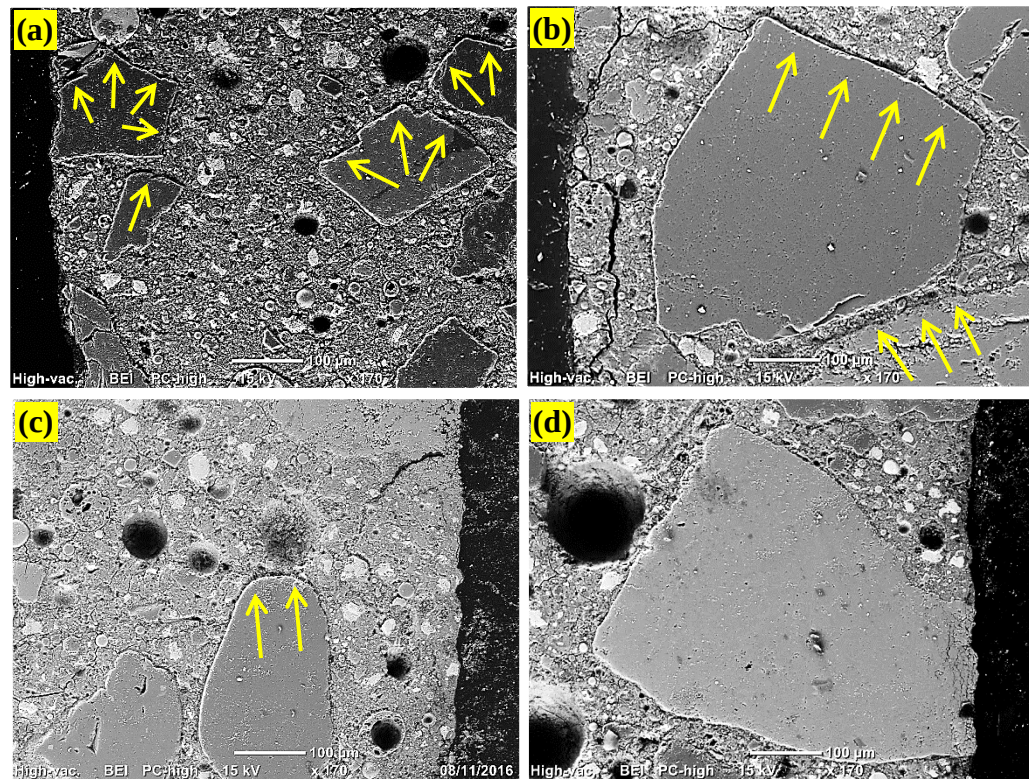


Figure 5.12- BSEM micrographs for thin sections at joint walls of the field cores: (a) early-cut with 1CC, (b) late-cut with 1CC, (c) early-cut with 1CC+J, and (d) late-cut with 1CC+J.

CHAPTER 6: DIELECTRIC RESPONSE OF CONCRETE: HARDENING AND HYDRATION DEVELOPMENT

This chapter investigates the optimum frequency needed to determine the dielectric response of concrete by gauging its sensitivity to different curing conditions of concrete and correlates the dielectric response of real-time sensor to setting time and strength/hydration development of concrete mixtures with a range of reactivity.

6.1 Introduction

Isothermal conduction calorimetry has been customarily used in the laboratory to assess the hydration process by measuring the heat flux liberated from small cement paste samples. However, since it only captures the processes involving significant changes in heat, it may give limited information during the induction period, which involves dissolution of ions from cement grains, nucleation and growth of hydration products and initial setting. After a few days of hydration, limited heat emission at the steady-state stage (ASTM C1679-14 2014) does not provide information about the development of hydration products. Moreover, the small size samples of cement paste, in the absence of fine and large aggregates which contribute to heat exchange processes, does not fully represent typical scenarios of concrete (McCarter and Afshar 1988). In the field, concrete at the fresh state (especially elements with a high surface-to-volume ratio) may experience thermal gradients and heat exchange due to ambient conditions resulting in different levels of maturity at the surface relative to the inner core. Indeed, this is different from isothermal conditions of conduction calorimetry. Continuous techniques such as electrical methods may be used to study the hydration of cement. For example, several attempts have been reported in the literature to study dielectric properties of fresh or hardened cement

paste/concrete in the laboratory (McCarter and Afshar 1988; Camp and Bilotta 1989; Gu and Beaudoin 1996; Zhang et al. 1996; van Beek et al. 1997; Sun 2008; Lee and Zollinger 2012). For example, McCarter and Afshar (1988) showed that the temporal change in dielectric response of cement paste during the first day of hydration can be used to monitor the hydration progress. Gu and Beaudoin (1996) used dielectric measurements of one year old hardened cement paste to provide a rapid estimation of the water-to-cement ratio (w/c). Zhang et al. (1996) reported the increase of dielectric constant of cement paste with w/c, within the first 30 hours of hydration. Also, Lee and Zollinger (2012) used dielectric properties of fresh concrete to estimate the volume fraction of the different phases in hardened concrete.

Thermogravimetric analysis (TGA) is commonly used to evaluate the degree of hydration of cement paste by determining the chemically bound water which corresponds to the mass loss of a sample typically heated up between 105°C and 1050°C (Halamickova et al. 1995; Cook and Hover 1999; Feng et al. 2004). The mass loss occurs through dehydration of chemically bound water in calcium silicate hydrate (C-S-H), calcium hydroxide (CH) and other hydration products (e.g. ettringite), as well as decarbonation of calcite (CaCO₃). Different references have considered the endothermic range of up to 105°C for evaporable water (Wang et al. 2004; Cassagnabère et al. 2009). There are several aspects which affect the accuracy of the obtained degree of hydration from TGA. For example, it was reported that drying samples at 105°C to eliminate evaporable water may cause severe drying shrinkage of the hydrated cement paste and decomposition of some hydration products [e.g. ettringite] (Bai et al. 2002; Snoeck et al. 2014). In addition, although the average bound water values of different types of fully hydrated portland cement pastes are in the narrow range of 0.23 to 0.25 g/g of ignited sample (Pane and

Hansen 2005; Neville 2011), the hydration process in blended cement pastes comprising supplementary cementitious materials (SCMs) is more complex as hydration and pozzolanic reactions are involved in bounding water exchange. In general, thermogravimetric analysis can be used as a reasonable indication of hydration development of concrete prepared from portland and/or blended cements (Scrivener 2015).

From an electrical perspective, dielectric materials can sustain a charge from an applied electric field and then transmit that energy (Halliday et al. 2013). The dielectric constant (or relative permittivity) is defined (Halliday et al. 2013) as the ratio of a material's electrical permittivity to that of free space (vacuum). As the dielectric property of water is very high, the dielectric properties of building materials in a wet-state are mainly governed by the amount of water preserved (free, adsorbed or bound) in the system (van Beek et al. 1997). The dielectric constant of water at 0°C changes from about 77 in liquid state to 3.4 in a solid state (ice). This constant will be different in the states intermediate between water and ice, such as gels, hydrates, and interfacial water (Gu and Beaudoin 1996). Water molecule with its large dipole moment has the ability to orient in an applied electric field. During cement hydration, the water molecules change from free to bound water in several states of hydration or crystallization. As the water molecule passes from one bonding state to another, the intensity of this ability will change and affect the dielectric relaxation of cement paste (Zhang et al. 1996). Setting/hardening of cement paste is a consequence of chemical reactions and phase transition on both grain surface and in aqueous phase. Continual monitoring of these processes may give insight of hydration and microstructural development of concrete. Dielectric response of cement paste/concrete during hydration may reflect the changes of bonding state of water; yet, in the previous laboratory studies in this area (McCarter and Afshar 1988; Camp and Bilotta 1989) sealed paste specimens were

used to avoid loss of water during the curing period. However, in-situ concrete at fresh state is vulnerable to adiabatic conditions and perhaps uncontrolled evaporation. For example, field concrete may suffer from some evaporation through the exposed surface, even if the rate of evaporation is below a critical threshold [e.g. 0.50-0.75 kg/m²/h] (ACI 308R-16 2016). In addition, to effectively study the dielectric response of cement paste/concrete during the hydration process, understanding its correlation with key indicators such as setting time, strength and degree of hydration is necessary. However, this area remains largely unexplored.

6.1.1 Research Significance

Field concrete may experience evaporation of moisture, thermal gradients and heat exchange due to ambient conditions. Continuous monitoring of the dielectric response of concrete may be used as an invaluable field indicator for the evolution of the hydration process. However, this has not been substantiated, due to dearth of research information in this area. Hence, the motive of the present study, which aimed at investigating and correlating the response of a developed real-time dielectric sensor embedded in concrete with key parameters of the hydration process and strength development, with a focus on the example of concrete pavement joints.

6.2 Experimental Program

6.2.1 Materials and Mixtures

General Use (GU) portland cement and fly ash (Class F), as a supplementary cementitious material (SCM), meeting the requirements of CAN/CSA-A3001 (2013) standard were used in the concrete mixtures. Also, a commercial nanosilica sol (50% solid content of SiO₂ dispersed in an aqueous solution) was incorporated in two mixtures. Table 6.1 shows the chemical composition and physical properties of the GU cement, fly ash and nanosilica.

An air-entraining admixture complying with ASTM C260/C260M (2016) was used in concrete mixtures to achieve a fresh air content of 5 to 8%. Also, a high-range water reducing admixture (HRWRA) complying with ASTM C494/C494M (2013) Type F was used to obtain a target slump of 50 ± 20 mm. Both air content and slump represent typical values for concrete pavement mixtures in Manitoba, Canada (CW3310–R17 2015). Locally available natural gravel coarse aggregate (with a maximum size of 20 mm) with specific gravity and absorption of 2.65 and 2%, respectively was used. The fine aggregate was well graded river sand with a specific gravity, absorption, and fineness modulus of 2.53, 1.5% and 2.9, respectively. To simulate curing of concrete pavements in most North American jurisdictions, a white compound made of high-grade hydrocarbon resins in a water-based emulsion conforming to ASTM C309 (2011) Types 1, 2 and 1-D, Class B was used.

Table 6.1: Chemical composition and physical properties of GU cement, fly ash, and nanosilica

	GU	Fly Ash	Nanosilica
<u>Chemical Composition</u>			
SiO ₂ (%)	19.8	56.0	99.17
Al ₂ O ₃ (%)	5.0	23.1	0.38
Fe ₂ O ₃ (%)	2.4	3.6	0.02
CaO (%)	63.2	10.8	--
MgO (%)	3.3	1.1	0.21
SO ₃ (%)	3.0	0.2	-
Na ₂ O (%)	0.1	3.2	0.20
<u>Physical Properties</u>			
Specific Gravity	3.17	2.12	1.40
Fineness, m ² /kg	410*	290*	80000**
Mean particle size, μ m	13.15	16.56	35×10^{-3}
Viscosity, mPa.s	--	--	8.0
pH	--	--	9.5

*Blaine fineness.

**Determined by titration with sodium hydroxide as reported by the manufacturer.

In this study, five different concrete mixture designs were prepared (Table 6.2). In addition to the reference pavement mixture (GU), four mixtures were designed to comprise SCMs,

providing a range of setting behaviours and reactivity. The total binder content and water-to-binder ratio (w/b) were kept constant at 320 kg/m³ and 0.40, respectively, which represent typical concrete pavement mixture designs in Manitoba, Canada (CW3310–R17 2015). Constituent materials were mixed in a mixer with a speed of 60 rpm. To attain homogenous dispersion of components, a specific sequence of mixing was adopted based on experimental trials. First, approximately 15% of the mixing water was added to the aggregate while mixing for 30 seconds. The cement and fly ash were then added to the aggregate and mixed together for 60 seconds. The nanosilica and the admixtures (air-entraining admixture, and HRWRA) were added to the remaining water while stirring vigorously for 45 seconds to obtain a liquid phase containing well-dispersed nanoparticles and admixtures. Finally, the liquid phase was added to the mixture and mixing continued for 2 minutes. After mixing and casting the concrete, a vibrating table with frequency of 3600 vibrations per minute (60 Hz) was used for compaction of specimens until air bubbles appearing on the surface stopped.

Table 6.2: Proportions of concrete mixtures per cubic meter

Mixture ID	Cement (kg)	Fly Ash (kg)	Nanosilica (kg)	Water* (kg)	Coarse aggregate (kg)	Fine aggregate (kg)	HRWRA (l/m ³)	Air entrainment, (l/m ³)
GU	320	0	0	128	1213	679	0.8	0.18
GUF15	272	48	0	128	1213	660	0.7	0.22
GUF30	224	96	0	128	1213	640	0.6	0.24
GUS	301	0	38	109	1213	659	1.2	0.18
GUF15S	253	48	38	109	1213	640	1.0	0.22

*Adjusted amount of mixing water considering water content of nanosilica (aqueous solution with 50% solid content of SiO₂).

6.2.2 Procedures

The experimental program was conducted in three stages to correlate the dielectric response of concrete to key properties of hydration development as described below.

6.2.2.1 Stage I

Previous studies in the literature (Camp and Bilotta 1989; Gu and Beaudoin 1996) did not report on appropriate frequency ranges for dielectric response of concrete. Also, these investigations were done on sealed specimens; thus, the effect of different curing conditions on the dielectric response of concrete was uncertain. Thus, the first stage of this work was designed to address these aspects. To study the effect of evaporation and curing on the dielectric response of concrete, specimens were saw-cut using a diamond saw to simulate joints in concrete pavements, which are deemed critical locations. Creating and exposing joint walls in concrete pavements through saw-cutting make joints susceptible to uncontrolled evaporation. Later during service, these locations are most vulnerable to higher degree of infiltration through two-dimensional absorption (from vertical joint wall and top surface near the joint) at each side of the joint (Tiznobaik and Bassuoni 2017c). Six replicate specimens (100×100×350 mm) were prepared from mixture GUF15, as this mixture represents typical concrete pavement in Manitoba, Canada (CW3310–R17 2015). They were cast in plastic moulds with two Dielectric Moisture Sensors (DMS) [Figure 6.1]. Each sensor has two parallel copper electrodes attached in the same plastic tape. Each set of two prisms received one type of curing on the top surface: one coat of curing compound, one coat of curing compound plus filling the joint immediately after saw-cutting with the curing compound, and no curing compound, referred to hereafter as 1CC, and 1CC+J, and NC respectively. To simulate joint locations in concrete pavements, the prisms were demoulded and saw-cut (dry-cut), using a diamond saw, at a depth of 50 ± 3 mm and width of 3 to 4 mm after 18 hours of casting. Subsequently, each specimen was sealed with a plastic wrap around the side surface and at the bottom to simulate surrounding pavement media. The prisms were kept under laboratory conditions ($23\pm2^{\circ}\text{C}$ and $55\pm5\%$ RH) during testing.

The capacitance was measured by Agilent LCR U1733C meter using three different frequencies (1 kHz, 10 kHz and 100 kHz) starting one hour after casting for every six hours in the day one, and one reading per day afterwards up to 91 days.

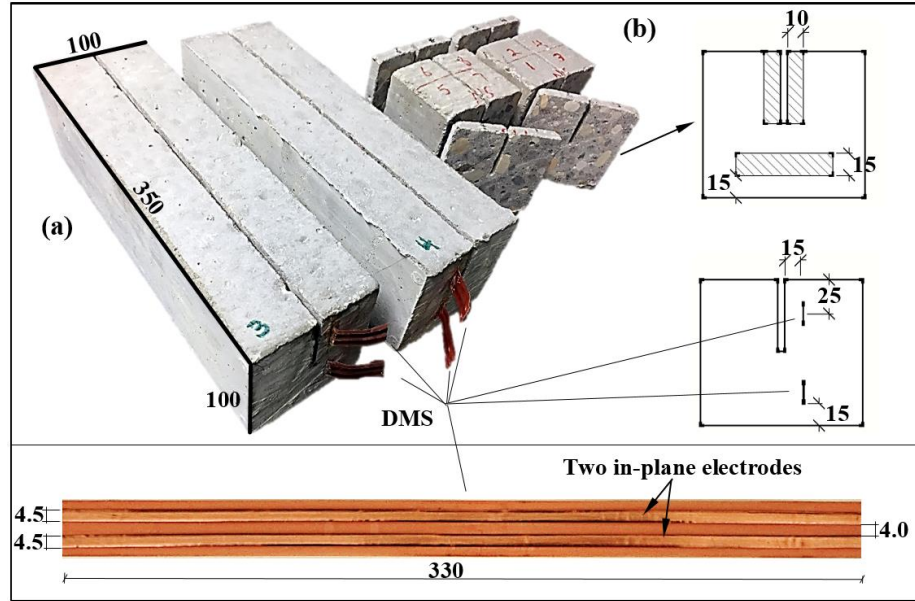


Figure 6.1- Configuration of specimens: (a) with DMS, and (b) without sensors, showing locations of DMS and TGA samples. (Note: all dimensions are in mm)

6.2.2.2 Stage II

The hydration trends of the concrete mixtures were characterized by isothermal conduction calorimetry. Paste samples with identical proportions to the paste fraction in the concrete mixtures (Table 6.2) were prepared to determine the heat released from the hydration reactions by an isothermal calorimeter kept at 23°C following the general guidelines of ASTM C1679 (2014). The rate of heat generated was monitored and recorded every minute continuously for 100 hours, and it was normalized by the mass of the sample. Also, the cumulative heat released was determined for each mixture. To study the correlation between the dielectric response of concrete during hydration and hardening limits, the mortar fraction of each mixture (portion passing sieve No. 4) was extracted from each

mixture and placed in a container at room temperature as specified by ASTM C403 (2016). At regular time intervals, the penetration resistance was determined by standard needles. Also, two replicate prisms (100×100×350 mm) from each mixture were cast in plastic moulds with two DMS. The capacitance response of concrete was measured every 15 min starting from casting using 1 kHz frequency (identified from *Stage I*) to establish the relationship with setting time.

6.2.2.3 Stage III

In this stage, correlations between real-time dielectric response of concrete and compressive strength as well as quantity of hydration products up to 91 days after casting were investigated. The compressive strength was measured at ages of 1, 3, 7, 28, 56 and 91 days according to ASTM C39/C39M (2014). For each mixture, triplicate concrete cylinders (100×200 mm) were cast in PVC moulds according to ASTM C192/C192M (2016). The top surfaces of specimens were left with no curing and specimens were kept in moulds under laboratory conditions (23±2°C and 55±5% RH) until testing. As curing in the field may range from absence of curing (in critical locations such as joints in pavements) to continuous moist-curing, selection of the curing condition was driven by adopting the worst-case scenario (i.e. no curing on the top face of specimens), which intensified the effect of moisture evaporation from the surface of concrete. In addition, four prisms (100×100×350 mm) were cast in PVC moulds, two with DMS, and two without sensors [Figures 6.1(a) and 6.1(b)].

Similar to cylinders, the top surface of the prisms were not cured, the sides and bottom were sealed with a plastic wrap to simulate uncontrolled evaporation at joints. Starting immediately after casting Agilent LCR U1733C meter was used to measure capacitance using 1 kHz frequency based on the findings of *Stage I*, up to 91 days. To

assess the evolution of hydration products in the cementitious matrix at different ages (1, 3, 7, 14, 21, 28, 56 and 91 days), the prisms without sensors were unwrapped and four 15±2 mm thick vertical slices were cut using a dry diamond saw [Figure 6.1(b)]. Subsequently bigger chunks (8-12 mm) were extracted from the slices, visually inspected and an effort was made to remove large aggregate. The result of this process produced the best four samples (each 10 g of 40-50 chunks with the size of 4-7 mm, representing the concrete microstructure at specific locations. These chunks were extracted immediately at the joint walls for joint samples and at the bottom 15 mm of the slices for bottom samples. Each sample was dried in the oven at 45±2°C and 40% RH for approximately three days until reaching a constant mass. Each two of four samples were crushed and ground manually together to a fine powder passing sieve #200 (75µm) providing two samples per location for thermogravimetric analysis (TGA). The TGA tests were conducted in nitrogen gas environment at a heating rate of 10°C/min up to 1050°C.

6.3 Experimental Results and Discussion

6.3.1 Sensitivity of dielectric response to curing conditions

Since the exact frequency of the LCR meter to sensitively determine the capacitance of concrete has not been established in the literature, three different frequencies (1, 10 and 100 kHz) were used to record the capacitance readings from one hour after casting up to 91 days. Figure 6.2 shows the dielectric response, in terms of average capacitance (readings from two bottom sensors in duplicate specimens with 1CC application), of concrete mixture GUF15. It was observed that the capacitance response with a frequency of 1 kHz showed the highest coefficient of determination ($R^2=0.99$) relative to the other frequencies, exhibiting a power function declining with time. Moreover, the capacitance data with 1 kHz was generally higher by one to two orders of magnitude relative to corresponding data

at other frequencies. For example, at seven days, the capacitance with 1, 10 and 100 kHz were 21.43, 0.85, and 0.22 nF, respectively. In addition, the rates of reduction of capacitance at these frequencies were 386.22, 10.11, and 0.11 nF/day within seven days of casting. Subsequently, the rate of reduction was small (0.227, 0.006, and 0.001 nF/day, respectively) between seven and 91 days, corresponding to the rate of the hydration process at this age. This implies that the capacitance response at 1 kHz was the most sensitive to the state of moisture in the cementitious system, and thus it was selected for the rest of experimental work to provide clear distinction among concrete systems with variable qualities.

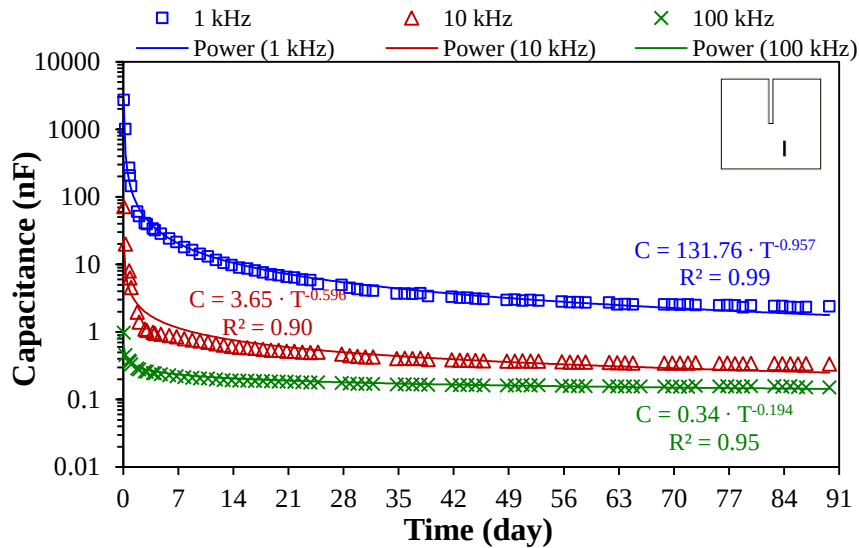


Figure 6.2- Average capacitance response of GUF15 concrete at different frequencies.

Figure 6.3 shows the capacitance of GUF15 specimens at 1 kHz, in terms of average capacitance (readings from two joint sensors in duplicate specimens). Irrespective of the curing method implemented, all the trends showed continuous reduction of capacitance within 28 days. Although the capacitance of specimens with NC and 1CC were within a close range (i.e. NC was 84% that of 1CC) at the first day, specimens with NC exhibited a

steeper reduction of capacitance afterwards which continued up to the fourth day, resulting in three-fold higher capacitance of the 1CC specimens. Higher capacitance indicates higher level of water preserved in the cementitious system (Lee and Zollinger 2012), owing to the application of the curing compound. Comparatively, the capacitance responses of specimens with 1CC+J (top section) and the average of bottom sections of all specimens (NC, 1CC and 1CC+J) did not show significant differences, yielding values at least about two-fold higher than that of corresponding specimens with 1CC.

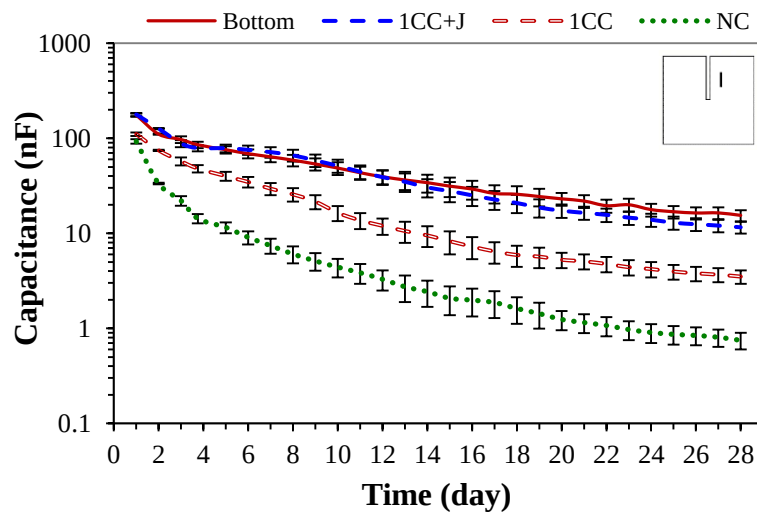


Figure 6.3- Effect of curing on the capacitance of GUF15 concrete in joint locations with reference to average bottom capacitance [1 kHz frequency]. (Note: error bars represent standard deviations)

This indicates the retention of higher moisture level in these concrete systems (top section of 1CC+J and bottom sections of all specimens) up 28 days. These trends conform to the findings of a recent field study by the authors (Tiznobaik, and Bassuoni 2017b) in the sense that concrete pavement sections at joint locations with 1CC+J and bottom sections had the highest efficiency of curing and microstructural development. The trends herein suggest that the dielectric response of concrete can be used as a sensitive indicator for the efficiency

of curing, which is beneficial to monitoring field concrete especially in locations exposed to uncontrolled evaporation (e.g. joints in concrete pavement).

6.3.2 Relationship between hardening behaviour and dielectric response

Isothermal calorimetry was conducted on paste samples with the same proportions as the paste fraction of the concrete mixtures tested to characterize the trends of hydration. For comparison purposes, the heat flow data presented herein are shown over 28 and 80 hours; however, the steady-state stage was developed up to 80-100 hours. The normalized heat flow and cumulative heat released at 23°C for the pastes mixtures are shown in Figure 6.4.

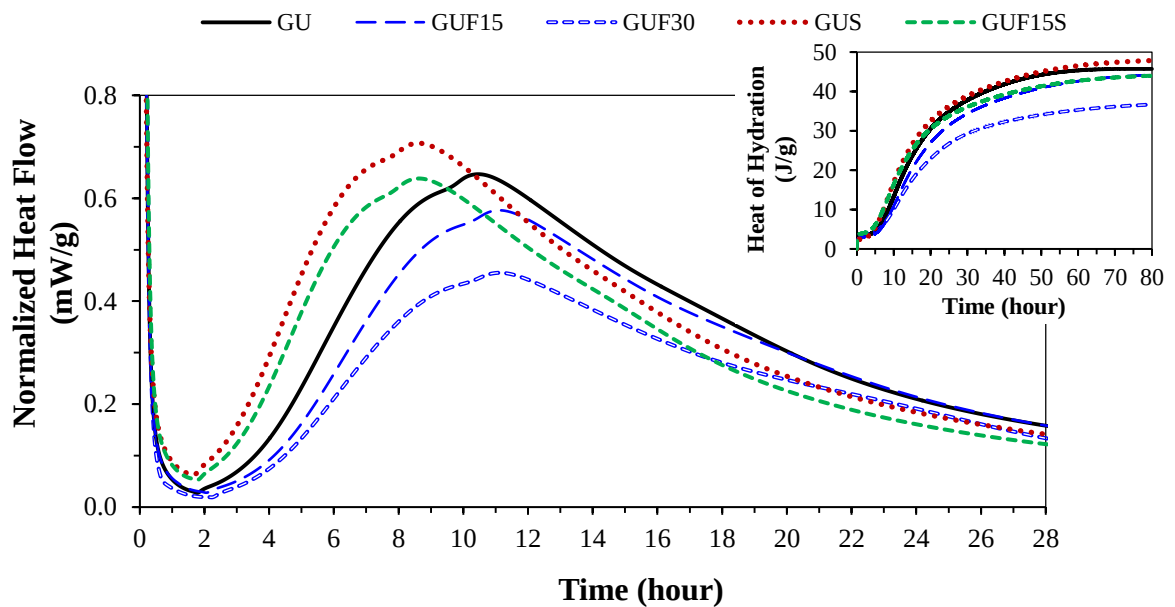


Figure 6.4- Isothermal calorimetry curves for pastes at 23°C.

The trends show that the mixtures had a range of reactivates as targeted in the experimental design. Rapid rise in the first section of the heat flow curve (end of the dormant period) may coincide with the initial setting of the paste, whereas the peak of the curve may indicate its final setting (Mehta and Monteiro 2014) [end of acceleration stage]. However, the exact values of the initial and final setting times for the concrete mixtures were determined by

the penetration resistance test. The heat flow trends showed that mixtures comprising nanosilica, even with 15% fly ash, had accelerated hydration (shifted to left side), whereas mixture incorporating fly ash without nanosilica had delayed hydration (shifted to the right side) relative to the GU mixture, especially with increasing the dosage of fly ash to 30%. For instance, the end of the dormant period and peak of the heat flow curve for mixture GUS occurred 72 and 111 minutes, respectively earlier than that of the GU mixture. Conversely, the end of the dormant period and peak of hydration curve for mixture containing 30% fly ash (GUF30) took place at 36 and 40 minutes longer than that of the reference mixture (GU). Correspondingly, the total heat evolved over 80 hours for GUF30 was approximately 20% less than that of the reference paste.

The results of penetration resistance of the mixtures are given in Figure 6.5, and they conformed to the heat of hydration trends. The penetration resistance trends distinctively showed the hardening behaviour of the mixtures tested, as the nano-modified mixtures (GUS and GUF15S) had accelerated setting, and the mixtures comprising fly ash without nanosilica (GUF15 and GUF30) had retarded setting relative to the reference mixture (GU). For instance, the initial and final setting times of mixture GUS were 58 and 68 minutes, respectively shorter than that of mixture GU. This is attributed to the addition of ultrafine silica particles ($80,000 \text{ m}^2/\text{kg}$) that accelerated the kinetics of hydration and pozzolanic reactions. It has been suggested that even agglomerates of nanosilica particles (silica aggregates) can speed up the kinetics of cement hydration by nucleation and creation of additional sites for early precipitation of hydration products, resulting in shortening of setting time (Kong et al. 2013). Also, it has been shown that early (within 24 h) pozzolanic reactivity is possible in the presence of this type of nanosilica sol. (Ghazy et al. 2016). The effect of higher dosages of fly ash on retarding the hydration and consequently the setting

times of concrete is well-documented (Malhotra et al. 2000, Huang et al. 2013), as the mixture containing 30% fly ash (GUF30) exhibited initial and final setting times that were 46 and 111 minutes, respectively longer than that of the reference GU mixture (GU). Excessive delays were not observed in this study, since the fly ash used had a relatively moderate CaO content (11%), but it is still classified as Class F (CaO less than 15%) according to CAN/CSA-A3001 (2013).

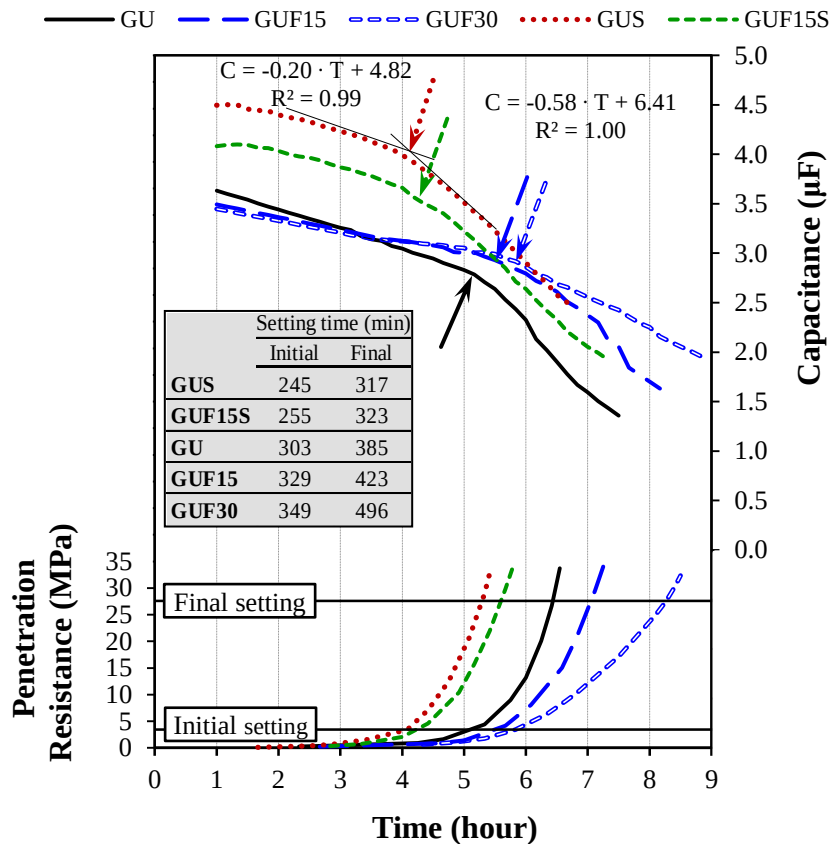


Figure 6.5- Capacitance vs. hardening behaviour of concrete; arrows indicate time corresponding to the knee point on the curves.

When the average dielectric responses of the concrete mixtures were plotted against the elapsed time (Figure 6.5), curves of similar descending trends were obtained. Each curve had a knee point representing the intersection of a bilinear behaviour, with two distinctive

slopes of fitted lines before and after the knee. The steeper slope for each mixture occurred right after the knee point. For instance, the first and second slopes of fitted lines of mixture GUS were -0.20 and -0.58 $\mu\text{F/h}$, respectively (Figure 6.5). At the initial stages of hydration, there are varying degrees of charge mobility where their polarization can induce large dipole moments giving large dielectric responses (McCarter and Afshar 1988). Later, as the viscosity of the paste increases, an irrotational binding of charges reduce the dielectric responses (McCarter and Afshar 1988). It can be observed that the knee point for each mixture coincides with the initial setting time depicted in Figure 6.5. Although the initial setting is defined as an arbitrarily chosen stage referring to phase transition in the matrix from a viscous liquid to the beginning of solidification (Neville 2011), the dielectric response trends signify the initial setting as a specific time in the cement hydration process. Hence, by continually keeping records of dielectric responses with a real-time sensor and determining the slope as a time-dependent coefficient, the hardening threshold of concrete can be acquired.

6.3.3 Relationship between strength/hydration development and dielectric response

Figure 6.6 shows the compressive strength results of all concrete mixtures at different ages, with no curing application at the surface of cylinders. Mixture GUS at one, three and seven day(s) had compressive strengths of 10, 12 and 10%, respectively higher than that of the control mixture (GU). This is in compliance with the heat of hydration and penetration resistance trends, which reflected accelerated hydration and hardening of this mixture due to the presence of nanosilica. Similarly, for GUF15S mixture the results (up to 7 days) were slightly higher than that of GU, due to the dilution of the portland cement component with 15% Class F fly ash. The difference between mixtures with nanosilica and the GU mixture diminished with ages, as no significant difference observed among these mixtures at 56

and 91 day. Therefore, the effect of nanosilica on the evolution of compressive strength resembles that of accelerating admixtures; similar observations were reported by Sonebi et al. (2015), and Ghazy et al. (2016). As expected, the mixtures incorporating fly ash (GUF15 and GUF30) showed significantly lower strengths at early-age compared to that of GU due to retarding effect of fly ash as previously reflected by the hydration and hardening trends. Afterwards, while mixture GUF15 showed 25% increase from 28 to 91 days, the control mixture yielded 15%, and the compressive strength of GUF15 was 94% of mixture GU at 91 days. This increase in the rate of strength gain of GUF15 was due to additional pozzolanic activity beyond 28 days (Neville 2011), especially with the lower content of Class F fly ash (15%). Although the lower compressive strength of GUF30 relative to GU would have been expected to be compensated by the pozzolanic activity at later ages, this was not the case herein.

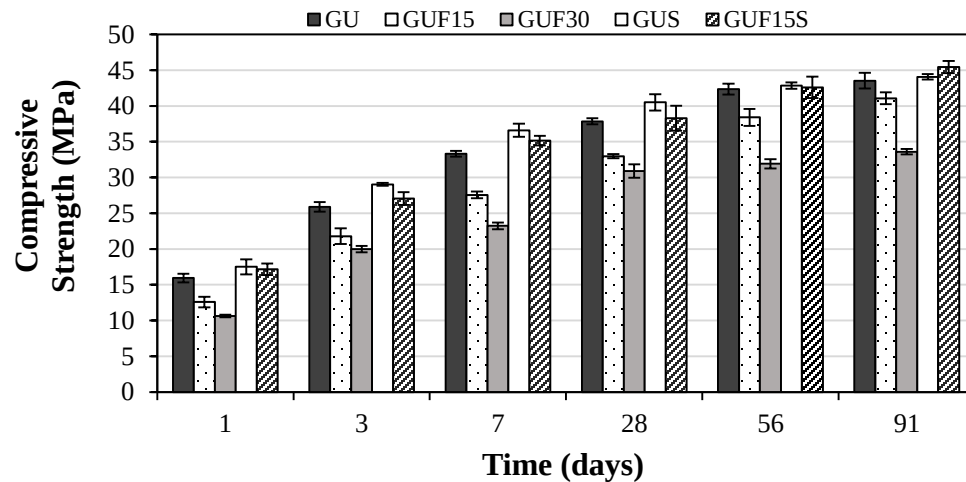


Figure 6.6- Compressive strength of concrete mixtures at different ages. (Note: error bars represent standard deviations)

It seems that worst-case curing scenario (no curing at the top surface of specimens while keeping specimens in moulds), implemented in this stage led to less developed pore

structure at early-age of the mixture comprising 30% Class F fly ash, resulting in evaporation and limiting the progress of pozzolanic activity. This signifies the importance of extending the curing period for mixtures with high volumes of fly ash (Mehta and Monteiro 2014).

The results of capacitance of the concrete mixtures for the bottom and joint locations in prismatic specimens are shown in Figure 6.7. Similar to the previous trends in Figures 6.2 and 6.3, it can be noted that the capacitance of concrete mixtures exponentially decreased with elapsed time irrespective of the sensor location. However, the readings at the bottom of specimens were constantly higher than that of joint locations in the same specimens. For instance, in GUS specimens, the capacitance values of bottom and joints locations at 91 day were 0.885 and 0.541 nF, respectively. This implied a higher degree of hydration efficiency, in the bottom of specimens relative to the joint location, which was exposed to evaporation as discussed earlier.

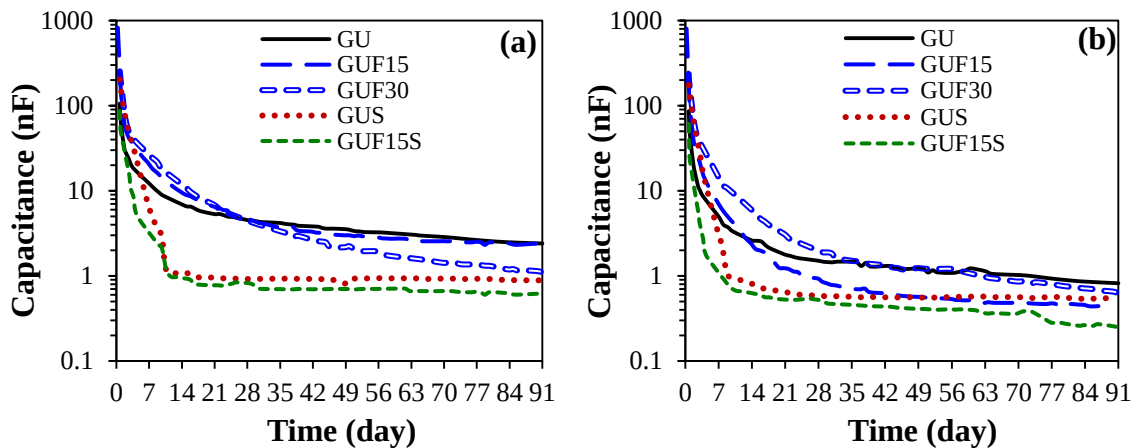


Figure 6.7- Capacitance response of concrete at 1 KHz frequency: (a) bottom, and (b) joint locations.

In addition, for each sensor location the capacitance showed variable trends for the concrete mixtures tested. For the bottom sensor, mixtures incorporating nanosilica (GUS, and

GUF15S) had sharper drop in capacitance within the first 10 days, whereas fly ash mixtures (GUG15, and GUF 30) had higher capacitance relative to the GU mixtures. Subsequently, the rate of change of capacitance with time became small or zero after 10, 28 and 56-75 days for the nanosilica, GU and fly ash mixtures respectively. These trends reflect the faster reactivity and hydration development of the mixtures comprising nanosilica, and slower reactivity of fly ash mixtures relative to the control GU mixture. These trends were generally replicated for the top sensor, but the concomitant effects of evaporation at joints and hydration development affected the magnitude of capacitance (as discussed earlier) and rate of capacitance reduction with time among the five mixtures.

The relationship between strength development and dielectric response of concrete is shown in Figure 6.8. The regression analysis showed a series of strong negative logarithmic correlations between the capacitance response of different concrete mixtures and their corresponding compressive strengths at different ages, as the coefficients of determination were greater than 0.95. According to this relationship, the compressive strength of concrete increased as the capacitance response decreased, which alludes to the dependence of capacitance on the degree of hydration of cementitious paste as discussed below.

As shown in Figure 6.9, the amount of water loss corresponding to portlandite (calcium hydroxide: CH) was determined at the endothermic range of 420-500°C (Loukili et al. 1999), to trace the evolution of hydration and pozzolanic reactions. The GU mixture showed the highest CH contents up to 91 days (Figure 6.9(a)) as it comprised 100% GU cement. Although the contents of CH were lower in GUF15, the rate of CH formation was high up to seven days. The filler/nucleation effect of the finer portion of fly ash particles on improving the hydration of clinker (Pane and Hansen 2005; Sakai et al. 2005), coupled

with the effect of high CaO content (10.8%) in the fly ash (Table 6.1) and dilution of clinker with only 15% fly ash, led to moderate delay in setting time and reduction of compressive strength relative to the GU mixture. After 21 days, CH slightly decreased in the system and stayed almost unchanged, indicating pozzolanic activity in the system, conforming to the improvement in strength of GUF15 up to 91 days. The inclusion of high dosage of fly ash (30%) slowed down the hydration of GUF30 up to seven days in comparison with the GU binder, conforming to the delay in setting and early-age strength. Subsequently, a decreasing trend in the amount of CH in GUF30 indicated some pozzolanic activity, which was insufficient to significantly improve strength at later ages, due to lack of extended moist curing and formation of coarse microstructure as discussed earlier. In the GUS mixture, even after one day, the amount of CH was lower than that of the GU binder, which almost remained unchanged up to seven days, followed by a marginal drop up to 91 days, reflecting intense pozzolanic activity especially at early-age due to the presence of nanosilica, conforming to the hardening and strength results. Also, the trend observed for the GUF15S mixture was quite comparable to that of GUF15, except that in the former higher pozzolanic activity because of nanosilica led to lower amounts of CH. Figure 6.9(b) shows that that irrespective of the type of binder, lower CH contents were found at the vicinity of the joint than that of the corresponding samples at the bottom of specimens due to the adverse effect of drying, which affected the development of hydration and pozzolanic reactions at this location. Such an effect was also captured by the lower capacitance readings at this location [Figure 6.7(b)].

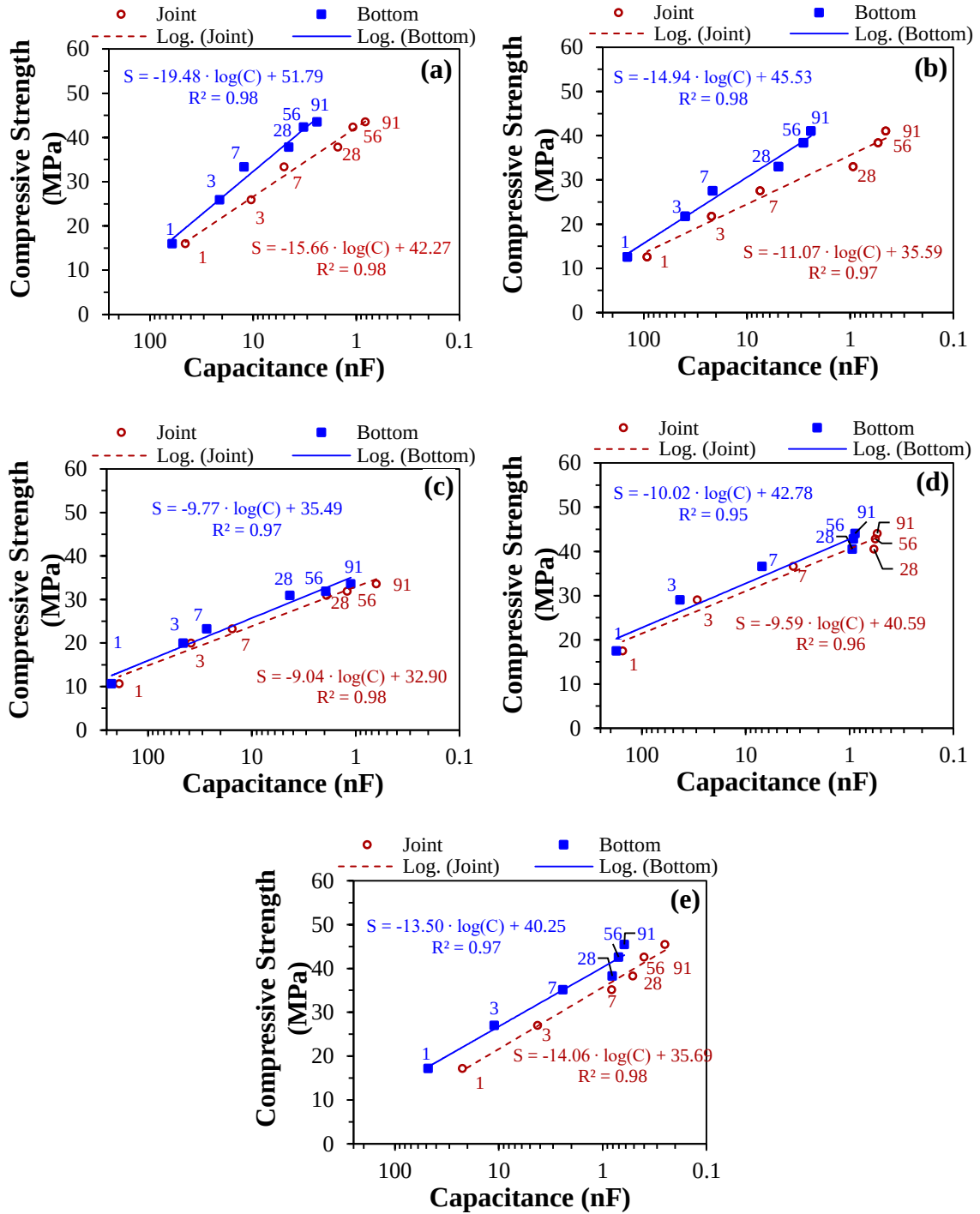


Figure 6.8- Relationship between capacitance response and compressive strength of concrete: (a) GU, (b) GUF15, (c) GUF30, (d) GUS, and (e) GUF15S.

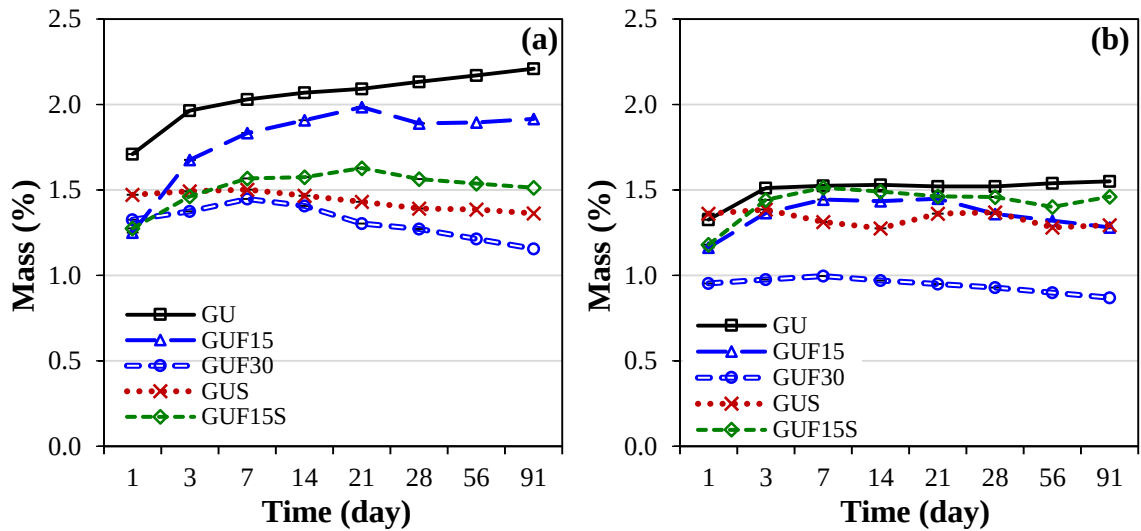


Figure 6.9- Mass loss from samples in TGA corresponding to CH: (a) bottom, and (b) joint locations.

Figure 6.10 shows the results of mass loss after TGA from 105°C to up to 91 days. As expected, all trends showed increase of mass loss with time, irrespective of the sample location, which is linked to progressive hydration development (i.e. formation of more hydration products) of cementitious paste. In Figure 6.10(a), the binary mixtures incorporating fly ash (GUF15 and GUF30) showed less mass loss with respect to that of GU at day one; this gap was gradually reduced up to day seven which may be related to the diluting effect of clinker and slower reactivity of Class F fly ash at early-age, conforming to the portlandite trends and strength results. The mass loss values of mixtures GUS and GUF15S were in a close range with GU up to three days, despite of lower content of clinker, which is likely due to presence of ultrafine nanosilica that accelerated the kinetics of hydration as discussed earlier. From day seven to 91, the mass loss of GU was in a close range and about 1.5% higher than that of all mixtures incorporating SCMs (Figure 6.10(a)). This may be attributed to the presence of higher amounts of CH in GU than that in the other mixtures with SCMs, due to the pozzolanic reactivity Figure 6.9(a). However,

CH in a hydrated system contributes much less to strength relative to C-S-H (Sonebi et al. 2015).

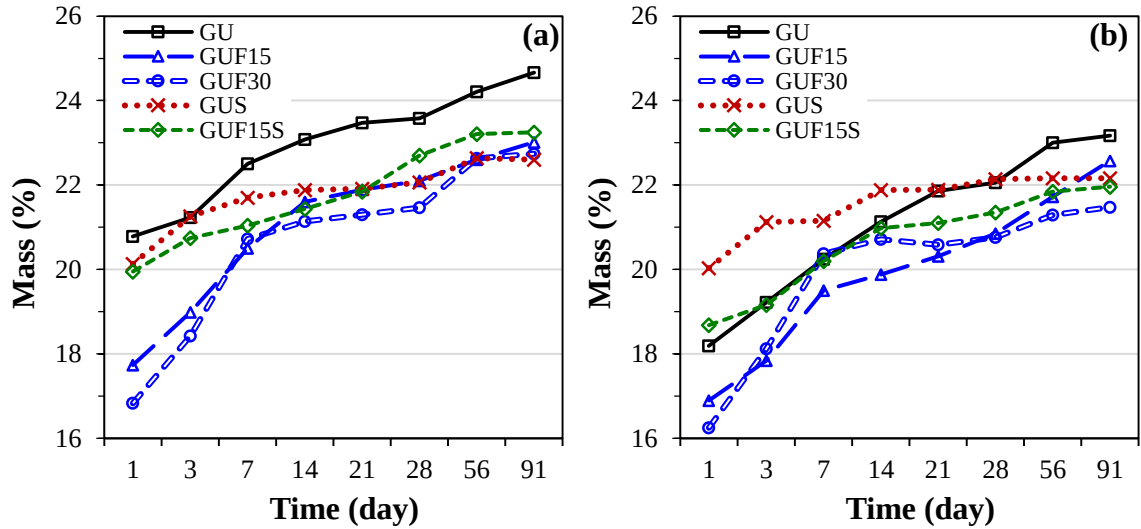


Figure 6.10- Mass loss of samples after TGA: (a) bottom, and (b) joint locations.

In addition, the temperature at which the decomposition of C-S-H initiates is not well established, however, C-S-H is reported to be partially decomposed at 105°C (Gallé 2001; Snoeck et al. 2014), which cannot be differentiated from the evaporable water at this range (up to 105°C). Also, the physical filler effect of either nanosilica or fly ash particles cannot be captured by TGA. Therefore, Figure 6.10, (mass loss from 105 to 1050°C) represents an approximation for the hydration products in the cementitious system, but it does not cover all the aspects contributing to compressive of concrete. The trends at the joint locations in Figure 6.10(b), show lower mass loss values than corresponding samples in the bottom of specimens in Figure 6.10(a). Again, this related to drying effect at the joint location which impaired the hydration and pozzolanic activity, and in turn negatively affected the development of microstructure in that zone.

The results of regression analysis between capacitance response of concrete and mass loss from TGA are presented in Figure 6.11.

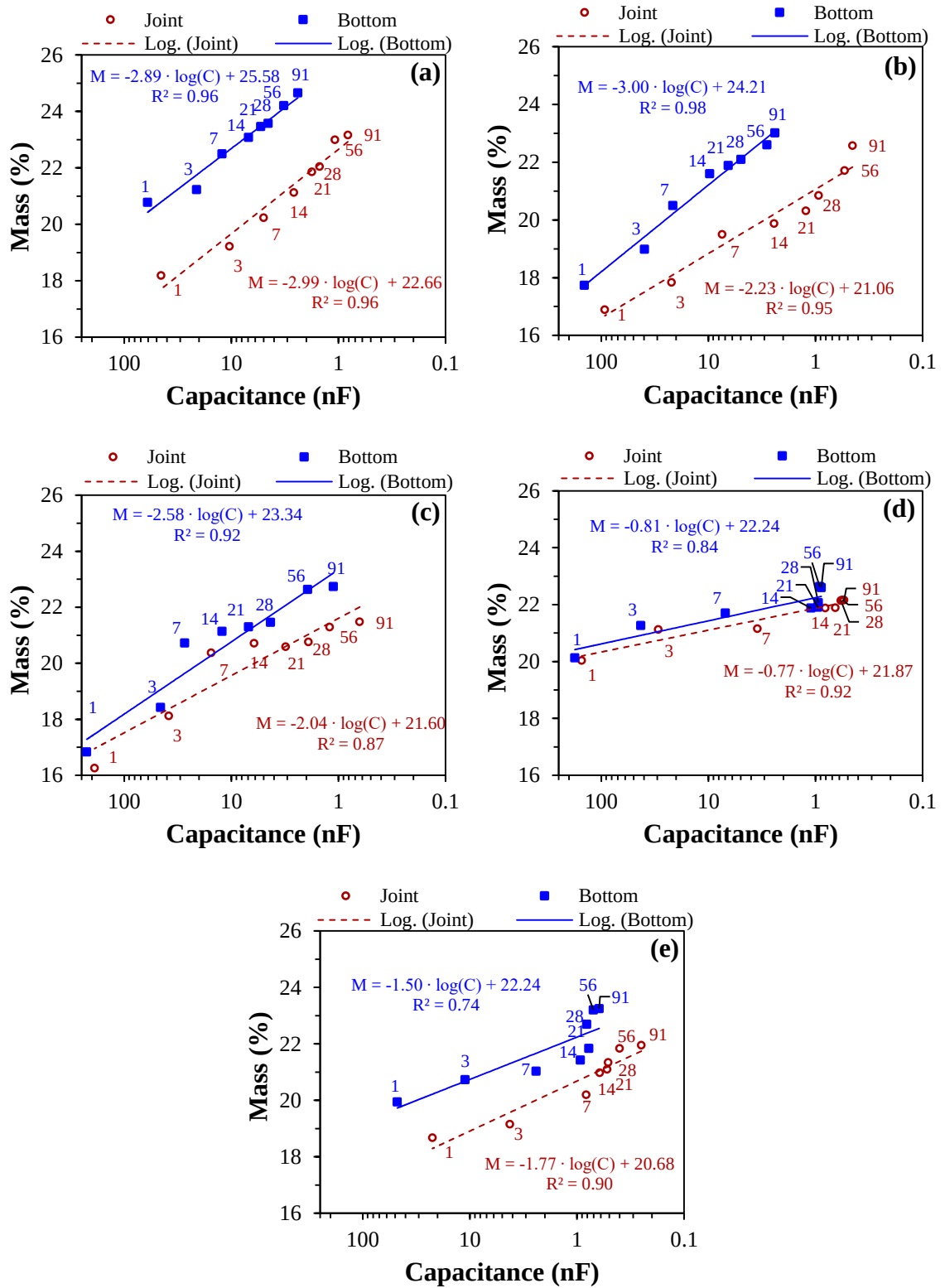


Figure 6.11- Relationship between capacitance response of concrete and mass loss from

TGA: (a) GU, (b) GUF15, (c) GUF30, (d) GUS, and (e) GUF15S.

This showed a series of negative logarithmic correlations where all mixtures gave coefficient of determination (R^2) in the range of 0.77 to 0.98. These relationship suggest that the capacitance response of concrete can reasonably estimate the hydration development of concrete with or without SCMs, in compliance to the strength relationships established in Figure 6.8. These trends suggest that calibration curves can be prepared for individual concrete mixtures to predict their in-situ strength/hydration development in the field from capacitance measurements in a similar manner to the concept of maturity, but with sensitivity to curing efficiency, especially at early-age.

CHAPTER 7: EFFECT OF CURING METHODS ON QUALITY OF CONCRETE JOINTS: EXPERIMENTAL AND MODELING

This chapter focuses on developing an analytical model based on Katz-Thompson relationship to determine the absorption capacity of joints in concrete pavements according to the absorption test customized to joint geometry of pavements developed in Chapter 4.

7.1 Introduction

7.1.1 Overview

Joint deterioration in concrete pavements is linked to penetrability of concrete (MDOT 1996; Chen et al. 2011) which is highly affected by the efficacy of curing and saw-cutting operations as well as environmental conditions such as freeze-thaw cycles, especially with the application of de-icing salts in cold regions (Litvan 1976). Joints are vulnerable locations to higher degree of infiltration since they facilitate two-directional absorption (from vertical joint walls and top surface near the joint) at each side of the joint (Tiznobaik and Bassuoni 2017c). Joints in concrete pavements are created by crack-induced saw-cut to prevent random cracking due to high tensile stresses that may develop during the first 72 hours after casting (FHWA 2007). However, saw-cutting creates two joint walls at early-age making them susceptible to uncontrolled evaporation. This may lead to coarse microstructure at joint locations with respect to the rest of the pavement, and consequently potential for premature joint distress during service.

Capillary absorption is considered representative for understanding the fluid transport, and in turn durability performance of porous building materials (Bentz et al.

2001; Neithalath 2006). Water sorptivity in porous building materials is typically evaluated by gravimetric measurements through determining the sample mass at progressive time intervals using unidirectional flow (ASTM C1585 2015) or immersion (ASTM C642 2013) tests. Also, advanced methods have been applied to demonstrate fluid transport experimentally in porous building materials such as X-ray attenuation and Nuclear Magnetic Resonance (NMR) (Leech et al. 2008). In addition, numerical simulations have been introduced to understand the effect of fundamental properties of medium and fluid on unsaturated flow (i.e. flow occurring through partially saturated media) through complex geometries (Wallach and Parlange 1998; Dershowitz and Fidelibus 1999). In many of these studies, the concept of sorptivity developed from the theory of unsaturated flow originally developed for water transport in soil physics (Richards 1931), has been used to describe the process of capillary water absorption in porous building materials such as concrete (Hall et al. 1984; Carpenter et al. 1993). Richard (1931) showed that the unsaturated flow equation of water can be derived from continuity equation as follows:

$$\nabla \cdot [K(\theta)\nabla\psi(\theta)] = \frac{\partial\theta}{\partial t} \quad \text{Eq. 7.1}$$

where, θ is moisture content per unit volume of dry medium (mm^3/mm^3), $K(\theta)$ is unsaturated capillary conductivity (mm/h), $\psi(\theta)$ is capillary potential (mm), and t is elapsed time (h). Since $K(\theta)$ is strongly dependent on water content (Hall and Hoff 2012) and it is usually nonlinear for most porous materials (Hall and Hoff 2012), Eq. (7.1) has no analytical solution.

Mualem (1976) proposed an analytical model to predict the unsaturated conductivity curve by using the moisture content-capillary potential curve and saturated hydraulic conductivity:

$$K(\theta) = K_s \theta^I \left(\int_0^\theta \frac{d\theta}{\psi(\theta)} / \int_0^1 \frac{d\theta}{\psi(\theta)} \right)^2 \quad \text{Eq. 7.2}$$

$$\theta = \frac{\theta - \theta_r}{\theta_s - \theta_r} \quad \text{Eq. 7.3}$$

where, K_s is saturated permeability (mm/h), θ is normalized moisture content, θ_s is saturated moisture content (mm^3/mm^3), θ_r is residual moisture content (mm^3/mm^3), and I is empirical parameter accounting for the interconnectivity and tortuosity of pores. In order to calculate the integral in Eq. (7.2), different models were proposed for relating the moisture content to the capillary potential (Brooks and Corey 1964; van Genuchten 1980; van Genuchten and Nielson 1985; Kosugi 1994) mainly for soil physics, as for example shown below (van Genuchten 1980; van Genuchten and Nielson 1985):

$$\theta = \left(\frac{1}{1 + (\alpha\psi)^n} \right)^{\left(1 - \frac{1}{n}\right)} \quad \text{Eq. 7.4}$$

where, both α (mm^{-1}) and n (dimensionless) are curve fitting parameters. This numerical model was recently adopted to study capillary suction in mortar and concrete as unsaturated porous media (Smyl et al. 2016; Smyl et al. 2017).

7.1.2 Water Retention Curve (WRC) and Permeability

Concrete durability at an unsaturated state essentially depends on its moisture transport characteristics. The water retention curve (WRC) for concrete shows its capability to retain moisture at different levels of humidity which is crucial in modeling complex unsaturated media. However, determination of complete wetting WRC is difficult. It was reported that the method of water sorption isotherms used to determine WRC for concrete (Baroghel-Bouny et al. 1999) is unreliable at high humidity levels (Gregg and Sing 1982). In addition, it is time-consuming procedure (about 2 years) for concrete samples to reach equilibrium

moisture content (Leech et al. 2006) which makes it impractical for evaluating the transport characteristics of concrete at early-age. However, it is possible to determine the complete WRC based on Van Genuchten parameters, α and n (van Genuchten 1980) from mercury intrusion porosimetry (MIP) test (Leech et al. 2006).

Permeability is defined as the ability of a saturated porous material with a fluid medium to allow flow of fluid passing through, under the action of a pressure differential. This steady-state rate of flow in porous material is directly related to the hydraulic gradient according to Darcy's law (Neville 2011):

$$Q/A = \frac{k}{\eta} \left(\frac{dP}{dL} \right) \quad \text{Eq. 7.5}$$

where, Q is flow rate (m^3/s), A is cross-sectional area of flow (m^2), k is intrinsic permeability (m^2), η is dynamic viscosity of fluid (Pa.s), dP is pressure loss (Pa) over a flow path of length, dL (m). Intrinsic permeability is the concept of permeability that depends on the porous medium characteristics, and it is independent of fluid characteristics that govern the flow, such as viscosity. Permeability can be measured by different methods such as permeability cell, triaxial method, pressure relaxation technique, and beam bending; among all, permeability cell is the most common method. In this method, permeability is determined by establishing a pressure gradient through a specimen to measure the flux by using a device such as permeameter. There are several parameters that must be addressed while using this method. First, whether the specimen has been dried before or it is at an undried conditions. This is critical as a single drying cycle may increase the concrete permeability as much as two times that of undried sample due to microcracking originating from the drying procedure (Powers et al. 1959). Second, for concrete mixture designs with low water-to-binder ratios (w/b), it is challenging to make

the specimen saturated prior to the test. Third, applying adequate confinement pressure is necessary to prevent leakage that may result in loss of exerted pressure (Scherer et al. 2007). Forth, capillary pressure at the outlet side of the sample that is exposed to atmospheric pressure may distort the permeability results if the exerted pressure is not high enough. Even by addressing all aforementioned factors, the variation in permeability results is reported to be in the magnitude of $\pm 50\%$ (Scherer et al. 2007). Furthermore, water permeability tests are laborious to perform in terms of time and required equipment, which makes other testing techniques (e.g. absorption) appealing.

7.1.3 Katz-Thompson Theory

Percolation theory was applied by Katz and Thompson (1986) on sedimentary rocks to develop a relationship between critical pore diameter and air permeability. They suggested a correlation between intrinsic permeability of saturated porous media to critical pore diameter and the ratio of bulk to pore solution conductivity based on the following equation:

$$k = \frac{1}{226} d_c^2 \frac{\sigma}{\sigma_0} \quad \text{Eq. 7.6}$$

where, d_c is critical pore diameter (m), σ is bulk conductivity (S/m), and σ_0 is pore solution conductivity (S/m). To evaluate the validation of Katz-Thompson equation (Eq. 7.6), studies have been conducted using water permeability. The published water permeability and pore diameter data for cement paste and concrete were analysed by El-Dieb and Hooton (1994). Instead of measuring the formation factor (σ_o/σ) directly, they calculated the ratio from pore parameters of the equation proposed by Katz and Thompson (1987) based on mercury intrusion porosimetry (MIP):

$$\frac{\sigma}{\sigma_o} = \frac{d_{max}^e}{d_c} \varphi S(d_{max}^c) \quad \text{Eq. 7.7}$$

where, $d_{max}^e = 0.34d_c$, φ is total porosity (%), and $S(d_{max}^c)$ is fraction volume of pores larger than d_{max}^e . These researchers reported a poor correlation between experimentally measured permeability and the one obtained from Eq. 7.6. Christensen et al. (1996) experimentally measured conductivity of cement pastes with different w/b and correlated published permeability data with Eq. 7.6, and reported a good agreement. Tumidajski and Lin (1998) measured conductivity and permeability of concrete directly and found a poor correlation with Eq. 7.6. However, they reported a good correlation between measured formation factor and the one obtained from Eq. 7.7, according to MIP results. Also, Halamickova et al. (1995) supported this relationship for paste with w/b of 0.50. Nokken and Hooton (2008) stated that the previous published data that reported poor correlations suffered from experimental deficiencies in measuring permeability as described in section 1.2. They tested concretes with a wide range of w/b (0.30-0.90) and reported a high correlation between measured water permeability and Katz-Thompson method (Eq. 7.6). Based on the aforementioned analogy, an effort was made in the current study to develop an analytical model according to Katz-Thompson relationship to determine the absorption capacity of joints in concrete pavements with respect to a customized absorption test protocol developed by the authors for concrete joints (Tiznobaik and Bassuoni 2017c).

7.2 Experimental Program

7.2.1 Materials and Mixtures

General Use (GU) portland cement and fly ash (Class F), as a supplementary cementitious material (SCM), meeting the requirements of CAN/CSA-A3001 (2013) standard were used in the concrete mixtures. Table 7.1 shows the chemical composition and physical properties

of the GU cement and fly ash. An air-entraining admixture complying with ASTM C260/C260M22 (2016) was used in concrete mixtures to achieve a fresh air content of 5 to 8%. Also, a high-range water reducing admixture (HRWRA) complying with ASTM C494/C494M23 (2013) Type F was used to obtain a target slump of 50 ± 20 mm. Both air content and slump represent typical values for concrete pavement mixtures in Manitoba, Canada (CW3310–R17 2015). Locally available natural gravel coarse aggregate (with a maximum size of 20 mm) with specific gravity and absorption of 2.65 and 2%, respectively was used. The fine aggregate was well graded river sand with a specific gravity, absorption, and fineness modulus of 2.53, 1.5% and 2.9, respectively. To simulate curing of concrete pavements in most North American jurisdictions, a white compound made of high-grade hydrocarbon resins in a water-based emulsion conforming to ASTM C309 (2011) Types 1, 2 and 1-D, Class B was used.

Table 7.1: Chemical composition and physical properties of GU cement and fly ash

	GU	Fly Ash
<u>Chemical Composition</u>		
SiO ₂ (%)	19.8	56.0
Al ₂ O ₃ (%)	5.0	23.1
Fe ₂ O ₃ (%)	2.4	3.6
CaO (%)	63.2	10.8
MgO (%)	3.3	1.1
SO ₃ (%)	3.0	0.2
Na ₂ O (%)	0.1	3.2
<u>Physical Properties</u>		
Specific Gravity	3.17	2.12
Fineness, m ² /kg	410*	290*
Mean particle size, µm	13.15	16.56

*Blaine fineness.

7.2.2 Procedures

In this study, two different concrete mixtures were used, which represent typical concrete pavement mixture designs in Manitoba, Canada [Table 7.2] (CW3310–R17 2015). Laboratory specimens were prepared from the two ready-mixture concretes delivered to the laboratory by a certified supplier, approved for providing concrete for pavements by

the City of Winnipeg (COW), Manitoba. For each mixture design, four 500 mm×520 mm×150 mm concrete slabs were cast in wooden formwork, [Figure 7.1(a)]. Also one Dielectric Moisture Sensors (DMS) for each slab was installed prior to casting [Figure 7.1(b)]. DMS is a capacitance based sensor designed to measure moisture content in masonry or concrete. Each sensor has two parallel copper electrodes attached in the same plastic tape. The sensors were installed in a fashion to be parallel to the joint after saw-cutting. Each set of four slabs received four different types of curing: no curing, one coat of curing compound as per the COW specifications, one coat of curing compound plus filling the joint immediately after saw-cutting with the curing compound, and two coats of curing compound referred to hereafter as NC, 1CC, 1CC+J, and 2CC, respectively. After excess moisture due to bleeding had evaporated (30 to 45 min after the finishing operations) the curing compound was applied using portable sprayers on the designated 1CC, 1CC+J, and 2CC sections. The coat of curing compound was applied at the rate of 0.23 l/m² according to the manufacturer's specifications.

Table 7.2: Proportions of concrete mixtures per cubic meter

Constituent	Unit	Mixture 1	Mixture 2
20 mm Coarse Aggregate	kg	1170	1213
Fine Aggregate	kg	655	679
Air Entrainment	l	0.25	0.21
Fly Ash (Class F)	kg	57	48
Cement (GU)	kg	323	272
Water	kg	133	128
Water Reducer	l	1.33	1.12
Water-to-Binder (w/b)		0.35	0.40
Slump		50±20 mm	
Air Content		5-8%	

The formwork was removed after 7 h and the curing compound was applied to the side surfaces of the slabs. The slabs were saw-cut to establish the contraction joints with the dimensions of 3 to 4 mm wide and 50±3 mm deep. Three saw-cuts were applied on each slab at about 22 h, after casting. It is reported the conventional sawing of concrete

pavements generally begin at about the final setting time (Krstulovich et al. 2011) and may extend up to 24 h after casting (Zollinger 2001). This saw-cutting time was chosen corresponding to concrete maturity/strength of about 12.3 MPa, when the cutting activities (conventional wet-cut) in concrete pavements are still feasible without much risk of random cracking (Tiznobaik and Bassuoni 2017b). The slabs were kept under laboratory conditions ($23\pm 2^{\circ}\text{C}$ and $55\pm 5\%$ RH). The capacitance was measured by Agilent LCR U1733C meter using 1 kHz frequency. At 28 days, eight cores (75 mm in diameter) per each slab were extracted directly at the joints. The location of cores and testing scheme are illustrated and defined in Figure 7.1(a) and Section 2.3, respectively.

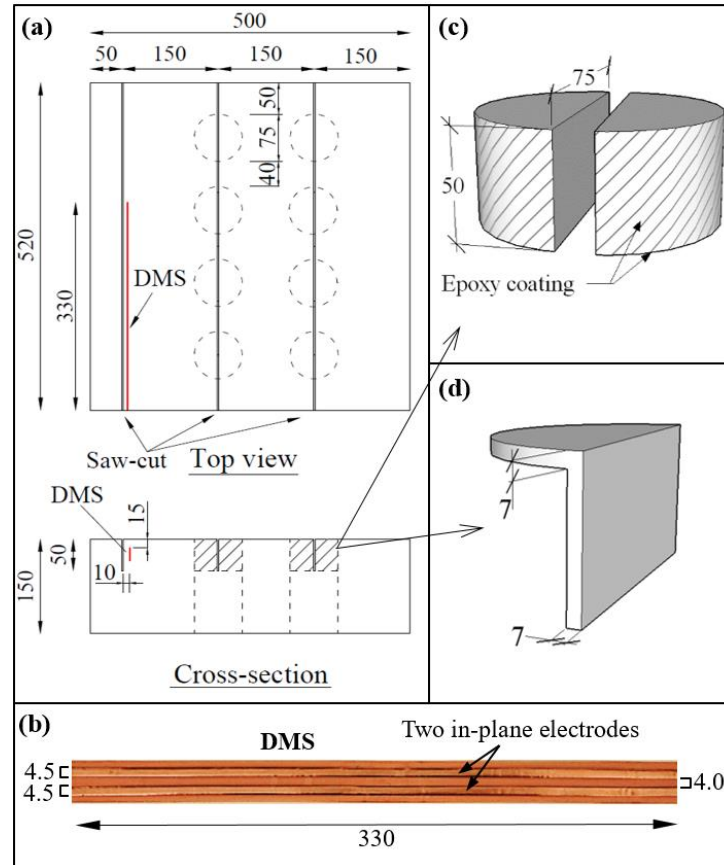


Figure 7.1- A schematic diagram of a slab specimen showing: (a) the location and number of the cores extracted, (b) DMS, (c) top joint specimen, and (d) specimen for MIP and quantifying saturated moisture content. (Note: all dimensions are in mm)

7.2.3 Tests

A customized absorption test was performed on the cores extracted from laboratory slabs, as it was found to characterize concrete of variable microstructural qualities with high repeatability (Tiznobaik and Bassuoni 2017c). In this method, three full-depth 75 mm cores are extracted from concrete pavement slabs and cut into 50 ± 2 mm slices parallel to the surface of concrete. The curing compound residue was gently cleaned with a brass brush, and 50 ± 2 mm thick slices were cut from the top part of cores at joints [Figure 7.1(c)]. The concrete specimens were placed in an environmental chamber at $50 \pm 2^\circ\text{C}$ and 40% RH until reaching a constant mass (difference in mass in two successive days is less than 0.1%). All surfaces except the joint walls and top surfaces were sealed by a rapid setting epoxy [Figure 7.1(c)]. The specimens were then placed in a sealed desiccator under vacuum pressure (~ 85 KPa) for 6 hours. Afterwards, the specimens were removed from the desiccator and the initial dry mass to the nearest 0.01 g was recorded. Each specimen was freely immersed in 4% calcium chloride (CaCl_2) solution for up to 360 min, and the amount of absorption after 1, 5, 10, 20, 40, 80, 160 and 360 minutes were recorded to the nearest 0.01 g. These results were presented as the absorption of concrete specimens by:

$$I_a = \frac{m_t}{a \cdot \rho} \quad \text{Eq. 7.8}$$

where, I_a is absorption depth of concrete at a certain point of time (mm) m_t is difference between wet mass and dry mass at a specific point of time (g), a is cross sectional area of the exposed surfaces (mm^2), and ρ is density of the fluid [i.e. 4% CaCl_2 solution at 23°C] (g/mm^3).

The experimental results of the absorption test were statistically evaluated by the Analysis of Variance (ANOVA) method at a significance level (α) of 0.05. ANOVA is a

statistical model used to analyze the differences between group means and their variation among and between groups. The variance of a specific parameter is separated into components attributed to different sources of variation. ANOVA tests the hypothesis that the means of several groups are comparable via a *t*-test, and it can generalize the test to more than two groups. According to ANOVA, exceeding the F_{cr} for an *F*-distribution density function indicates that the variable tested has a statistically significant effect on the average results (Montgomery 2012). The ANOVA results were further evaluated by the Least Significant Difference (*LSD*) post hoc test (Fisher's test), when applicable, to declare pairwise comparisons between the means of each two groups. *LSD* post hoc test declares two means significantly different if the absolute value of their sample differences exceeds the least significant difference, *LSD* (Montgomery 2012).

The effect of curing compound application on the development of microstructure in concrete was assessed by mercury intrusion porosimetry (MIP). To capture the characteristics of pore structure, the MIP technique was conducted on laboratory concrete samples at 28 days. To prepare samples for MIP, bigger chunks about 7 mm were extracted from the specimens of two cores illustrated in Figure 7.1(d), which visually inspected and an effort was made to remove large aggregate. The result of this process was 16-22 chunks with the size of 4-7 mm, representing the concrete microstructure at specific locations. The samples were oven dried for 72 h at a temperature of 55 ± 2 °C; they were then kept in a desiccator containing calcium sulfate for 24 h. This method of drying was adopted to avoid decomposition of calcium silicate hydrate (C-S-H) and formation of micro-cracks, which may occur at higher drying temperatures. The contact angle and the surface tension of mercury were taken as 130° and 485 dynes/cm, respectively (Shi and Winslow 1985; Said et al. 2012).

7.2.4 Modeling of Moisture Transport in Concrete

7.2.4.1 Material parameters

The intrinsic permeability, k was measured using MIP results according to Katz-Thompson theory (Eqs. 7.6 and 7.7). Then saturated permeability, K_s (mm/h) was calculated as:

$$K_s = \frac{k \cdot \rho \cdot g}{\eta} \cdot 3.6 \times 10^6 \quad \text{Eq. 7.9}$$

where, g is 9.81 (m/s²); ρ is density of ($\rho=1010$ kg/m³), and η is dynamic viscosity ($\eta = 9.6 \times 10^{-4}$ Pa·s) of the fluid [i.e. 4% CaCl₂ solution at 23°C] (Conde 2004). Also, factor 3.6×10^6 was multiplied to change the unit from m/s to mm/h. The saturated moisture content (θ_s) was measured from concrete samples shown in Figure 7.1(d). Thickness of the sample (7 mm) was chosen to reflect the curing zone effect according to Gowriplan et al. (1990). For each data point, samples were cut from three cores, oven dried for 72 h at a temperature of 55 ± 2 °C, and consequently submerged in 4% calcium chloride solution for five days. Then, θ_s was calculated as an average ratio of the mass gains divided by dry masses. To simplify the case for concrete modeling, residual moisture content was reported to be considered as zero (i.e., $\theta_r=0$), which does not represent a significant effect on unsaturated flow models (Pour-Ghaz et al. 2009; Smyl et al. 2016). While Mualem (1976) proposed the value of I (power value of θ in Eq. 7.2) for most soil types to be 0.5, it was reported that I does not represent a material property, and hence it should be considered as a fitting parameter (Kosugi 1994). Smyl et al. (2016) calculated the value of I through a model training by using maximum likelihood approach as -7.0 and -9.0 for concrete and mortar, respectively. This value was reported to be in the range of -7 to -5 and -9.0 to -8.0 for damaged concrete and mortar, respectively (Smyl et al. 2016). Hence, in this study I was assumed as an average reported value of -6.0. Also, a least square procedure using

MATLAB was applied to MIP results through curve fitting to Eq. 7.4 to determine van Genuchten model parameters (α and n).

7.2.4.2 Numerical simulation

To simulate unsaturated flow of 4% CaCl_2 solution in concrete specimens extracted at joints, finite element software HYDRUS (2D/3D) (Šimůnek et al. 2008) was used. The software used Galerkin finite element method with linear basis functions to obtain a solution of the flow equation (Eq. 7.1) with respect to none-linear permeability function of Eq. 7.2, subject to imposed initial and boundary conditions. For the epoxy coated surfaces [Figure 7.1(c)] zero flux boundary conditions were applied while for the exposed surfaces to absorption, saturated moisture contents (θ_s) were applied. The finite element mesh was structured as space discretization by tetrahedral elements with a maximum dimension of 1.0 mm, and model geometries are illustrated in Figure 7.2.

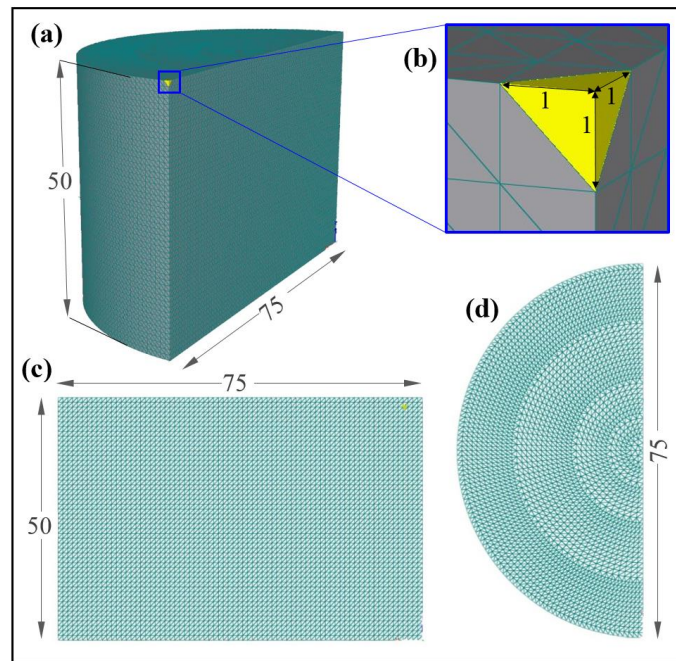


Figure 7.2- Finite element of the analytical model: (a) 3D mesh, (b) tetrahedron element, (c) joint wall, and (d) top view of finite element mesh. (Note: all dimensions are in mm)

The materials parameters were identified based on the details described in Section 2.4.2, and used as input parameters to the model. The presented model was solved in terms of moisture content.

7.3 Results and Discussion

7.3.1 Experimental

7.3.1.1 Dielectric response of concrete

Figure 7.3 shows the dielectric response of concrete, in terms of capacitance. Irrespective of mixture designs and curing schemes implemented, all the trends showed continuous reduction of capacitance within 28 days, exhibiting a power function declining with time and a series of high coefficients of determination ($R^2 > 0.91$). This implies that the capacitance response at 1 kHz was sensitive to the level of moisture in the cementitious system. Although the capacitance of specimens with NC and 1CC were within a close range (i.e. NC was 80% and 84% that of 1CC for w/b of 0.35 and 0.40, respectively) at the first day, slabs with NC exhibited steeper reductions of capacitance afterwards which continued up to the fourth day, resulting in three- and four-fold higher capacitance of the 1CC slabs for w/b of 0.35 and 0.40, respectively. Higher capacitance indicates higher state of water retention in the cementitious system (Lee and Zollinger 2012) due to the curing compound application, and or higher w/b of the mixture design.

While the capacitance responses of slabs with 1CC and 2CC for each w/b showed comparable trends and values, the 1CC+J results indicated significantly higher values with respect to that of 1CC and 2CC. This implies that covering the exposed joint walls with curing compound immediately after saw-cutting provides a higher moisture retention level up to 28 days. The trends herein show that the dielectric response of concrete can be used as a sensitive parameter to indicate curing efficiency that can be used as a measure to

monitor field concrete especially in locations subjected to uncontrolled evaporation (e.g. joints in concrete pavement).

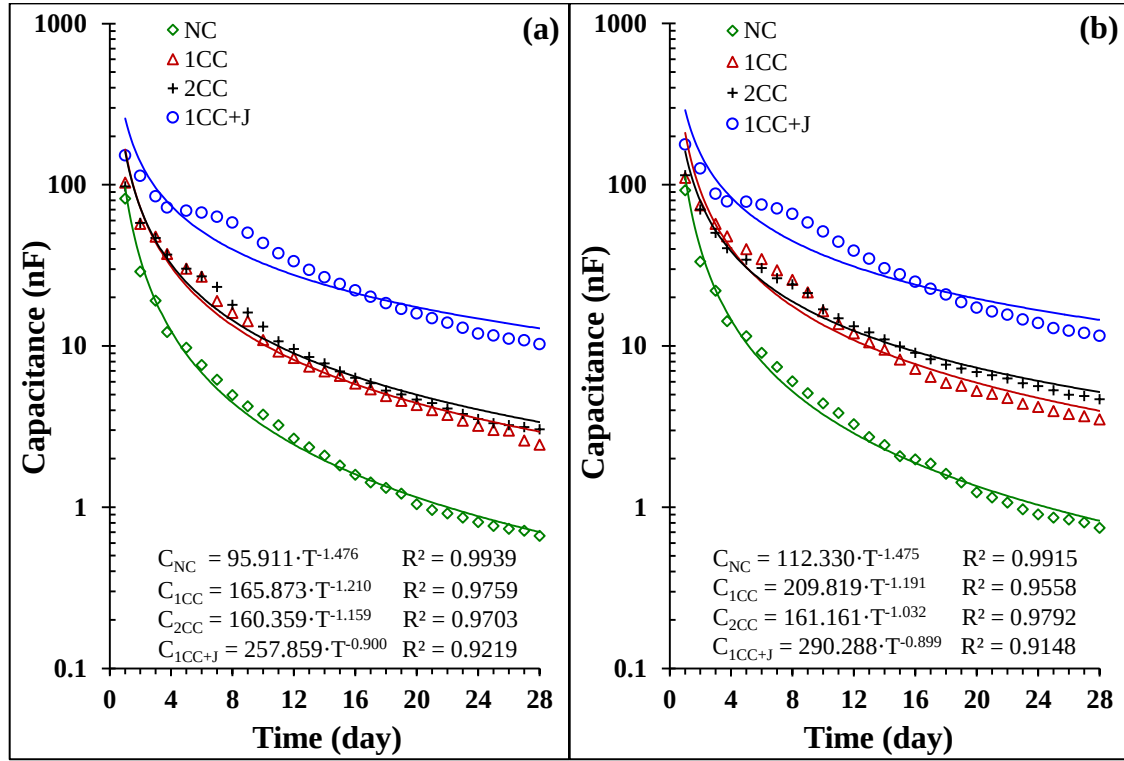


Figure 7.3- Effect of curing on capacitance of concrete: (a) w/b 0.35, and (b) w/b 0.40.

(Note: continuous lines indicate trend lines)

7.3.1.2 Absorption of concrete

Concrete absorption was determined according to the test described in section 2.3. Concrete absorption (Figure 7.4) was expressed in terms of depth considering the density of 4% CaCl_2 and exposed surface area (Eq. 7.8). Each series of data refers to the three samples tested from each location which represent absorption of the top part of the cores at joint locations. Table 7.3 shows ANOVA results for absorption that determined the statistical significance among each of four groups of joints. All the F values were greater than the F_{cr} of 4.07, representing significant differences among the data from 1 min up to 360 min.

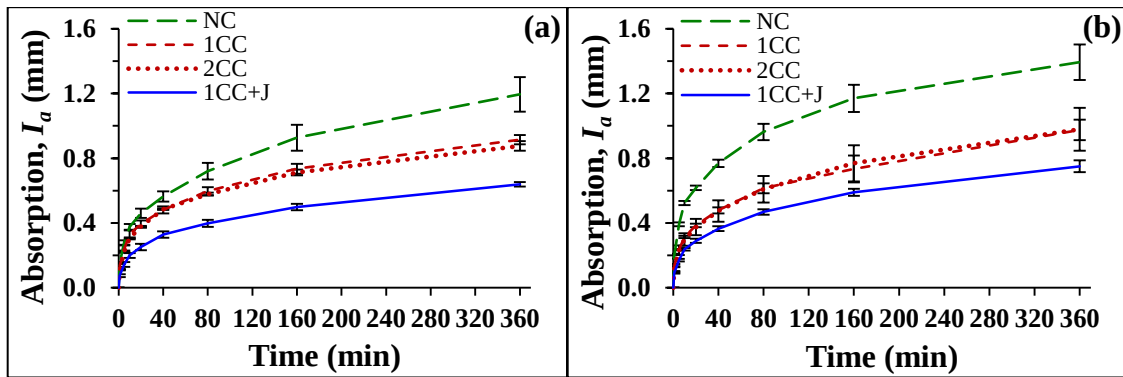


Figure 7.4- Absorption of concrete: (a) w/b 0.35, and (b) w/b 0.40. (Note: error bars represent standard deviations)

Table 7.3: ANOVA results for absorption of laboratory cores

Comparison Groups	w/b	<i>F</i>								<i>F_{cr}</i>
		1(min)	5	10	20	40	80	160	360	
NC, 1CC, 2CC, and 1CC+J	0.35	20.82 ^a	41.85*	56.58*	53.09*	60.45*	54.35*	44.81*	47.35*	4.07
	0.40	41.05*	96.85*	105.41*	78.35*	65.43*	51.26*	27.43*	24.64*	

*Indicates statistical significance.

The ANOVA results were further evaluated by post hoc Fisher's test to state pairwise comparisons and the results are presented in Table 7.4. The results specify significant differences between the absorption values of NC and 1CC starting from 1 min up to 360 min for both w/b. These were supported by Fisher's test at 360 min as one coat of curing compound on the top surface caused absorption reduction of 0.282 and 0.419 mm which were greater than *LSD* of 0.106 and 0.173 mm for w/b of 0.35 and 0.40, respectively (Table 7.4). This may indicate that the main difference between NC and 1CC was due to higher absorption through the top surfaces of joints in NC that were exposed to laboratory conditions (23±2°C and 55±5% RH). The case for 1CC and 2CC was quite different where the results show no significant difference between the two methods of curing. For instance, Fisher's test results showed difference in absorption of 1CC and 2CC as 0.037 and 0.004 mm which were less than *LSD* of 0.106 and 0.173 mm for w/b of 0.35 and 0.40, respectively

(Table 7.4). Hence, it can be concluded that the second coat of curing compound in 2CC did not markedly improve the microstructure in the top surface zone of concrete joints relative to 1CC. The results for both w/b indicate significant differences between the absorption values of 1CC and 1CC+J starting from 1 min up to 360 min. These were supported by Fisher's test as covering the joint walls with curing compound on 1CC+J caused absorption reduction of 0.277 and 0.224 mm which were greater than *LSD* of 0.106 and 0.173 mm for w/b of 0.35 and 0.40, respectively (Table 7.4). As the top surface of concrete in both 1CC and 1CC+J received one coat of curing compound, the differences in the absorption values originated from the absorption through joint walls. Conforming to the capacitance trends, this suggested that covering the joint walls with curing compound immediately after saw-cutting prevented uncontrolled evaporation resulting in better microstructural development in the 1CC+J slab.

Table 7.4: Fisher's test for absorption of cores at 360 min

w/b	Comparison Aspect	Absorption (mm)	Difference (mm)			LSD** (mm)
			1CC	2CC	1CC+J	
0.35	NC	1.195	0.282*	0.319*	0.559*	0.106
	1CC	0.913	-	0.037	0.277*	
	2CC	0.876	-	-	0.240*	
	1CC+J	0.636	-	-	-	
0.40	NC	1.394	0.419*	0.415*	0.643*	0.173
	1CC	0.975	-	0.004	0.224*	
	2CC	0.979	-	-	0.228*	
	1CC+J	0.751	-	-	-	

*Indicates statistical significance.

***LSD* is calculated as; $LSD = t_{(\alpha/2, v)}(2 \cdot MS_E/n)^{0.5}$, *t* is the critical value from *t*-distribution, *MS_E* is the mean of all groups' variances, *n* is number of data in each group.

7.3.1.3 Pore structure

The MIP test results at 28 days are shown in Figure 7.5 and Table 7.5. The trends generally complied with the absorption results, where NC had a relatively coarser pore structure than that of 1CC [Figures 7.5(a), 7.5(b) and Table 7.5]. For example, the cumulative intrusion decreased from NC to 1CC by 13 and 17% for w/b of 0.35 and 0.40, respectively. Also the

threshold pore dimeters were reduced by 55 and 36% for w/b of 0.35 and 0.40, respectively. The MIP results also indicated 1CC and 2CC had similar trends as cumulative intrusions and threshold pore diameters for both samples were in close ranges (Table 7.5), which represent comparable microstructural development at the joint locations. The MIP results for the 1CC+J showed reduction in cumulative intrusion and threshold pore diameter with respect to 1CC. For instance, the threshold pore diameters for 1CC+J and 1CC were 0.090 and 1.250 μm for w/b of 0.35, and 0.150 and 2.070 μm for w/b of 0.40, respectively, which complies with the absorption results.

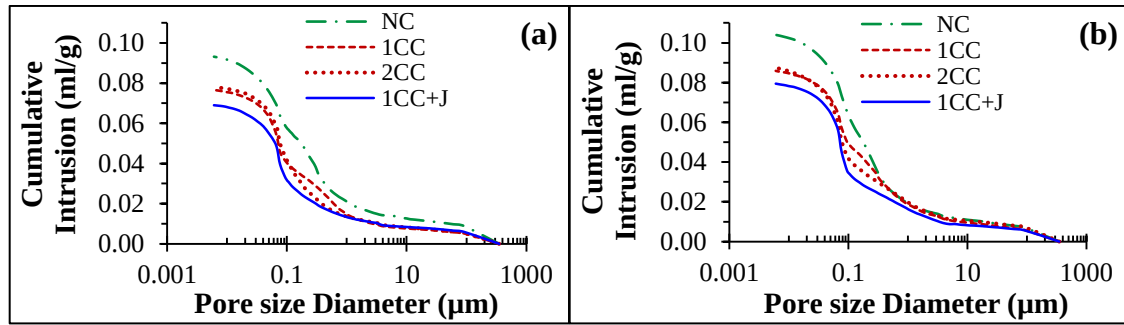


Figure 7.5- Pore size distribution of concrete at 28 days: (a) w/b 0.35, and (b) w/b 0.40.

Table 7.5: MIP test results for cores at 28 days

w/b	Scheme	Total Intrusion $\times 10^{-2}$ (ml/g)	Threshold Pore Diameter (μm)	Apparent Total Porosity (%)	Critical Pore Diameter (μm)
0.35	NC	9.3	2.790	19.2	0.077
	1CC	7.7	1.250	16.2	0.069
	2CC	7.8	1.450	16.4	0.069
	1CC+J	6.9	0.090	14.5	0.069
0.40	NC	10.4	3.220	21.4	0.077
	1CC	8.6	2.070	17.9	0.077
	2CC	8.7	2.100	18.1	0.069
	1CC+J	7.9	0.150	16.8	0.069

7.3.2 Modeling

Table 7.6 shows saturated permeability K_s , saturated moisture content (θ_s), and van Genuchten model parameters (α and n) values for different mixture designs and curing

applications. As expected K_s and θ_s decreased with improvement in the curing regime, lowering w/b, or both. For instance, when the curing changed from NC to 1CC, K_s decreased by 41 and 42% for w/b of 0.35 and 0.40, respectively. This indicated better microstructural development of concrete. Figures 7.6(a) and 7.6(b) show the predicted absorption of concrete based on the solution of the numerical model for the both w/b. It can be noted the values and trends are in close range to the experimental results [Figures. 7.4(a) and 7.4(b)]. For instance, at w/b of 0.35, the predicted absorption values of NC, 1CC, and 1CC+J at 360 min were 1.195, 0.857, and 0.635 mm from the numerical model and 1.195, 0.915, and 0.638 from experimental results, respectively.

Table 7.6: Permeability, saturated moisture content, and van Genuchten model parameters values for cores at 28 days

w/b	Scheme	$S(d_{max}^e)$ (%)	σ/σ_0 $\times 10^{-3}$ (%)	k $\times 10^{-19}$ (m ²)	K_s $\times 10^{-6}$ (mm/h)	θ_s (mm ³ /mm ³)	α $\times 10^{-5}$ (1/mm)	n
0.35	NC	17.5	11.4	2.999	11.165	0.130	1.314	1.405
	1CC	15.2	8.4	1.775	6.610	0.110	1.545	1.402
	2CC	15.6	8.7	1.836	6.836	0.110	1.216	1.352
	1CC+J	13.2	6.5	1.382	5.146	0.102	1.656	1.336
0.40	NC	19.7	14.3	3.753	13.972	0.155	1.507	1.408
	1CC	16.8	10.2	2.161	8.048	0.125	1.997	1.403
	2CC	16.7	10.3	2.168	8.071	0.127	2.041	1.397
	1CC+J	15.7	9.0	1.900	7.075	0.113	1.561	1.344

Figures 7.7(a) and 7.7(b) show response of numerical model in predicting absorption of cores at different time intervals up to 360 min. The points are located on or slightly under/above the line of equality between the experimental and predicted absorption values. The performance of the numerical model for absorption at joints was assessed based on the average absolute error (AAE) to reflect the accuracy of the predictions:

$$AAE = \frac{100}{n_D} \sum_{i=1}^n \frac{|m_i - c_i|}{m_i} \quad \text{Eq. 7.10}$$

where, m_i and c_i are the measured and calculated absorption values (mm), respectively, and n_D is the number of data points. The AAE is defined as the average magnitude of the errors, which indicates closeness of the measured and calculated values. The AAE for w/b of 0.35 and 0.40 were 9.20 and 7.42%, respectively. Also, the coefficients of determination (R^2) for the measured and calculated absorption data were 0.9919 and 0.9955 for w/b of 0.35 and 0.40, respectively, indicating strong association between the measured and calculated datasets. Therefore, it can be substantiated the numerical model reliably predicted the absorption values starting from 1 to 360 min (Figures. 7.6 and 7.7).

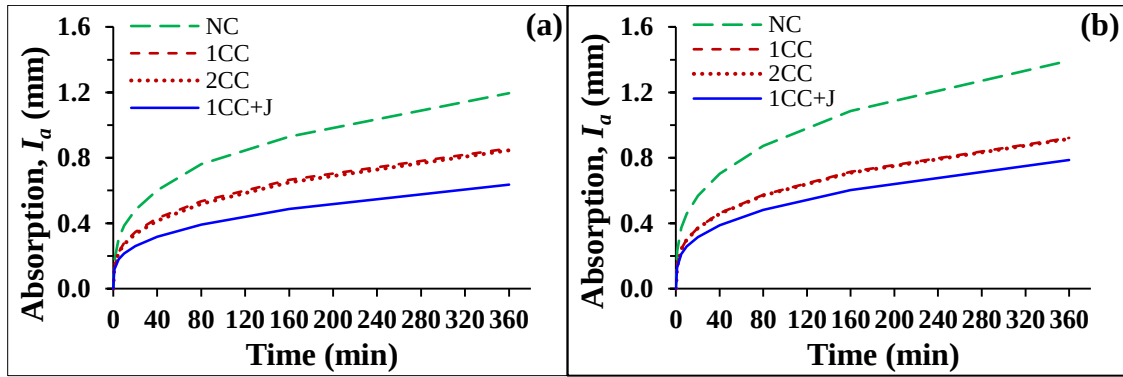


Figure 7.6- Predicted absorption of concrete based on the numerical model: (a) w/b 0.35, and (b) w/b 0.40.

Also, the model may have adequate generalization capability for predicting the absorption behaviour of similar concrete mixtures, within this range of w/b, cured under the regimes implemented herein. In addition, as the material parameters used to construct the numerical simulation were acquired from the MIP test and saturated moisture content (θ_s) results, the predicted moisture stratification from 1 to 360 min can be a valid measure to represent absorption into concrete joints. It can be observed in Figure 7.8 that the absorption up to 5 min was mainly one-directional (i.e. on the top and joint wall surfaces) while afterwards the effect of two-directional absorption became more distinctive.

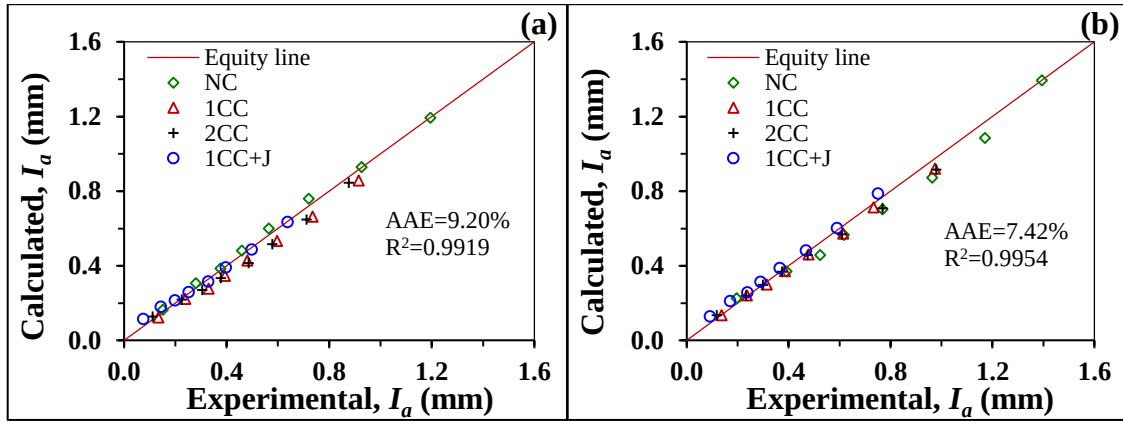


Figure 7.7- Response of numerical model in predicting absorption of cores from 1 up to 360 min: (a) w/b 0.35, and (b) w/b 0.40.

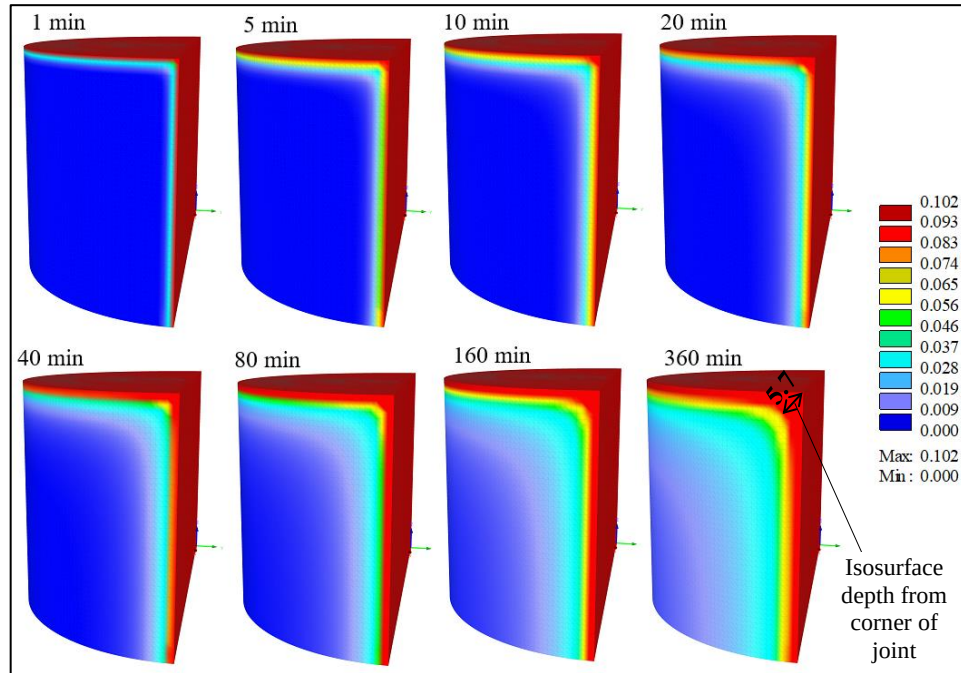


Figure 7.8- Predicted absorption of cores with respect to moisture content (θ) at w/b 0.35 for 1CC+J.

Moreover, the effect of two-directional absorption at w/b of 0.35 (1CC+J) indicated the isosurfaces with 80% of saturated moisture content (θ_s) were at the depth of 2.1, 3.3, and 5.7 mm from corner of the joint at 10, 80, and 360 min, respectively (Figure 7.8). This

effect was also reflected for different curing applications (Figures 7.9 and 7.10). For example, for w/b 0.35 at 360 min, the isosurfaces with 80% of θ_s were at depths of 8.9, 7.0, 7.1, and 5.7 mm from the corner of the joint for NC, 1CC, 2CC, and 1CC+J, respectively (Figure 7.9). Thus, the overall numerical results suggested that the unsaturated flow model reliably simulated fluid transport at joint locations in concrete pavement slabs with accurate predictions relative to experimental results.

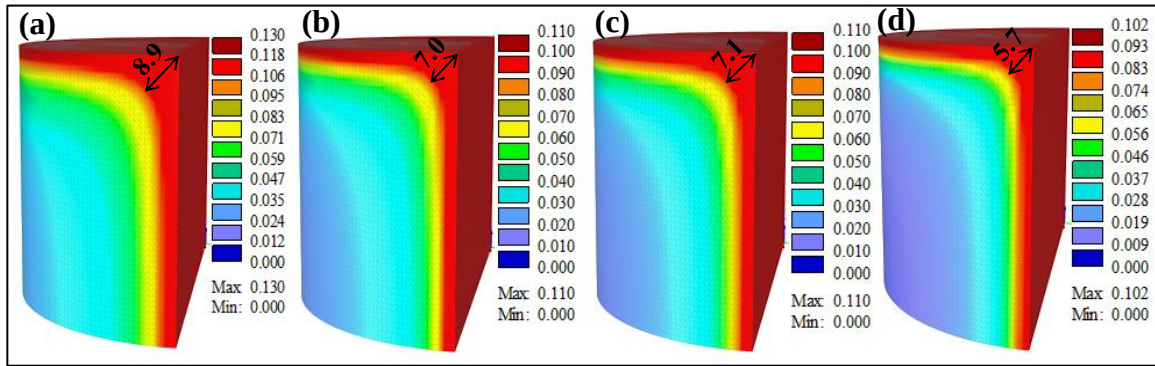


Figure 7.9- Predicted absorption of cores with respect to moisture content (θ) for w/b 0.35 at 360 min: (a) NC, (b) 1CC, (c) 2CC, and (d) 1CC+J. (Note: “ $\leftrightarrow$ ” indicates isosurface depth [mm] with 80% of θ_s)

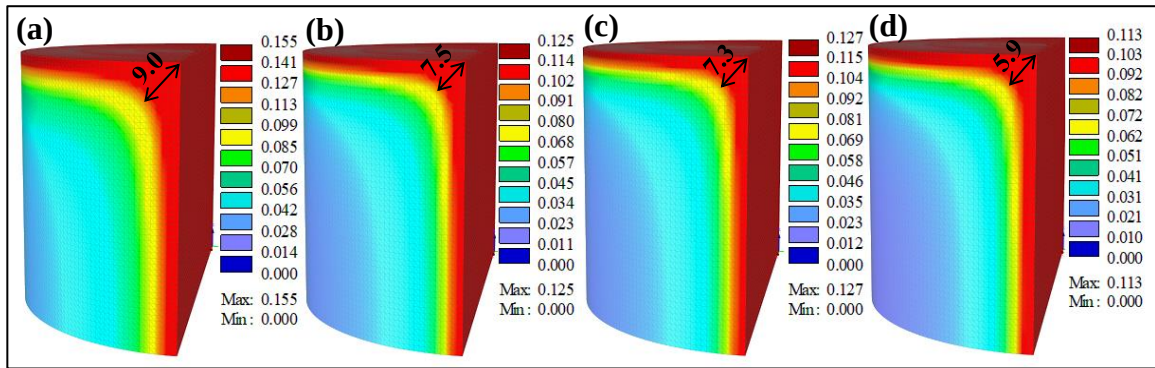


Figure 7.10- Predicted absorption of cores with respect to moisture content (θ) for w/b 0.40 at 360 min: (a) NC, (b) 1CC, (c) 2CC, and (d) 1CC+J. (Note: “ $\leftrightarrow$ ” indicates isosurface depth [mm] with 80% of θ_s)

CHAPTER 8: SUMMARY, CONCLUSIONS AND RECOMMENDATIONS

8.1 Summary

This thesis involved field, experimental and modeling studies in order to evaluate and enhance the effect of different curing compound applications on microstructural development at the joint locations in concrete pavements. The experimental analysis was conducted on cores/specimens through absorption, rapid chloride penetrability, compressive strength, maturity, thermogravimetry, mercury intrusion porosimetry, and scanning electron microscopy tests. Also, the results of absorption were thoroughly evaluated based on statistical analysis such as ANOVA, Tukey's test, Fisher's test, Welch's *t*-test, and *F*-test.

In Chapter 3, the effect of three different curing compound applications (NC, 1CC, 2CC) on the transport properties and microstructural development of concrete pavements were investigated. In Chapter 4, the efficacy of different procedures for evaluating the absorption of concrete was investigated to develop a customized test protocol for determining the absorption capacity of joints in concrete pavements. In Chapter 5, the effect of curing compound application (one coat of curing compound [1CC] and one coat of curing compound plus filling the joint immediately after saw-cutting [1CC+J]) and two different saw-cutting methods, early-entry dry cut (early-cut) and conventional wet cut (late-cut), on the transport properties and microstructural development of concrete pavements were investigated which comprised field sections and laboratory slabs. The study in Chapter 6, was implemented to investigate the optimum frequency needed to determine the dielectric response of real-time sensor embedded in concrete and correlate

setting time and strength/hydration development of concrete mixtures with a range of reactivity. In Chapter 7, the study consisted of experimental investigations and analytical model based on the unsaturated flow theory and Katz-Thompson relationship with 3D finite element software to determine the absorption capacity of joints in concrete pavements according to the absorption test customized to the joint geometry of pavements.

8.2 Conclusions

8.2.1 Application of curing compounds on concrete pavements

- The general trend of the rate of absorption test (ASTM C1585 2013) showed some reduction of the total absorption of concrete with the application of curing compound (especially 2CC) relative to NC due to the improvement in the hydration process near the surface of the concrete.
- For laboratory specimens and field cores obtained from the center of the slab, the curing method had a significant effect on reducing the absorption ratio (COW test), conforming to the general trends of the ASTM C1585 test. The reduction of the absorption capacity of concrete with 1CC and 2CC was due to the retention of moisture within the surface layer of concrete, which led to efficient hydration and microstructural development, with the close performance of concrete with 1CC and 2CC.
- In the COW test, the higher absorption values of the cores at the joints was attributed to increasing the exposed surface area in contact with water and weakened planes from the saw-cutting operations at early- and later-ages. However, the dominant effect of the vertical faces of the cores made it difficult to distinctively reflect the quality of

microstructure at joint locations, since high variability was observed among the results of different curing schemes.

- The RCPT results for laboratory and field concrete showed that specimens with 1CC and 2CC had slightly lower migration coefficients than that of corresponding specimens with NC, but without statistical significance. The low w/b (0.36 to 0.38) of this concrete led to a dense and disconnected microstructure that decreased diffusion of chloride ions.
- The trends of TG, MIP and BSEM were confirmatory to that of the absorption and penetrability tests in the sense that there was better microstructural development of concrete and densification of the ITZ in the increasing order of 1CC and 2CC, relative to corresponding concrete without curing (NC), even though the latter was exposed to intermittent rainfalls.

The overall results of Chapter 3 indicate that applying 2CC is highly efficient at densifying concrete microstructure and reducing its absorption and penetrability, especially if the second coat properly covers joint walls resulting from saw-cutting. In addition, 2CC may be recommended for patch repairs which usually comprise vigorously reactive powders for efficient hydration. Adding 2CC will make it more difficult for curing compound surface to wear out due to attrition by vehicle tires, especially when quick opening to traffic is required. Whereas applying 1CC with visual inspection afterward to cover ‘holidays’ at the surface of concrete pavement may lead to efficient microstructural development, applying 2CC maybe be more desirable from practical and technical perspectives. At joint locations, saw-cutting operations and lack of homogenous curing and drying of vertical surfaces

within the joint seem to critically affect the pore structure characteristics and absorption capacity of concrete. This suggests that joints may benefit from an additional overfilling operation with a curing compound after saw-cutting, which can be applied with the second coat of curing.

8.2.2 A test protocol for evaluating absorption of joints in concrete pavements

- The results from Phase I showed that procedure I (inadequate drying and immersion for one minute in water) with its criterion (absorption ratio) was not adequately reliable to estimate the absorption properties of concrete. The same trend was obtained for procedure II (a modified version of the procedure I) despite the addition of a drying period and extending the test duration to 360 min. In procedures, I & II, a bulk volume of well cured (hydrated) concrete were included in the test specimens, which may mask the true characteristics of the pore structure of the critical faces at joints.
- Procedure III highlighted the importance of epoxy coating to isolate the critical faces and capture their absorption trends and substantiated that the joint geometry (vertical joint walls and top surfaces) in concrete pavements facilitates a higher degree of saturation, which may be linked to the vulnerability of these locations to faster deterioration in the field.
- Procedure IV (adequate drying and vacuuming steps, isolating the critical faces and immersion of specimens in 4% CaCl_2 solution) was the most effective protocol in providing a clear/reliable distinction among concrete with variable qualities. The sensitivity of procedure IV, without the need for modifications, was also verified in Phase II for concrete specimens prepared with a close range of w/b (0.35 and 0.40).

Findings from Phase III further validated the robustness and repeatability of procedure IV on cores from field pavement sections extracted at joints and mid-slabs, as reflected by the high conformity of absorption trends with the physical features of the microstructure of concrete determined by MIP.

- The overall results from both laboratory specimens and field cores in Chapter 4 indicated that procedure IV is robust and reliable in reflecting the microstructural characteristics in concrete pavements, especially at joint locations.

8.2.3 Investigation into enhancing and evaluating curing efficiency of joints in concrete pavements

- For concrete with 1CC, creating and exposing the joint walls using both early- and late-cut methods made them susceptible to uncontrolled evaporation up to 28 days, which led to diminishing any significant differences among their absorption capacities.
- The RH results showed that overfilling the joints with a curing compound provided better moisture retention at these locations, irrespective of the saw-cutting time.
- The general trends of the absorption test showed significant reduction of the absorption rates and capacities of concrete extracted from joints filled with the application of curing compound (1CC+J), especially with the late-cut method due to the improvement in the hydration process at and near the joint walls.
- BSEM micrographs showed that saw-cutting inevitably caused intermittent micro-cracking at joint walls aggregates, which signifies the importance of improving the

microstructural development of surrounding paste (more than 50% of these critical zones).

- The MIP and BSEM trends were confirmatory to that of the absorption test in the sense that there was the better microstructural development of concrete at the joints location by applying curing compound after saw-cutting (1CC+J), relative to the corresponding joints without curing (1CC), especially with the late-cut method. The application of curing compound at the joints led to efficient pore size refinement and densification of the ITZ, which highlights the importance of this procedure (remedial curing of joints after late saw-cutting) for achieving the improved long-term performance of concrete pavements; thus, it is recommended for field practice.

8.2.4 Dielectric response of concrete: hardening and hydration development

- A frequency of 1 kHz was shown to be highly sensitive to variations in hydration development of concrete up to 91 days, exhibiting a power function declining with time.
- The dielectric response of concrete can be used as a sensitive indicator for the efficiency of curing, which is beneficial to monitor field concrete especially in locations exposed to uncontrolled evaporation such as joints.
- It was shown that the hardening threshold of concrete obtained from penetration resistance (laboratory test) coincided with a distinct change in the slope of capacitance response curve of concrete. Each capacitance curve had a knee point at the intersection of a bilinear behaviour, representing the hardening threshold, which can be used for in-situ concrete to guide finishing and saw-cutting activities.

- A strong set of negative logarithmic correlations were established between the capacitance response of concretes with a range of reactivity and their strength/hydration products, signifying the functionality of this dielectric property.
- The trends from Chapter 6 highlights the beneficial use of the dielectric response of concrete to estimate key characteristics of the hydration process. Calibration curves can be prepared for individual concrete mixtures to predict their in-situ strength/hydration development in the field from capacitance measurements with sensitivity to curing efficiency.

8.2.5 Effect of curing methods on quality of concrete joints: experimental and modeling

- Similar to results from Chapter 6, it was substantiated that dielectric response of concrete can be used as a sensitive parameter to indicate curing efficiency when monitoring field concrete especially in critical locations subjected to uncontrolled evaporation (e.g. joints in the concrete pavement).
- Similar to Chapter 5, the trends of absorption test suggested that covering the joint walls with curing compound immediately after saw-cutting prevented uncontrolled evaporation resulting in better microstructural development in the 1CC+J slab section.
- The MIP trends were confirmatory to that of the absorption test in the sense that there was the better microstructural development of concrete at the joints location cured with curing compound after saw-cutting.

- The numerical model has adequate generalization capability for predicting the absorption behaviour of concrete mixtures cured by different regimes. The numerical results showed that the absorption at the joint location was mainly one-directional up to 5 min while afterward the effect of two-directional absorption became more distinctive.
- The predicted stratification of moisture content from 1 up to 360 min simulated the progressive absorption of concrete cured by different methods, and the overall results suggested that the unsaturated flow model reliably simulated fluid transport at joint locations in the concrete pavement with accurate predictions relative to experimental results.

8.3 Recommendations for Future Work

Based on the results obtained from this doctoral research, the following points are recommended for future work;

- The results of both laboratory specimens and field cores indicated that procedure IV (customized absorption test for joints geometry) is robust and reliable in reflecting the microstructural characteristics in concrete pavements, especially at joint locations. Indeed, a round robin test is still needed to standardize this protocol.
- The remedial application of curing compound at the joints after late saw-cutting led to efficient pore size refinement and densification of the ITZ, which highlights the importance of this procedure for achieving durability of concrete pavements. Thus, it

is recommended that transportation agencies standardize this remedial action in the field practice, after performing multiple field trials across Canada.

- As this study highlights the beneficial use of the dielectric response of concrete in estimating the key features of the hydration process, field studies are still needed to further validate these findings.
- Further experimental and analytical studies are needed to investigate and predict stratification of moisture content for later stages of progressive absorption of concrete cured by different methods.

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Appendix A: Detailed statistical analysis results of Chapter 4

Table A.1: Tukey's test results for the absorption of the top part of concrete specimens from procedures II, III, and IV

Time (min)	Procedure II				Procedure III				Procedure IV		
	w/b	Absorption (%)	Difference	q_{cr}	Absorption (%)	Difference	q_{cr}		Absorption (%)	Difference	q_{cr}
1	0.3	0.86	0.86*		0.47	0.75*			0.47	0.61*	
	0.5	1.72	1.54*	0.23	1.22	1.06*	0.60		1.08	0.79*	0.25
	0.6	2.40	0.68*		1.53	0.31			1.26	0.18	
5	0.3	1.38	1.75*		0.84	1.41*			0.83	1.08*	
	0.5	3.13	2.55*	0.48	2.25	1.88*	1.12		1.91	1.51*	0.31
	0.6	3.94	0.81*		2.72	0.47			2.34	0.43*	
10	0.3	1.84	2.24*		1.14	1.99*			1.14	1.50*	
	0.5	4.08	3.11*	0.90	3.13	2.54*	1.58		2.64	2.23*	0.35
	0.6	4.95	0.87		3.68	0.55			3.37	0.73*	
20	0.3	2.16	2.58*		1.33	2.36*			1.32	1.70*	
	0.5	4.74	3.44*	1.13	3.69	3.06*	1.91		3.02	2.75*	0.33
	0.6	5.60	0.86		4.39	0.71			4.07	1.05*	
40	0.3	2.38	2.70*		1.62	2.83*			1.64	2.01*	
	0.5	5.09	3.49*	1.23	4.45	2.72*	2.30		3.65	3.27*	0.28
	0.6	5.88	0.79		5.34	0.88			4.91	1.26*	
80	0.3	2.62	2.78*		1.99	3.41*			1.97	2.47*	
	0.5	5.40	3.46*	1.27	5.41	4.43*	2.81		4.45	3.90*	0.26
	0.6	6.08	0.68		6.42	1.02			5.87	1.42*	
160	0.3	2.83	2.71*		2.36	3.85*			2.30	2.82*	
	0.5	5.55	3.31*	1.28	6.21	4.82*	3.43		5.12	3.88*	0.46
	0.6	6.14	0.60		7.18	0.98			6.18	1.06*	
360	0.3	3.02	2.59*		2.72	3.92*			2.73	2.77*	
	0.5	5.61	3.18*	1.28	6.65	4.64*	3.71		5.50	3.51*	0.45
	0.6	6.20	0.59		7.37	0.72			6.24	0.75*	

*Indicates statistical significance.

Table A.2: Welch's *t*-test results for absorption of the top part of Phase II specimens (w/b of 0.35 and 0.40)

Time (min)	Proc. IV: 6 h vacuum, 4% CaCl ₂			Proc. IV: no vacuum, 4% CaCl ₂			Proc. IV: 6 h vacuum, 20% CaCl ₂		
	<i>t</i>	<i>t_{cr}</i>	<i>p</i> -value	<i>t</i>	<i>t_{cr}</i>	<i>p</i> -value	<i>t</i>	<i>t_{cr}</i>	<i>p</i> -value
1	2.93	4.30	0.080	2.78	3.18	0.078	2.33	3.18	0.096
5	4.10	3.18	0.022*	3.96	3.18	0.040*	1.94	2.78	0.134
10	4.24	2.78	0.029*	4.08	4.30	0.037	0.90	2.78	0.418
20	5.40	4.30	0.007*	10.39	3.18	0.003*	0.63	2.78	0.566
40	5.05	3.18	0.024*	3.51	4.30	0.063	0.55	2.78	0.612
80	5.55	3.18	0.015*	3.46	4.30	0.069	0.15	3.18	0.894
160	5.96	3.18	0.009*	3.78	4.30	0.063	0.05	3.18	0.960
360	5.33	3.18	0.012*	2.94	4.30	0.097	0.25	3.18	0.822

*Indicates statistical significance

Table A.3: Welch's *t*-test for absorption of the top part of field cores and *F*-test results for the associated variances

Time (min)	Welch's <i>t</i> -test			<i>F</i> -test		
	<i>t</i>	<i>t_{cr}</i>	<i>p</i> -value	<i>F</i>	<i>F_{cr}</i>	<i>p</i> -value
1	2.17	3.18	0.129	5.64	19.00	0.301
5	2.56	3.18	0.890	4.79		0.345
10	0.83	4.30	0.490	31.92		0.061*
20	4.02	3.18	0.036*	6.51		0.266
40	4.80	3.18	0.023*	6.10		0.282
80	5.61	3.18	0.017*	6.74		0.258
160	7.18	3.18	0.006*	4.17		0.387
360	8.52	4.30	0.011*	28.99		0.067*

*Indicates statistical significance.

Table A.4: Welch's *t*-test for absorption depth of field cores at 360 min and *F*-test results for the associated variances

Comparison Aspect for Proc. IV (Absorption at 360 min)	w/b	Welch's <i>t</i> -test			<i>F</i> -test		
		<i>t</i>	<i>t_{cr}</i>	<i>p</i> -value	<i>F</i>	<i>F_{cr}</i>	<i>p</i> -value
Top, Joint vs. Bottom, Mid-slab	0.35	20.70	2.78	0.000*	1.75	19.00	0.726
	0.40	15.07	3.18	0.000*	2.71		0.539

*Indicates statistical significance.