UNIVERSITY OF CAPE TOWN

# Critical Review of the South African Concrete Durability Index Tests

## MSc(Eng) Dissertation

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# DECLARATION

I know the meaning of plagiarism and declare that all the work in the document, save for that which is properly acknowledged, is my own. This work has not been submitted previously at any other institution for any degree. This dissertation is in submission of partial requirements for the degree of Master of Science in Engineering at the University of Cape Town.

Zaahir Mukadam

January 2014

# ACKNOWLEDGEMENTS

The completion of a MSc level dissertation is not an easy task, and while I may be the author of this work, it would be impossible to achieve it by my efforts alone. I would like to thank the following people for having helped me through it:

- My supervisor Professor Mark Alexander, for his continuous supervision, challenging questions and willingness to help; and my co-supervisor Associate Professor Hans Beushausen for also providing guidance and feedback. It has been a privilege to work under them.

- The UCT Concrete Materials laboratory staff, for always being there to help out when needed in the laboratory.

- The Concrete Materials and Structural Integrity Research Unit (CoMSIRU) for providing financial support over the last two years.

- Mr Eike von Guerard for his assistance in providing the new chloride conductivity rig that was tested in this work.

- My fellow postgraduate colleagues who provided me with critical feedback for my work as well as the many fun times

Lastly, to my family, especially my mum and sister, who have always been there and supported me in times of ease and hardship.

بِسْمِ اللهِ الرَّحْمنِ الرَّحِيمِ

**“In the Name of God, The Most Gracious, The Most Merciful.”**

# ABSTRACT

The Durability Index (DI) approach is a performance-based approach used in South Africa for specifying the durability of reinforced concrete structures with regard to service life and concrete quality. The approach comprises three DI tests which are used to measure the durability-related transport processes of permeation, absorption and ionic diffusion. These tests are the oxygen permeability index (OPI), water sorptivity index (WSI), and chloride conductivity index (CCI) tests, respectively. Much work has been done over the past two decades in developing the DI tests to the present stage, where they are being, and have been, used in large projects such as the Gauteng Freeway Improvement Project, which made use of the OPI and WSI tests as a basis for checking the quality of the concrete used in the major road upgrade project. Non-compliance with target values can result in rejection of the material or penalties imposed on the concrete supplier or contractor. This highlights the need for the DI tests to be accurate and reliable so that engineers have confidence in their results.

This dissertation presents a critical review of the DI tests. The overall objectives were to critically examine the present DI testing regimes and identify and address areas of concern with the tests in order to make them more reliable. One of the major areas of concern related to specimen preconditioning: the 7-day oven drying regime and specimen saturation techniques have been called into question. Consequently, this work investigated the influence of the drying method and saturation processes on the DI results. The experimental set up involved casting twelve concrete mixes using four binders (CEM I 52.5N, CEM II B-M (L-S) 42.5N, 50/50% CEM I 52.5N /GGBS, 70/30% CEM I 52.5N /FA) and three w/c ratios (0.4, 0.5 and 0.65). Specific aspects tested were the influence on OPI, WSI and CCI results when specimens were dried to constant mass compared to 7 days; the feasibility of the use of an alternative drying regime involving solvent replacement using isopropanol; the saturation condition of test specimens involved in the CCI and WSI tests; and the impact of using a newly designed CCI rig as opposed to the traditional one used in the UCT laboratory.

Statistical analysis of the drying results showed that the oven drying method is adequate in removing 75%-90% of the moisture within a 7-day period, with the remaining moisture only accounting for between 0.5 – 0.8% of the total mass of the specimen. Despite this, extended drying to constant mass, which took up to 17 days, generally has an effect on the DI tests, with the CCI test results being most affected. It was unclear whether this is due to the excess loss of moisture or due to microstructural changes within the concrete. It was recommended that the drying duration should be capped at 7 days, after which specimens should be placed in a desiccator, with a desiccant kept at a maximum 60% relative humidity for up to 1 week until the relevant test is performed. This is on the basis of arresting further moisture changes in the specimens.

The saturation of specimens with NaCl solution showed that, particularly for slag-extended concretes, it was difficult to fully saturate the specimens. This could impact on the CCI test results and highlights the importance of the saturation step in the CCI test. The WSI saturation method was found to be adequate in producing fully saturated specimens.

The newly designed CCI rig consistently produced higher results than the UCT rig. Thus, the design of the rig is important in terms of obtaining consistent results. This prompted an investigation into the use of different materials as a calibration disc. A ceramic disc was shown to have good promise in this regard. A redesign of the CCI test rig was recommended to address issues including the need for the cells to be completely filled with solution and the need for the potential difference to be taken exactly across the specimen faces and to be more user-friendly than the current version.

Non-experimental aspects of the DI tests were also discussed in the dissertation. These included, among others, the reformulation of the OPI calculation method to have a sounder statistical basis and aspects surrounding calibration for the OPI, as well as the importance of maintaining the correct NaCl solution concentration in the CCI.

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# 1. INTRODUCTION

## 1.1. Background

The governing criterion in the design of reinforced concrete structures in the past, and to a large degree in the present, has been the strength requirement. The result of this is that, fortunately, most structures do not fail and collapse due to a lack of strength. The aspect of the durability of the concrete was not afforded as much attention. Consequently, many structures have shown signs of deterioration earlier than expected for their service life, which has resulted in the need for repair and rehabilitation of these structures, often at great cost (Alexander et al., 2008). The cost of repair has led to owners of structures beginning to insist on assurances of a repair free service life for the concrete structures. Awareness of the negative economic impact caused by durability related failures has increased and as a result the last two decades have seen significant strides in the field of concrete and reinforced concrete durability. However, there is still significant development that needs to take place to address all the durability issues with concrete structures.

Concrete has been a construction material of choice for many decades. Many concrete structures dating as far back as the Roman era are still standing today. This attests to the inherent durability of concrete. This is taken for granted by many structural engineers. However, even the newest concrete is not immune to the effects of weathering and corrosion from natural exposure to the environment (Ballim et al., 2009). The use of steel as reinforcement has introduced an issue where corrosion of the steel rebar results in loss of serviceability of the structure. Durability performance can be defined as the ability of a structure to perform its intended function over a specified period of time under the influence of agents anticipated in service (ISO 15686-8:2008). This implies that the durability is related to the material performance and environment and cannot be thought of as being an inherent property of the concrete (Ballim et al., 2009). Thus, using strength as a guarantee of the concrete durability is not acceptable any longer.

Different countries have different approaches in addressing the issue of reinforced concrete durability. In South Africa, the Durability Index (DI) approach has been developed to characterize the potential durability of new concrete. It has gained credit for following a more performance based approach rather than the traditional prescriptive, or ‘recipe’ type approach. The prescriptive approach specifies limiting values for certain material properties (w/c ratio, cement type, cover etc.) depending on environmental conditions and expected service life of the structure without adequately addressing the issue of the concrete cover quality. Such limiting values may be out-dated as they were developed based on past experience and significant changes have occurred with the materials used in modern concrete, such as the use of extenders. Another problem with the prescriptive approach has to do with quality control. While the design may place restrictions on values for certain properties, there is often no way of verifying that the construction practices have adhered to these values since they are difficult to measure.

On the other hand, the performance based approach provides a rational method to design for durability. The desired acceptable performance criteria of the structure are defined and the concrete is specified and designed accordingly to meet these criteria. The DI approach provides tools to help define the performance criteria. It makes use of three tests, namely the Oxygen Permeability Index (OPI) test, the Chloride Conductivity Index (CCI) test and the Water Sorptivity Index (WSI) test, to characterise and quantify different mechanisms of deterioration (permeation, migration and absorption). The results obtained from the OPI and CCI tests can be used as input parameters to service life prediction models which aid the designer to make provision for the durability requirements of a particular structure in particular environmental condition. Designers and contractors can use the approach to help specify the required quality and cover thickness of the concrete for a given environment and binder system (Ballim et al., 2009).

The DI tests have been progressively modified and improved (Grieve et al., 2004; Stanish et al., 2006). However, there is still room for improvement surrounding the test methods, particularly around the aspects of the test variability, preconditioning of test specimens and practical issues concerning performing the tests.

## 1.2. Problem Statement

The South African Durability Index Approach has become fairly well established in South Africa. However, concerns still exist around certain aspects of the test methods, test variability and reliability, and applications of the tests. These aspects need to be adequately identified and addressed if the tests are to be used with confidence in industry.

## 1.3. Research Objectives

This work will critically examine current DI testing regimes in South Africa, namely the OPI, WSI and CCI tests. These have been in use for some time and are intended to be introduced as National Standards. Certain areas of concern with the test methods have been identified. This work will aim to address these issues in order to make the test methods more reliable. This dissertation will:

- Investigate the effect of the preconditioning methods currently used in the DI tests on their results for a range of w/c ratios and binders. Specifically, these include:
    - The oven drying preconditioning method, and a comparison of this with an alternative method involving the use of isopropanol as drying agent

  - The pre-saturation of specimens with NaCl solution before undergoing chloride conductivity testing
  - The saturation time required for the water sorptivity test

- Investigate the effect on chloride conductivity results when different test rigs are used
- Refine the Standard Test Method Statements as appropriate

In general, many areas are investigated for all three DI tests. The goal is to highlight certain areas with the DI tests that may be cause for concern, with the hope that further work can then be carried out to address each issue more rigorously.

### 1.4. Research Significance

Figure 1.1 summarises the South African durability approach in a framework that uses material indexes as a means of determining potential durability and on-site quality control. The key stages in formulating this approach were developing test methods that could characterise a range of concretes, studying in-situ performance, and applying the results to practical construction (Alexander et al., 2006). This dissertation feeds directly into the framework by aiming to address some concerns with the DI tests, as mentioned in the objectives. Ultimately, it is expected that the test methods be the standard tools with which the performance of reinforced concrete can be confidently measured.

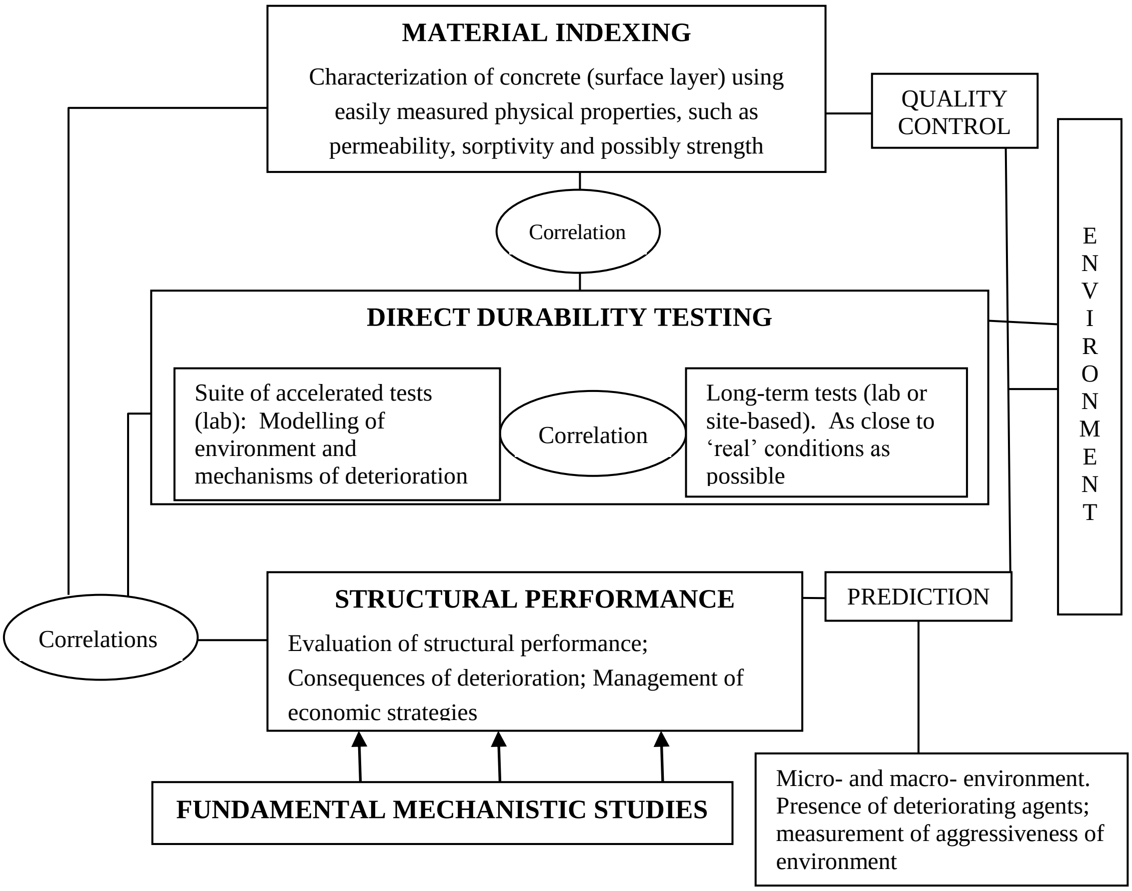

**Figure 1.1: Framework for carrying out durability studies (Alexander et al., 2006)**

## 1.5. Scope and Limitations

- There exist many binder options for concrete in South Africa. Portland cement is becoming less frequently used as a stand-alone binder. It is more common for the inclusion of extenders such as ground granulated blast furnace slag (GGBS) and fly ash (FA) to be used to enhance the concrete properties. The binders used in this work were limited to CEM I 52.5N, a blend of 50/50% CEM I 52.5N/GGBS, 70/30% CEM I 52.5N/FA and CEM II B-M (L-S) 42.5N.
- The w/c ratios were limited to 0.4, 0.5 and 0.65 as this provided a range over which typical structures are likely to be designed.
- The work was limited to laboratory cast and cured concrete cubes. Test specimens from existing concrete structures were not used as it is difficult to control the variables used in site concrete.
- The preconditioning methods investigated in this study were limited to the oven drying method (currently used in the DI tests) and the solvent replacement method using isopropanol. Although there are a variety of options available to remove moisture from concrete specimens, these two were chosen as being the most viable for practical purposes.
- A new chloride conductivity rig has been designed. Part of this study aims to test the results from it compared with those from the current UCT rig. The testing of different equipment to be introduced to the market is usually accompanied by many tests to assess the repeatability and reproducibility of the test. This was not conducted in this work due to constraints on time and other factors associated with conducting full scale inter-laboratory exercises. The work assumes the new chloride conductivity rig tested in this work rig is of acceptable standards to be used. The intended tests were to identify whether generally comparable results were achieved.

## 1.6. Layout of the Dissertation

This work will be laid out in the following manner:

- *Chapter 1* provides an introduction and gives background to the dissertation.
- *Chapter 2* comprises the literature review and provides a theoretical background to the study as well as highlight important issues regarding the Durability Index tests.
- *Chapter 3* describes the experimental methods used in this study. It gives a detailed account of the work that was carried out in the laboratory as well as the statistical tools used to analyse the results.
- *Chapter 4* presents the results and discussion. These are analysed and discussed with the hope of obtaining insights from the experimental work which was performed. A general

discussion follows which provides thoughts on aspects which were not tested in the laboratory on the DI tests.

- *Chapter 5* is made up of the conclusions and recommendations. Conclusions are drawn and recommendations are made with regards to further work that may be beneficial in the field.
- *Appendices* are provided including information about the detailed concrete mix specifications that were used, detailed DI test results and photographs, the Durability Index Manual, statistical tables that were used to calculate critical values in the statistical analysis, and a conceptual design for a possible new chloride conductivity rig.

# 2. LITERATURE REVIEW

## 2.1. Introduction

Durability related performance of structures has been receiving increasing attention in recent times. Durability, as related to a structure, is defined in ISO 13823 (2008) as ‘the capability of a structure or component of the structure to satisfy the design performance requirements over a specified period of time, with planned maintenance, under the influence of the environmental actions in a given area’.

Durability of concrete and reinforced concrete structures is influenced by mechanisms that involve the ingress of ions or molecules from surrounding liquids and gases into and through the material (Richardson, 2002). These mechanisms and their rate are controlled by many variables including the paste microstructure, fracture strength, and environmental factors such as freeze-thaw and temperature changes (Long et al., 2001). The most important factor is that of the ease of ingress of moisture into the concrete, which depends largely on the nature of the paste microstructure (Long et al., 2001). If the microstructure is well characterised, then an idea of the resistance to ingress of deleterious substances can be obtained. However, characterising the physical microstructure is difficult, if not impossible.

Thus, an alternative method which is based on transport processes, or transport mechanisms, within the concrete is used to characterize the concrete durability. There are three main transport mechanisms by which substances can penetrate and move within the concrete. They are the diffusion of ions under a concentration gradient, permeation of a solution under hydrostatic head and absorption of liquids under capillary action (Mikulic et al., 2001). Good correlations have been shown to exist between these transport mechanisms and the deterioration mechanisms in concrete (RILEM, 1995; Gouws, 2003). It therefore makes sense to test and quantify the transport mechanisms as measures of potential durability of the concrete for design and quality control purposes. Thus, permeability, diffusivity, porosity and sorptivity are measured to represent the transport mechanisms through the pore structure of the concrete (Long et al., 2001).

This chapter presents some of the background theory on transport mechanisms and concrete deterioration science. This is followed by a description of some of the test methods that are used to quantify these mechanisms. Focus will be given to the South African Durability Index tests and their issues as these forms the basis of the dissertation.

## 2.2. The Role of the Concrete Pore Structure

Pores in concrete are commonly classified as macropores, capillary pores and gel pores (CEB, 1992). The size of pores vary over several orders of magnitude as described by Ballim (1994), summarising the work carried out by Mindess and Young (1981), shown in Table 2.1.Macropores (large capillary pores in Table 2.1) are visible to the naked eyes and occur due to entrained air voids and entrapped air bubbles. Capillary pores are the residue from original water-filled spaces in the fresh paste. Gel pores form within the cement paste and are on the nano-scale size. Any water trapped in gel pores is tightly bound and requires large amounts of energy to remove. Capillary pores on the other hand hold water which is relatively free and as a result, are critical to durability related transport processes. These pores provide the pathways for movement of fluids. The moisture content of the capillary pores has an influence on the ease and rate at which the fluids can be transported. A representation of this is shown in Figure 2.1.

The nature of the pore structure has an influence on its transport properties. The size and interconnectivity of the pores are significant factors and relate to the penetrability of the concrete, which can be defined as the ease with which liquids and gases may ingress and move through the material. The degree to which the pores are connected typically lies in the region of 20-30% in the case of cement paste (CEB, 1992). For concrete, this will vary throughout the material, especially at the interfacial transition zone which is more porous than with the rest of the paste. The interfacial transition zone (ITZ) is the area between the hydrated cement paste and the aggregate. These areas have properties which are different to the bulk cement paste in part due to the accumulation of bleed water around coarse aggregate particles leading to a higher porosity and lower density in these regions.

**Table 2.1: Classification of pore sizes in hydrated cement pastes (Ballim, 1994)**

| **Pore type** | **Diameter (m $\times 10^{-10}$)** | **Description** | **Paste properties affected** |
|---|---|---|---|
| Capillary | 100000-500 | large capillaries | strength; permeability |
| | 500 – 100 | medium capillaries | strength; permeability; shrinkage at high RH |
| Gel | 100 -25 | small (gel) capillaries | shrinkage to 50% RH. |
| | 25~5 | micropores | shrinkage;<br>creep |
| | <~5 | micropores “interlayer” | shrinkage;<br>creep |

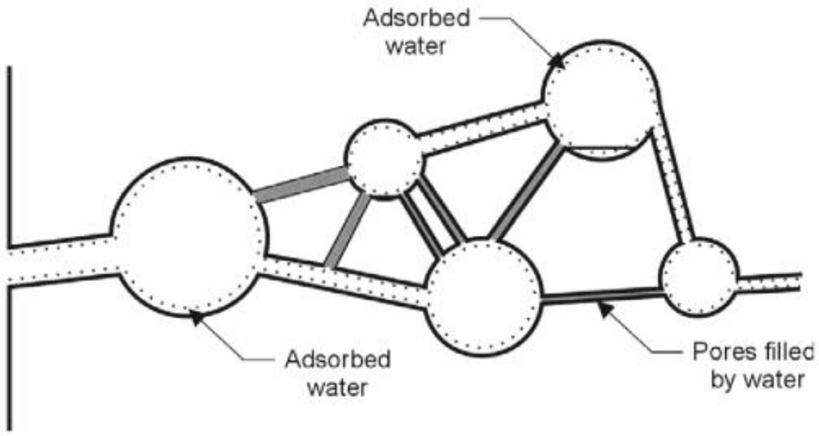

**Figure 2.1: Representation of water in capillary pores in concrete in equilibrium with a nonsaturated atmosphere (Bertolini et al., 2013)**

Several researchers have shown that the porosity of concrete generally decreases with depth into the concrete (Valenta and Modry, 1969; Mamillan, 1973; Kreijger, 1983). The higher porosity of the surface layer of the concrete is important to durability related issues since it is this area that is exposed to the external environment and hence potentially harmful substances. For this reason the cover depth for reinforced concrete is important from a durability point of view.

## 2.3. Transport Mechanisms Relevant to Concrete Durability

While pores provide pathways for the movement of substances through concrete, the movement itself is governed by a number of transport mechanisms. The mechanisms are important for durability predictions since ingress of harmful agents are linked to the ability and ease with which they can move through the porous concrete microstructure. Most deterioration mechanisms in reinforced concrete structures are linked to the movement of fluids into and/or out of the concrete pore structure. The main transport processes involving the passage of potentially deleterious substances through the material are permeation, sorption, diffusion, convection and migration (Richardson, 2002). Bertonlini et al., (2013) summarised the factors leading to the phenomenon on corrosion deterioration of reinforced concrete based on the ease of the transport of deleterious substances. Corrosion of the reinforcement will be discussed later. This is shown in Figure 2.2.

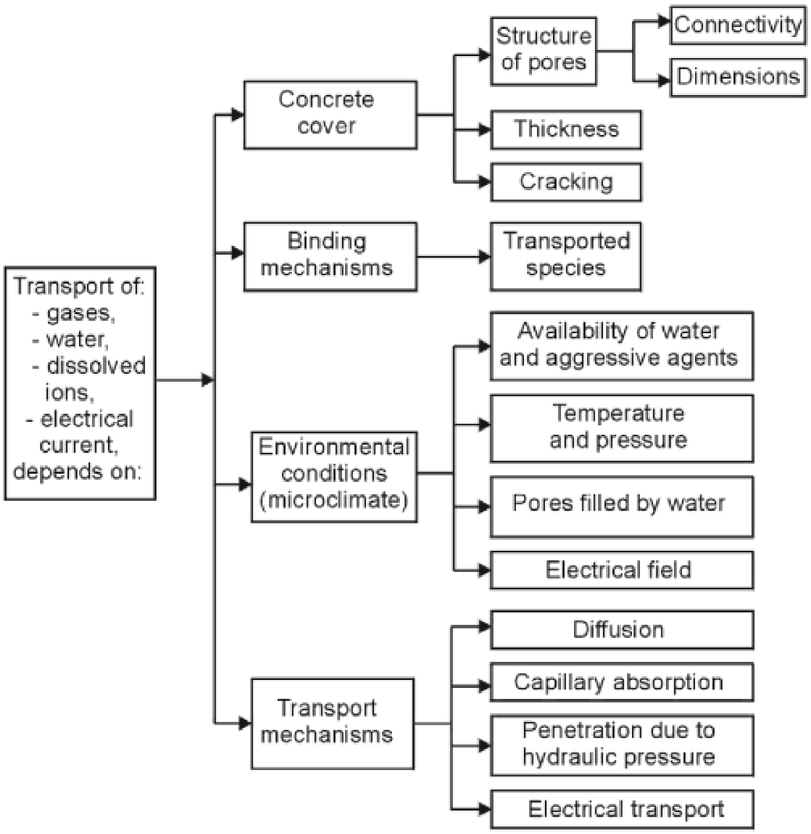

**Figure 2.2: Principal factors involved in the transport processes in concrete (Bertolini et al., 2013)**

### 2.3.1. Permeation

Permeation is the process of movement of fluids through the concrete pore structure under an applied pressure while the pores are saturated with the fluid (Claisse, 2005). It is worth noting that a porous material is often permeable. However porosity and permeability are not synonymous. Unless a porous sample has pores which are interconnected, it will not be permeable. The permeability of the concrete material is dependent on its microstructure, moisture condition and the characteristics of the permeating fluid (Ballim et al., 2009). A dense microstructure where the pores are poorly connected will have a low permeability since the movement of the permeating fluid has a difficult passage through the material.

The movement of the permeating fluid through concrete is considered to be slow and laminar, so permeation can be described using described using D'Arcy's Law. Under steady state conditions D'Arcy's law states that the rate of flow is directly proportional to the hydraulic gradient (Basheer et al., 2001). From a concrete durability perspective, water is the most important liquid that would penetrate hence the equation for incompressible flow is considered, as shown in Equation 2.1.

$$v = \frac{Q}{A} = -K\left(\frac{dh}{dL}\right) \tag{2.1}$$

where: $v$ = mean flow velocity (m/s)
$K$ = coefficient of permeability (m/s)
$dh$ = change in head across specimen (m)
$dL$ = path length (m)
$Q$ = flow rate ($m^3$/s)
$A$ = cross sectional area ($m^2$)

This may be expressed as:

$$\frac{dq}{dt} = -KA\left(\frac{\Delta h}{L}\right) \tag{2.2}$$

where: $\frac{dq}{dt}$ = flow rate ($m^3$/s)
$K$ = coefficient of permeability (m/s)
$L$ = thickness of specimen (m)
$A$ = cross sectional area of sample ($m^2$)
$\Delta h$ = hydraulic head drop across specimen (m)

The above equations are applicable to incompressible fluids such as liquids. For gases, the viscosity as well as the compressibility must be considered. The Hagen-Poiseuille relationship for laminar flow, shown in Equation 2.3, is applicable in this case and can be used to determine coefficient of permeability.

$$K_g = \eta \frac{Ql}{tA} \frac{2P}{\left(p_1^2 - p_2^2\right)} \tag{2.3}$$

where: $K_g$ = coefficient of permeability of the gas ($m^2$)
$\eta$ = viscosity of the gas ($Ns/m^2$)
$Q$ = volume of gas flow ($m^3$)
$l$ = thickness of penetrated section (m)
$A$ = penetrated area ($m^2$)
$p$ = pressure at which Q is measured ($N/m^2$)
$p_1$ = pressure of gas at entry ($N/m^2$)
$p_2$ = pressure of gas at exit ($N/m^2$)
$t$ = time over which gas flow occurs (s)

### 2.3.2. Diffusion

Diffusion is the process by which liquids, gases or ions move through a porous material from an area of high concentration to an area of low concentration. High surface concentrations on the concrete are initially developed through absorption of the diffusant, setting up a concentration gradient. This allows it to move towards the internal part of the material where the concentration is low. Diffusion occurs at a much faster rate in gases than in liquids and solids. Diffusion in solids is the slowest process. Diffusion is dependent on the following (Alexander and Mackechnie, 2001):

- Temperature – diffusion will occur at higher rates at increased temperatures
- Moisture content – gases will diffuse more rapidly through open pores than through pores filled with water. Diffusion in water occurs up to 4-5 orders of magnitude slower than in air (Bertolini et al., 2013)
- Type of diffusant
- Inherent diffusability of the material

Fick’s first law of diffusion is commonly used to model ionic and gaseous diffusion in concrete in the steady state. It states that the rate of transfer of mass through a unit area of a section is proportional to the concentration gradient and a diffusion coefficient. It can be used to describe the diffusion of a liquid or ion in a material of uniform permeability (Richardson, 2002).

$$J = -D\frac{\delta C}{\delta x} \tag{2.4}$$

where: $J$ = mass transport rate (change in concentration/change in time) (g/m$^2$s)
$D$ = effective diffusion coefficient (m$^2$/s)
$C$ = concentration (g/m$^3$)
$x$ = distance (m)
$\frac{\delta C}{\delta x}$ = concentration gradient (g/m$^3$/m)

The negative sign is due to diffusion occurring in the opposite direction to that of increasing concentration. A practical implication for using this relationship for testing of concrete properties is that concretes with low porosity may take a long time to reach steady-state conditions. As a result, typical steady-state tests, such as one shown in Figure 2.3, are usually long term (Bertolini et al., 2013).

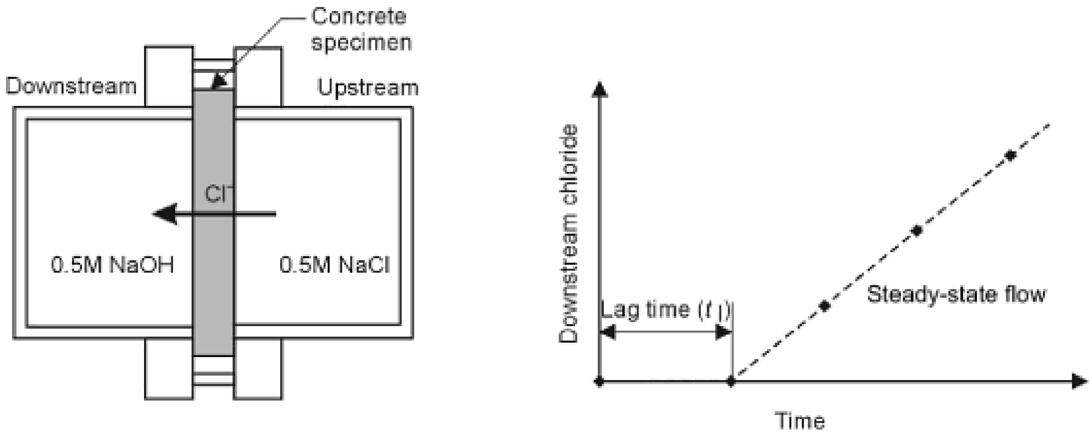

**Figure 2.3: Setup for steady-state diffusion cell and example of typical results (Bertolini et al., 2013)**

It is more common for non-steady state conditions to occur i.e. when the concentration gradient changes over time. Nonsteady-state conditions typically involve a setup shown in Figure 2.4. An example is when ionic diffusion occurs. In such a case diffusion is normally modelled using Fick's second law of diffusion which describes how diffusion causes changes in concentration with time:

$$\frac{\delta C}{\delta t} = D\frac{\delta^2 C}{\delta x^2} \tag{2.5}$$

where: $D$ = diffusion coefficient ($m^2/s$)
$t$ = time parameter (s)

In this case, *D* may be constant or varying depending on factors such as time, temperature, concentration, location etc. (Basheer et al., 2001). If *D* is constant then the solution to the above equation becomes:

$$C_{(x,t)} = C_s = \left(1 - erf\left(\frac{x}{2(Dt)^{1/2}}\right)\right) \tag{2.6}$$

With the boundary conditions

$C_x = 0$ at $t = 0$ and $0<x<\infty$
$C_x = C_s$ at $x = 0$ and $0<t<\infty$

where: $C_x$ = chloride concentration at depth $x$ and time $t$ ($g/m^3$)
$C_s$ = surface chloride concentration ($g/m^3$)
$erf$ = error function

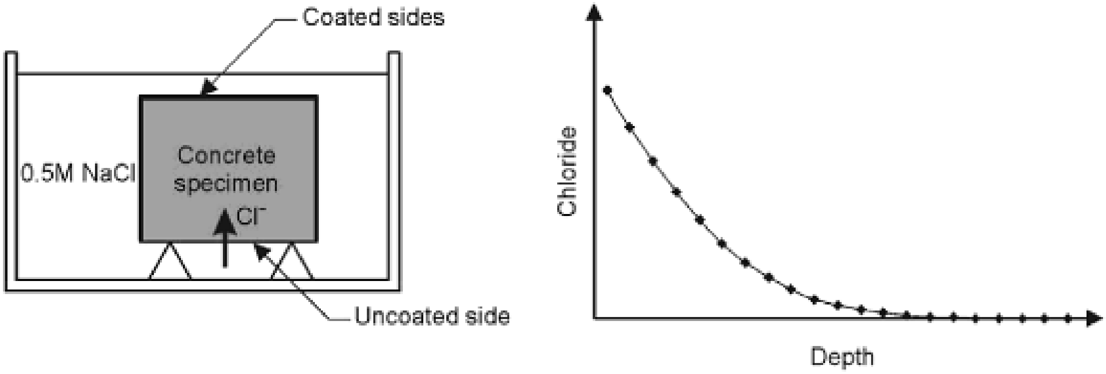

**Figure 2.4: Setup for nonsteady-state diffusion and example of typical results (Bertolini et al., 2013)**

In addition to the diffusion process, there may be chemical reactions taking place between the penetrating substances and the concrete, such as physical and chemical binding of chloride ions to the hydration products in the concrete (Basheer et al., 2001). The above equations do not account for chemical processes and may lead to an error in the estimate of the diffusion coefficient. Figure 2.5 shows the effect of diffusion combined with chemical reactions, such as chloride binding on the chloride concentration (Verbeck, 1987). Diffusion is a dominant process for concrete which occurs in areas high in salts, for example in structures submerged in the sea or salt-contaminated soil (Ballim et al., 2009).

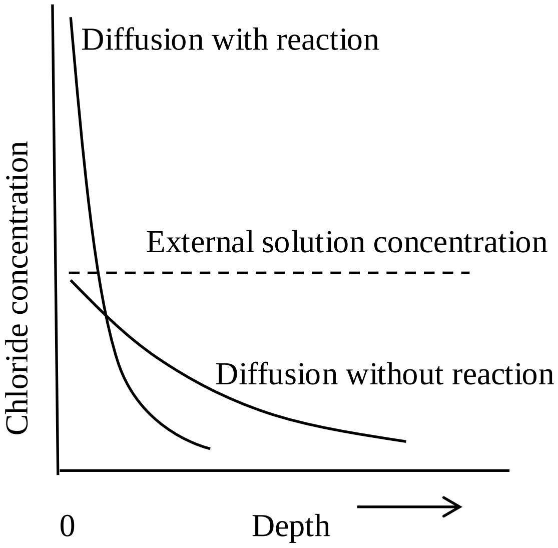

**Figure 2.5: Diffusion with and without chemical reaction (Verbeck, 1987)**

### 2.3.3. Absorption

Absorption is the process by which fluids are taken up into a porous, unsaturated solid under capillary action. This occurs in the absence of an external pressure (Kropp and Alexander, 2007). Dissolved substances such as chlorides and sulphates can enter the concrete as the water is absorbed. Water absorption caused by wetting and drying cycles of the concrete surface is an important transport mechanism near the concrete surface but is less significant as the depth into the structure increases (Ballim et al., 2009). Capillary suction is more prominent in dry or partially saturated concrete than in concrete with a saturated pore structure (Elsener et al., 2000). Water absorption into dry concrete is considered to have two basic parameters, namely porosity and sorptivity.

Sorptivity is the rate at which a water front advances through a porous material. Porosity is also used to characterise the absorption properties and provides a measure of the total volume of pores in concrete that can be filled with water. The fundamental relationship governing the initial absorption of water sorptivity was shown by Hall (1989) to be proportional to the square root of wetting time:

$$A = S\sqrt{t} \tag{2.7}$$

where $A$ = water intake
$S$ = sorptivity
$t$ = elapsed time

The sorptivity can therefore be calculated by taking the slope of the linear part of the $A$ versus $t^{1/2}$ curve.

Since the filling up of pores and the uptake of water via capillary action occur simultaneously, a combined effect of the above two parameters is usually observed. To account for this effect, a modified version of the above relationship which includes an additional term was proposed by Basheer (1991).

$$A = C + S\sqrt{t} \tag{2.8}$$

where $A$ = water intake
$C$ = initial absorption
$S$ = sorptivity
$t$ = elapsed time

### 2.3.4. Migration

Migration is also known as accelerated diffusion, electro-diffusion or conduction. It is the movement of ions under an electric field. Since diffusion tests are time-consuming and impractical, laboratories often use migration to accelerate the diffusion process in accelerated chloride tests (Ballim et al., 2009). Migration is governed by the Nernst-Planck equation (Equation 2.9), which takes into account the movement of ions by combined actions due to a concentration gradient (diffusion), an electric field (migration) and convection. Figure 2.6

illustrates the different reactions occurring when an electric potential is applied to concrete with NaOH and NaCl electrolytes (Andrade, 1993).

$$J = D\frac{dc}{dx} + \frac{zF}{RT}DC\frac{dE}{dx} + CV_e \tag{2.9}$$

where $J$ = mass flux (g/m$^2$s)
$D$ = diffusion coefficient (m$^2$/s)
$C$ = concentration (g/m$^3$)
$z$ = electrical charge
$F$ = Faraday constant (J/V.mol)
$R$ = gas constant (J/mol.K)
$T$ = absolute temperature (K)
$E$ = electrical potential (V)
$V_e$ = velocity of solution (m/s)

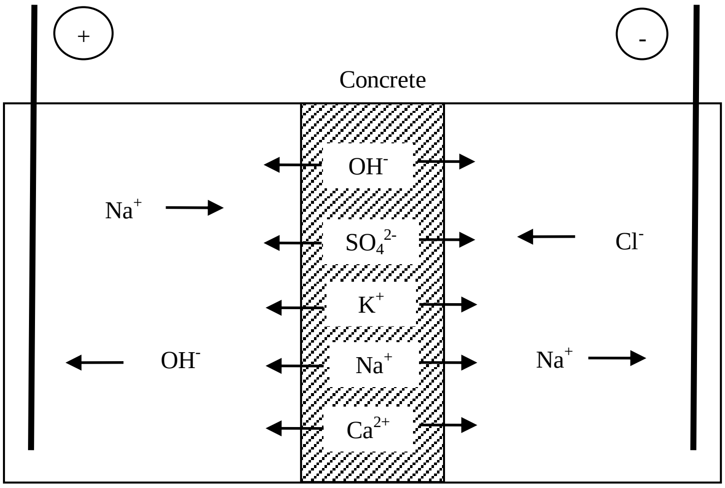

**Figure 2.6: Diffusion and Migration Processes (After Andrade (1993))**

The above relationship assumes ideal conditions with low ionic concentrations and does not take into account interactions with other ions (Streicher, 1997). At high concentrations ion-solvent and ion-ion interactions have an increased influence on the diffusion and conduction processes. When other ions are present, their mobility will also influence the diffusion and conduction processes since the more mobile ions will tend to diffuse faster. Most accelerated tests used to predict the chloride diffusion coefficient, such as the chloride conductivity test, involve setting up an electrochemical cell to accelerate the diffusion of ions.

## 2.4. Factors Affecting Durability

As has been described previously, the various transport mechanisms are used as indicators ease of access of fluid. The combination of these transport mechanisms and the chemical interaction between the ingressed fluid and the hydrated cement paste (HCP) provides an indication of the durability or potential durability of the concrete (Ballim, 1994). The material properties of the concrete have an influence on both of these and consequently will have an influence on the durability. This section will provide an overview of these properties as reviewed by Ballim (1994) and Bauwer (1998).

### 2.4.1. Water/cement Ratio

Permeability increases considerably with an increase in w/c ratio due to the increasing porosity and interconnectivity of the pores associated with increasing w/c ratio (Ballim, 1994)

Figure 2.7 illustrates how the chloride conductivity, linked to the migration transport mechanism, increases with an increase in w/c ratio for a range of binder types wet cured for 28 days. The permeability is also shown to increase as the w/c ratio increases in Figure 2.8 for Portland cement (PC) pastes (Powers et al., 1954).

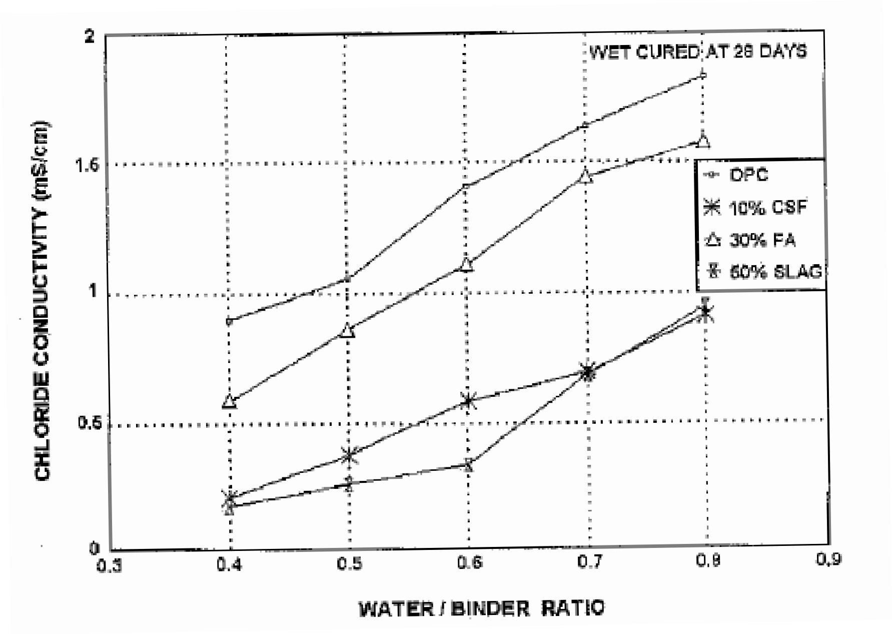

**Figure 2.7: Chloride conductivity versus water/binder ratio (Alexander et al., 1999)**

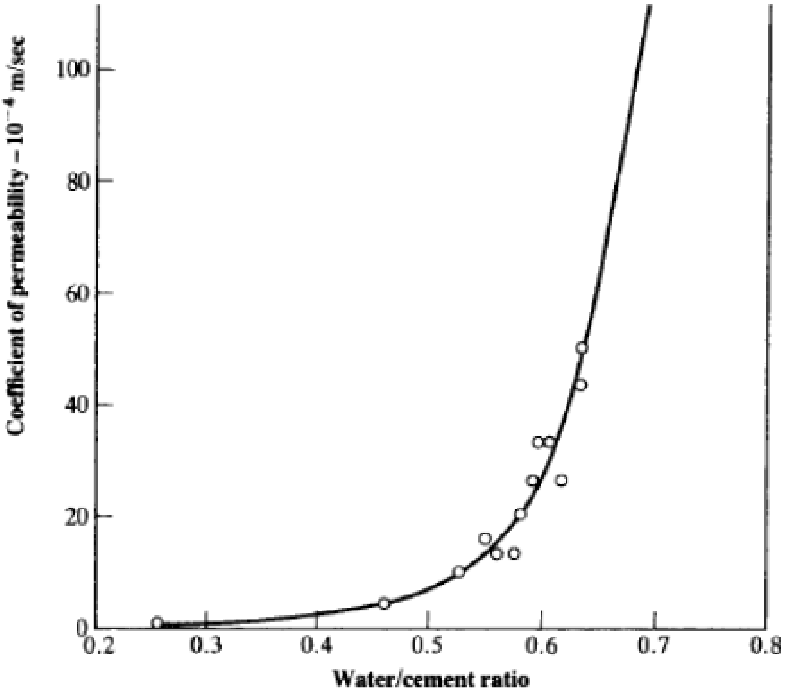

**Figure 2.8: Effect of w/c ratio on permeability for mature cement paste (Powers et al., 1954)**

### 2.4.2. Maximum Aggregate Size

In general, the permeability of concrete increases with an increase in aggregate size (Fulton, 1986). This is likely explained by the effects of the interfacial transition zone (ITZ) between the aggregate and the HCP, which tends to be of higher porosity than the rest of the cement matrix. Fulton (1986) compared the effect of aggregate size on the concrete permeability for a range of w/c ratios and aggregate sizes. Table 2.2 suggests that using smaller aggregate sizes will increase its impermeability. While this is true, practically it becomes uneconomical to do so.

**Table 2.2: Relationship between permeability, w/c ratio and nominal maximum aggregate size (Fulton, 1986)**

| w/c ratio | 0.476 | 0.526 | 0.558 | 0.667 |
|---|---|---|---|---|
| Max. Aggregate Size | Relative Permeability | | | |
| 9.5 mm | 1 | 1.3 | 7 | 30 |
| 13.2 mm | 1 | 2 | 8 | 32 |
| 19.0 mm | 1* | 3 | 12 | 48 |
| 37.5 mm | 2 | 6 | 20 | 66 |

* used as the datum for comparisons

Another point of view has been put forward which contradicts the above. If the permeability of the aggregates is much lower than the permeability of the HCP then the total permeability will be reduced (Alexander et al., 2008). This effect is due to the increase in the tortuosity of the system, making it more difficult for the ions to move freely through the concrete.

### 2.4.3. Water Content

According to Fulton (1986), the permeability of a concrete will be low provided a low enough w/c ratio is used, irrespective of how much water there is in the mix. Ballim, (1994) states that this may only be the case for mixes which are well proportioned. If the water content is increased while keeping w/c ratio constant then a bleeding effect will tend to occur leaving voids around aggregate particles thereby increasing the transport properties in these areas. This highlights the importance of good mix proportioning to achieve the desired properties.

### 2.4.4. Cement Content

The cement content has an effect on the deterioration mechanisms involving chemical interactions between the HCP and the ingressed reagents, such as carbonation and soft water attack. The rate of the change is determined by the amount of hydration products available for reaction. Thus a concrete with a higher cement content than another will show a slower rate of deterioration even if their permeability is the same (Ballim, 1994).

Wasserman et al (2009) showed that the capillary absorption and chloride penetration increased with increases in cement content at 160 kg/m$^3$, 180 kg/m$^3$ and 200 kg/m$^3$. This is shown in Figure 2.9. The rationale is that an increase in cement content increases the total paste volume which is the largest contributor to the total concrete porosity.

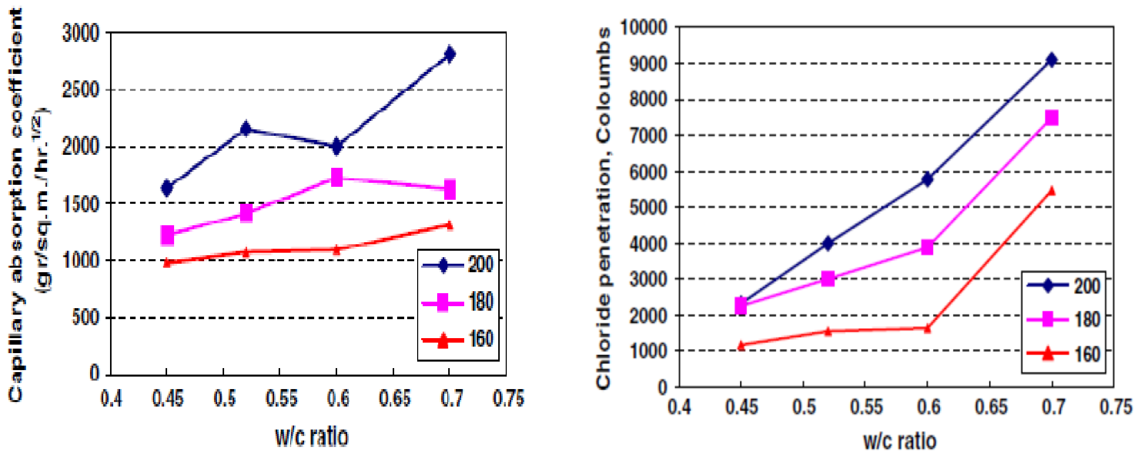

**Figure 2.9: Effect of cement content on absorption (left) and chloride penetration (right) (Wasserman et al., 2009)**

### 2.4.5. Degree of Hydration

Powers et al. (1954) showed that the permeability of concrete significantly reduces as the hydration time increases. It was shown that the water permeability of a cement paste with w/c ratio 0.7 reduces from 2x10$^{-6}$ m/s in the fresh state to 1x10$^{-12}$ m/s after 24 days of hydration (Powers et al, 1954). Only a further 40 % reduction in permeability occurred thereafter. Ballim (1994) suggests that the reason for this is continued hydration after casting has an effect on reducing the fluid transport properties of the concrete.

### 2.4.6. Curing

Du Preez and Alexander (2004) investigated the effect of curing on the oxygen permeability, water sorptivity and chloride conductivity test values for concrete in marine conditions. They showed that there was a noticeable variation between site curing (air curing, compound curing, Burlap cloth and sand curing), and wet curing at 23 °C, with wet curing producing better results. Ballim (1994) summarised Powers (1959) estimation of the duration of moist curing required to achieve discontinuity of the capillaries. Curing is thus a critical component in developing good durability properties due to a well hydrated cement paste. However it is often neglected in practice to save time. Curing especially has an effect on the near-surface properties of the concrete, which is of paramount importance to the protection of the reinforcement steel. Figure 2.10 demonstrates how the permeability is affected by the curing time. The required amount of time required to achieve capillary discontinuity for a range of w/c ratios is shown in Table 2.3. From this it can be seen that using a w/c ratio over 0.60 is not conducive to achieving good durability because it becomes increasingly difficult to achieve discontinuity in the capillary pores.

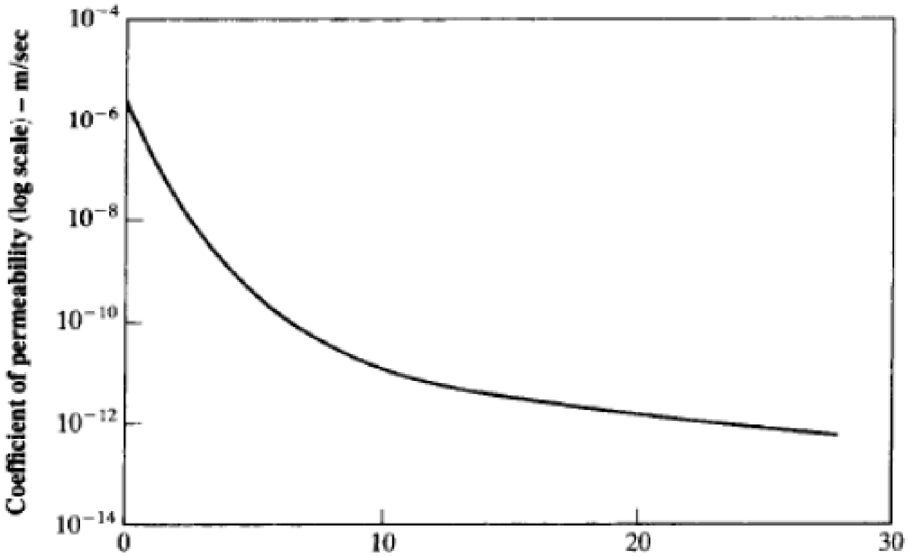

**Figure 2.10: Effect of hydration on concrete permeability (Powers et al., 1959)**

**Table 2.3: Required duration of moist curing for capillary discontinuity [After Powers (1959)]**

| w/c ratio | Degree of hydration (%) | Duration moist curing for capillary discontinuity (days) |
|---|---|---|
| 0.40 | 50 | 3 |
| 0.45 | 60 | 7 |
| 0.50 | 70 | 14 |
| 0.60 | 92 | 180 |
| 0.70 | 100 | 365 |
| >0.70 | 100 | Impossible |

### 2.4.7. Binder Type

The type of binder used has an influence on the microstructure of the HCP. Different binders include plain PC, as well as binders containing a combination of OPC and a supplementary cementitious material such as slag (GGBS), FA and condensed silica fume (CSF). Ballim (1994) showed that the degree, to which a binder is ground i.e. its fineness, has an impact on the concrete permeability. As an example of this refinement effect, Manmohan and Mehta (1981) showed that the addition of FA results in a significant reduction in the porosity of the concrete.

Slag has cementitious properties and can form CSH compounds similar to Portland Cement, but only in the presence of sufficient amounts $Ca(OH)_2$. It is commonly mixed with OPC which provides the required levels of $Ca(OH)_2$ to “activate” the cementitious properties of slag. GGBS is a by-product of the iron and steel-making industry. The advantage of using GGBS lies in its chloride binding capabilities and has shown to have a lower chloride diffusion coefficient than either OPC or FA extended mixes (Ballim, 1994). Du Preez and Alexander, (2004) noticed that in marine conditions, concrete with GGBS and FA extended binders showed similar chloride conductivity values at 120 days. In South Africa, and particularly in the Western Cape, Corex slag (GGCS) has become available as an alternative slag binder. It is different to GGBS in the way that it is produced. A monograph by Alexander et al., (2003) demonstrated a similar improvement in durability performance as that of GGBS. Figure 2.11 shows the chloride conductivity performance of different binders. It can be seen that the binders containing supplementary cementitious materials perform better than the stand-alone PC mixes.

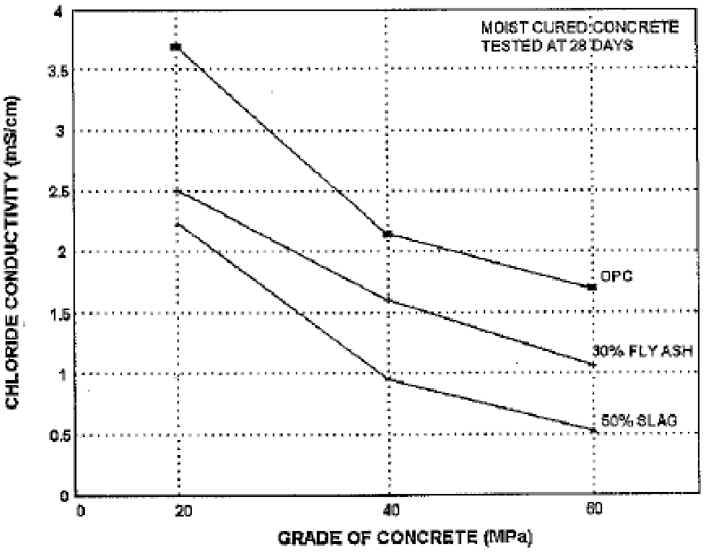

**Figure 2.11: Chloride conductivity results for different binders (Mackechnie, 1996)**

Recently, there has been a new PC binder introduced in the South African market called CEMI 52.5N. It is more finely ground than other Portland cement binders. Angelucci (2013) performed several durability tests using different binders available in the South African market and found that this binder behaved differently than expected. The permeability of CEMI 52.5N binder was relatively insensitive to changes in w/c ratio. The results are shown in Figure 2.12.

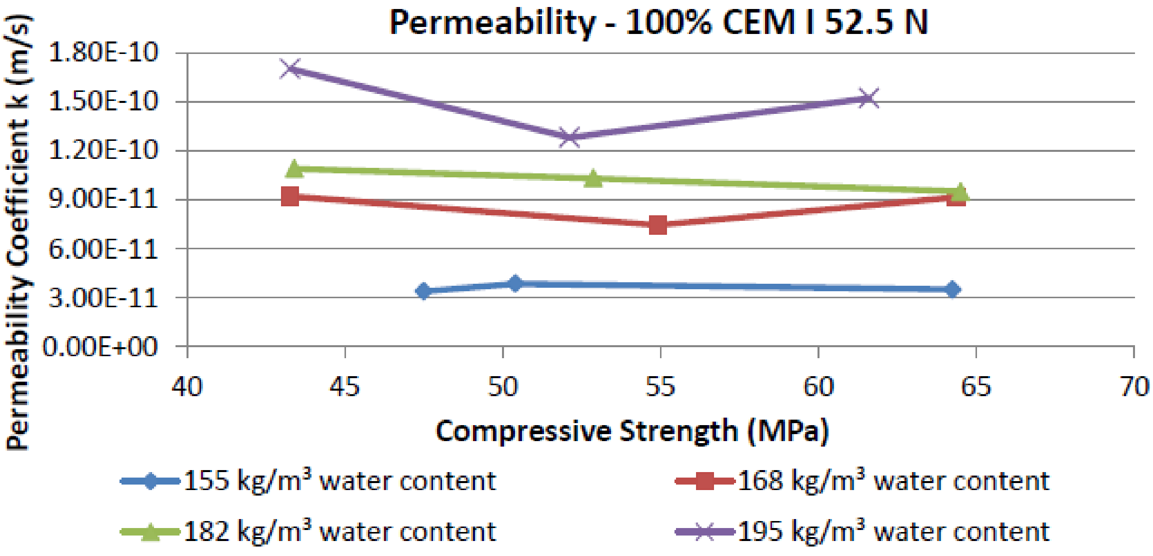

**Figure 2.12: Influence of w/b ratio and paste volume on permeability of CEMI 52.5N concretes (Angelucci, 2013)**

## 2.5. Test Methods Available for Durability Characterisation

### 2.5.1. Gas Permeation Tests

#### Cembureau Permeability Test

The Cembureau method was developed by Kollek (1989). The test comprises of a 50 mm thick and 150 mm diameter specimen fitted into a rubber collar which is placed inside a cell. Samples are preconditioned at 20 °C and 65% relative humidity for a period of 28 days. Alternatively they can be dried at 105 °C for 7 days, although this can lead to significant damage of the concrete microstructure (Romer, 2005). A constant pressure is applied at one end of the specimen and the gas flow is measured. The gas flow depends on the pressure difference, testing area, specimen thickness, and viscosity of the test gas (Beushausen and Alexander, 2008). The Cembureau test has been found to provide reliable and repeatable results (Holmes et al., 2009). The schematic set up for the test is shown in Figure 2.13.

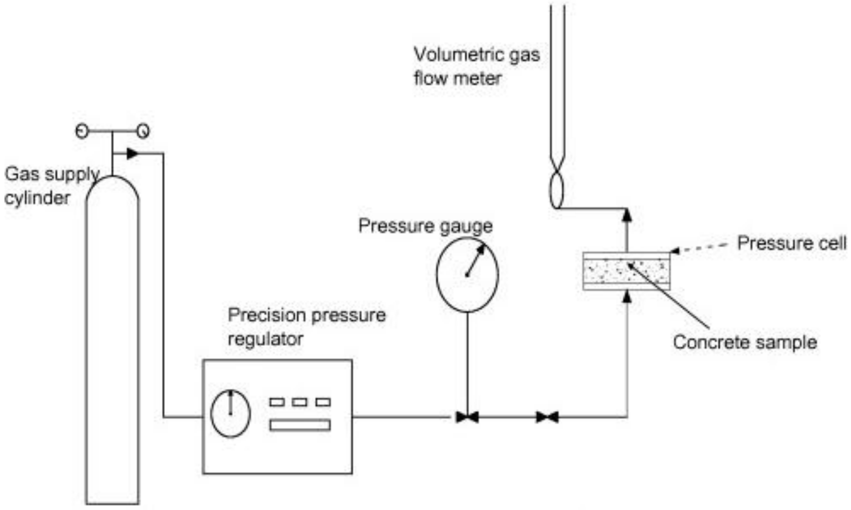

**Figure 2.13: Schematic of the CEMBUREAU permeability test set up**

#### Torrent Permeability Test

The Torrent Permeability Test (TPT) is a Non-Destructive Test (NDT) applied to the surface of the concrete structure (see Figure 2.14). It consists of a two-chamber vacuum cell and a regulator which balances the pressure in the inner chamber with the pressure in the outer chamber, as shown in Figure 2.15. The outer chamber (guard ring) prevents air from the surrounding atmosphere from entering the test setup. The cell is placed on the concrete surface and a vacuum is produced with a pump which forces the outer ring to seal onto the surface. The pressure in the inner chamber increases as the air from the underlying concrete is drawn. The rise in pressure in the inner chamber is recorded as and is directly related to the permeability of the concrete (Torrent et al., 2007). The TPT is usually used in accordance with the Swiss Standard SIA 262/1 (2003).

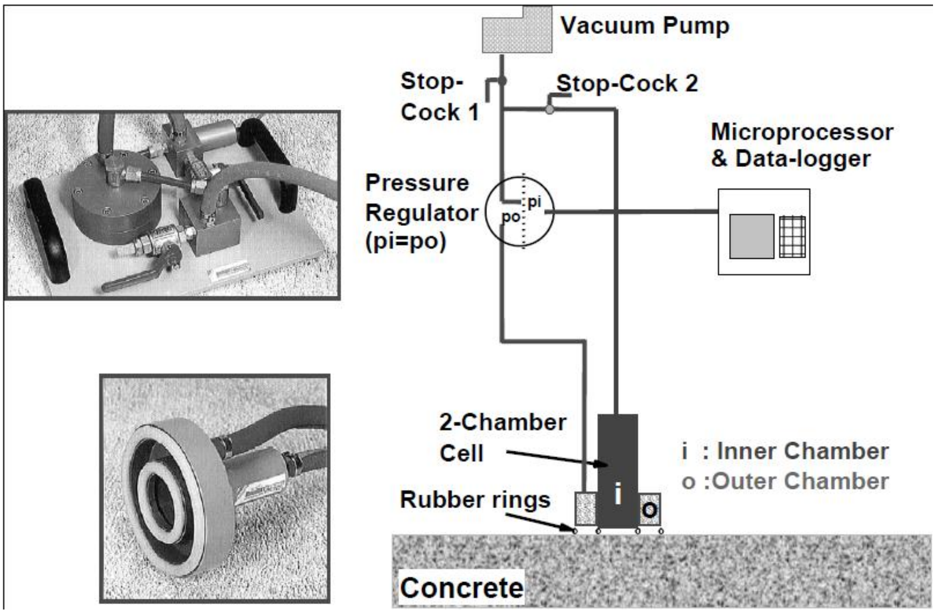

**Figure 2.14: Torrent's Test setup (Torrent et al., 2007)**

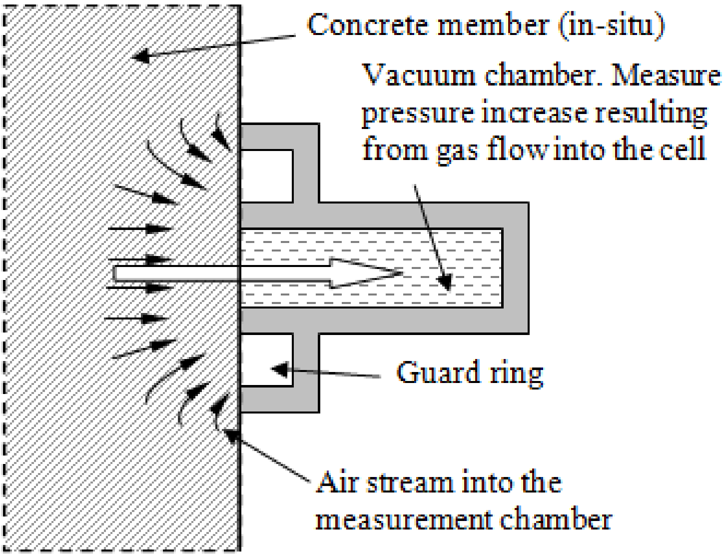

**Figure 2.15: Pressure application for the Torrent Permeability Test (Beushausen and Alexander, 2008)**

## Oxygen Permeability Index Test

The Oxygen Permeability Index test forms part of the three Durability Index tests used in South Africa and will be discussed in Section 2.6, as it is one of the tests focussed on in this dissertation.

### 2.5.2. Chloride Penetration Tests

#### Long Term Tests (Diffusion)

Considered the most fundamental chloride test, the **90-day salt ponding test** (AASHTO T259) involves a combination of absorption, diffusion and wicking mechanisms. Permeability, diffusion and absorption are the important processes in the test. As the name suggests, it is a long term test and as a result is used as a reference test. Slabs, 305 x 305 x 76 mm, are moist cured for 14 days after which they are dried for 28 days in an environment at 50 % relative humidity. A dam of 3% NaCl solution is maintained on the top surface for a period of 90 days. The test set up is shown in Figure 2.16. The chloride ingress is then measured by grinding off 13 mm slices and analysing the chloride content for each slice. Other rapid tests are correlated with this long term test (Pfeifer et al., 1994). A big drawback of this test is the long time it takes to complete. It is more suitable for research purposes than as a quality control test for real projects. This 90 day period however has been shown to be too short to produce significant levels of chloride ingress in high strength concretes (Hamilton et al., 2007).

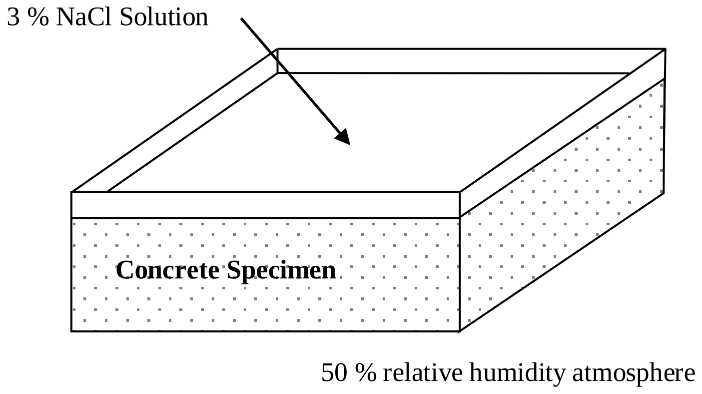

**Figure 2.16: 90-Day Salt Ponding test (AASHTO T259)**

The **Bulk Diffusion Test (BDT)** is similar to the ponding test, but with some improvements. After a 28-day moist curing period, cylindrical specimens are saturated with limewater placed in a solution of NaCl for a minimum of 35 days and up to 90 days for high quality concretes. This is followed by grinding off 0.5 mm layers then analysing the chloride contents of each layer to obtain a chloride profile with depth in the specimen. The layers are much smaller than the 13 mm layers used in the 90-day salt ponding test so the chloride profile obtained has a clearer resolution. Figure 2.17 shows the set up for the BDT.

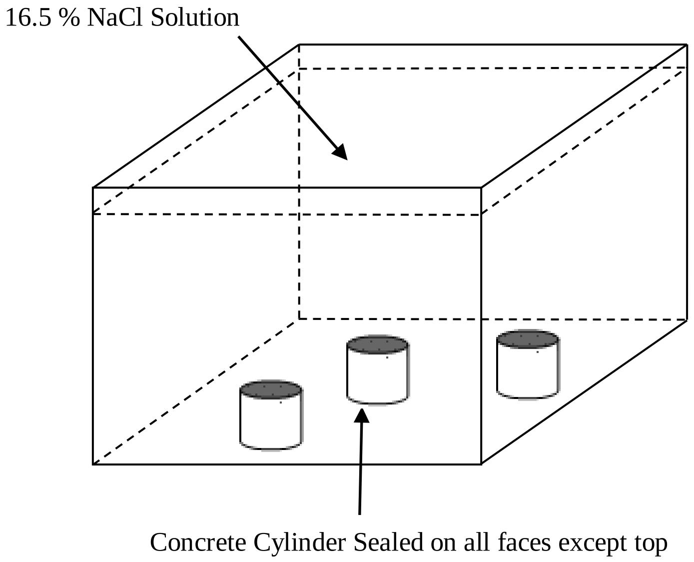

**Figure 2.17: Bulk Diffusion Test (NordTest NTBUILD 443)**

### Accelerated Tests (Migration)

### Rapid Chloride Permeability Test

The rapid chloride permeability test (RCPT, ASTM C 1202) makes use of a 51mm thick x 102 mm diameter sample coated with epoxy on the side. One surface of the face is exposed to sodium chloride solution while the other face is exposed to sodium hydroxide solution. A 60 V DC potential is applied and the current reading is taken in 30 minute intervals for a period of 6 hours. The test provides a measure of the charge passed, measured in coulombs, as a representation of the current through the concrete specimen. The test set up is shown in Figure 2.18.

This method is widely used due to its simplicity and speed. Whiting (1981) showed that there is a reasonable correlation to the more fundamental 90-day salt ponding test. However, Pfeifer et al. (1994) concluded that reliable correlations do not exist between the RCPT and the 90-day chloride ponding test. They show that concrete made with only Portland cement exhibit coulomb values up to fifteen times higher than the same concretes with silica fume added, whereas the corresponding 90-day chloride ponding test is only up to two times higher (Pfeifer et al., 1994). Another study has also found that the general correlation between chloride ingress and coulomb values given in the ASTM C1202 test procedures appears invalid for use with concretes containing silica fume, fly ash and high-range water-reducing admixtures (Shane et al, 1999). In addition, the test has also been criticized for its harsh testing conditions. The applied voltage of 60 V is high and can lead to high temperatures which influence the diffusion process. A further limitation is that the test does not provide a measure of the diffusion process, but rather the electrical conductivity of the concrete.

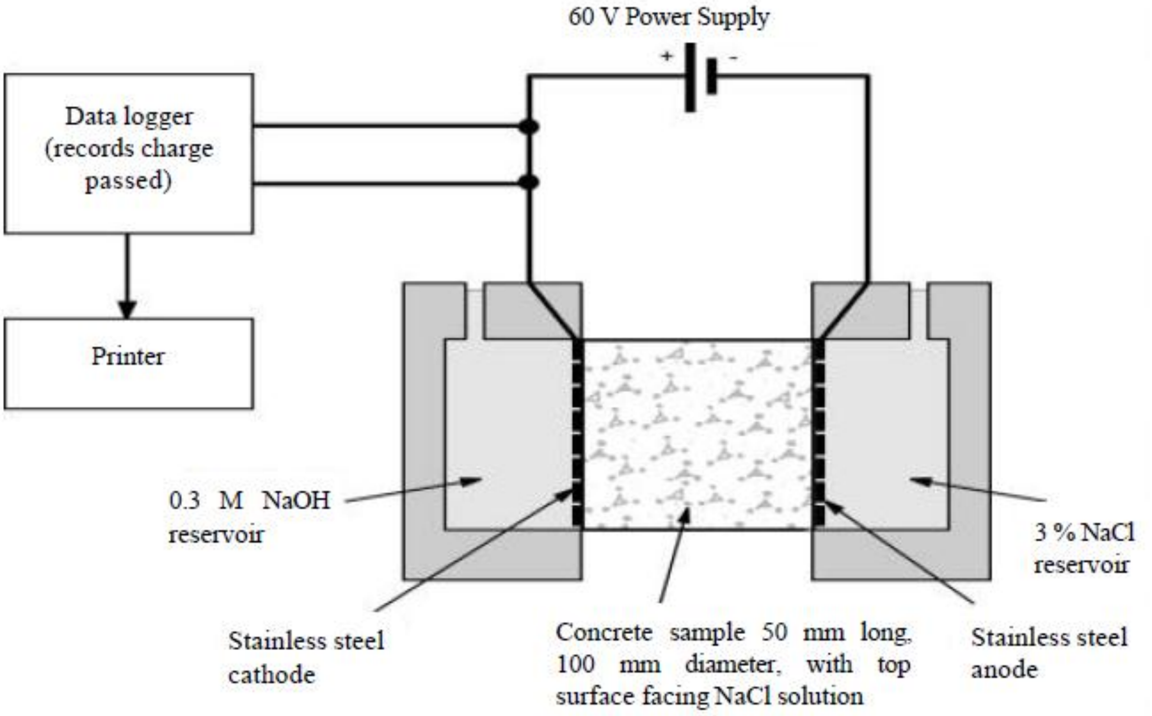

**Figure 2.18: Schematic of test set up for rapid chloride permeability test (Hooton et al, 2001)**

### Rapid Migration Test - Nord Test (NTBuild 492)

The Nord Test method described in the guideline NTBuild492 (Figure 2.19) is another rapid test utilising an applied voltage under non-steady state conditions where a concentration gradient exists. The test was initially developed by Tang and Nilsson (1992) as an alternative to the much criticized RCPT (Tang and Nilsson, 1992). It generally has better correlations with the long-term diffusion tests than the RCPT (Hamilton et al., 2007). The RMT method makes use specimens with a diameter of 100 mm and a thickness of 50 mm obtained from cast cylinders or cores drilled from a larger area. The cylinders are preconditioned by vacuum saturation in limewater and are then placed in a cell with the catholyte and anolyte being 10 % NaCl and 0.3 N NaOH respectively. An external 30 V electrical potential is applied for a period of between 6 and 96 hours, forcing chloride ions to migrate into the specimen from the surrounding solution. After this the specimens are axially split and silver nitrate is sprayed on the inner surface providing a visible white colour corresponding to the penetration depth of the chloride ions. The chloride penetration depth is measured allowing the chloride migration coefficient to be calculated (NTBuild492, 1999).

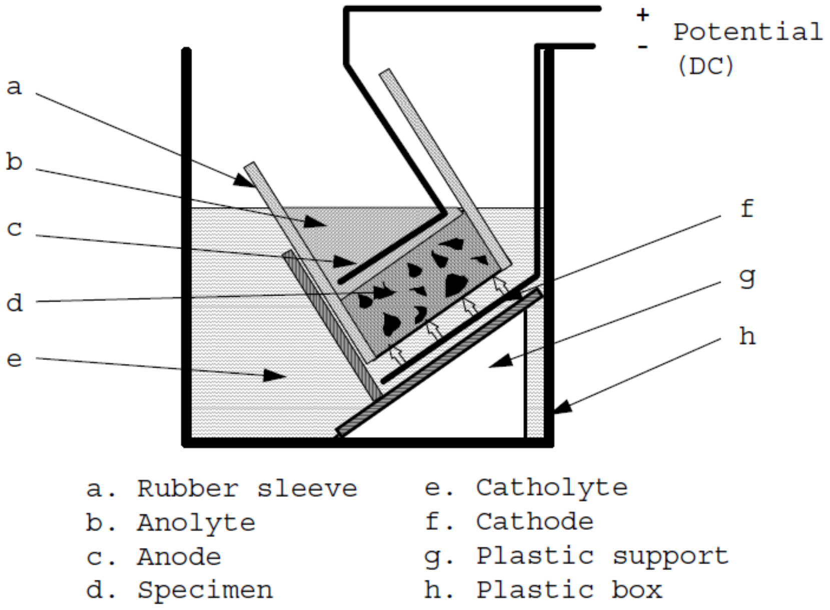

**Figure 2.19: Arrangement of the Nord Test (NTBuild 492, 1999)**

In the test, voltages are adjusted depending on the initial current reading obtained with an applied potential difference of 30 V. This is done so that a reasonable penetration depth can occur within the specimen regardless of its permeability. A specimen with low permeability would otherwise have a penetration depth that is too shallow to be measured, while on the other hand the chloride ions might penetrate right through specimens of high permeability, so a penetration front would not be visible.

Speisz and Brouwers (2012) investigated the influence of the adjustable voltages on testing conditions which may result in different conditions during the test duration for different concrete samples. It was found that the voltages did affect the migration coefficient of the ‘traditional’ theoretical model but not for an alternative model which they propose, which takes into account the non-linear chloride binding isotherm and non-equilibrium conditions between free and bound chloride ions in the concrete (Speisz and Brouwers, 2012).

Although this test was an improvement on the RCPT, it still had other problems especially to do with the extended test duration of up to 4 days in some cases (Hamilton et al., 2007). Hooton et al. (2001) proposed the duration be limited to 18 hours for practicality. The accuracy of the colorimetric technique used has also been called into question as being qualitative and not distinct (Andrade et al. 1999). In the work conducted by Speisz and Brouwers, (2012) it was noticed that a dark discolouration occurred during the splitting phase of the experiments prior to spraying with silver nitrate solution, which matched the chloride profile that was observed after its application. This effect was possibly caused by incomplete liquid saturation or water transport due to the electrical field.

### Chloride Conductivity Test

The Chloride Conductivity Test forms part of the Durability Index tests used in South Africa and will be discussed in detail in Section 2.6 as it forms a large component of the focus of this dissertation.

### 2.5.3. Absorption Tests

Capillary suction tests are generally performed in such a way to remove the effect of a pressure differential. This means exposing bottom face of a specimen to a few millimetres of the liquid under investigation (Kropp and Alexander, 2007). Hall (1989) states the importance of sorptivity measurements as being more representative of the concrete near surface properties than permeation measurements. Most of the following tests measure the uptake of water using similar procedures but varying in the specimen size.

### Autoclam Permeability System

The Autoclam permeability system is a versatile non-destructive test that allows the measurement of both air-permeability as well as water-permeability and water sorptivity. For sorptivity testing, water is introduced to the test area by means of a priming pump with the air being allowed to escape through a bleed pipe (Basheer et al., 2007).Once the water chamber is filled the priming pump switches off and the micropump pressurises the test area to 0.02 bar above atmospheric pressure, at which pressure the transport of water into capillaries is thought to be by absorption as opposed to pressure induced flow (Basheer et al., 2007). The pressure inside the chamber is controlled to be constant as the water is absorbed. The volume of water taken up is recorded over a period of 15 minutes. The volume of water absorbed is plotted against the square root of time to give a line with slope reported as the sorptivity index ($m^3/\sqrt{min}$). The schematic of the test is shown in Figure 2.20.

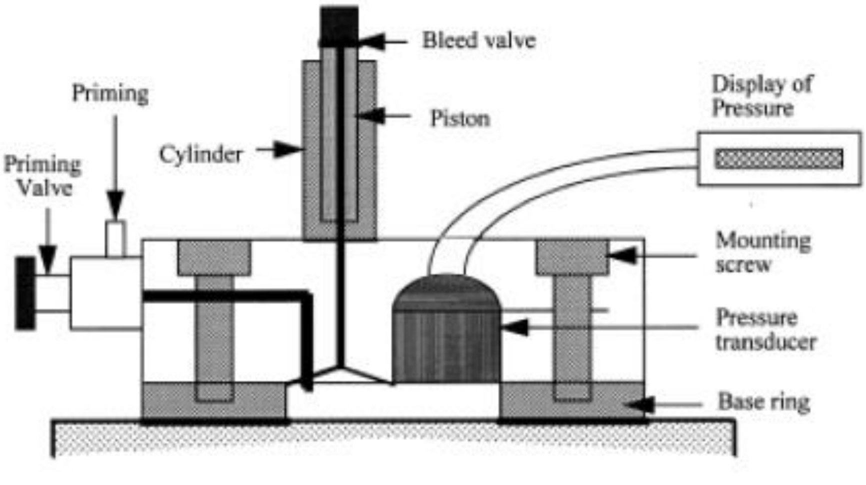

**Figure 2.20: Schematic of the operation of the Autoclam System (Basheer et al., 1991)**

### Kelham Sorptivity Test

In this test developed by Kelham (1988), a circular specimen is sealed on the sides with waterproof adhesive tape and then placed in water. The mass gain of the specimen is recorded until the specimen reaches near saturated conditions observed by small increments of mass gain. The test set up is shown in Figure 2.21.

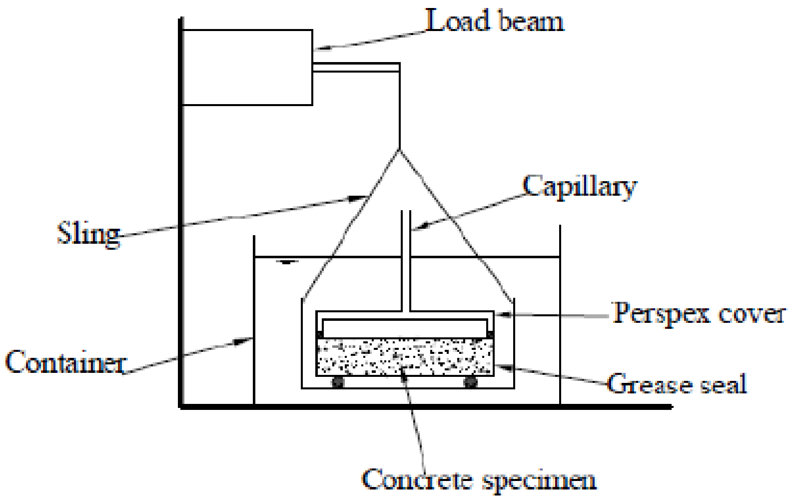

**Figure 2.21: Test set up of the water absorption test used by Kelham (1988)**

### Sabir et al. (1998) Water Sorptivity Test

This test measures the uptake of water and is similar to the Kelham (1988) test except that it only exposes the bottom face of the test specimen to a reservoir of water. The apparatus consists of a rigid copper frame which is attached to a sensor of an electronic balance. The other end of the frame is connected to an aluminium tray containing a 45 mm diameter hole. The specimen is placed on the tray which is suspended on the surface of the water. The water is absorbed by the specimen and the mass gain is automatically monitored and recorded over a period of one hour, after which enough data points are obtained to calculate the sorptivity, which is given as the slope of the graph of absorption versus time. This makes the test easy to perform as the test operator can set up the equipment and obtain readings automatically. A drawback of this test is that only one specimen can be tested at a time. The apparatus used in this test is shown in Figure 2.22.

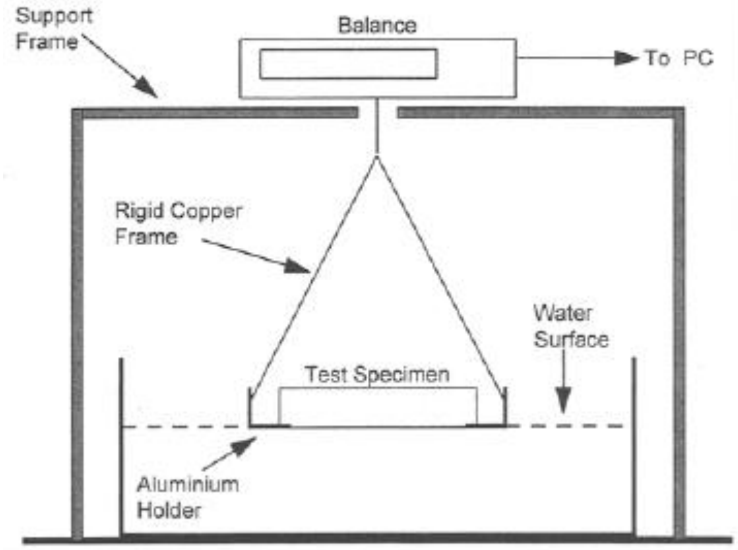

**Figure 2.22: Sorptivity Apparatus used by Sabir et al., (1998)**

### ASTM C 1584-04 Water Absorption Test

The specimens used in this test are 100 mm diameter and 50 mm thick and sealed along the sides. The specimen is placed on rollers or layers of blotting paper and exposed to about 1 - 3 mm of water. The mass gain is recorded over time as with other sorptivity tests. The seven day duration of the test is lengthy compared with other water absorption tests. The test set up is displayed in
Figure 2.23.

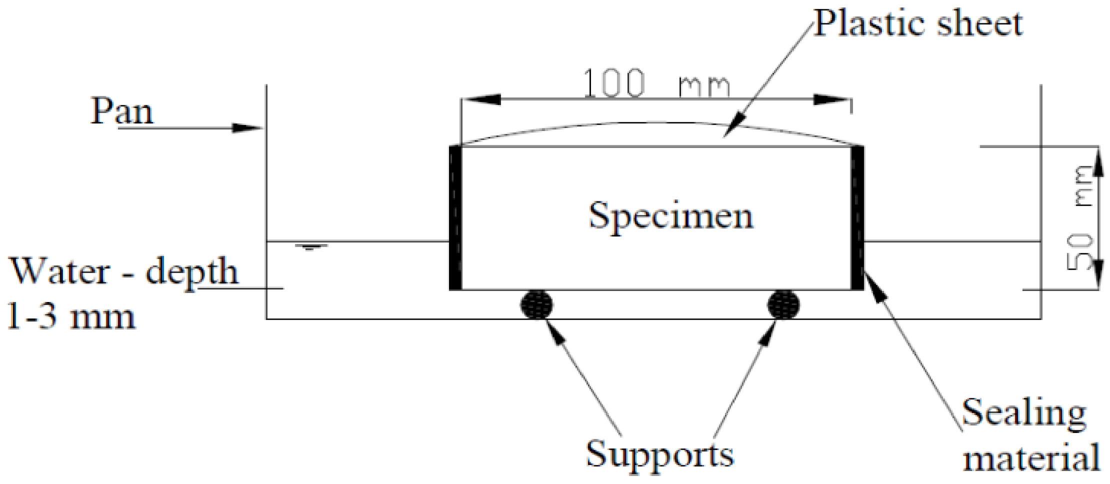

**Figure 2.23: ASTM C1584-04 water absorption test setup**

### Initial Surface Absorption Test (ISAT)

The ISAT is a simple test where a square or circular acrylic cap is fixed to the test surface and filled with water until a pressure head of 200 mm is set up (see Figure 2.24). The water absorption rate is then measured over a period of time by following the rate of retraction of the meniscus in the capillary tube (Basheer et al., 2007). The biggest shortcoming of this test is that the flow is not unidirectional but the water spreads laterally as the wetting front advances. This test is at a disadvantage compared to water sorptivity tests which measure unidirectional flow in the laboratory due to the lateral spreading of the water. In the field the initial moisture state is difficult to control (Hall, 1989). The test is reported as the ISA-Value given by Equation 2.10.

$$ISA = 0.6D / \delta t \qquad (2.10)$$

where: $ISA$ = rate of water suction (ml/m$^2$/s)
$D$ = number of scale units covered by retraction of meniscus
$\delta t$ = time taken for the meniscus to move D scale units of the capillary tube (s)

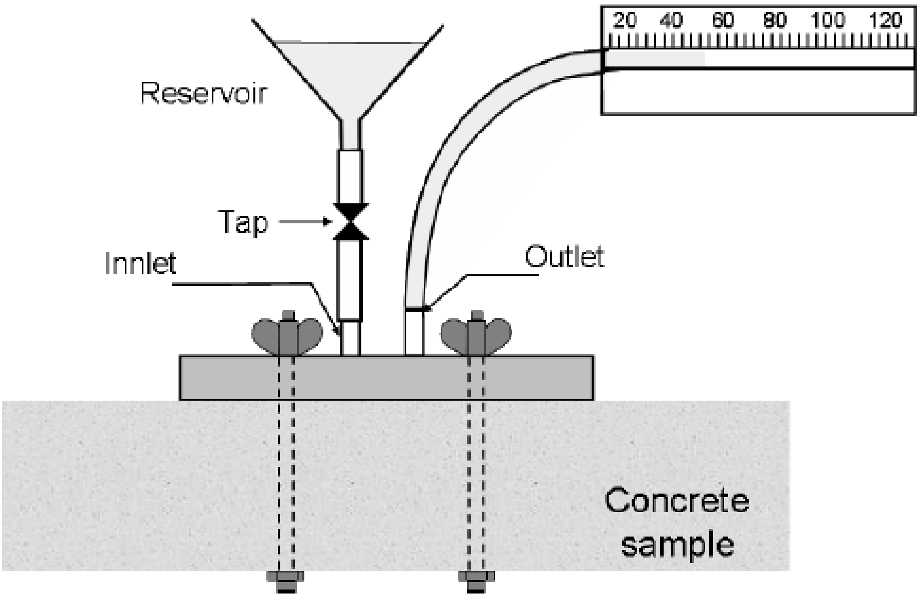

**Figure 2.24: Sketch of ISAT method (Basheer et al., 2007)**

## 2.6. South African Durability Index Test Methods

Researchers in South Africa have focussed on developing a Durability Index Approach which aims to characterize the concrete potential for durability (Alexander and Mackechnie, 2001). The approach makes use of three tests - the Oxygen Permeability Test (OPI), Chloride Conductivity Test (CCI) and the Water Sorptivity test (WSI). These are used to measure the permeability, conductivity and sorptivity of the concrete in the aim that suitable engineering parameters can be obtained to inform design and quality control of concrete construction. Concrete can be tested and checked against recommended values as a quality control (Table 2.4). The three tests are described in the following sections.

**Table 2.4: Suggested ranges for durability classification (in terms of quality of the cover concrete) using index values (Alexander et al., 2001)**

| Durability class | OPI (log scale) | Sorptivity (mm/√h) | Conductivity (mS/cm) |
|---|---|---|---|
| Excellent | >10 | <6 | <0.75 |
| Good | 9.5-10 | 6-10 | 0.75-1.50 |
| Poor | 9.0-9.5 | 10-15 | 1.50-2.50 |
| Very poor | <9.0 | >15 | >2.50 |

SANRAL has since used different specifications with which to classify the in-situ concrete quality based on environmental exposure conditions and cover depth (SANRAL, 2009; Nganga, 2012). The exposure class is an estimate, from which a value of the OPI, WSI and CCI for different concrete binders can be obtained. Table 2.5 provides a summary of the minimum values for compliance.

**Table 2.5: Concrete Durability Specification Targets (Civil Engineering Structures only) (adapted from SANRAL, 2009)**

| Test | Binder | Recommended minimum values<br>*(Based on exposure type and cover depth)* |
|---|---|---|
| OPI (log scale) | All | 9.00-9.60 |
| Sorptivity (mm/hr$^{0.5}$) | All | 10.00 |
| Chloride conductivity (mS/cm) | 70:30 CEMI:FA | 0.65-2.60 |
| | 50:50 CEMI:GGBS | 0.85-2.70 |
| | 50:50 CEMI:GGCS | 1.00-3.40 |
| | 90:10 CEMI:CSF | 0.25-0.65 |

### 2.6.1. Oxygen Permeability Index Test

The OPI test involves measuring the pressure drop across a sample of concrete. A 30 mm thick, 70 mm diameter sample is preconditioned in an oven at 50 °C for seven days to obtain a dry test specimen. It is then placed in a rubber collar fitted into a rigid sleeve and placed in a falling head permeameter.

Oxygen gas is passed through the sample under an initial pressure of 100 ± 5 kPa and is allowed to decay to 50 kPa or up to a period of 6 hours, whichever occurs first. The rate at which the pressure decreases is measured and a permeability coefficient is calculated.

$$\mathrm{k} = \frac{\omega \mathrm{Vgdz}}{\mathrm{RA}\theta} \quad ; \quad \mathrm{z} = \frac{\Sigma[\ln(P_0/P_t)]^2}{\Sigma\,[\ln(P_0/P_t)t]} \qquad (2.11)$$

where: $k$ = coefficient of permeability of test specimen (m/s)
$\omega$ = molecular mass of oxygen (kg/mol)
$g$ = acceleration due to gravity (9.81m/s$^2$)
$z$ = slope of line determined in regression analysis
$R$ = universal gas constant (Nm/K.mol)
$A$ = area of concrete specimen (m$^2$)
$\theta$ = absolute temperature (K)
$t$ = time (s)
$P_0$ = initial pressure at start of test (kPa)
$P$ = pressure at time t (kPa)

An average of four test results for coefficients of oxygen permeability (k) is obtained and an oxygen permeability index (OPI) is calculated as the negative logarithm of the coefficient of oxygen permeability. The purpose of using the negative logarithm is to present the OPI in a simple way.

$$OPI = -log_{10}[\frac{1}{4}(k_1 + k_2 + k_3 + k_4)] \qquad (2.12)$$

This formula makes use of the arithmetic mean of four permeability values. Gouws (2011) has questioned the rationale behind using this format to represent OPI values as it has no scientific basis. There has been question whether to derive the OPI from the mean k-values, or to obtain individual OPI and then take the mean of those ($k_{mean}$) (Gouws, 2011). The results are not equal as shown in Table 2.6.

**Table 2.6: Calculation of OPI (Gouws, 2011)**

| | **OPI** | **k (m/s) × $10^{-10}$** |
|---|---|---|
| | 9.7 | 2.0 |
| | 10.0 | 1.0 |
| | 10.3 | 0.5 |
| **Mean** | **10.0** | **1.17** |
| **OPI derived from $k_{mean}$** | | **9.93 ≠10** |

For tests conducted on samples from the same concrete, treated in the same manner, permeability values are not normally distributed but follow a log-normal distribution (Torrent, 2010; Nganga, 2011). A log-normal distribution is one where data is distributed in a way such that the logarithms of the values follow a normal distribution. Using an arithmetic mean as a measure of central tendency of data which is not normally distributed is incorrect. Since OPI values are taken as logarithms of permeability values, resulting in a normally distributed sample, it may be acceptable to use the arithmetic mean when comparing many OPI values.

There are other methods which provide a more representative measure of central tendency for such distributions, such as the geometric mean or the median (Neves et al., 2012; Neave, 1988). The geometric mean of a sample of population *n* is given by:

$$GM = \sqrt[n]{x_1 x_2 \dots x_n} \tag{2.13}$$

The geometric mean has the drawback that a coefficient of variation cannot be easily calculated when comparing permeability (k) values. As a result, determining the repeatability and reproducibility of comparative tests would prove to be difficult. Navidi (2008) makes use of the following formulae to calculate the mean and variance for data that is log-normally distributed:

$$E(y) = e^{\mu + \frac{\sigma^2}{2}} \tag{2.14}$$

$$V(y) = e^{2\mu + 2\sigma^2} - e^{2\mu + \sigma^2} \tag{2.15}$$

where:

$E$ = mean

$V$ = variance

$m$ = arithmetic mean of log-values

However, applying the above formulae in OPI calculations may be difficult to implement in practice.

The OPI test method is useful in assessing the characteristic concrete structure since it is sensitive to cracks and openings which provide an easy pathway for permeating gas (Alexander and Mackechnie, 2001; Beushausen and Alexander, 2009). There is a good correlation between the OPI values for concretes tested at an age of 28 days and carbonation depths due to natural exposure making OPI a good indicator of carbonation resistance (Mackechnie and Alexander, 2002). The schematic set up for the OPI test is shown in Figure 2.25.

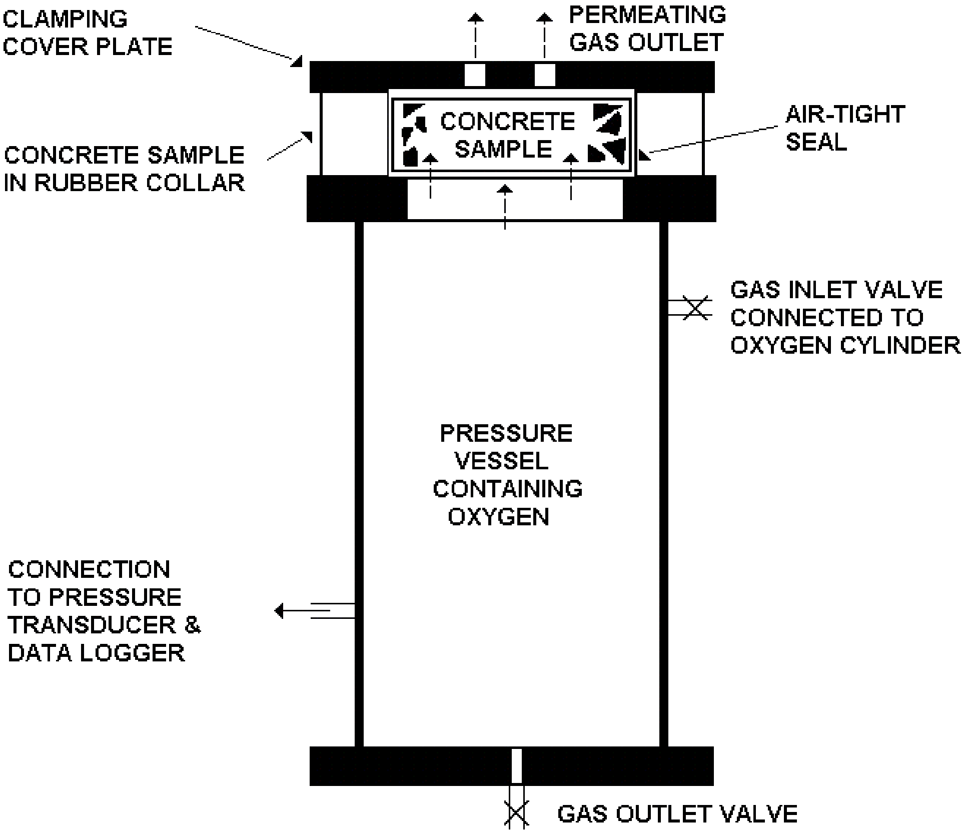

**Figure 2.25: Oxygen permeability index test apparatus (Alexander and Mackechnie, 2001)**

### 2.6.2. Water Sorptivity Index Test

There exist several tests to measure bulk absorption of water as described in Section 2.5.3. The water sorptivity index (WSI) test used as part of the DI tests is a modified version of Kelham's sorptivity test (Kelham, 1988). The test was simplified and compromised so that it could be easy to use without overly sacrificing accuracy.

Specimens are prepared in the same way as they are done for the OPI test. Since they are identical to those used in the OPI test, the same specimens can be used for the water sorptivity test. The sides of the specimens are sealed with epoxy or tape to ensure absorption occurs in one direction only. The initial mass of the sample is determined and then it is placed on a paper towel saturated with calcium hydroxide solution. The test set up is shown in Figure 2.26.

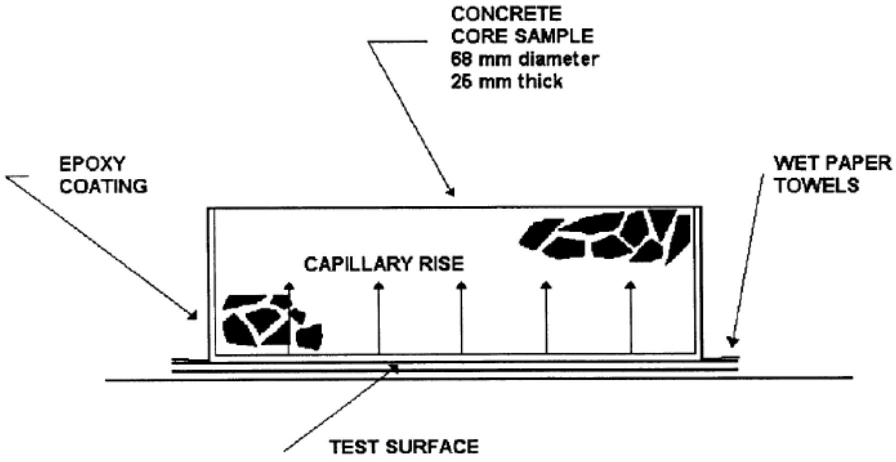

**Figure 2.26: Water sorptivity index (WSI) test**

The concrete samples are placed face down and exposed to a few millimetres of water. The concrete sample is removed from the water and weighed at intervals of 3, 5, 7, 9, 12, 16, 20 and 25 minutes. The mass of water absorbed by the specimen is plotted against the square root of time, with the slope being used to calculate the final sorptivity.

$$\mathrm{M_{wt}} = \mathrm{F}\sqrt{\mathrm{t}} \qquad (2.16)$$

where: $M_{wt}$ = mass gained determined as, ($M_{st}$ – $M_{so}$)

$M_{st}$ = mass at a particular time, $M_{so}$ – mass at initial time

$F$ = slope of line of best fit (g/√hr)

$t$ = time (hours)

$$S = \frac{Fd}{M_{sv} - M_{s0}} \times 100 \qquad (2.17)$$

where: $M_{sv}$ = vacuum saturated mass (g)

$M_{so}$ = mass at start of the test (g)

The sample is then vacuum-saturated in a calcium hydroxide solution for a period of 18 ± 3 hours. This is done to determine the porosity of the concrete and is obtained by comparing the saturated mass with the oven dried mass. Strictly speaking, the porosity is not required for the calculation of sorptivity. However, it is a useful result providing information on the concrete microstructure.

$$\eta = \frac{M_{sv} - M_{s0}}{Ad\rho_w} \times 100 \qquad (2.18)$$

where: $M_{sv}$ = vacuum saturated mass (g)
$M_{so}$ = mass at start of the test (g)
$A$ = cross-sectional area of specimen ($mm^2$)
$d$ = thickness of specimen (mm)
$\rho_w$ = density of water ($10^{-3}$ $g/mm^3$)

Typical sorptivity values vary from 5 $mm/hr^{0.5}$ for well cured Grade 30 – 50 concrete to 15-20 $mm/hr^{0.5}$ for Grade 20 poorly cured concrete (Alexander et al., 2008). The water sorptivity test is particularly sensitive to the cover properties of the concrete and therefore reflects the degree of curing (Alexander et al., 2006). An advantage is that more than one specimen can be tested at a time depending in the test operators capability of measuring the mass of specimens on time.

### 2.6.3. Chloride Conductivity Index Test

Several diffusion tests have been successful at measuring the diffusion coefficient where the concrete is exposed to high concentrations on the one face and low concentrations on the opposite face as shown in Section 2.5.2. However, these rely on the natural diffusion process to take place in normal time. Since the diffusion process is naturally slow, it may take months and even years to obtain meaningful results. Thus, there was a need to develop more rapid forms of testing. Accelerated test methods make use of an applied potential difference to speed up the diffusion process. One such method is the South African chloride conductivity index (CCI) test developed at the University of Cape Town by Streicher (Streicher and Alexander 1995). It was developed as a modified version of the Dundee Test (Dhir et al., 1990), which comprises of a de-ionised water-saturated concrete specimen adjacent to a 5 M NaCl solution and a de-ionised water solution. The increase in chloride concentration downstream of an applied 10 V potential difference is measured at regular intervals and plotted against time. There were problems identified with this test including the following (Streicher, 1997):

- Hydrogen generation would be generated and get trapped under the concrete specimen thereby hindering diffusion.

- Acid generation inside the cell during the test would damage the concrete specimens
- Chlorine gas evolution at the anode would affect the chloride concentration measurements.
- An inappropriate test relationship – the use of Fick's First Law in the case of a rapid test is incorrect.
- Potential losses in the cell – The potential difference across the electrodes was kept constant at 10V, so the potential difference across the specimen would vary depending on the potential losses at the electrode-electrolyte interfaces.

Modifications to the above were proposed and the CCI test was developed (Streicher, 1997). The setup is shown in Figure 2.27. The test apparatus is made up of two cells filled with 5M NaCl. The core test sample is preconditioned by oven drying at 50 °C for seven days, in the same way as for the other two DI tests. It is then followed by a 24 hour vacuum saturation with 5M NaCl solution. This is done to standardise the pore water solution ensuring no concentration gradient is present. A 10 V potential difference is applied to accelerate the movement of chloride ions. An improvement on the Dundee test was to apply a potential difference of 10 V across the actual specimen and not at the electrodes. This meant that a higher potential difference is applied across the electrodes due to potential losses in the electrode and electrode-solution interfaces. The 10 V potential difference across the specimen is ensured by the inclusion of luggin capillaries which are used to measure the potential difference across the specimen.

Since the sample is vacuum-saturated with NaCl solution, there should be no chloride concentration gradient within the specimen. As a result, the movement of ions is only due to the applied potential difference and not due to diffusion processes. It follows that the vacuum saturation process is an important step in the test, as non-uniform saturation will introduce a chloride concentration gradient and allow for diffusion to take place. Furthermore areas within the specimen which are not saturated with NaCl solution are areas which will hinder the migration of chloride ions, since the ions can only move through solution.

The chloride conductivity is determined by measuring the current flowing through the system. While the test is being conducted the voltage is steadily increased until the voltmeter shows a reading of 10 V, at which stage the current reading is recorded. This makes the test appealing as it is particularly rapid.

The conductivity can then be calculated as:

$$\sigma = \frac{\mathrm{it}}{\mathrm{VA}} \tag{2.19}$$

Where: $\sigma$ = chloride conductivity (mS/cm)
$i$ = current (mA)
$V$ = voltage (V)
$t$ = specimen thickness (cm)
$A$ = cross sectional area (cm$^2$)

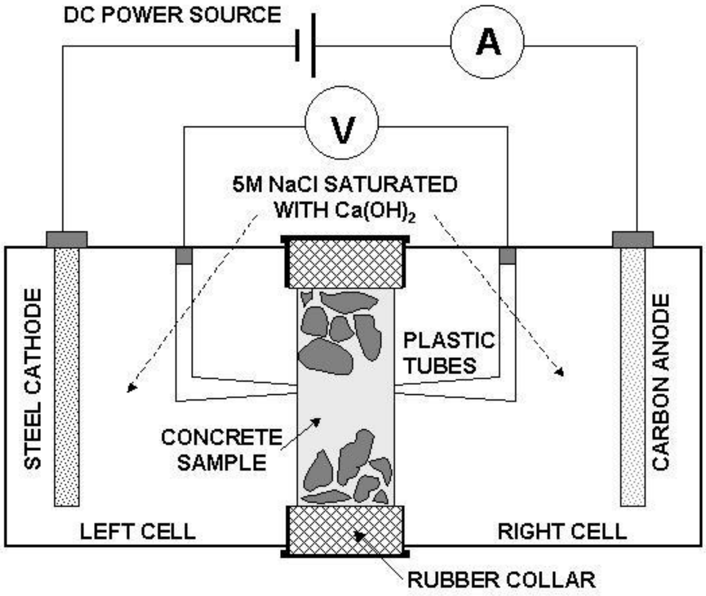

**Figure 2.27: Chloride conductivity test (Streicher and Alexander, 1995)**

More recent test rigs make use of a stainless steel anode instead of a carbon anode. The reason that carbon was used as the anode material was due to its successful use in the Dundee test apparatus (Streicher, 1997). Dhir et al. (1990) reported that metal anodes dissolved and deposited metal oxides on the test specimen surface.

The CCI test has a sound theoretical basis, and has shown to correlate well with long term diffusion tests (see Figure 2.28) (Alexander et al., 199). However there are practical issues that arise when using the test. Stanish et al. (2006) noted that there were problems experienced by test operators mainly due to tight tolerances with the apparatus. The specified diameter of the specimens used with the test apparatus is 68 ± 2 mm. This poses a problem when larger samples need to be placed in the core barrel. Smaller samples also might not seal properly and slip out. An incorrectly sealed specimen will allow solution from one cell to the other and register a higher current reading on application of the potential difference which would not be representative of the specimen. Modifying the test rig to include rubber collars of different internal diameter which would be able to hold different sized specimens was suggested as a practicable solution (Stanish et al., 2006).

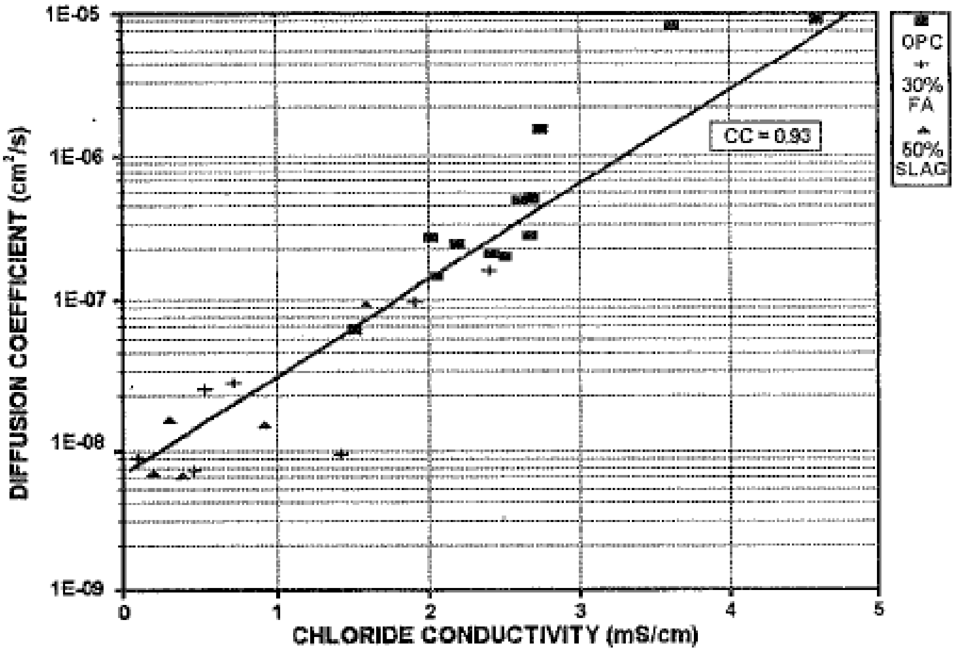

**Figure 2.28: Chloride conductivity versus diffusion coefficient, after two years marine exposure (Alexander et al., 1999)**

Another practical inefficiency stems from the fact that the test takes longer to set up than to actually perform; the test itself is almost instantaneous. Although it is much quicker than other similar accelerated tests for chloride resistance characterisation, when there are many specimen to be tested most of the time is spent on repeatedly having to remove and place the specimen in the central section and re-filling the cells to make sure there is enough solution in them. This leads to a lot of spillage and as a result it is considered to be a ‘messy’ test.

A new version of the CCI apparatus has been designed by Secundum Engineering Services (referred to as the “New Rig” in this study) which aims to address the shortcomings of the previous version. This rig is shown in Figure 2.29. It comprises of stainless steel electrodes as opposed to carbon anode that the UCT version uses. It also makes use of telescopic luggin capillaries which allow the potential difference reading to be taken exactly across the specimen faces, unlike the UCT rig which has luggin capillaries which are fixed in place. The internal cell volumes are the same for both versions of the test rigs so they both hold the same amount of solution. The New Rig has bleed holes for each section of the cell which allow the removal of air. The UCT version does not have these features which theoretically should enable more accurate results to be obtained. However, because the UCT rig is widely used, the New Rig needs to be compared with it before it can be confidently used. This will form part of this study.

Colour-coded cells indicating cathodic (red) and anodic (yellow) sections of the

**Figure 2.29: New chloride conductivity rig**

## 2.7. Variables in DI Tests

### 2.7.1. OPI

Ballim (1994) developed the OPI test method and performed several tests to determine the effect of changing various parameters on the permeability results. Govender (2010) also conducted a study on the OPI test to determine the variables that could influence the test results. The findings are presented below:

- Curing condition and duration – As alluded to earlier in the literature review, the curing time has an effect on the permeability of the specimen (Ballim, 1994). This was picked up by the OPI test. At the time of writing the curing time for laboratory tests is 28 ± 3 days. This allows a range of 6 days within which the test can be conducted. It was suggested that this window period be reduced (Govender, 2010).

- Specimen thickness – Ballim (1994) suggested that specimen thickness should be greater than 15 mm as specimens below this thickness showed significant variation of permeability results. Govender (2010) found that, for thicknesses varying from 28 -32 mm, there was no significant effect on OPI results.
- Specimen preconditioning – the duration of drying in the oven had an effect on some of the OPI results for some mixes. Ballim (1994) found the 50 °C temperature to be mild enough for minimal microstructural damage.

### 2.7.1. WSI

The water sorptivity test is a relatively simple test, with not much reliance on apparatus; a tray to contain the $Ca(OH)_2$ solution and paper towels or roller pins to elevate the specimens are all that is needed. With that, the following variables have been identified to influence the sorptivity:

- Sealing of sides – the sides of the specimen need to be sealed to have the fluid uptake occur from the exposed face only.
- Temperature – the temperature of the specimens is important to obtain repeatable sorptivity results (Alexander and Mackechnie, 1999).
- The water sorptivity test has been shown to be sensitive to changes in cement type and w/c ratio, more so than the OPI test (Ekolu 2012).
- Specimen preconditioning – Ballim (1994) found the WSI test to be less sensitive to changes in drying than the OPI test.

### 2.7.2. CCI

The chloride conductivity test is influenced by many variables which are controlled during the test. The effect of oven drying and the saturation method have been discussed in Section 2.11.2 and Section 2.11.5. When developing the test, Streicher (1997) performed several tests to determine the ruggedness of the test. The following variables were investigated:

- Specimen Thickness – varying the specimen thickness between 20 and 30 mm has a negligible effect on chloride conductivity (Alexander at al., 1999). However if the size of the aggregate is comparable to the thickness of the specimen then the conductivity will be affected due to the effect of the more porous ITZ (Hooton et al., 2001). Figure 2.30 shows the variation of chloride conductivity with specimen thickness for samples vacuum saturated for 5 hours then immersed in chloride solution 1, 3 days and 7 days.

- Curing – OPC mixes are less sensitive to curing duration than mixes containing GGBS and FA
- W/c ratio – The CCI is sensitive to changes in w/c ratio for OPC mixes, but less so for GGBS and FA mixes which had substantially lower conductivity values.
- The nature and concentration of the solution – The type and concentration of the solution in the cell have an effect on the chloride conductivity test. The higher the concentration, the higher the conductivity will be due to more available ions for migration.
- Soaking time – after vacuum saturation, a longer soaking time in the NaCl solution will result in chloride binding occurring with the concrete pore structure thereby reducing the specimen conductivity.

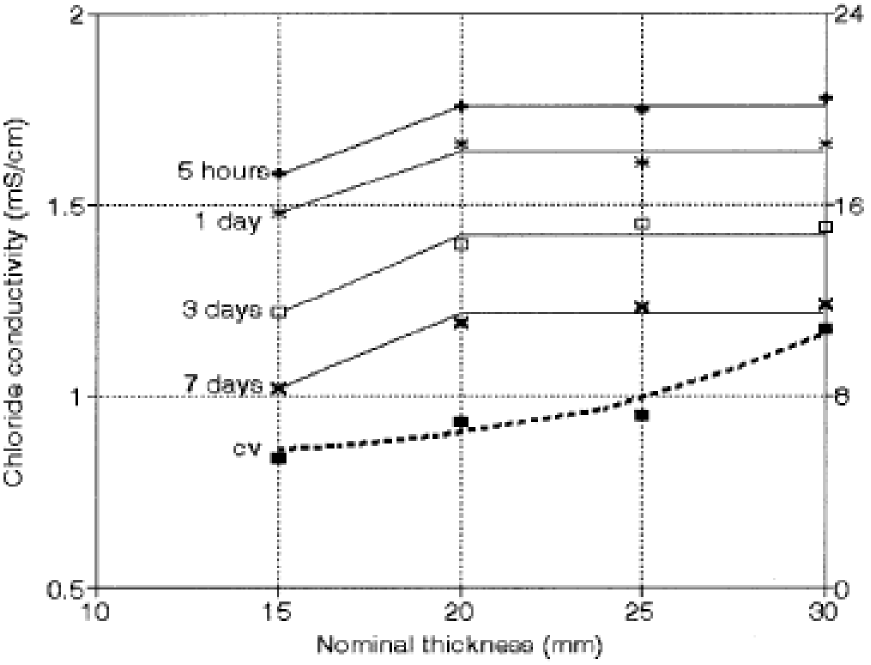

**Figure 2.30: Effect of specimen thickness on conductivity and variability (Streicher and Alexander, 1999)**

## 2.8. Comparisons of the DI tests with International Methods

In order for the South African DI tests to be useful and to facilitate exchange of knowledge, they need to be comparable to the relevant international test methods. With this in mind a series of international tests were carried out to determine the correlations between various test methods (Torrent and Fernandez Luco, 2007). Results obtained with the OPI test method were compared with those from the Cembureau and Torrent Permeability Test. The CCI test was compared with the RCPT as well as the bulk diffusion test. The WSI test was compared with the RILEM (RILEM TC116-PCD 1999) test method. The RILEM Test method is similar to the WSI method except in the way that results are analysed.

It was found that there was a very good correlation between the different gas permeability tests as well as the chloride conduction tests as shown in Figure 2.31-Figure 2.32. Results were not available for the comparative tests involving sorptivity measurements because the WSI test did not differentiate between certain concrete mixes. This was unusual and went against previous experience with the WSI test. It was thought to have occurred as a result of incorrect vacuum-saturation of specimens (Beushausen and Alexander, 2008). As a result, correlations could not be made between the WSI and the RILEM test method.

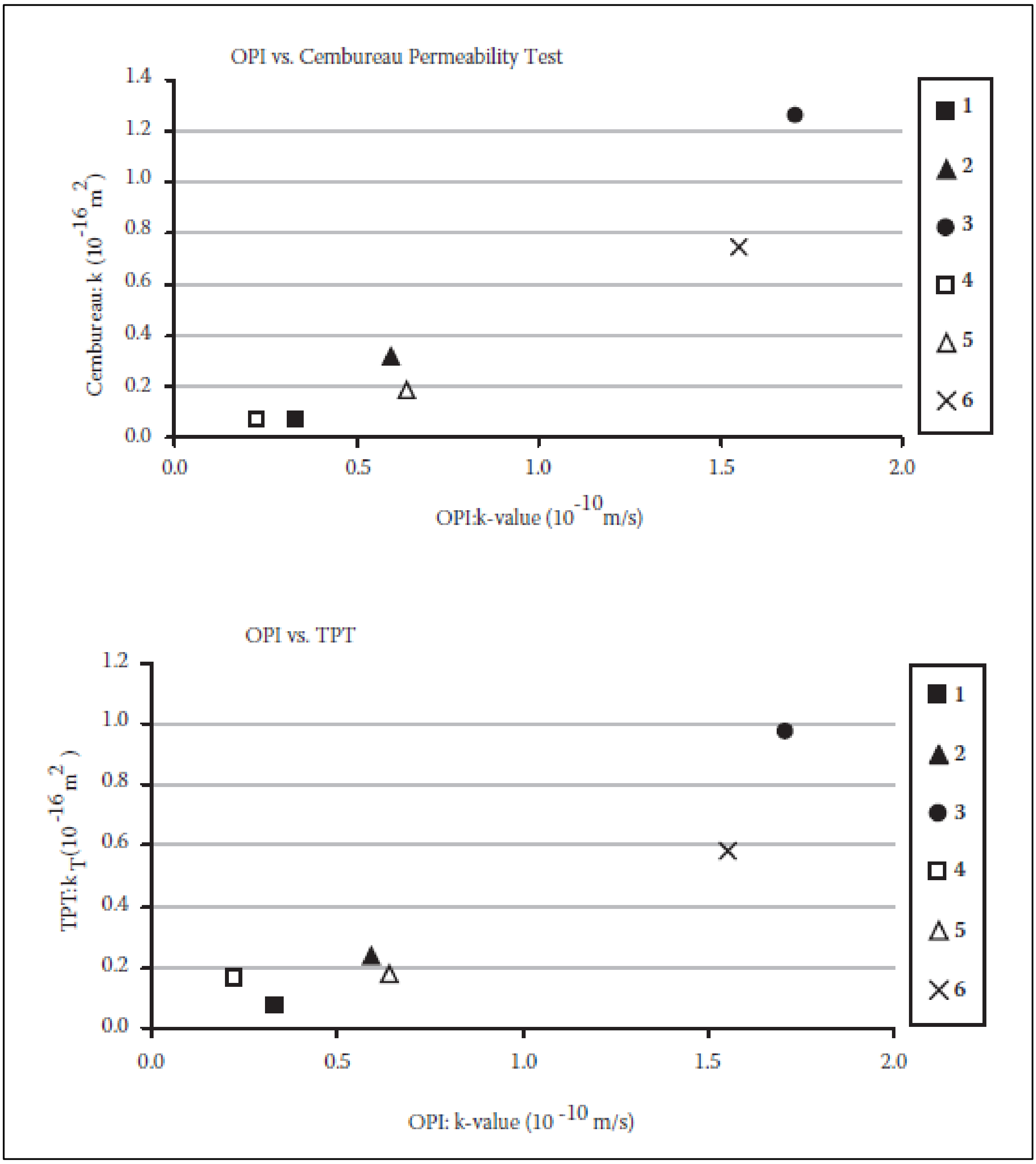

**Figure 2.31: Correlation between the OPI and Cembureau (top) and OPI and TPT (bottom) for six concrete mixes (After Beushausen and Alexander, 2008)**

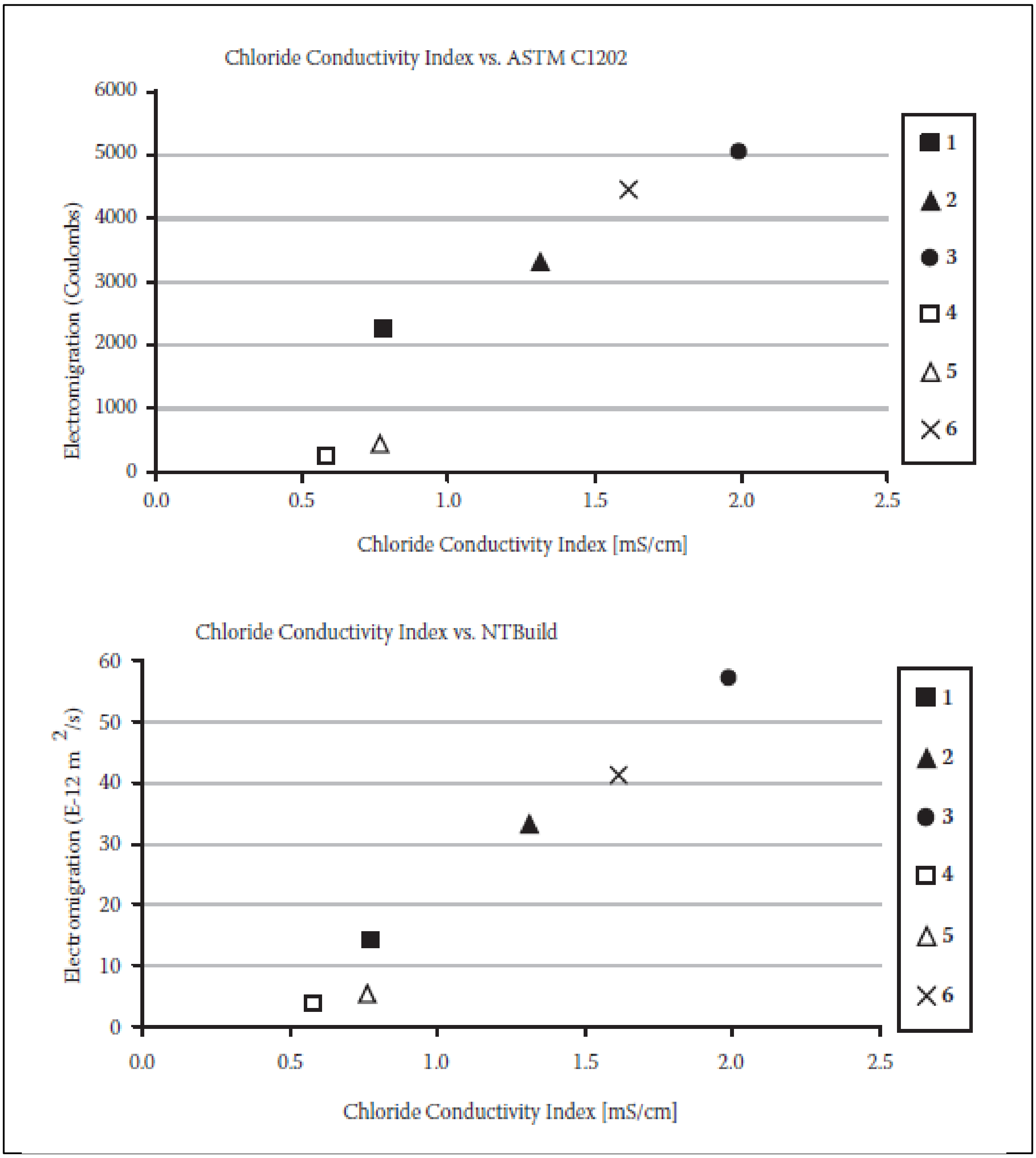

**Figure 2.32: Correlation between the CCI and ASTM C1202 (top), and CCI and NTBuild (bottom) for six concrete mixes (After Beushausen and Alexander, 2008)**

## 2.9. Repeatability and Reproducibility of DI Tests

Repeatability is associated with the variability of results obtained from repeated measurements on the same item by the same person each time. Reproducibility is the variability of results obtained by different people testing the same item. While the South African DI tests show favourable correlations with international test methods, the DI tests themselves need to be robust and practical if they are to be successfully implemented in industry. With this in mind, a series of round robin tests on the DI test methods were conducted between various laboratories in the South Africa. After each round, the test methods were refined to be more accurate or simpler to use. The results of the initial round robin test were summarised by Grieve et al (2003).

Following the initial round, the test methods were rewritten in more detail to reduce the possibility of misinterpretation. The updated tests underwent a second round of inter-laboratory testing but this round again was deemed to be unsuccessful at producing adequate levels of repeatability and reproducibility of results, especially with regards to the sorptivity and chloride conductivity tests (Grieve, 2004). Once more, the methods were reviewed and rewritten and a third round of testing was carried out. This time the OPI and the WSI tests proved to be less problematic than the CCI test. The results are presented below in Table 2.7.

It was concluded that the OPI and WSI tests showed acceptable levels but the CCI test was still problematic, particularly with the reproducibility of results (Stanish et al., 2006).

**Table 2.7: Test Repeatability and Reproducibility (Stanish et al., 2006)**

| | OPI | k | Sorptivity | Sorptivity-Porosity | Chloride Conductivity | C.C. - Porosity |
|---|---|---|---|---|---|---|
| Repeatability | 1.4 % | 32.2 % | 9.9 % | 5.5 % | 9.1 % | 5.5 % |
| Reproducibility | 1.8 % | 36.6 % | 12.8 % | 6.4 % | 21.1 % | 8.9 % |

The OPI test is taken as a log of permeability, and so will tend to dilute the effect of variability in actual permeability (k-values). The variability of the k-values was expectedly higher due to the inherent sensitivity to any defects in the test specimens. This will be discussed further in the next section.

Problems experienced with the chloride conductivity test included tight tolerances with the apparatus. The specified tolerance with the test apparatus is 68 ± 2 mm. This poses a problem when larger samples need to be placed in the core barrel, while smaller samples might not seal properly. Core barrels used by some laboratories were 68 ± 0. 5 mm while others were 70 ± 0.5 mm in diameter, thus there was a mismatch between the size of the cores able to be obtained and the ones able to be used in the test. The 2010 UCT DI manual specifies that the specimens be 70 ± 2 mm in diameter. It was recommended that the chloride conductivity test apparatus be redesigned (Stanish et al., 2006). Modifying the test rig to include rubber collars of different internal diameter which would be able to hold different sized specimens was suggested as a practicable solution (Stanish et al., 2006). A newer version of the CCI apparatus has been designed and is to be commercially available. However, the repeatability and reproducibility of this version has not been verified.

## 2.10. Variability of OPI Results

Of the three DI tests the OPI test appears to be the most robust, showing good correlations to international methods as well as being repeatable and reproducible and posing few problems with its practical use (Beushausen and Alexander, 2008). However, there are still a few areas which need to be addressed in order to improve the test further. The main issue derives from the high variability in test results.

As mentioned previously, variability of actual OPI results is low (1.4% repeatability and 1.8% reproducibility). However, the variability of the permeability (k-value) was seen to be around 32.2% and 36.6% repeatability and reproducibility respectively (Stanish et al, 2006). OPI results are given as the negative logarithm in order to make it simpler to use in practice. This means that small differences in OPI correspond to permeability values which may be orders of magnitude apart. This high variability is illustrated in Figure 2.33. An audit was carried out in 2011 and the conclusion was that the test was being performed to a satisfactory standard (Raath, 2011). However, there are still issues remaining around the issue variability, particularly that introduced by the drying preconditioning. The OPI test has been sent to the South African Bureau of Standards (SABS) to be published as a National Standard, but this was put on hold until the issue of variability can be addressed (Gouws, 2011).

The relatively high variability in results is generally accepted as being due to the inherent flaws within concrete which affects permeability. This is supported by Stanish et al. (2006), where the high variability was experienced by multiple laboratories testing the same concrete. It is not fully certain that the inherent flaws within the concrete is the only source of variability and it is not in part down to variability introduced by the testing apparatus and methods. This means that the outliers generally have to be examined carefully before deciding to not include them since valid test results might be discarded. Gouws (2011) believes that a source of the variability issue lies in the way that the coefficient of variability is calculated for the OPI values. She suggests that the correct way to do it is to obtain the mean and standard deviation of the coefficient of permeability and use those values to determine the coefficient of variability (Gouws, 2011).

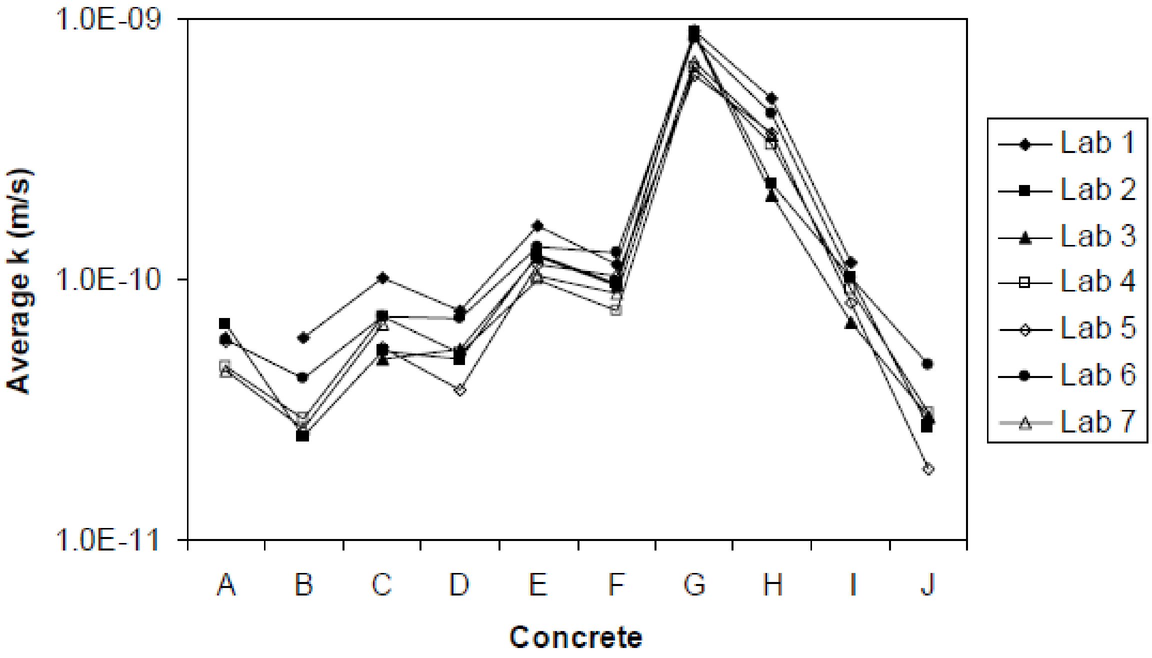

**Figure 2.33: Average permeability values from round-robin testing (Stanish et al., 2004)**

## 2.11. Sources of Variability

This section will focus on the potential sources of variability for the DI tests. Particular attention will be focussed on the specimen conditioning parameters, and possible alternatives to the procedures which are currently used on the DI tests.

### 2.11.1. The Need for Specimen Conditioning

Permeability results are known to be sensitive to the moisture state of the concrete (Whiting, 1988; Kearsely and Wainwright, 2001). Water is held in the HCP at two levels; within the pores and within the gel. Pore water, or capillary water, is “free” water and is the water that is available for reaction. Gel water, which is held tightly by capillary tension and hydrogen bonds, contributes to the microstructure and can cause considerable microstructural shrinkage if removed (Collier et al., 2008). The capillary water essentially blocks the pathways for gas movement through the paste matrix. Thus, the excess water within the specimen needs to be removed to produce a moisture reference state from which meaningful measurements can be taken. The way this is done will have an effect on the results obtained (Abbas et al., 1998; Gardner et al., 2005; Bahador and Jong, 2006).

Sorptivity is also affected by the moisture state of the concrete. Lampacher (1992) showed that, for the ISAT (described in Section 2.5.3), the covercrete absorption is dependent on the moisture condition of the concrete. Concrete specimens were dried for different durations, up to 90 days, in the oven at 50 °C and the absorption was tested. Figure 2.34 shows that the absorption typically increased as the drying time increases.

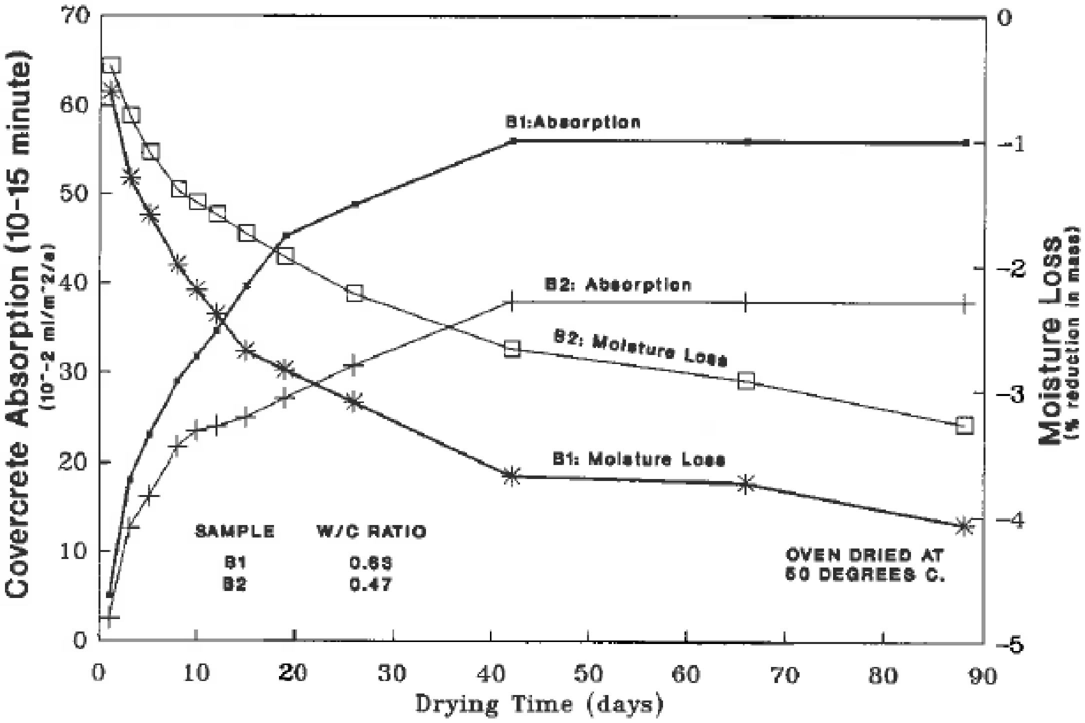

**Figure 2.34: Variation in the covercrete absorption test results with changes in moisture content of the sample (Lampacher 1992)**

As described in Section 2.6, four concrete test specimens are required per test in each of the three DI tests. These are obtained from cores which have been cut from test cubes or from the actual structure with the ends cut or grinded to the required thickness. The same specimens may be used for the OPI test and the WSI test while the CCI test requires four new specimens. They are preconditioned to remove moisture in an oven at 50 °C for a period of 7 days after which they are cooled to 23 °C. The challenge is to have a procedure which is practical while still removing sufficient moisture to produce reliable results.

### 2.11.2. Drying Techniques

#### Oven Drying

The preconditioning stage for the OPI and WSI test involves oven drying the specimen at 50 °C for a period of 7 days after which they are cooled to 23 °C. Oven drying at 105 °C is also widely used to remove moisture (Nyame, 1985; Hampton, 1993; Laskar et al., 1997). Galle (2001) compared the effect of different drying methods on the pore structure of the hardened cement paste. The methods included oven drying methods at 60 °C and 105 °C as well as vacuum drying and freeze drying. The results indicated that oven drying at 105 °C significantly degraded the structure and increased its porosity (and permeability if the pores are connected). Oven drying at 60 °C for 7 days and vacuum saturation had similar results, but could provide a more realistic porosity value. Ballim (1993) found the most effective method to be oven drying at 50 °C for a period of 7 days, causing minimal microstructural damage, while Mackechnie (2000) showed that drying at 50 °C produces different levels of microstructural change in high quality concretes containing silica fume. This method of drying involves the phase transformation of water from liquid to vapour. As this occurs, capillary suction is experienced which can induce microcracking and the collapse of the paste microstructure (Zhang & Scherer, 2011).

### Freeze Drying

Freeze drying is performed by immersing the specimen directly into liquid nitrogen at -198 °C, and then placing it in a vacuum freeze drier at low temperature (-78 °C) and low pressure (4 MPa). According to Galle (2001), this is the best drying method for hydrated cement pastes because it does not alter the pore microstructure. However, this method does generate significant damage related to the thermomechanical stress within the CSH gel (Galle, 2001). Furthermore, freeze drying has the effect of leaving water in the cement (Collier et al., 2008; Konecny & Naqvi, 1993). It is also mainly used on small specimens and is not practical for use in drying large specimens.

### Solvent Replacement Methods

Solvent replacement methods involve replacing the pore water with organic liquids and then removing the solvents to obtain dry concrete sample. Samples are soaked in the solvent which diffuses into paste and replaces the pore solution. The mass change of the sample is monitored while the process is taking place. The solvent exchange is assumed to be complete once the mass stabilises. The solvent is then removed by evaporation at ambient or elevated temperature (Zhang & Scherer, 2011). Solvents are miscible with the pore water and have a lower surface tension than water so their expulsion from the pores does not cause significant capillary suction as experienced with straight oven drying (Knapen et al., 2006). Solvents include acetone, methanol and isopropanol, among others. Solvent exchange with isopropanol is considered to be the most effective technique at preserving the microstructure (Zhang & Scherer, 2011).

There are mixed views on the use of solvents. Solvent replacement with isopropanol produced samples with the least damage to fine pores when compared to oven drying and freeze drying (Konecny and Naqvi, 1993). Collier et al. (2008) agree with these results and showed that the pore sizes of acetone-quenched concrete samples were also least affected, although only slightly compared to other drying methods. They proposed that removal of pore water be done by immersion in acetone followed by drying in a vacuum desiccator (Collier et al., 2008). Gardner (2006) showed that drying with propanol resulted in a considerable decrease in chloride conductivity compared to oven dried results, although it was uncertain whether the solvent replaced specimens were completely dry when tested. If they were not completely dry, the remaining pore water in the specimens would contribute to the decrease in conductivity. Zhang and Scherer (2011) concluded that the solvent replacement technique is more effective at preserving the microstructure than the oven drying method. Some authors believe that the solvents may react with the cement hydration products thereby altering the microstructure.

A drawback of this method is that it takes a much longer time to complete for larger specimen sizes compared to oven drying. Samples with thickness of 10 mm took nearly 3 weeks for drying to complete (Gran & Hansen, 1998). The specimens used in the DI tests are 30 mm thick so can be expected to take significantly longer. In addition, the quality of the concrete would likely have an effect on the diffusion process, with denser pore structures taking a longer time for full replacement to occur. This raises the question of the practicality and efficiency of this method for use as part of the DI test specimen preparation, where it is quite conceivable to expect a large number of specimens to be tested in a limited amount of time.

**Vacuum Drying**

Vacuum drying involves placing the specimen in a vacuum cell to eliminate the moisture. This method has been found by Galle (2001) to be similar to that of oven drying at 60 °C, producing similar stresses and microcracks. This method has detrimental effects on the concrete microstructure and should be avoided.

### 2.11.3. RILEM TC116-PCD Preconditioning Method

Special mention is made to The RILEM TC116-PCD (1999) method, hereafter referred to as the RILEM method as it is a widely used preconditioning method for permeability measurements (Carcasses et al., 2002; Castro-Gomes et al.,2011 ) . The method is carried out on specimens at an age of 28 days or older. The method produces specimens not conditioned to a completely dry state, but to a state with an intermediate known moisture content. The method is as follows.

A specimen from the group of specimens to be tested is dried at 105 °C until the mass change between 24 hours is less than 0.5 g in order determine the evaporable water content.

$$w_e = \frac{W_e}{m_d} \tag{2.20}$$

where: $W_e$ = $m_o - m_d$ [i.e. initial mass – dry mass] (g)

$m_d$ = dry mass

Once dried, the specimen is placed in a desiccator and conditioned at 75% RH and 20 °C, over a saturated solution of NaCl with excess salt, until moisture equilibrium is reached (when two successive weights over a 24 hour period are less than 0.1 g ). Specimens can be crushed into smaller pieces to accelerate the moisture absorption process.

$$w_{e,75} = \frac{W_{e,75}}{m_d} \tag{2.21}$$

where: $W_{e,75} = m_{e75} - m_d$ (g) [i.e. mass at 75% RH – dry mass]

After determining the total and intermediate moisture concentrations, the required weight loss for each specimen to be tested is calculated by:

$$\Delta m = \left(\frac{w_e - w_{e,75}}{1 + w_e}\right) m_0 \tag{2.22}$$

The specimens to be tested are then dried at 50 °C until the observed mass loss is within 5% of the calculated required mass loss $\Delta m$. This produces samples with a moisture condition equal to a moisture condition at 75% RH. However, the distribution of the moisture within the specimen is not considered to be uniform.

To produce a uniform moisture state, a moisture redistribution step is carried out involving placing the specimens in sealed containers and then in an oven at 50 °C for two weeks after which the specimens are cooled down and ready for the gas permeability test to be performed.

This method produces a test specimen with a uniform moisture distribution, however the time required to precondition the specimens is lengthy. The total time taken for the drying of specimens plus the two week moisture redistribution stage can take about a month. There are modified versions of the above approach proposed by Castro-Gomes et al. (2011) and Anton et al. (2012). However, they are still time consuming.

This process may be acceptable for research purposes but to incorporate such a method in tests which are to be used in industry is unfeasible as it may delay projects. In the DI tests, the much shorter 7 day duration of drying is used which does not include the additional steps involving moisture redistribution; specimens are merely placed in an oven until DI testing commences. The specimens used in the DI tests are required to be dry and not at a defined moisture concentration, as is required by the RILEM procedure. As a result, the DI method is much shorter since the moisture redistribution step is avoided, assuming all the pore water has been removed. However, the question also arises whether the drying is completely uniform throughout the specimen.

### 2.11.4. Investigation of the Influence of Specimen drying on the DI Tests

When developing the CCI test, Streicher (1997) investigated the influence of sample conditioning with regards to the drying of specimens. Two drying temperatures were considered – 50 °C for 14 days, followed by 100 °C for a further 7 days. Two Portland Cement concrete mixes (40 MPa and 60 MPa) were tested. It was determined that up to 95 % of the moisture had been removed within the 7 day period while drying at 50 °C, while a further 1 % reduction in mass occurred when drying at 100 °C, as shown in Figure 2.35.

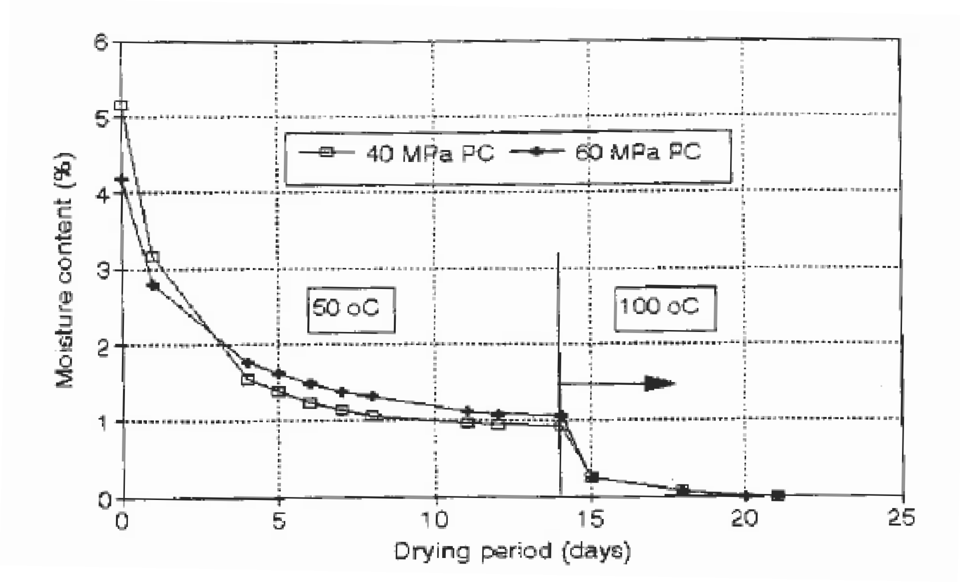

**Figure 2.35: Drying at 50 °C and 100 °C (Streicher, 1997)**

Further tests were conducted on a 40 MPa 50 % slag concrete which was wet cured for 12 months. The curing type and duration would result in a good quality concrete. The ‘standard’ curing duration is 28 days for the DI tests. Two oven drying temperatures were used - 50 °C for 7 days and 105 °C for 7 days. The specimens were vacuum saturated, left to soak in a solution of $Ca(OH)_2$ and the conductivity was measured on different days. This is shown in Figure 2.36. It can be seen that the drying temperature has an effect on the conductivity at 7 days, due to the microstructural damage caused at the higher temperature. The convergence of the conductivities as time went on was put down to autogenous healing of the concrete (Streicher, 1997).

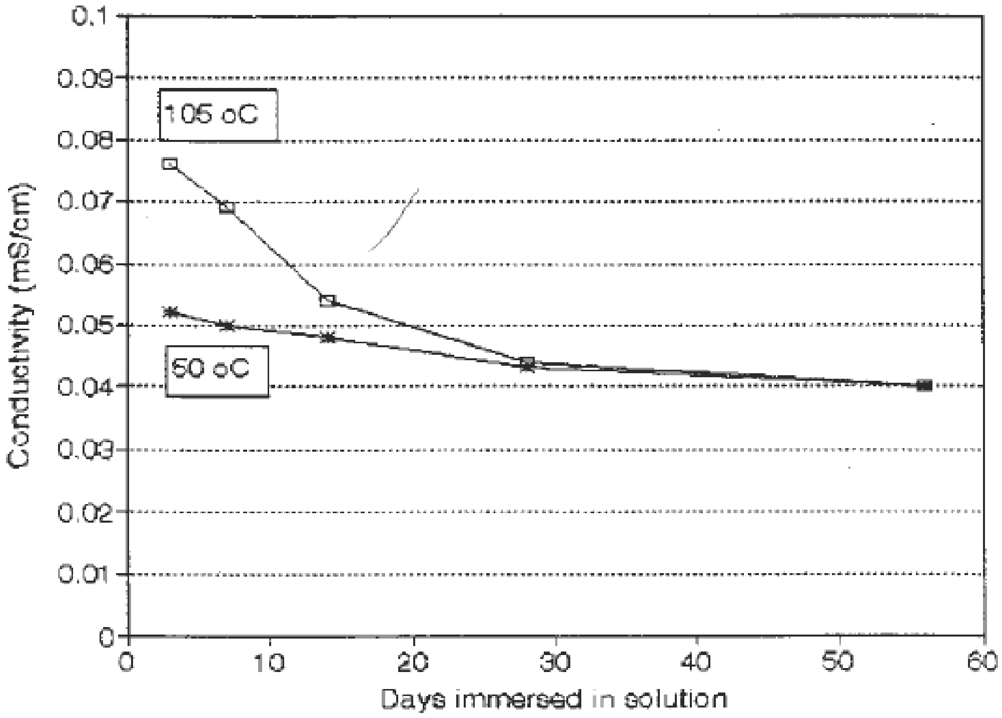

**Figure 2.36: Conductivity of concrete saturated with sat. $Ca(OH)_2$ solution after drying at 50 °C and 100 °C plotted against time (Streicher, 1997).**

The 50 °C was selected as a mild enough temperature to induce minimal microstructural damage. The next step was to determine the duration of drying required. Statistical tests were performed on conductivity results from oven drying durations of 7 days and 14 days at 50 °C for a 40 MPa OPC mix. The outcome was that the increase in drying duration did not significantly impact the conductivity (Streicher, 1997). This test was part of a series of tests to determine the ruggedness of the CCI test. Several variables were tested and changed at the same time, following a “Plackett-Burman” design (ASTM E 1169-89). A statistical t-test is then performed to determine if the result is statistically significant. While the drying durations were deemed to be statistically not significant, this was only done on one mix while varying other factors such as the concentration of the saturating solution and the temperature. There was no testing done on a variety of mixes

Ballim showed that on a 45 MPa OPC concrete wet cured for 12 months, it took an average of 8.65 days to reach constant mass (as defined by a 0.1% change in mass over 24 hours). It was decided that specimens greater than 17 mm should be dried for a period of 14 days at 50 °C.

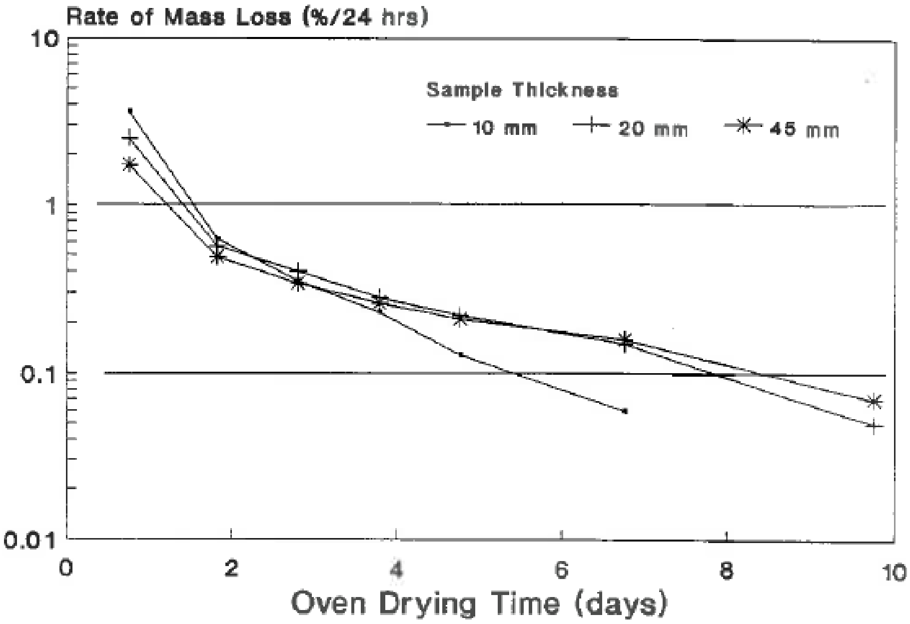

**Figure 2.37: Variation in the rate of mass loss for samples dried at 50 °C (Ballim, 1994)**

The influence of drying temperature on the OPI was also investigated for three binder types (OPC, OPC/FA and OPC/GGBS) and two w/c ratios (0.63 and 0.53) for each binder type. The specimens were exposed to the following drying sequence:

a) Over silica gel or in a vacuum chamber for 3 months;
b) Over phosphorous pentoxide ($P_2O_5$) for 7 days
c) In an oven at 50 °C for 7 days
d) In an oven at 100 °C for 24 hours

The permeability at each phase was measured and is shown in Figures 2.38 to 2.40. The permeability is shown relative to the results from the 100 °C drying method. The actual permeability is shown in parentheses in the legend of the figures. The sample code containing D and V correspond to the initial drying using silica gel (D) and vacuum drying (V) respectively.

**Table 2.8: Mix identification for drying tests used by Ballim (1994)**

| **Binder** | **w/c** | **Mix Identification** |
|---|---|---|
| OPC | 0.63 | PC1 |
| OPC | 0.53 | PC2 |
| OPC/FA | 0.63 | FC1 |
| OPC/FA | 0.53 | FC2 |
| OPC/GGBS | 0.63 | SC1 |
| OPC/GGBS | 0.53 | SC2 |

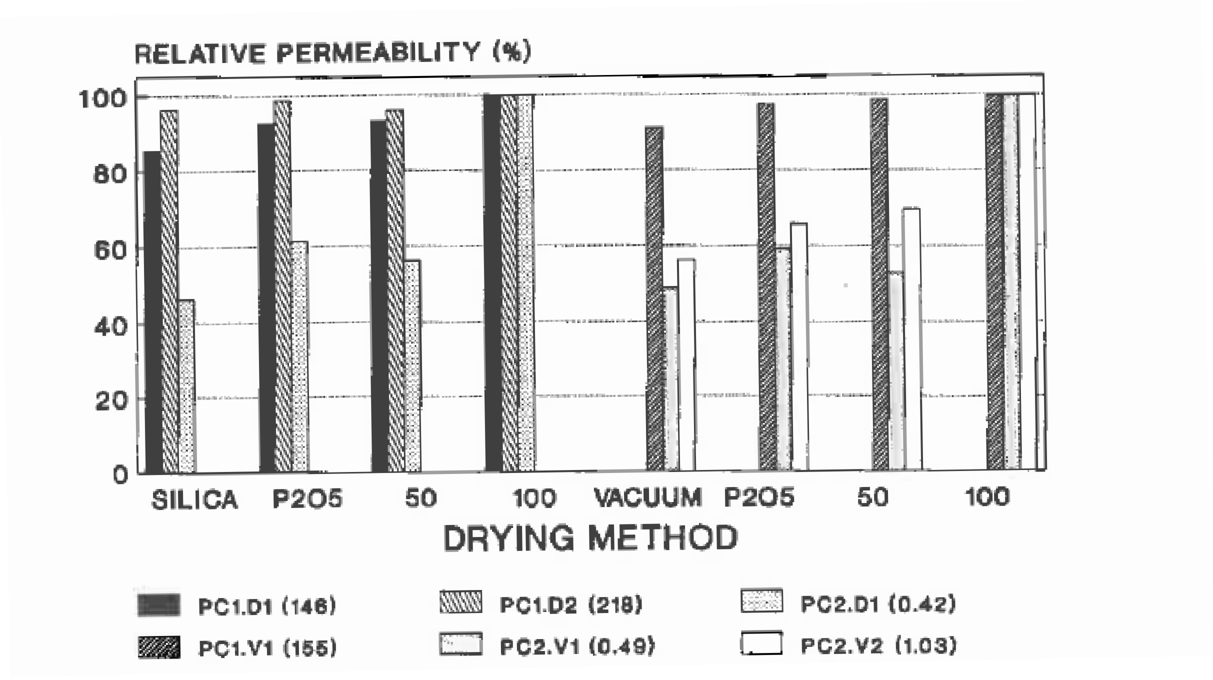

**Figure 2.38: Permeability of samples after various drying methods relative to permeability after 100 °C drying of the OPC samples (Ballim, 1994)**

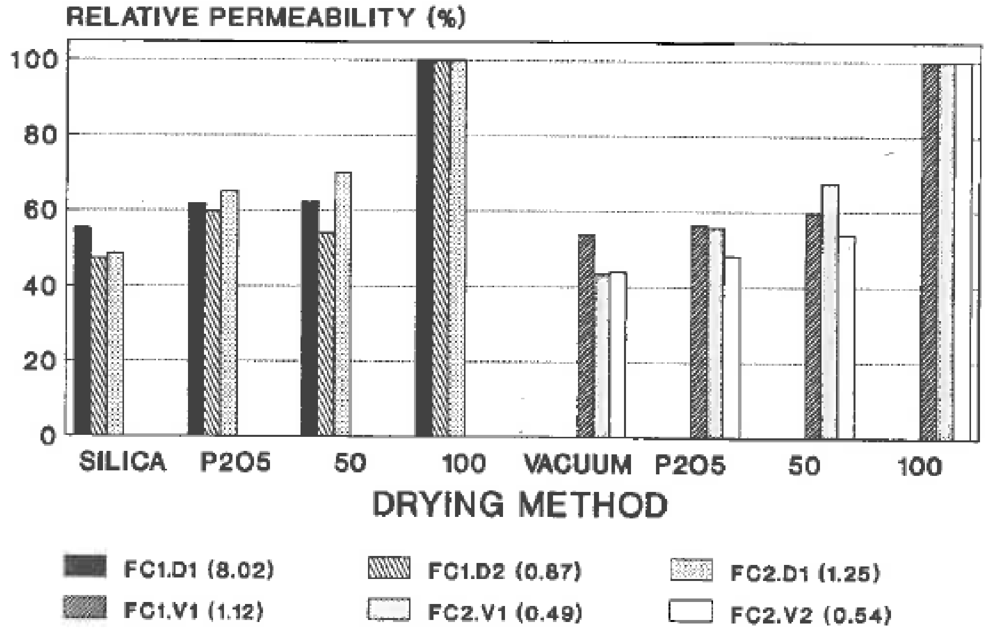

**Figure 2.39: Permeability of samples after various drying methods relative to permeability after 100 °C drying of the OPC/FA samples (Ballim, 1994)**

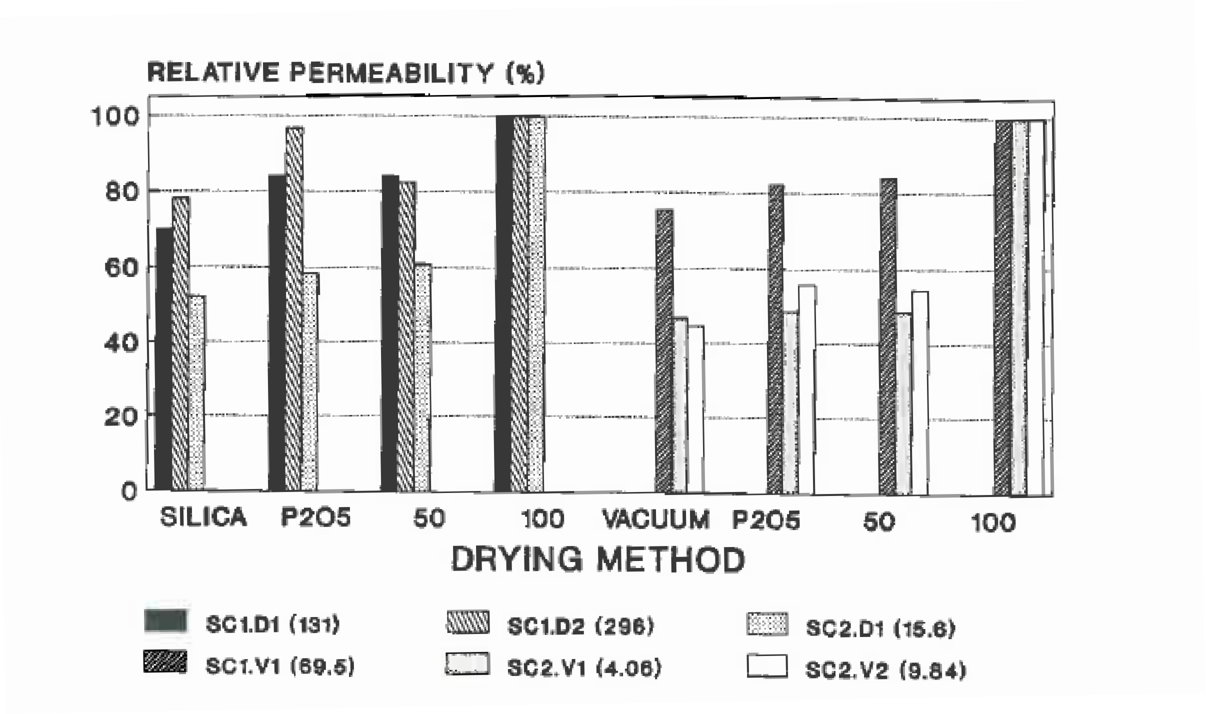

**Figure 2.40: Permeability of samples after various drying methods relative to permeability after 100 °C drying of the OPC/GGBS samples (Ballim, 1994)**

The results showed that, while some microstructural damage may have occurred, drying at 50 °C was comparable to using more gentle methods such as the vacuum drying and silica gel drying methods. Drying at 100 °C caused significant alterations in the concrete microstructure, especially with low permeability concretes.

The sorptivity test is not as sensitive to changes in drying as the OPI test (Ballim, 1993). This conclusion resulted from similar tests conducted by Ballim (1993) on sorptivity measurements as the permeability tests described above. The same drying regimes were selected as used for the OPI test. Specimens dried at 50 °C actually showed lower dryness compared to the silica gel counterparts. Despite this, they exhibited an increase in sorptivity, although this was very small. Figures 2.41 and 2.42 show the relative sorptivity resulting from the various drying methods. It was concluded by Ballim (1994) that for specimens 15 mm thick, the drying duration should be a minimum of 7 days at 50 °C.

Unlike the CCI test, the drying tests performed by Ballim (1994) were conducted on a range of concretes. However, new binders have since been introduced in the market which may be influenced differently. The drying conditioning of specimens therefore remains an area of interest in the DI tests as the moisture condition of the specimens is significant in all the DI tests.

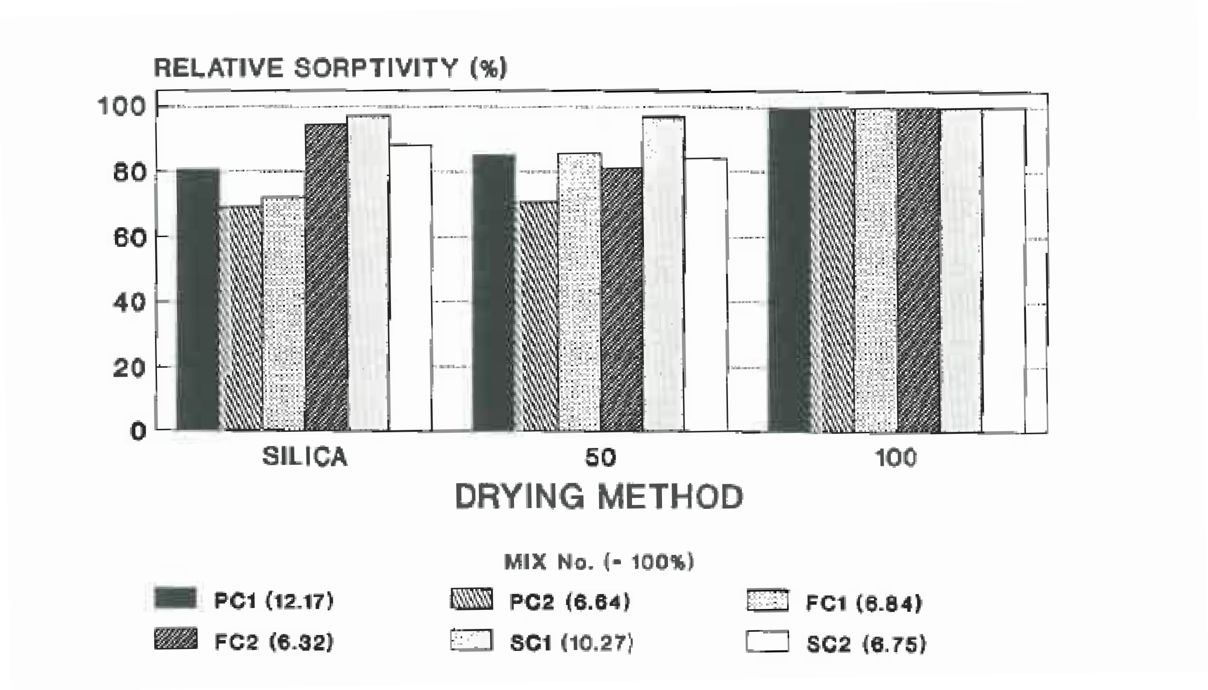

Figure 2.41: Relative sorptivities after various drying phases for samples initially dried over silica gel (Ballim, 1994)

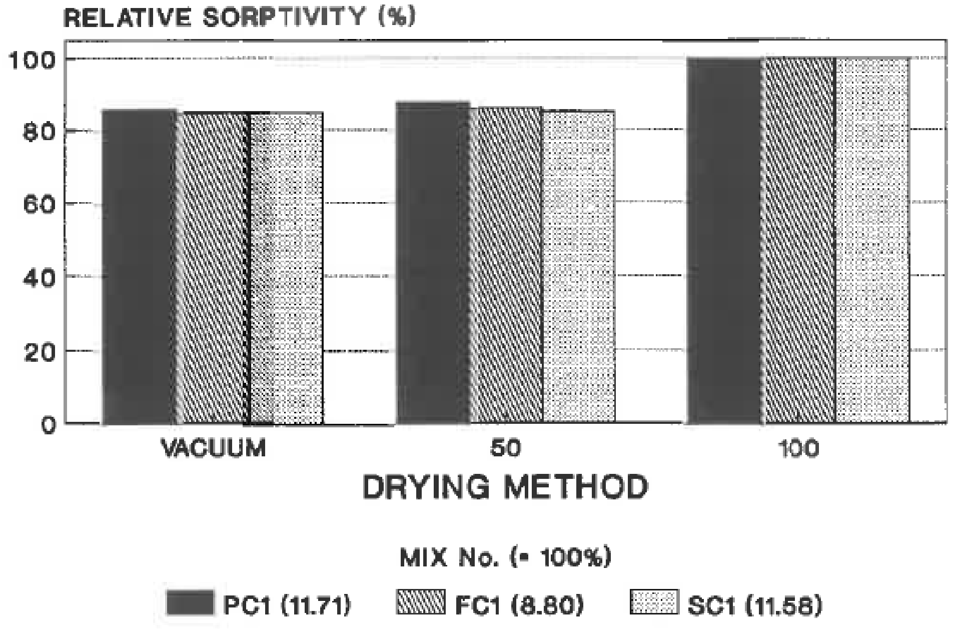

Figure 4.18(b) Relative sorptivities after various drying phases for samples initially vacuum dried

Figure 2.42: Relative sorptivities after various drying phases for samples initially vacuum dried (Ballim, 1994)

### 2.11.5. Specimen Saturation Techniques

The porosity of a specimen is an important property that is useful to characterise for durability related issues. In order to determine the porosity, a dry specimen can be saturated with a liquid (typically water), with the volume of liquid taken up relating to the specimen porosity. This can occur by soaking the specimen in the liquid, which is time consuming, or by forcing the liquid in the pores by some other method. Safiuddin and Hearn (2005) compared various saturation techniques specified in ASTM C1202, namely the vacuum saturation method, cold-water saturation and boiling water saturation. To fully saturate a sample, all the voids need to be filled with the saturating liquid. Thus, all the air needs to be pushed out of the voids first. Vacuum saturation was shown to be the most effective method to achieve this compared to the methods of cold water saturation and boiling water saturation, which were not able to expel all the air-bubbles in the pores (Safiuddin and Hearn, 2005).

Saturation of $Ca(OH)_2$ and NaCl solution is required for the WSI and the CCI tests respectively. This is an important step in both the tests. In the CCI test, if the specimen is not saturated or partially saturated, there will be fewer pathways for the ions to migrate which will result in the specimen exhibiting a lower conductivity. The current DI method makes use of vacuum saturation to achieve the saturated specimens. However, difficulties in practice have been experienced with this method leading to inadequately saturated samples which contribute to increased variability in results (Nganga, 2011; Beushausen and Alexander, 2008; Stanish et al., 2006). In the round robin tests conducted on the WSI test some laboratories experienced consistently low porosity values which indicated that the vacuum saturation was not done up to the standard that other laboratories had conducted it (Stanish et al., 2006).

The CCI test requires a fully saturated specimen for the test to produce accurate results. The ingress of the saturating liquid (5.0 M NaCl) is time dependent, and will take longer for the higher quality concrete with a dense pore structure. However, if the specimen is left too long in the saturating liquid, then there is a chance that chloride binding will occur and therefore there will be a decrease the amount of chloride ions available for transport. This effect is shown in Figure 2.43, where the conductivity decreases after a few days in solution, regardless of the solution concentration due to a decrease in the amount of mobile chloride ions available.

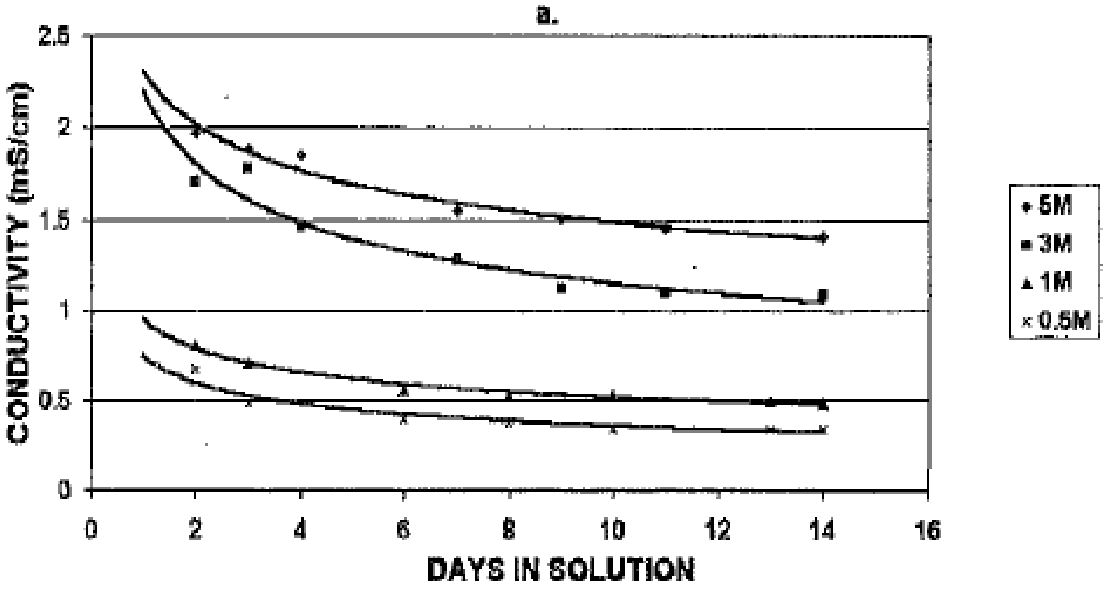

**Figure 2.43: Conductivity reductions with time for OPC binder when using various solution concentrations (Alexander et al., 1999).**

### 2.11.6. Obtaining DI Discs from Cores

As part of the sample preparation procedure, the outer 5 mm of the specimen is required to be removed in order to eliminate the wall effect that creates a higher mortar fraction on the edges. The 2009 Durability index manual states that this can be done by either grinding or cutting the faces of the specimens. As noted in the round robin report by Stanish et al. (2004), some specimens used for inter-laboratory tests were prepared using the grinding method and others using the cutting method. This could introduce a source of variability when comparing test results between laboratories since the two methods are different. Grinding the samples results in them having a higher mortar fraction on the exterior face than on the interior (unground) face. This, to an extent, defeats the purpose of removing the exterior 5 mm because it presents a non-uniform material where the outer face has a mortar fraction that extends for some unknown length (Stanish et al. 2004). This aspect can be eliminated by simply choosing the cutting method to produce “clean” samples. The 2010 DI manual has reflected this and the option for grinding has been omitted. However, the 2009 version is still widely used, so the grinding of specimens may still be an issue in some laboratories.

## 2.12. Summary

Reinforced concrete durability has often been found to be inadequate in the past, resulting in much expense for repair and rehabilitation of reinforced concrete structures. In recent times, however, there has been in increase in awareness of the need to adequately design for durability and not just strength. The main issue results from corrosion of the reinforcement which leads to spalling of the covercrete. Thus, the nature of the covercrete is important to protect the underlying steel from harmful substances that can enter through a number of transport mechanisms such as permeation, diffusion and absorption and migration, among others.

The transport mechanisms are affected by several material factors of the concrete. These include the w/c ratio, aggregate size, water and cement content, curing and binder type. There exist many tests to measure the various transport mechanisms, both in South Africa and international methods. The South African approach makes use of the oxygen permeability index test (OPI), water sorptivity index test (WSI) and chloride conductivity index test (CCI). These measure the permeation, absorption and migration mechanisms respectively. The tests have been developed over the years and have been shown to be repeatable and reproducible, as well as being rugged. However, there are still some concerns around the tests. These include the preconditioning method which removes moisture from the specimens to provide a reference moisture state from which the DI tests can be measured. The current oven drying method has been questioned as it may be damaging to the concrete microstructure. Alternative drying methods such as solvent replacement and vacuum drying were reviewed. The WSI and CCI also require vacuum saturation to take place in addition to the oven drying. It is unclear whether the current saturation procedure is adequate. An additional concern related to the CCI test is the uncertainty whether different rigs produce comparable results.

The above aspects provided the basis for further investigation, the details of which are presented in Chapter 3.

# 3. EXPERIMENTAL METHODOLOGY

## 3.1. Introduction and Research Aims

This chapter describes the work carried out as part of the experimental component of this study. The experimental work was performed to provide a greater understanding of the variables involved in the DI tests, particularly regarding the sample preconditioning methods. The experimental design and the statistical tools used to analyse the results are explained.

This study provides a critique of the Durability Index tests in an attempt to identify possible areas of improvement. Through an evaluation of the literature in Chapter 2 it is clear that the test methods have had considerable work done to improve them over the years. However, several areas of possible improvement have been identified that could be addressed in the laboratory. These mainly are around the preconditioning phase of the tests rather than the actual tests themselves. Aspects which could be investigated in the laboratory are:

- The oven-drying technique currently used to dry the specimens
- The saturation condition of the specimens before chloride conductivity testing
- Differences in chloride conductivity test rigs and the effect on measured values

An experimental strategy was devised to determine the extent to which the above issues affected the DI results.

## 3.2. Details of Concrete Constituent Materials and Mix Designs

The experimental work carried out in the laboratory required concrete cubes to be cast. In this work, the term w/c will be used to denote both the water/cement ratio as well as the water/binder ratio. Three w/c ratios were investigated i.e. 0.4; 0.5; and 0.65, providing mixes over a strength and durability spectrum. Table 3.1 shows the binders and w/c ratios used. The difficulty with changing the w/c ratio lies in whether to change the water content or binder content. In this study, to limit the number of variables, the water content was kept constant at 170 L/$m^3$ for each mix. The binder content was then adjusted to obtain the required w/c ratio. The mixes were designed following the C&CI method (Addis and Goodman, 2009). The quantity of material required was calculated by determining the volume in $m^3$ and then converting to mass using the relative density of the material. The stone content was calculated based on the Compacted Bulk Density (CBD) of the stone and the Fineness Modulus (FM) of the sand respectively. The sand content was calculated by subtracting the sum of the calculated volumes of the binder, water and stone from 1000, thereby obtaining the mix proportions for a cubic metre mix.

### 3.2.1. Binder Properties

Different binders were chosen for testing to provide results across a range of concretes. Four commonly used binders were selected namely CEM I 52.5N and CEM II B-M (L-S) 42.5N (containing GGCS and ground limestone), referred to in this work as CEM II (B) 42.5N, supplied by PPC Cement; Ground Granulated Blast Furnace Slag (GGBS) supplied as Slagment by Afrisam; and Fly Ash (FA) supplied by Ash Resources. GGBS and FA were used as extenders combined with CEM I 52.5N.The different binders and w/c ratios used in this work are shown in Table 3.1. The chemical composition of each binder is shown in Tables 3.2 - 3.4.

**Table 3.1: Binder type and w/c ratio used**

| Binder | w/c ratio |
|---|---|
| CEM I 52.5N | 0.4, 0.5, 0.65 |
| 50% CEM I 52.5N, 50% GGBS | 0.4, 0.5, 0.65 |
| 70% CEM I 52.5N, 30% FA | 0.4, 0.5, 0.65 |
| CEM II (B) 42.5N | 0.4, 0.5, 0.65 |

**Table 3.2: Chemical composition of CEM I 52.5N and CEM II B-M (L-S) 42.5 N (data from PPC)**

| Oxide | CEM I 52.5N | CEM II B-M (L-S) 42.5N |
|---|---|---|
| | % by mass of cement | |
| $SiO_2$ | 21.10 | 22.30 |
| $Al_2O_3$ | 4.00 | 7.40 |
| $Fe_2O_3$ | 3.35 | 3.00 |
| CaO | 65.80 | 53.50 |
| MgO | 0.87 | 4.30 |
| $SO_3$ | 2.30 | 2.00 |
| $K_2O$ | 0.70 | 0.60 |
| $Na_2O$ | 0.10 | 0.30 |

**Table 3.3: Typical composition of South African GGBS (Grieve, 2009)**

| Oxide | % by mass in GGBS |
|---|---|
| $SiO_2$ | 34-40 |
| $Al_2O_3$ | 11-16 |
| CaO | 32-37 |
| MgO | 10-13 |
| FeO | 0.3-0.6 |
| MnO | 0.7-1.2 |
| $K_2O$ | 0.8-1.3 |
| S | 1.0-1.7 |
| $TiO_2$ | 0.7-1.4 |

**Table 3.4: Typical composition of South African FA (Grieve, 2009)**

| Oxide | % by mass in FA |
|---|---|
| $SiO_2$ | 48-55 |
| $Al_2O_3$ | 28-34 |
| CaO | 4-7 |
| MgO | 1-2 |
| $Fe_2O_3$ | 2-4 |
| $Na_2O + 0.658K_2O$ | 1-2 |

### 3.2.2. Aggregate Properties

Greywacke stone was used for the coarse aggregate with a nominal maximum aggregate size kept constant at 19 mm. The sand was a 50/50 blend of Philippi Dune sand and Klipheuwel sand. Workability of the mixes, particularly at the lower w/c ratios, was a factor that needed to be controlled. The sieve analysis and grading curves are shown in Figure 3.1 and Figure 3.2.

### 3.2.3. Workability of the Mixes

To ensure adequate workability of the mixes, a high-range water reducing admixture (Sikament N-N) was added where needed in sufficient mass to obtain a slump within 80 – 120 mm. The amount of admixture to be added was determined in trial mixes that were carried out where progressive amounts were added until the required workability was achieved. The final amount of admixture used for each mix is shown in Table 3.5.

**Table 3.5: Amount of admixture added and workability of mixes**

| Binder | w/c | Admix added (mL/20 L mix) | Admix added (% by mass binder) | Slump (mm) |
|---|---|---|---|---|
| CEM I 52.5N | 0.40 | 45 | 0.6 | 80 |
| | 0.50 | 30 | 0.5 | 80 |
| | 0.65 | 0 | 0.0 | 85 |
| CEM I 52.5N/GGBS | 0.40 | 45 | 0.6 | 85 |
| | 0.50 | 35 | 0.6 | 80 |
| | 0.65 | 0 | 0.0 | 80 |
| CEM I 52.5N/FA | 0.40 | 35 | 0.5 | 85 |
| | 0.50 | 20 | 0.3 | 85 |
| | 0.65 | 0 | 0.0 | 120 |
| CEM II (B) 42.5N | 0.40 | 40 | 0.5 | 85 |
| | 0.50 | 30 | 0.5 | 90 |
| | 0.65 | 0 | 0.0 | 95 |

**Table 3.6: Sieve analysis for klipheuwel and Philippi dune sand**

| Sieve opening (mm) | Klipheuwel Sand | Philippi Dune Sand |
|---|---|---|
| | Cumulative % passing (%) | |
| 4.75 | 100.0 | 100.0 |
| 2.36 | 99.9 | 100.0 |
| 1.18 | 94.5 | 97.0 |
| 0.6 | 84.9 | 30.2 |
| 0.3 | 65.3 | 10.6 |
| 0.15 | 4.6 | 1.0 |
| 0.075 | 1.1 | 0.0 |
| PAN | | |

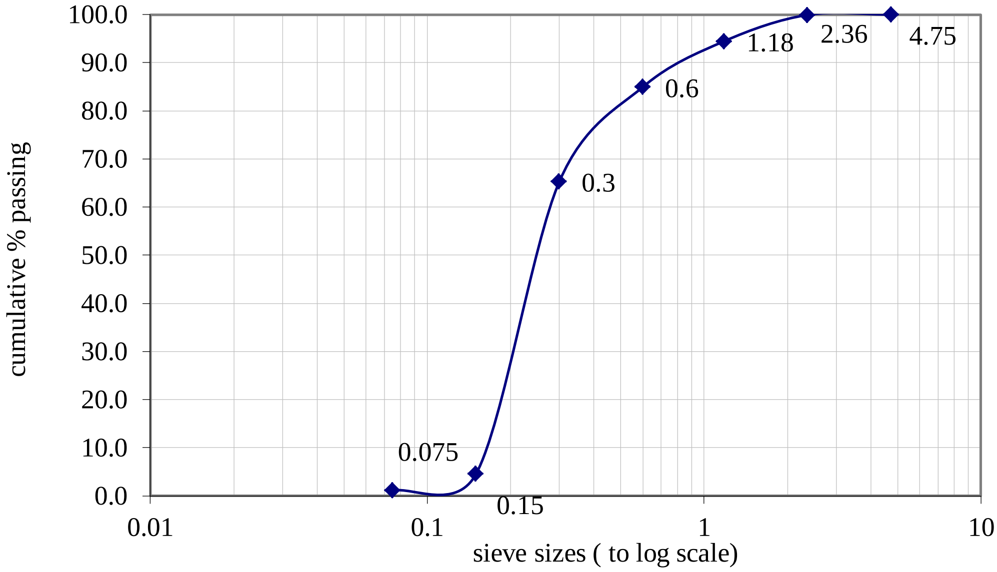

**Figure 3.1: Sieve analysis and grading curve for Klipheuwel sand**

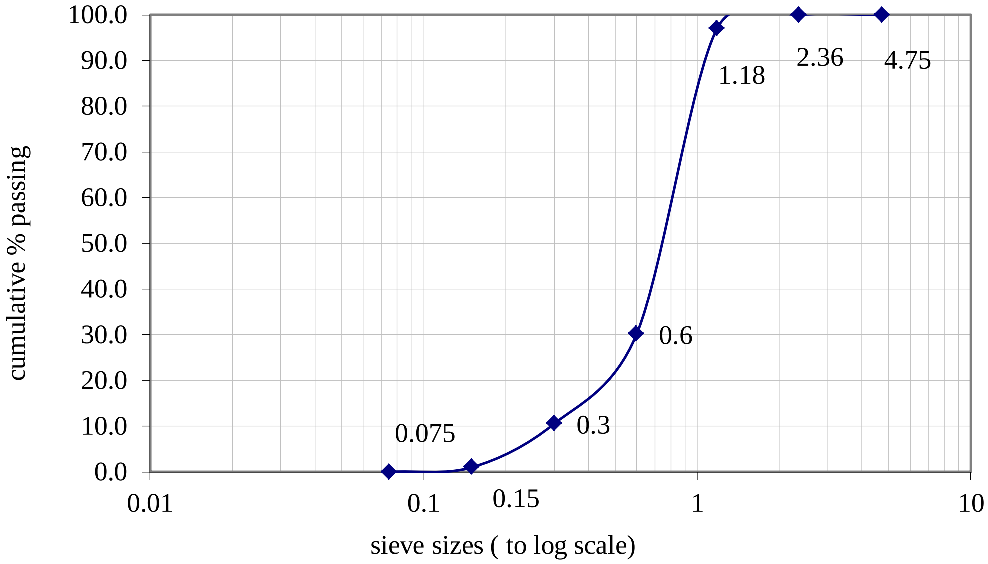

**Figure 3.2: Sieve analysis and grading curve for Dune sand**

### 3.2.4. Mix Proportions

A summary of the mix proportions is shown in Table 3.7.

**Table 3.7: Mix proportions**

| **Binder** | **w/c** | **Water *(L/m$^3$)** | **Cement (kg/m$^3$)** | **SCM (kg/m$^3$)** | **Stone (kg/m$^3$)** | **Sand (kg/m$^3$)** |
|---|---|---|---|---|---|---|
| CEM I 52.5N | 0.40 | 170 | 425 | - | 1020 | 840 |
| | 0.50 | 170 | 340 | - | 1030 | 910 |
| | 0.65 | 170 | 262 | - | 1150 | 850 |
| CEM I 52.5N/GGBS | 0.40 | 170 | 212 | 212 | 1020 | 840 |
| | 0.50 | 170 | 170 | 170 | 1030 | 910 |
| | 0.65 | 170 | 131 | 131 | 1150 | 850 |
| CEM I 52.5N/FA | 0.40 | 170 | 297 | 128 | 1020 | 840 |
| | 0.50 | 170 | 238 | 102 | 1030 | 910 |
| | 0.65 | 170 | 183 | 78 | 1150 | 850 |
| CEM II (B) 42.5N | 0.40 | 170 | 425 | - | 1020 | 840 |
| | 0.50 | 170 | 340 | - | 1030 | 910 |
| | 0.65 | 170 | 262 | - | 1150 | 850 |

**Admixture added as necessary, determined in trial mixes. See Table 3.5.*

## 3.3. Amount of Concrete Required

A total of 12 concrete mixes were cast. A minimum of 20 L of concrete was required for each mix. Extra cubes were cast to account for concrete losses and wastage. A grand total of 240 cubes were cast for the 12 mixes used in this study. Table 3.8 summarises the quantities required for the tests in the experimental program.

**Table 3.8. Number of discs and cubes per mix (100 mm cubes)**

| | **Test** | **No. of DI disc specimens*** | **No. of cubes (100 mm)** |
|---|---|---|---|
| 1 | Compressive strength (28 days) | - | 3 |
| 2 | DI tests on oven dried specimens (constant mass) | 8 | 4 |
| 3 | OPI and WSI tests on oven dried specimens (7 days) | 4 | 2 |
| 4 | DI tests on isopropanol-dried specimens | 8 | 4 |
| 5 | Chloride Saturation tests | 4 | 2 |
| 6 | New vs UCT CCI test rig | 4 | 2 |
| | Extra cubes for backup | | 3 |
| | Total | | 20 |

**The DI disc specimen is 30 mm thick x 70 mm diameter*

The specimens used to test the New CCI test rig against the UCT version (item 6 in Table 3.8) served two purposes. The results from the CCI tests performed on the UCT test rig were also used as the CCI results for oven dried specimens at 7 days (item 3 in Table 3.8). Separate cubes for CCI testing on specimens dried for 7 days were therefore not needed, justifying the need for only two cubes.

## 3.4. Curing Regime

The DI tests are known to be affected by the type and degree of curing (Ballim, 1994). To remove the curing effect as a variable, all cubes that were cast in this work were cured for a period of 28 days in water tanks kept at a constant temperature of 23 ± 2 °C.

## 3.5. Laboratory Tests Performed

### 3.5.1. Compressive Tests

Compression tests at 28 days were carried out as a means of quality control of the mixes. Cubes, 100 mm in dimension, were each weighed to verify that their density was within an acceptable range and then crushed to failure using an Amsler Compression Machine. The loading rate was maintained at 0.4 MPa per second as is standard according to SANS 5865.

**Figure 3.3:Test cube undergoing compressive strength testing in the Amsler Compression Machine**

### 3.5.2. Investigating the Effect of the Oven Drying Procedure on the DI Test Results

Part of the preconditioning stage of the DI tests is to place the specimens in an oven for a minimum of seven days at 50 °C to remove excess moisture in the concrete. There is concern that this procedure might cause some microstructural damage to the specimens. In developing the OPI test, Ballim (1994) investigated this aspect and concluded that a 50 °C oven temperature was sufficiently mild, however some researchers have advocated against the use of oven drying even at this temperature (Gouws, 2011). New binders have since been introduced and the effect that oven drying may have on concrete produced with them is unknown. Different methods for the removal of moisture were identified in the Chapter 2. For this study, two drying regimes were chosen for investigation:

i) Oven drying at 50 °C;
    - for seven days
    - until constant mass was achieved – defined as a change in mass of 0.05% over 24 hours

ii) Solvent replacement with isopropanol, followed by drying at 30 °C

The first regime was chosen to investigate the effect of the 50 °C temperature on the DI results. Specific attention was paid to prolonged exposure in the oven. The seven day drying time was chosen to be used as a control since this is specified in the DI test manual. It assumes that this time is adequate to produce specimens that are sufficiently dry for use in the DI tests. The question arises of what happens when the seven day drying time is exceeded? This is not uncommon, particularly when there are many specimens to be tested and there is not sufficient test equipment to conduct the tests. A logistical issue arises in this case since it may not be possible to test all the specimens at the same time, resulting in the samples having to remain in the oven for a longer duration. It follows that every day the specimens are allowed to remain in the oven is more time for any additional moisture that remains after the seven days to be removed. As a result, the specimens being tested would have an inconsistent moisture state if they are tested at different times. Thus there is the need for tests to be done on specimens left for longer periods in the oven to determine its impact on DI tests.

After placing the specimens under these two oven drying regimes, they were subjected to all three DI tests. This was done to try and answer two questions; (a) are the samples sufficiently dry after 7 days (shown by a negligible mass loss thereafter) and (b) does it make a difference to the DI Test results? The specimens were assumed to be completely oven-dry when the mass change over a 24 hour period was 0.05% or less.

The second drying regime used in this study was the solvent replacement method. The general mechanism at play is that as the solvent saturates the specimen it replaces the water in the pores with a solvent-water solution which has a lower surface tension and boiling point than the original water in the pores. The solvent solution can then be removed from the concrete pores at a mild temperature causing lower capillary suction forces within the HCP than oven drying at high temperatures, resulting in fewer microcracks in the specimens. Isopropanol was chosen as the solvent to be used as it is miscible with water, forming an azeotrope solution with a lower surface tension and boiling point than pure water. Furthermore, it is suggested to be the best at preserving the concrete microstructure (Zhang & Scherer, 2011). The specimens were vacuum saturated with isopropanol to speed up the process. Figure 3.4 shows the setup for the vaccum saturation of the specimens. The vacuum saturation procedure was performed as follows:

i) Specimens were placed in a vacuum desiccator and the air was evacuated for a period of 3 hours at a pressure of – 80 kPa to remove air in the chamber and specimens.
ii) Isopropanol was then introduced to the desiccator and the specimens were allowed to saturate under the vacuum for a period of 1 hour.
iii) The vacuum was released and the specimens were allowed to soak in the isopropanol for a period of 18 hours.
iv) The saturated specimens were then weighed to provide the initial mass.
v) They were then placed in an oven to provide a constant temperature environment at 30 °C with.
vi) The mass loss was monitored daily and specimens were assumed to be dry when the mass change over a 24 hour period of 0.05% or less.

**Figure 3.4: Vacuum desiccator used to vacuum saturate specimens with isopropanol**

The grade of isopropanol used was analytical grade (AR) propan-2-ol which was obtained from Kimix, a chemical supplier in Cape Town.

Eight (8) specimens for each mix were randomly selected and their mass loss was monitored and recorded daily until drying had been completed. The criteria for completion of drying was achieved when the mass loss over a 24 hour period was 0.05% or less. The percentage average mass loss for the eight specimens was plotted against time (days).

Once the drying stage was completed, the specimens were tested in the OPI, WSI, and CCI tests. The results were then analysed statistically to determine if the different methods and duration of drying produced similar DI test results.

### 3.5.3. Investigating the Saturation Condition of Specimens Used in the CCI Test

The chloride conductivity test requires the specimens to be saturated with NaCl solution before being tested. A saturated specimen 'completes the circuit' and allows the migration of chloride ions through the cell and specimen when a potential difference is applied across the cell. Unsaturated or partially saturated areas within the specimen represent areas where migration cannot readily take place. As a result, if a specimen is not fully saturated there are fewer pathways for migration to occur, and a lower conductivity than the 'true' conductivity of the specimen will be expected.

The saturation method currently used in the test is as follows:

i) Specimens are placed in a vacuum dessicator and the air is evacuated for a period of 3 hours at a pressure of -80 kPa.
ii) The NaCl solution is introduced into the vacuum chamber and then allowed to saturate the specimens under the -80 kPa vacuum for a period of 1 hour ± 15 minutes.
iii) The vacuum is released and the specimens are allowed to soak in the NaCl solution for a further period of 18 hours.

It is assumed that the above saturation method results in fully saturated specimens. This may be called into question particularly for concrete with a dense microstructure such as concretes at low w/c ratios or with the inclusion of supplementary cementitious components such as GGBS and FA.

To test if full saturation was occurring, certain specimens were subjected to the above saturation procedure and then split in the centre and sprayed with silver nitrate solution. Silver nitrate reacts with sodium chloride to produce a silver chloride precipitate. If full saturation with NaCl occurred then the internal face displayed a white colour from the silver chloride precipitate that forms as a result of the reaction between the chloride and silver ions. Unsaturated areas were represented by a brown colour that formed from the reaction with $OH^-$ ions. This colorimetric technique is similar to the one used in the Rapid Chloride Migration Test (NTBuild 492). This method provided a qualitative result as to whether saturation had occurred or not. However, it did not give the actual amount of chlorides present. It is unknown how sensitive this procedure is to chloride concentration, therefore it was difficult to be sure how much chloride was available in the 'brown' areas. To obtain an estimate of the degree of saturation, the treated surface was photographed and then the saturated (white areas) and unsaturated areas were calculated by importing the image into AutoCAD and using the area calculation tool (see Figure 3.5). The degree of saturation as a percentage could then be estimated as follows:

$$\text{Degree of Saturation (\%)} = \frac{\text{Saturated Area}}{\text{Total Area}} \times 100 \qquad (3.1)$$

A chloride profile could be obtained, however this was not conducted in this work as the primary aim was to investigate if the saturation procedure was an issue.

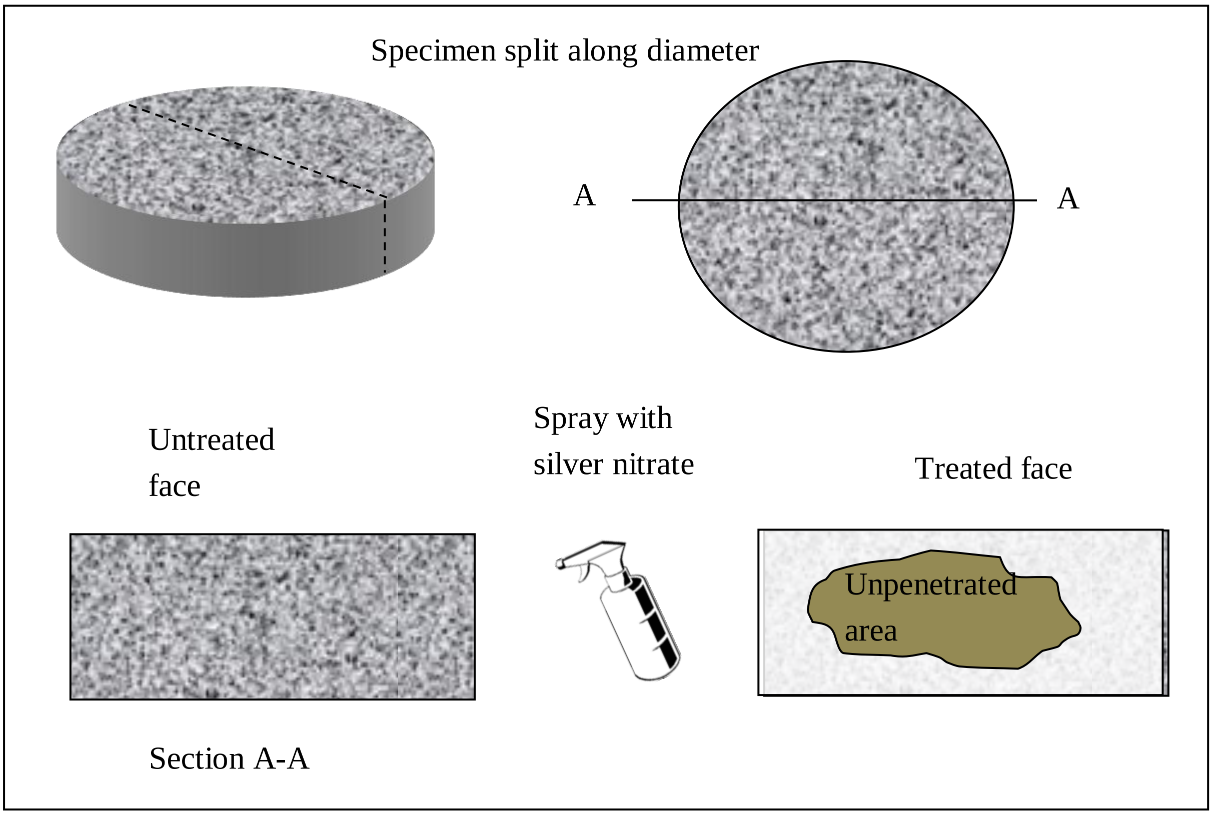

**Figure 3.5: Saturation test example**

### 3.5.4. Investigating the Saturation Condition of Specimens used in the Sorptivity Test

The saturation procedure has also been called into question in the WSI test. The WSI test also makes use of a similar saturation technique to the one used for the CCI test except that in this case the saturating liquid used is calcium hydroxide ($Ca(OH)_2$) and not NaCl. As mentioned previously, the current method assumes that the specimen is fully saturated after the saturation process is complete. The total time that saturation can take place under this method is 19 hours (1 hour vacuum saturation followed by 18 hours soaking). This saturation duration was chosen as a matter of practical convenience for test. To test if the duration of saturation was adequate, the 'standard' saturation procedure was followed:

iv) Specimens were placed in a vacuum dessicator and the air was evacuated for a period of 3 hours at a pressure of – 80 kPa.
v) The $Ca(OH)_2$ solution was introduced into the vacuum chamber and then allowed to saturate the specimens under the -80 kPa vacuum for a period of 1 hour.
vi) The vacuum was released and the specimens were allowed to soak in the NaCl solution for a further period of 18 hours.

The purpose of evacuating the air is to make it easier for the solution to enter the pores of the concrete. Upon completing the saturation procedure described above the following steps were performed:

- The saturated mass was recorded in the 'standard' way after the 18 hours soaking
- The specimens were placed back in the $Ca(OH)_2$ solution and allowed to soak for a further 24 hours.
- The mass was recorded to determine if it had increased during this period.

### 3.5.5. Using an 'Improved' Version of the Chloride Conductivity Index Test Rig

When the CCI test was developed, a schematic diagram of the rig was provided which can be interpreted differently by different manufacturers. A modified version of the CCI rig was designed, hereafter referred to as the 'New Rig' (shown in Figures 3.6 - 3.9), which uses the same fundamental theory as the standard UCT version, but proposes some improvements. These include a new aesthetic design, the use of colour-coded half-cells, and the introduction of telescopic luggin capillaries which should allow potential difference readings to be taken exactly across the faces of the specimen. The UCT rig does not have this feature so it may provide slightly different measures of the potential difference across the specimen depending on the specimen thickness. The inclusion of "bleed holes" in the New rig also allows for entrapped air to be removed. This is shown in Figures 3.6 to 3.9. The UCT rig does not have such a feature so there may be an issue with entrapped air in the system. The effect of these changes on the CCI results was investigated. The general practicality of the two tests was also considered.

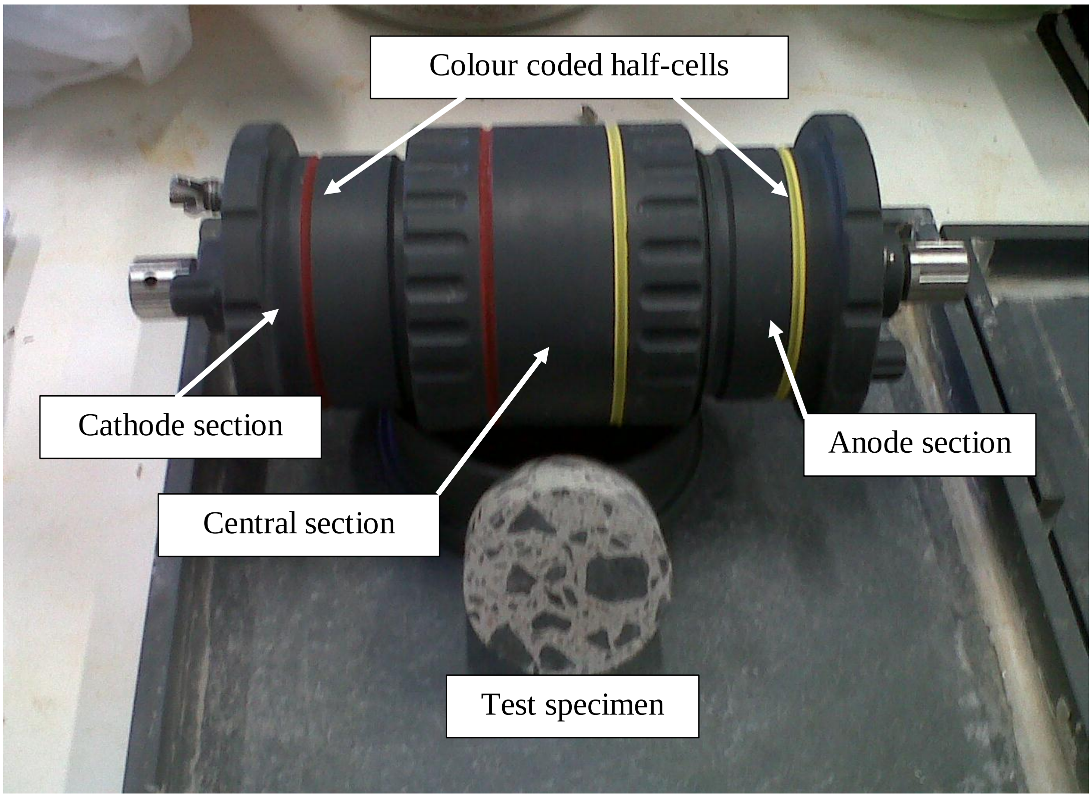

**Figure 3.6: Assembled version of the New chloride conductivity rig**

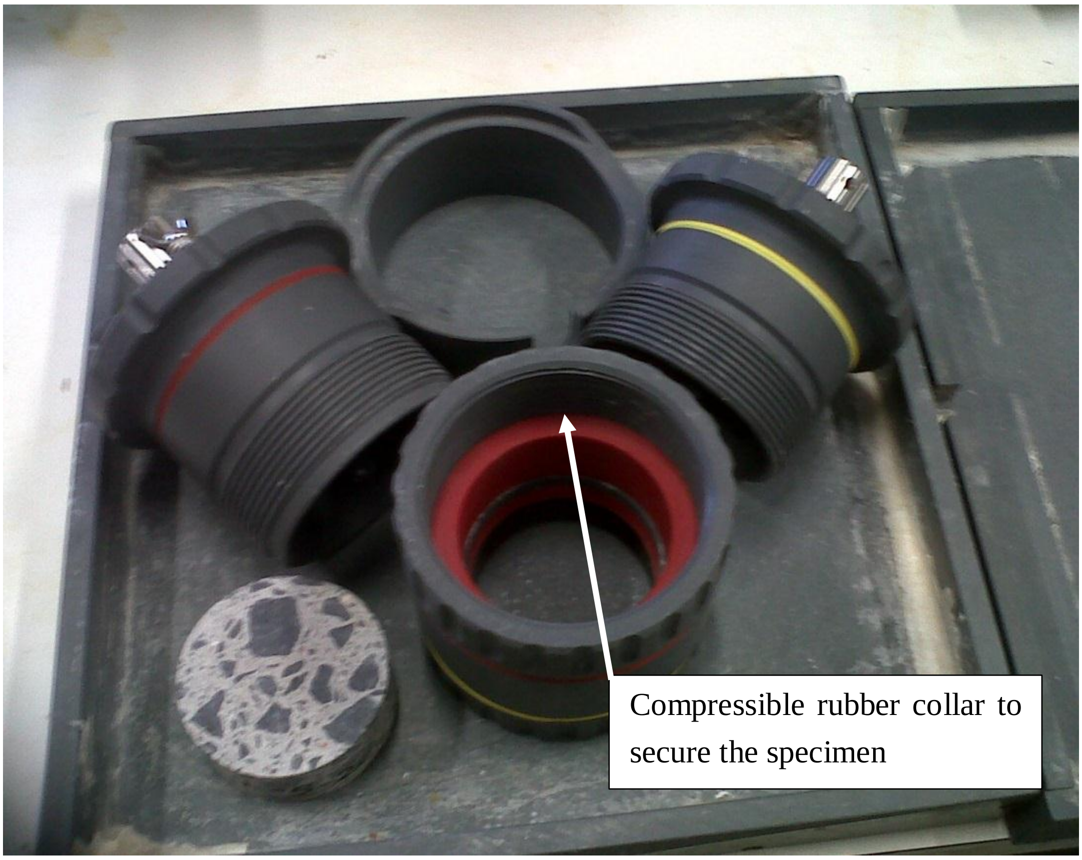

**Figure 3.7: New chloride conductivity rig showing central holding part and half-cell components**

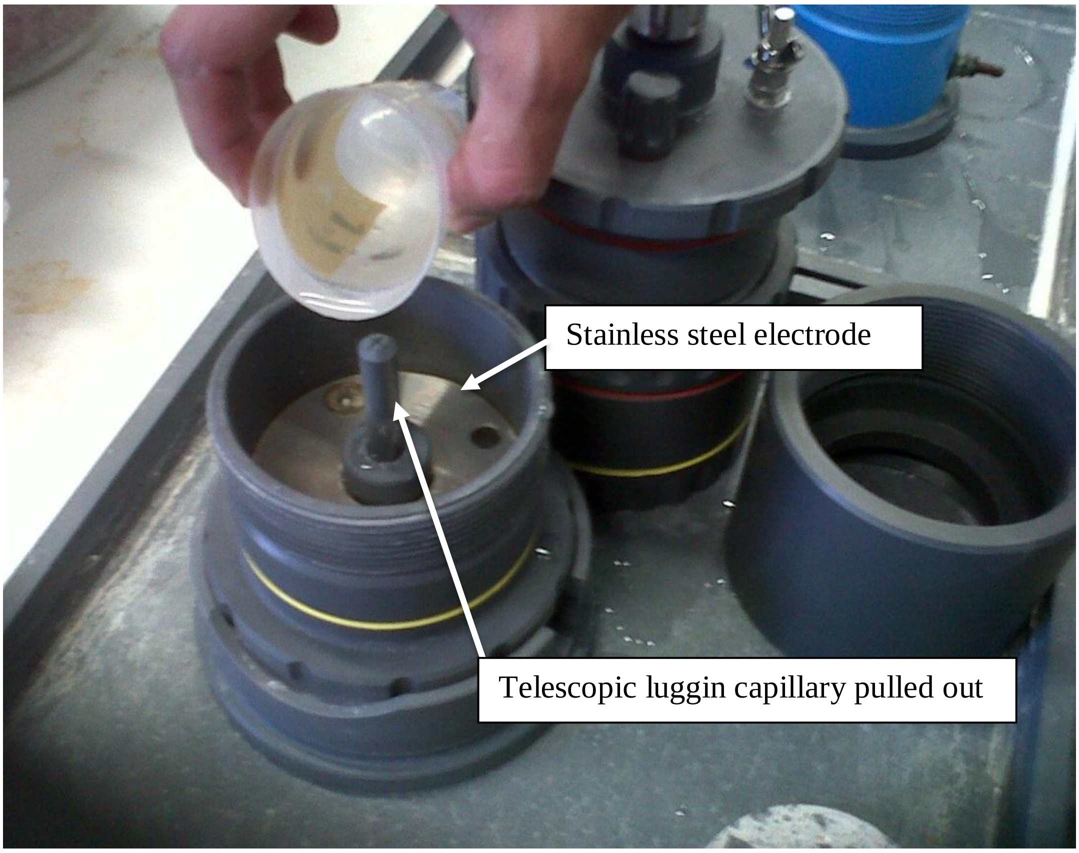

Figure 3.8: Filling up the New chloride conductivity rig with NaCl solution

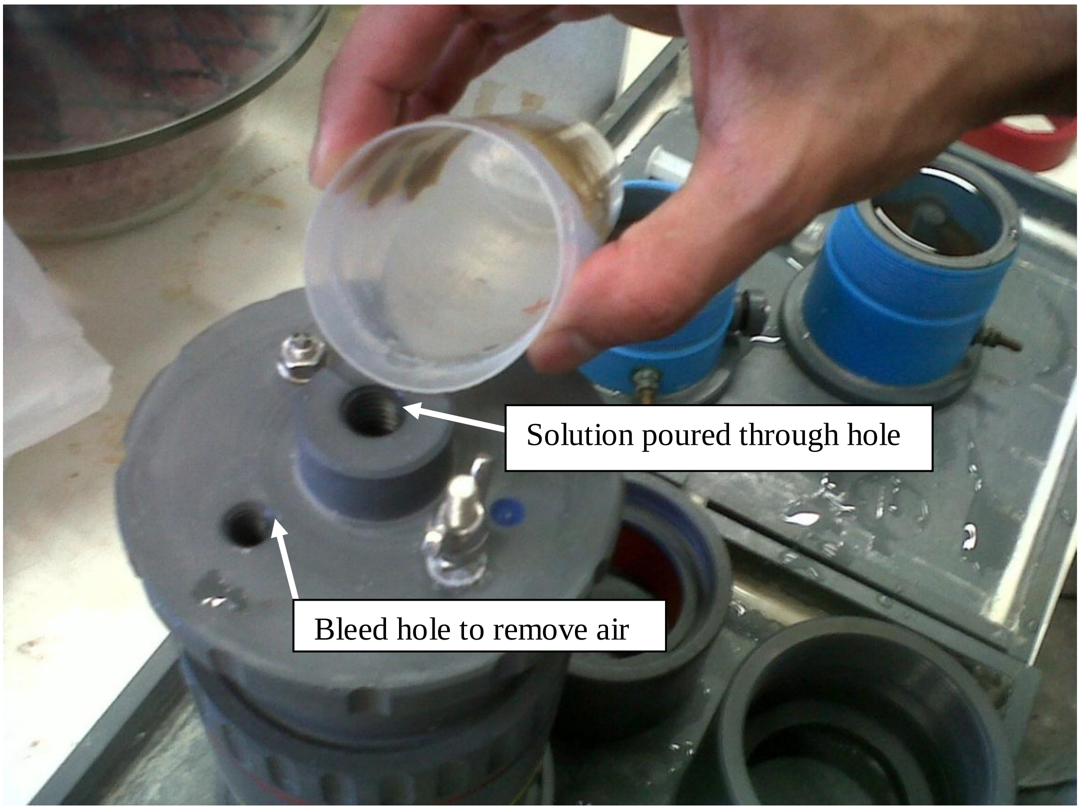

Figure 3.9: Topping up the New chloride conductivity rig with NaCl solution

Preparation of the test specimens was carried out as per the UCT 2010 DI Manual. The CCI test was then conducted. Four specimens were tested for each concrete mix. First, the specimen was placed in the UCT rig and readings taken. Immediately thereafter it was placed in the New rig and readings were taken. The next specimen underwent the same procedure but with the order of testing reversed, i.e. place the specimen in the New rig, take readings and immediately after, place the specimen in the UCT rig and take readings. This was done to minimise any effect of time between shifting the specimen from one rig to the other. In this way the apparati performed the tests on the same specimen. The time taken between shifting the specimen from one rig to another and taking readings was roughly within two minutes.

The results obtained were analysed statistically to determine any differences. This is important because different users need to be confident that the results they are getting with a particular test rig are comparable with others.

### 3.5.6. The Use of a Chloride Conductivity Calibration Disc

During the course of the CCI rig tests, it was noted that the different rigs were providing different results. The need for calibration of different rigs was apparent. At the time of experimentation, there was no standard way of ensuring that different conductivity rigs produce the same readings. As a starting point in addressing this issue it was decided to experiment with different materials to be used as a calibration disc in the hope that the readings obtained from this disc would not change over time.

The criteria for the choice of material were as follows:

- It needs to be readily available
- It needs to be inexpensive
- It needs to provide consistent readings over time
- It needs to be suitably porous and penetrable to produce conductivity results comparable to typical concretes (in the range of 0.5 – 2.5 mS/cm)

The specimen would need to be the same diameter as a typical concrete specimen that is tested in the chloride conductivity rig (70 ± 2 mm). The thickness of the specimen was not as important as the chloride conductivity rig is known to be less sensitive to variations in thickness of specimen between 20 – 30 mm (Streicher and Alexander, 1999).

Three materials were tested:

- A meranti hardwood – this is readily available for purchase and easily manufactured.
- A mortar mix with the following properties: CEM I 52.5N at a w/c 0.65.
- A ceramic tile – DICAR® manufactured by SMEG. This tile is typically used as a heat diffuser on stove tops to keep cooking vessels hot (see Figure 3.10).

**Figure 3.10: SMEG ceramic tile with 70 mm diameter disc specimen cut out - Top (left image) and Bottom (right image)**

These three materials were cored to obtain circular discs of diameter 70 mm. one disc per material was produced. They were then preconditioned according to the DI sample preparation method for the chloride conductivity test. This involved placing the specimens in an oven at 50 °C for seven days to remove excess moisture and evacuate the pores, then vacuum saturating them with 5M NaCl solution. They were then tested with the UCT chloride conductivity rig as well as the New chloride conductivity rig which was used in this work. After the initial tests it was determined that the meranti and mortar specimens were not suitable for use due to problems saturating the meranti disc and high conductivities for both materials. Therefore, further testing was performed on the ceramic disc only. It was then left to soak in a NaCl solution to ensure that it was always saturated chloride ions and was tested on a weekly basis in the UCT rig to determine its stability in a chloride solution over time.

## 3.6. Statistical Analysis of Results

Several aspects of this work required deeper analysis of the results because significant differences could not be identified by just observing trends in the graphs. The results obtained from the experimental work therefore needed to be analysed statistically to make decisions. Statistical tests were carried out on:

- The DI results from the different drying regimes to determine if drying method and time had a significant effect on results.
- The CCI results of the New and UCT rigs to determine if the different rigs produced equivalent results.

A brief overview of the statistical tools that were used is provided below from Walpole et al. (2012).

### 3.6.1. Statistical Tests Available

### 3.6.2. Hypothesis testing

A hypothesis test is done to test a claim about a particular sample. In the context of this study a sample represents a group of test determinations, for example the OPI results of the 4 test determinations represent a sample, with the sample size of 4. The sample mean and sample standard deviation can be calculated.

In this study the hypothesis to be tested is that the DI tests are influenced by the drying regime used on the specimens. A null hypothesis, $H_0$, refers to a hypothesis which is to be tested. The alternative hypothesis, $H_1$, is usually the logical complement of $H_0$. Therefore $H_0$ is that there is no difference in DI results obtained using different drying methods; $H_1$ is that there is a difference in DI results obtained using different drying methods.

A statistical test described in the following sections can then be used to test whether to accept the null hypothesis for a given significance level.

The significance level, $\alpha$, is the probability of rejecting a null hypothesis when it is true.

### 3.6.3. Analysis-of-Variance (ANOVA)

Where there are more than two samples being tested, such as the comparison of the isopropanol drying method with the two oven drying methods, the ANOVA technique may be employed to test the differences between means. This method assumes that the samples are normally distributed. The method tests the null hypothesis that all means are equal for the samples in question. If at least one of the means is not equal to the others then the null hypothesis is rejected. ANOVA does not identify which means are different.

### 3.6.4. Kruskal Wallis Test

As explained in the previous section, ANOVA is used to tests the equality of means when there are more than two samples being compared, as is the case when comparing the DI results for the specimens dried under the three methods - isopropanol, oven drying for 7 days and oven drying to constant mass. The ANOVA had the assumption that the test sample was normally distributed. When this does not hold, the non-parametric counterpart for the ANOVA test, called the Kruskal-Wallis or H-Test, can be used. A non-parametric method is one that does not assume a specific distribution of the underlying populations. The H-test is a generalisation of the rank-sum test (see Section 3.6.5) for the case where more than two samples are being compared. It is used to test the null hypothesis $H_0$ that the samples being tested are from identical populations.

Let $k$ be the total number of samples and $n_i$ be the number of determinations (i.e. sample size) in the $i$th sample, where $i = 1,2,\ldots,k$. The total number of determinations for all samples is given by $n = n_1 + n_2 + \ldots + n_k$. The determinations from all the samples are combined in ascending order and each determination is assigned a rank. The sum of the ranks for the *i*th sample is given by $R_i$. The *H*-statistic can then be calculated as:

$$H = \frac{12}{n(n+1)} \sum_{i=1}^{k} \frac{R_i^2}{n_i} - 3(n+1) \tag{3.2}$$

This is approximated well by a chi-squared distribution with $k$-1 degrees of freedom, shown in Table E.2 in Appendix E, from which $H_{crit}$ can be obtained. If $H > H_{crit}$ then $H_0$ is rejected in favour of $H_1$.

### 3.6.5. Rank-Sum Test

Where two samples that are non-normal are being tested, the rank-sum test is an appropriate non-parametric alternative to the t-test. The two samples are sorted into ascending order and ranks from 1 – 8 are assigned. The ranks for each sample are then added up. The u-values are then calculated from equations 3.3 and 3.4.

Let $n_1$ and $n_2$ be the number of observations from the smaller and larger sample respectively. In the case where sample sizes are equal, $n_1$ and $n_2$ can be used for either sample. The determinations from both samples are combined and ranked in ascending order. The ranks of the determinations from sample 1 are summed, and is denoted by $w_1$. Likewise, the ranks for sample 2 are summed and represented by $w_2$.

$$w_1 = \text{ sum of ranks of sample 1} \quad ; \quad w_2 = \text{ sum of ranks of sample 2} \tag{3.3}$$

$$u_1 = w_1 - \frac{n_1(n_1+1)}{2} \quad ; \quad u_2 = w_2 - \frac{n_2(n_2+1)}{2} \tag{3.4}$$

The smaller value of $u_1$ and $u_2$ is taken as U and compared with a critical value of $U_{crit}$ obtained from Table E.3 in Appendix E. If $U < U_{crit}$ then the null hypothesis is rejected.

### 3.6.6. t-Test

For small sample sizes (n<30) which have the underlying assumption of normality, the t-distribution may be used to describe the sample. The t-statistic for two samples is given by:

$$t = \frac{(\bar{x}_1 - \bar{x}_2)}{\sigma_p \sqrt{\frac{1}{n_1} + \frac{1}{n_2}}} \tag{3.5}$$

$$\sigma_p^2 = \frac{(n_1 - 1)\sigma_1^2 + (n_2 - 1)\sigma_2^2}{n_1 + n_2 - 2} \tag{3.6}$$

where: $\bar{x}_1 ; \bar{x}_2$ = mean for sample 1 and sample 2 respectively

$\sigma_1$ = sample 1 standard deviation

$\sigma_2$ = sample 2 standard deviation

$\sigma_p$ = pooled standard deviation

$n_1;n_2$ = size of sample 1 and sample 2 respectively

For the t-test, the value of $t$ is compared with a critical value $t_{\text{crit}}$ obtained from Table E.1 in Appendix E. If $|t| > t_{crit}$ then the null hypothesis is rejected.

### 3.6.7. Statistical Tests Chosen

A total of 12 mixes were cast, each varying some mix parameter (w/c ratio, binder type). Three treatments were employed in the preconditioning stage of the DI Tests. Each DI test had 4 specimens. So each test had a sample size of 4.

**Table 3.9: Test Sample Size**

| Sample | Treatment | Sample size $n$ |
|---|---|---|
| 1 | Oven dried for 7 days | 4 |
| 2 | Oven dried to constant mass | 4 |
| 3 | Isopropanol dried | 4 |

The permeability coefficient (k-value) obtained in the OPI test is known to follow a log-normal distribution (Neves et al., 2011; Nganga, 2012). This eliminates the suitability of statistical tests based on normality (ANOVA, t-test). Furthermore, the sample size for each test is less than 30, so it can be considered to be small. The appropriate statistical tools for this scenario are the non-parametric Kruskal-Wallis test and the Rank Sum test. For this study all the statistical tests were done using non-parametric tests.

It will be noted from the results in Chapter 4 that some of the results from the non-parametric tests were difficult to interpret. Therefore, it was decided to also test the OPI, CCI and WSI results using the t-test to verify the non-parametric test results. The OPI, CCI and WSI tests are assumed to be normally distributed, therefore it is acceptable to use the t-test.

### 3.6.8. Statistical Test Procedure

The following steps were performed when conducting the statistical tests on the data in question:

1. State null hypothesis; $H_0$: There is no difference between the DI results from drying for 7 days and drying to constant mass.
2. State alternative hypothesis $H_1$: There is a difference between the DI results obtained from drying for 7 days and drying to constant mass.
3. Select significance level; $\alpha = 0.05$ i.e. there is a 5% probability of rejecting the null hypothesis when it is true.
4. Test statistic:
    a. Kruskal-Wallis Test : H
    b. Rank-Sum test : U
    c. t-test: t
5. Critical region: obtained from Statistical Tables in Apendix
    - If $H < H_{crit}$ then fail to reject null hypothesis, $H_0$.
    - If $H > H_{crit}$ then reject null hypothesis in favour of alternative hypothesis, $H_1$.
    - If $U < U_{crit}$ then fail to reject null hypothesis, $H_0$.
    - If $U > U_{crit}$ then reject null hypothesis in favour of alternative hypothesis, $H_1$.
    - If $|t| < t_{crit}$ then fail to reject null hypothesis, $H_0$.
    - If $|t| > t_{crit}$ then reject null hypothesis in favour of alternative hypothesis, $H_1$.

### 3.6.9. Worked Examples

The following examples illustrate the above procedures performed. The results used are taken from the DI tests performed on the CEM I 52.5N binder at a w/c ratio of 0.40.

**Comparing k-values from the different oven drying treatments using the H-test.**

1. Null hypothesis, $H_0$, the means of the three samples are equivalent
2. Alternative hypothesis, $H_1$, the means of the three samples are not equivalent
3. $\alpha = 0.05$
4. Statistical test: H-test
5. Critical region: Reject H if $H > H_{crit} = 5.991$ (See Appendix E)

The total number of determinations is 12 and each is assigned a rank after sorting in ascending order. The ranks for each sample are then summed and squared ($R^2$) as shown in Table 3.10.

**Table 3.10: Statistical treatment of k-values for CEM I 52.5N w/c ratio 0.40 using the Kruskal-Wallis test**

| Treatment | Determination | k (m/s) | Rank | $R^2$ |
|---|---|---|---|---|
| **7 Day Oven Dry** | 1 | 4.48E-11 | 2 | 484 |
| | 2 | 5.40E-11 | 4 | |
| | 3 | 5.49E-11 | 5 | |
| | 4 | 1.24E-10 | 11 | |
| **Oven Dry to Constant Mass** | 1 | 1.17E-10 | 10 | 1296 |
| | 2 | 7.72E-11 | 8 | |
| | 3 | 5.70E-11 | 6 | |
| | 4 | 1.26E-10 | 12 | |
| **Isopropanol dried** | 1 | 5.24E-11 | 3 | 400 |
| | 2 | 8.90E-11 | 9 | |
| | 3 | 2.86E-11 | 1 | |
| | 4 | 5.91E-11 | 7 | |

$$H = \frac{12}{n(n+1)} \sum_{i=1}^{3} \frac{R_i^2}{n_i} - 3(n+1)$$

$$H = \frac{12}{12(13)} \left( \frac{484}{4} + \frac{400}{4} + \frac{1296}{4} \right) - 3(13)$$

$H = 2.92 < 5.991$ ; **$H_0$ is accepted.** i.e. the means of the 3 samples are equivalent.

### Comparing k-values from the different oven drying durations using the rank-sum test

Table 3.11 shows the permeability k-values used in this example. These results are for the CEM I 52.5N binder with a w/c ratio of 0.4.

1. Null hypothesis, $H_0$, the means of the two samples are equivalent
2. Alternative hypothesis, $H_1$, the means of the two samples are not equivalent
3. $\alpha = 0.05$
4. Statistical test: rank-sum test
5. Critical region: Reject $H_0$ if $U < U_{crit} = 1$

**Table 3.11: Statistical treatment of k-values for CEM I 52.5N w/c ratio 0.40 using the rank-sum test**

| Treatment | Determination | k (m/s) | Rank |
|---|---|---|---|
| **7 Day Oven Dry** | 1 | 4.48E-11 | 1 |
| | 2 | 5.40E-11 | 2 |
| | 3 | 5.49E-11 | 3 |
| | 4 | 1.24E-10 | 7 |
| **Oven Dry to Constant Mass** | 1 | 1.17E-10 | 6 |
| | 2 | 7.72E-11 | 5 |
| | 3 | 5.70E-11 | 4 |
| | 4 | 1.26E-10 | 8 |

$$w_1 = 1+2+3+7 = 13 \quad ; \quad w_2 = 6+5+4+8 = 23$$

$$u_1 = w_1 - \frac{n_1(n_1+1)}{2} \quad ; \quad u_2 = w_2 - \frac{n_2(n_2+1)}{2}$$

$$u_1 = 13 - \frac{4(5)}{2} \quad ; \quad u_2 = 23 - \frac{4(5)}{2}$$

$$u_1 = 3 \quad ; \quad u_2 = 13$$

U = 3 > 1; **$H_0$ is accepted.** i.e. the means of the 3 samples are equivalent.

**Comparing OPI results from the different oven drying durations using the t-test**

The OPI test will be used for the example here since the assumption of normality holds for OPI results.

1. Null hypothesis, $H_0$, the means of the three samples are equivalent
2. Alternative hypothesis, $H_1$, the means of the three samples are not equivalent
3. $\alpha = 0.05$
4. Statistical test: t-test
5. Critical region: Reject H if $|t|>t_{crit}=2.447$ (From Table E.1 $n_1+n_2-2=6$ degrees of freedom, α adjusted for two-tailed test)

Table 3.12 shows the OPI results used in this example. The CEM I 52.5N binder with a w/c ratio of 0.4 is again used for this example.

**Table 3.12:Statistical treatment of OPI results for CEM I 52.5N w/c ratio 0.40 using the rank-sum test**

| Determination | 7 Day oven dry | Oven dry to constant mass |
|---|---|---|
| 1 | 10.35 | 9.93 |
| 2 | 10.27 | 10.11 |
| 3 | 10.26 | 10.24 |
| 4 | 9.91 | 9.90 |
| Mean | 10.20 | 10.05 |
| Standard deviation | 0.1959 | 0.1597 |

$$\sigma_p^2 = \frac{(n_1 - 1)\sigma_1^2 + (n_2 - 1)\sigma_2^2}{n_1 + n_2 - 2}$$

$$\sigma_p^2 = \frac{(4-1)0.1959^2 + (4-1)0.1597^2}{4+4-2}$$

$$\sigma_p^2 = 0.0319$$

$$\sigma_p = 0.1787$$

$$t = \frac{(10.20 - 10.05)}{0.18\sqrt{\frac{1}{4} + \frac{1}{4}}}$$

$|t| = 1.207 < 2.447$; **$H_0$ is accepted**

### 3.7. Summary

This chapter explained the experimental programme undertaken in the laboratory aiming to investigate various issues surrounding the DI tests. In particular the issue of the preconditioning of the test specimens involving drying and vacuum saturation, and the use of different CCI rigs was investigated. The statistical tools used to analyse the results were also presented. It is hoped that the result of this leads to an improved understanding of these issues so as to improve the DI approach.

# 4. EXPERIMENTAL RESULTS AND DISCUSSION

## 4.1. Introduction

This chapter presents and discusses the detailed results from the experimental work described in Chapter 3. The compressive strength results are presented to show the consistency of the mixes used. The results relating to the DI performance of the specimens that were exposed to different drying regimes are presented. The DI tests performed on these specimens are analysed to infer relationships. The saturation procedures for the chloride conductivity rig and the WSI test are also discussed. The New chloride conductivity rig is tested against the UCT version to assess the differences in their results, if any. Following this, the need for a method of calibration of different chloride conductivity rigs is presented and various options to achieve this are investigated. The three DI test methods are then discussed to address outstanding issues.

## 4.2. Compressive Strength Tests on Concrete Mixes

A total of 12 mixes with various binders and w/c ratios were cast. The 28-day results obtained from the compressive strength tests are shown in Figure 4.1 and Table 4.1.

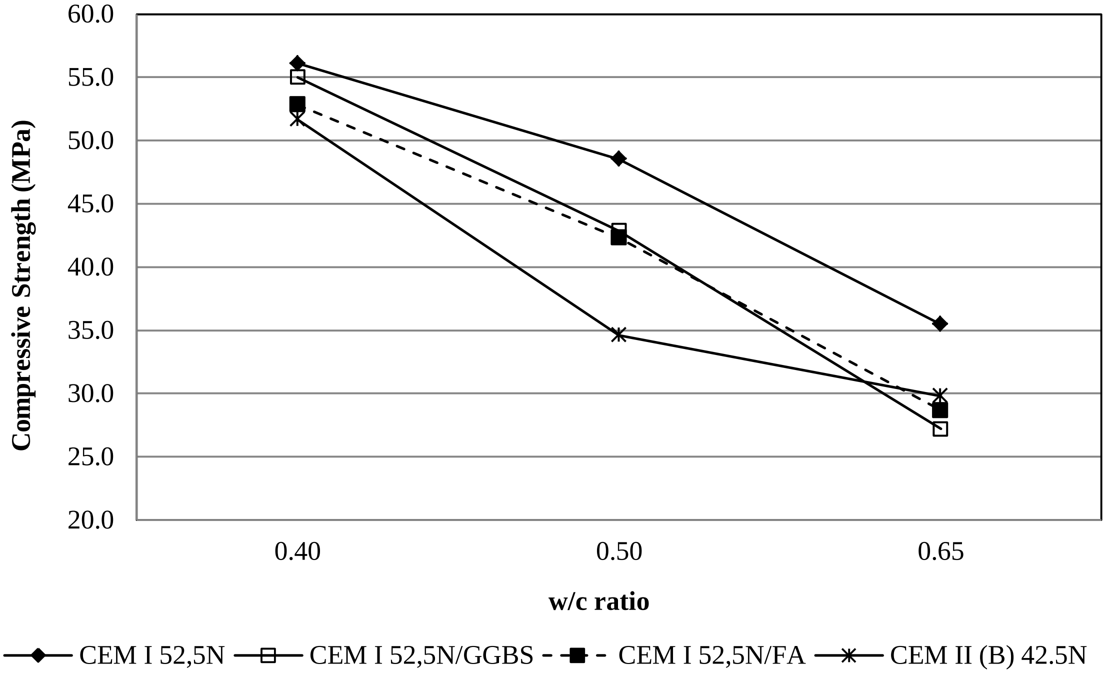

**Figure 4.1: 28-Day compressive strength results**

**Table 4.1: 28-day compressive strength values**

| Binder | w/c | Cube number | Density (kg/m$^3$) | $f_{cu}$ (MPa) | Mean $f_{cu}$ (MPa) | Standard Deviation (MPa) | CoV (%) |
|---|---|---|---|---|---|---|---|
| CEM I 52.5N | 0.40 | 1 | 2405 | 55.2 | 56.1 | 1.5 | 2.6 |
| | | 2 | 2440 | 57.8 | | | |
| | | 3 | 2405 | 55.3 | | | |
| | 0.50 | 1 | 2400 | 48.2 | 48.5 | 0.9 | 1.8 |
| | | 2 | 2415 | 49.5 | | | |
| | | 3 | 2405 | 47.9 | | | |
| | 0.65 | 1 | 2495 | 35.3 | 35.5 | 1.1 | 3.0 |
| | | 2 | 2375 | 36.6 | | | |
| | | 3 | 2490 | 34.5 | | | |
| CEM I 52.5N/ GGBS | 0.40 | 1 | 2445 | 55.2 | 55.0 | 0.2 | 0.3 |
| | | 2 | 2370 | 54.9 | | | |
| | | 3 | 2435 | 54.9 | | | |
| | 0.50 | 1 | 2465 | 42.3 | 42.9 | 0.5 | 1.2 |
| | | 2 | 2420 | 43.1 | | | |
| | | 3 | 2420 | 43.2 | | | |
| | 0.65 | 1 | 2410 | 27.5 | 27.2 | 2.0 | 7.2 |
| | | 2 | 2380 | 25.1 | | | |
| | | 3 | 2375 | 29.0 | | | |
| CEM I 52.5N/ FA | 0.40 | 1 | 2400 | 52.0 | 52.8 | 0.8 | 1.4 |
| | | 2 | 2390 | 53.0 | | | |
| | | 3 | 2485 | 53.5 | | | |
| | 0.50 | 1 | 2435 | 42.3 | 42.3 | 0.1 | 0.2 |
| | | 2 | 2426 | 42.4 | | | |
| | | 3 | 2435 | 42.2 | | | |
| | 0.65 | 1 | 2375 | 28.0 | 28.7 | 0.6 | 2.0 |
| | | 2 | 2415 | 28.9 | | | |
| | | 3 | 2425 | 29.1 | | | |
| CEM II (B) 42.5N | 0.40 | 1 | 2395 | 53.5 | 51.7 | 1.6 | 3.1 |
| | | 2 | 2400 | 51.0 | | | |
| | | 3 | 2400 | 50.5 | | | |
| | 0.50 | 1 | 2410 | 33.5 | 34.6 | 1.3 | 3.7 |
| | | 2 | 2405 | 34.3 | | | |
| | | 3 | 2410 | 36.0 | | | |
| | 0.65 | 1 | 2410 | 29.5 | 29.8 | 1.1 | 3.6 |
| | | 2 | 2420 | 31.0 | | | |
| | | 3 | 2435 | 28.9 | | | |

The compressive strength results show that the cube strength decreases for all binders as the w/c ratio is increased. There is an increased spread in compressive strength for the different binders at the w/c ratio of 0.5, particularly due to result of the CEM II (B) 42.5N binder which is lower than the other binders at the corresponding w/c ratio. The variability, as measured by the coefficient of variation, of the compressive strength and the densities of the cubes are all comparable, indicating good consistency of the mixes.

## 4.3. Drying Tests

### 4.3.1. Drying Regime

The Durability Index test procedures require the specimens to undergo a preconditioning treatment involving their placement in an oven for 7 days to remove the moisture. Chapter 2 made reference to the need for moisture to be removed from specimens before conducting permeability tests as moisture effectively blocks the transport of gas through the pore structure of the concrete. Furthermore moisture present in the concrete pores could also have a diluting effect of the NaCl solution used to saturate test specimens for chloride conductivity testing.

Chapter 3 described the methods used in this work to dry the specimens using the oven drying method as well as an alternative drying method using isopropanol. For each regime, the average mass of eight specimens was recorded during the drying period. This was appropriate given the low variability of the drying between the eight specimens, as shown in Table 4.2. The exception was the relatively high variability of the isopropanol-dried CEM I 52.5N specimens at a w/c ratio of 0.4. It is unclear what caused the relatively high variability in the mix. The coefficient of variability was based on the average of the percentage mass loss and standard deviation of the percentage mass loss of the eight specimens.

Figures 4.2 and 4.3 show the time taken for the moisture to be removed by the oven drying and the isopropanol drying methods for specimens made with different binders. The drying curves are represented by a mass loss relative to the un-dried mass at day 0 ($M_d/M_i$) rather than in absolute terms. This was done because the specimens would each have different initial masses due to their different densities resulting from variable amounts of coarse aggregate present in each specimen. Even with correct compaction, it is very unlikely that specimens 70 mm in diameter and 30 mm thick would have the same distribution of coarse aggregate. The slopes of the graphs indicate that in the first few days there is a high rate of moisture loss, followed by a decrease in the rate of moisture loss as time proceeds. This can be attributed to the moisture loss from the saturated pores close to the surface of the concrete specimens. The moisture that is held deeper in the specimens is more difficult to remove and therefore takes longer.

The CEM I 52.5N isopropanol drying curves for, both the 0.4 and 0.5 w/c ratios are similar. A larger amount of moisture loss would be expected at the higher w/c ratio. The rest of the curves for the other binder types all show different rates of mass loss, as would be expected since they would have different pore densities. The higher w/c ratio concretes generally lost more moisture as a result of the higher evaporable water content.

The drying curves indicate that there is a clear difference between the mass loss due to the two drying regimes. The isopropanol-dried specimens for all the mixes showed less total mass loss than their oven-dried counterparts. Both regimes reached a constant mass state (defined in this study as a mass loss of 0.05% or less over a 24 hour period) after at least 12 days. It is worth noting that there is still appreciable slope at this stage, indicating that drying was still taking place, albeit very slowly. The time to completion of drying is shown in Table 4.2.

**Table 4.2: Drying time and variability of the isopropanol and oven drying regimes**

| Drying Regime | Binder | w/c | Days to complete drying | [a]Average Mass-loss (%) | Standard deviation (%) | CoV |
|---|---|---|---|---|---|---|
| Oven Dried | CEM I 52.5N | 0.4 | 13 | 3.34 | 0.25 | 7.53 |
| | | 0.5 | 17 | 3.63 | 0.33 | 9.07 |
| | | 0.65 | 12 | 3.67 | 0.18 | 4.94 |
| | CEM I 52.5N/GGBS | 0.4 | 16 | 2.89 | 0.18 | 6.11 |
| | | 0.5 | 14 | 3.06 | 0.16 | 5.21 |
| | | 0.65 | 15 | 3.70 | 0.34 | 9.28 |
| | CEM I 52.5N/FA | 0.4 | 15 | 3.06 | 0.18 | 6.04 |
| | | 0.5 | 17 | 3.33 | 0.14 | 4.12 |
| | | 0.65 | 14 | 3.86 | 0.13 | 3.42 |
| | CEM II (B) 42.5N | 0.4 | 16 | 3.05 | 0.17 | 5.43 |
| | | 0.5 | 16 | 3.63 | 0.26 | 7.22 |
| | | 0.65 | 17 | 3.68 | 0.16 | 4.25 |
| Isopropanol Dried | CEM I 52.5N | 0.4 | 11 | 2.50 | 0.44 | 17.61 |
| | | 0.5 | 13 | 2.05 | 0.19 | 9.35 |
| | | 0.65 | 15 | 2.86 | 0.15 | 5.16 |
| | CEM I 52.5N/GGBS | 0.4 | 17 | 1.58 | 0.09 | 5.55 |
| | | 0.5 | 14 | 1.81 | 0.11 | 5.97 |
| | | 0.65 | 12 | 2.37 | 0.07 | 3.11 |
| | CEM I 52.5N/FA | 0.4 | 17 | 1.74 | 0.13 | 7.44 |
| | | 0.5 | 14 | 2.42 | 0.17 | 7.08 |
| | | 0.65 | 17 | 3.00 | 0.18 | 5.95 |
| | CEM II (B) 42.5N | 0.4 | 16 | 1.77 | 0.10 | 5.64 |
| | | 0.5 | 16 | 2.36 | 0.12 | 5.22 |
| | | 0.65 | 16 | 2.53 | 0.23 | 8.99 |

[a] *mass loss as a percentage of the initial mass.*

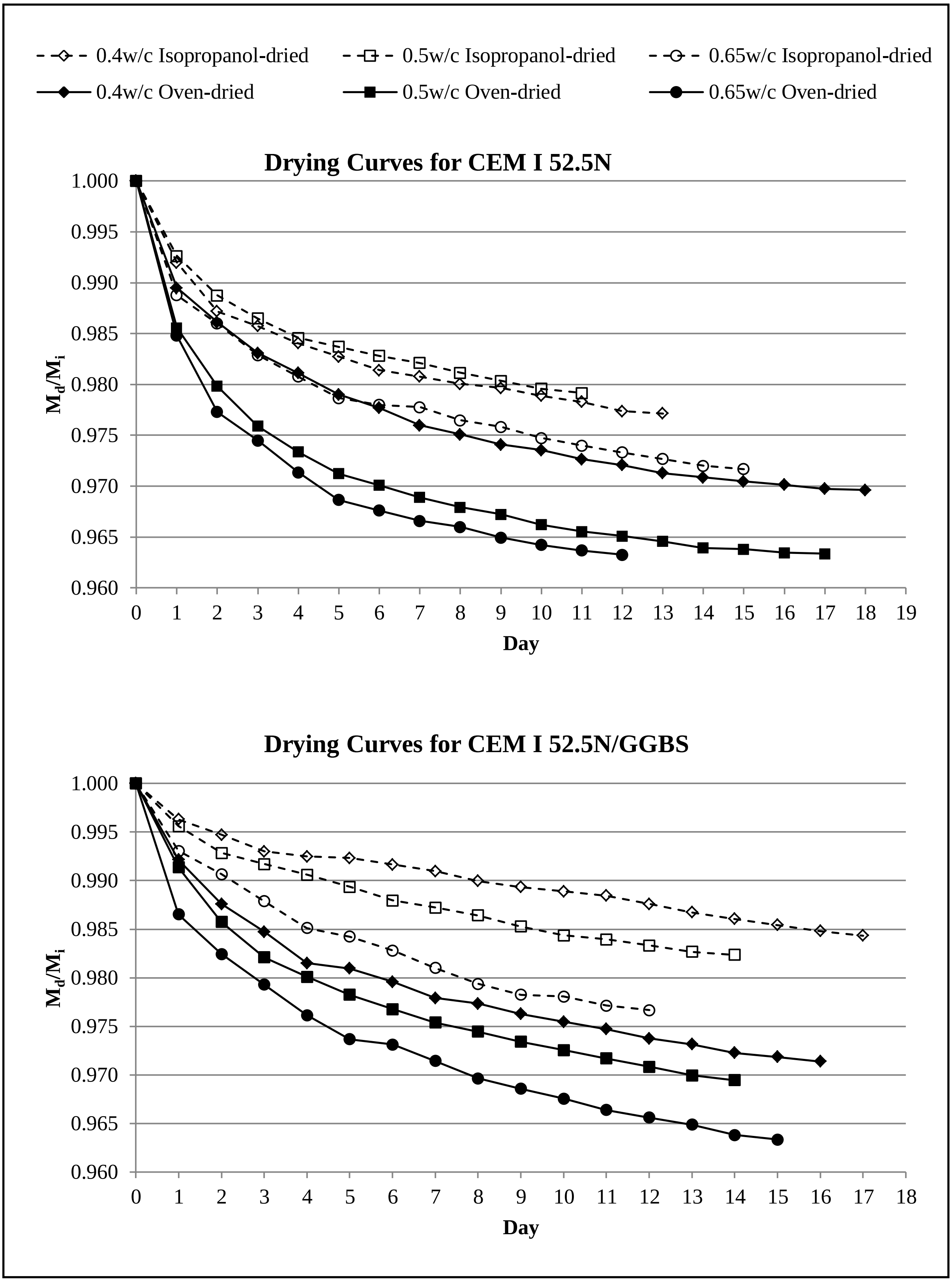

**Figure 4.2: Mass changes due to drying for CEM I 52.5N and GGBS extended binders at various w/c ratios**

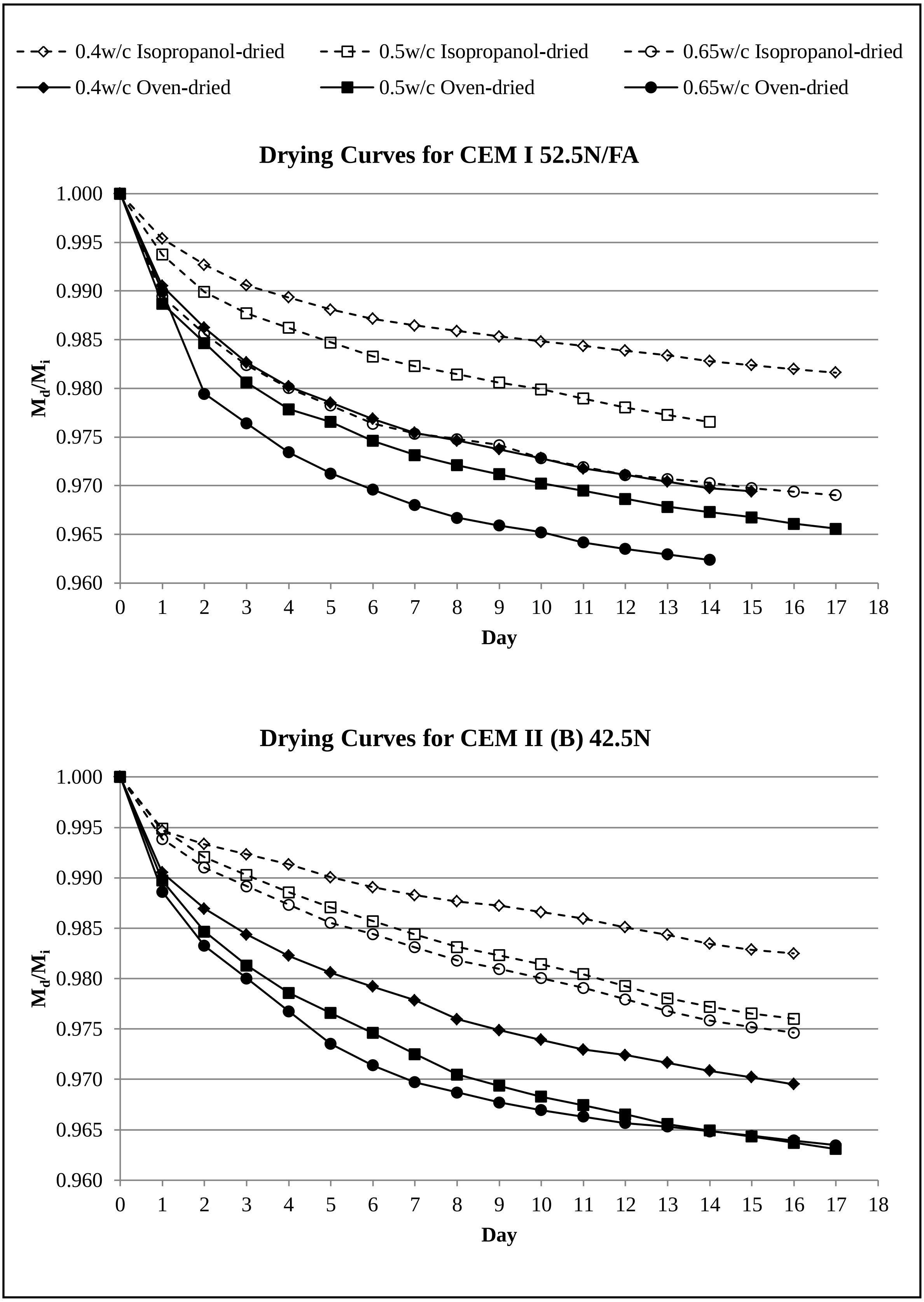

**Figure 4.3: Mass changes due to drying for CEM I 52.5N/FA and CEM II (B) 42.5N binders at various w/c ratios**

It may also be plausible that more micro-cracking could have been induced in the pore structure of the oven-dried specimens than the isopropanol-dried specimens thereby allowing excess moisture to be removed. The solvent replacement method has been shown in the Literature Review to be a more gentle method than oven drying. It was assumed that the saturation procedure to introduce the isopropanol into the concrete produced fully saturated specimens. As a result, all the water in the specimens would have mixed with the isopropanol before placing the specimens in the constant 30 °C environment. However, if full saturation did not occur, it may be possible that the isopropanol was not successful in replacing all the water in the concrete pores. Therefore the mild temperature that the specimens were kept at would be effective in evaporating the isopropanol but not sufficiently high enough to remove any residual water trapped in the pores.

### 4.3.2. Oven Drying Time

Comparisons were made between the average mass loss after 7 days and the mass loss after being dried to constant mass as defined by a 0.05% mass loss over a 24 hour period. In terms of this definition, Figures 4.2 and 4.3 show that the time taken to completely dry the specimens in the oven varies from 12 to 18 days depending on the binder type and w/c ratio. From Table 4.3 it can be seen that most of the moisture is removed within the 7 day period. For all the binders and w/c ratios, there was only between a 0.43 – 0.83% additional mass loss relative to the original mass of the specimen after the 7 day drying period, which translates to roughly 80% of the total moisture being lost within the 7 day period. The practical implication of this is that it appears that the 7 day period is sufficient for the majority of the drying to occur. The trade-off between having specimens which are completely dry and the additional time needed to achieve fully dry conditions seems uneconomical as it may cause delays in construction projects. However, it was still necessary to determine whether the additional moisture removed after the 7 day period affected the DI results.

The mean amount of additional mass loss after seven days drying for the different binders shows that CEM I 52.5N lost the least amount of moisture in this time. The GGBS and FA extended binders lost roughly 0.2% and 0.1% more mass respectively than CEM I 52.5N. The ratio of the additional mass loss to the 7-day mass loss (last column in Table 4.3) also shows that there is relatively constant post 7-day mass loss for the blended binders at all w/c ratios compared to the CEM I 52.5N and CEM II (B) 42.5N binders, which exhibit a decreasing trend. This trend can be explained by the higher w/c ratios having more evaporable water held less tightly in the specimens so the majority of it can escape in the early drying period.

**Table 4.3: Mass loss after 7 days oven drying vs. oven drying to constant mass**

| Binder | w/c | % Mass loss after 7 days | % Mass loss after Drying to Constant Mass | Additional % mass loss after 7 days | Mean of (3) | Ratio of (3):(1) % |
|---|---|---|---|---|---|---|
| | | (1) | (2) | (3) | | |
| **CEM I 52.5N** | 0.4 | 2.40 | 2.95 | 0.55 | 0.53 | 26 |
| | 0.5 | 3.11 | 3.62 | 0.51 | | 16 |
| | 0.65 | 3.34 | 3.77 | 0.43 | | 13 |
| **CEM I 52.5N/ GGBS** | 0.4 | 2.21 | 2.81 | 0.61 | 0.70 | 27 |
| | 0.5 | 2.46 | 3.14 | 0.68 | | 28 |
| | 0.65 | 2.86 | 3.67 | 0.81 | | 28 |
| **CEM I 52.5N/ FA** | 0.4 | 2.46 | 3.06 | 0.60 | 0.61 | 24 |
| | 0.5 | 2.69 | 3.32 | 0.64 | | 24 |
| | 0.65 | 3.20 | 3.80 | 0.60 | | 19 |
| **CEM II (B) 42.5N** | 0.4 | 2.22 | 2.98 | 0.76 | 0.70 | 34 |
| | 0.5 | 2.75 | 3.57 | 0.82 | | 29 |
| | 0.65 | 3.03 | 3.56 | 0.53 | | 17 |

A comparison between the two oven drying durations is represented in Figure 4.4. The trend shown in is that more mass loss occurs as the w/c ratio is increased. This can be explained by the lower density of the pore structure of the concretes at the higher w/c ratios. There is less water to be lost at a w/c ratio of 0.4 than 0.5 or 0.65. Figure 4.4 also shows that there is a trend in the blended binders losing more moisture after the 7 day period than the CEM I 52.5N. This is related to a more refined pore structure expected of the concrete made with blended binders.

There is a clear difference between the amounts of moisture lost for the two oven-drying durations. A possible explanation for the long time taken to completely dry the specimens could be due to the “ink bottle effect”. The “ink bottle” effect occurs due to the shape, size and interconnectedness of the pores being non-uniform. The drainage is limited by the smaller pore radius. Applying this to the drying of the concrete specimens, it would mean that most of the water, which is trapped in the larger pores, is easily lost while the little remaining moisture requires a longer time to escape because it is ‘locked’ in the smaller pores.

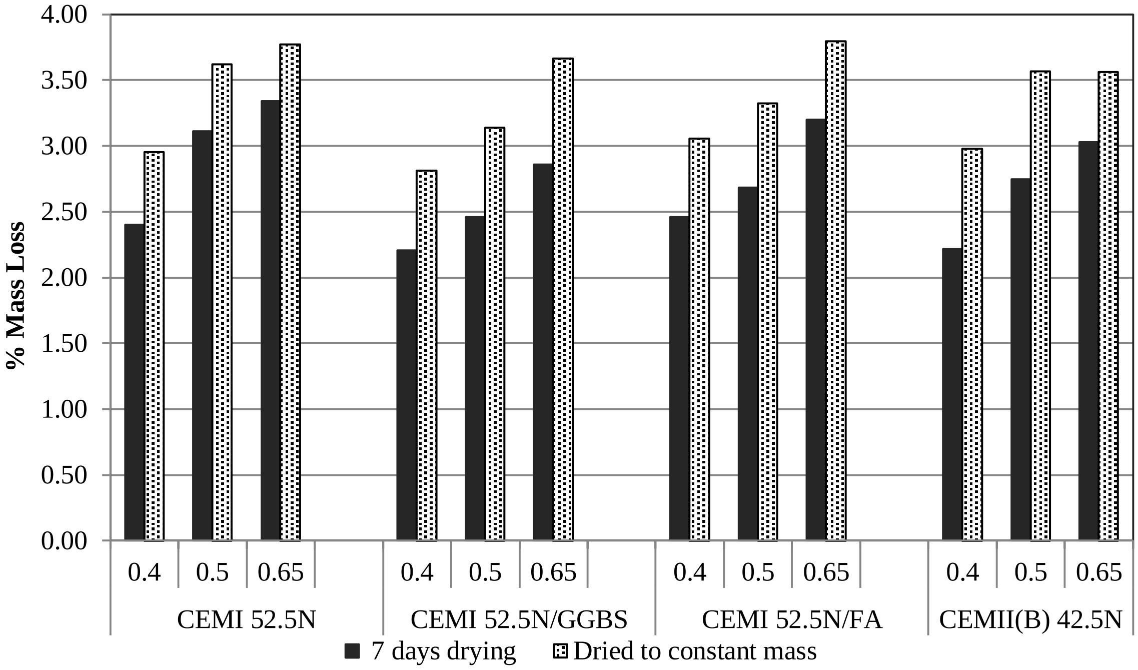

**Figure 4.4: Percentage mass loss for two 50 °C oven drying durations**

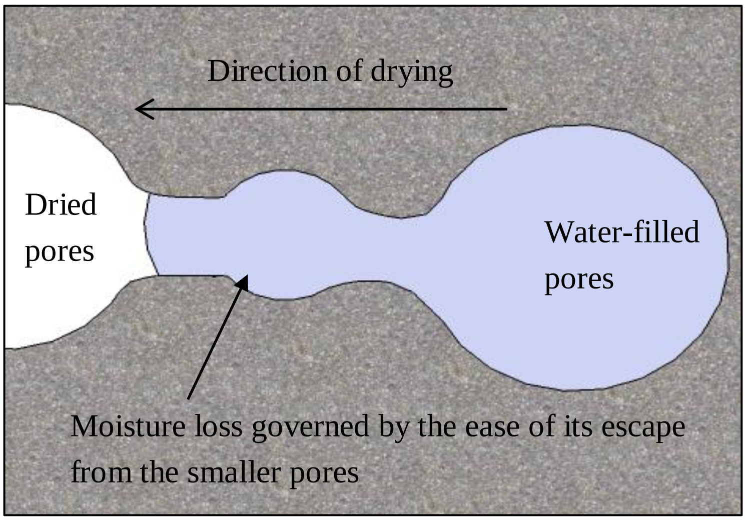

**Figure 4.5: Illustration of the "ink-bottle" effect**

### 4.3.3. Conclusions from Drying Tests

The drying tests performed show that there exist differences in the moisture loss between the isopropanol and oven drying regimes. The isopropanol drying regime results in less overall moisture loss compared to the oven drying method for a particular concrete. Regarding the oven drying regime - although there is some moisture loss after the 7 day period, the majority is lost within 7 days. Thus, from the results shown in the preceding sections, it appears that the current 7 day drying period is adequate. However, the effect on the DI tests of the excess loss of moisture after 7 days oven drying remains to be investigated.

## 4.4. Durability Index Test Results

The motivation for using different drying regimes in the previous section was to determine the effect that different methods of moisture removal had on the Durability Index test results. This required that the specimens that underwent the different drying regimes had to be tested using the OPI, WSI and CCI tests. After the specimens were dried, the procedures set out in the 2010 DI manual were followed. The results are displayed in the following sections.

### 4.4.1. Permeability Coefficient (k-values)

The OPI test provides a measure of permeability. It is calculated as the negative logarithm of the average permeability of the test specimens. The permeability coefficient provides the value of the actual permeability of the specimens. A summary of the k-values is provided in Figure 4.6 for general comparison, showing the results for all the mixes tested. The oven dried to constant mass result for the GGBS mix at the w/c ratio of 0.65 stands out as being particularly high. The concrete pore structure becomes less dense as the w/c ratio increases so a relatively high permeability would be expected for this mix. However, the magnitude of the increase is large compared to the corresponding change for other binders. The variability of the mixes is shown in Table 4.4, where it can be seen that, despite the relatively low coefficient of variation, the standard deviation of this mix is much higher than the other mixes. On this basis, this result will be omitted as a suspected anomaly. The isopropanol dried results, despite showing lower mass loss, still show comparable k-values to the 7-day oven dried results. The k-values for the different w/c ratios were plotted and are shown in Figures 4.7 to 4.10.

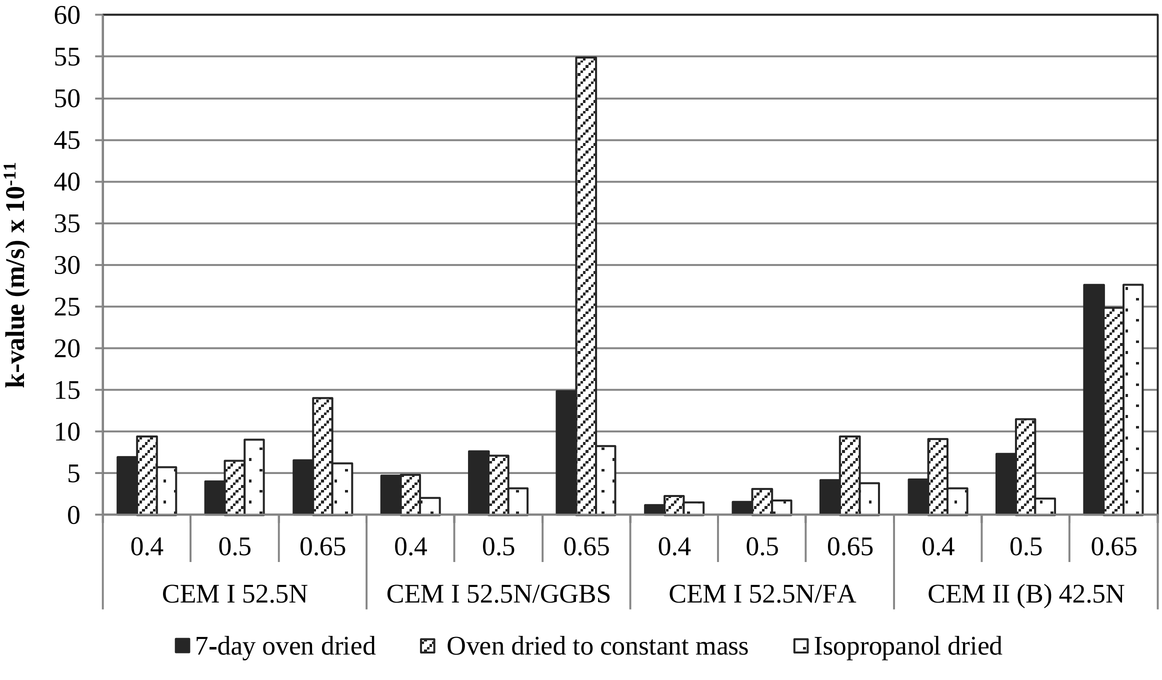

**Figure 4.6: Summary of the k-values**

**Table 4.4: Variability of the k-values**

| Binder | w/c | 7-day oven dried | | | Oven dried to constant mass | | | Isopropanol dried | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | Mean k (m/s) $\times10^{-11}$ | SDEV (m/s) $\times10^{-11}$ | CoV (%) | Mean k (m/s) $\times10^{-11}$ | SDEV (m/s) $\times10^{-11}$ | CoV (%) | Mean k (m/s) $\times10^{-11}$ | SDEV (m/s) $\times10^{-11}$ | CoV (%) |
| CEM I 52.5N | 0.4 | 7 | 3.6 | 53 | 9 | 3.3 | 35 | 6 | 2.5 | 43 |
| | 0.5 | 4 | 0.8 | 19 | 7 | 2.0 | 31 | 9 | 2.3 | 25 |
| | 0.65 | 7 | 2.2 | 34 | 14 | 5.7 | 39 | 6 | 2.2 | 35 |
| CEM I 52.5N/ GGBS | 0.4 | 5 | 2.3 | 49 | 5 | 0.6 | 50 | 2 | 1.4 | 68 |
| | 0.5 | 8 | 2.6 | 34 | 7 | 3.6 | 48 | 3 | 1.2 | 39 |
| | 0.65 | 15 | 4.3 | 29 | 55 | 32.0 | 38 | 8 | 4.6 | 55 |
| CEM I 52.5N/ FA | 0.4 | 1 | 0.3 | 26 | 2 | 1.3 | 37 | 1 | 0.1 | 7 |
| | 0.5 | 2 | 0.4 | 26 | 3 | 0.6 | 16 | 2 | 0.8 | 43 |
| | 0.65 | 4 | 1.4 | 34 | 9 | 1.6 | 65 | 4 | 1.5 | 40 |
| CEM II (B) 42.5N | 0.4 | 4 | 2.2 | 51 | 9 | 6.7 | 74 | 3 | 2.3 | 71 |
| | 0.5 | 7 | 3.9 | 53 | 11 | 6.5 | 57 | 2 | 0.5 | 25 |
| | 0.65 | 28 | 13.7 | 50 | 25 | 16.2 | 65 | 28 | 12.8 | 46 |

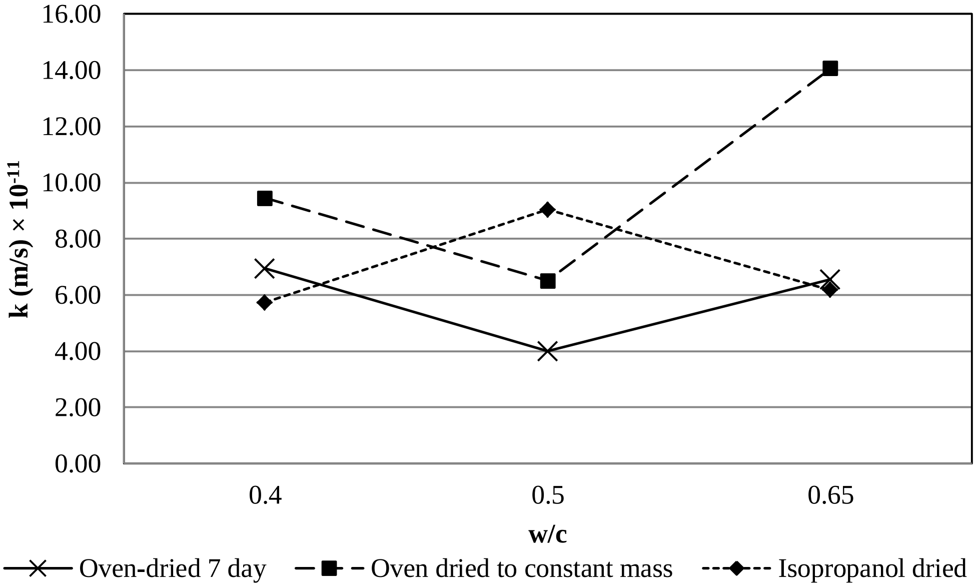

**Figure 4.7: Permeability coefficient results for CEMI 52.5N specimens**

The k-values for the CEM I 52.5N specimens decrease from the 0.4 - 0.5 w/c and then increase again at the 0.65 w/c ratio. The isopropanol dried samples show the opposite trend. This could be a consequence of incomplete saturation with isopropanol of the 0.5 w/c ratio specimens with the, thereby causing incomplete drying. This explanation is supported by Table 4.2 and Figure 4.2 which show the drying results for the isopropanol dried specimens for the two w/c ratios. It can be seen that the drying curves for the 0.4 and 0.5 w/c ratios were very similar. There was also less moisture loss for the 0.5 w/c ratio than the 0.4 w/c ratio mix. This was not the case for the oven dried specimens, or for isopropanol dried specimens from the other binders. The isopropanol results for the 0.5 w/c ratio may therefore be called into question.

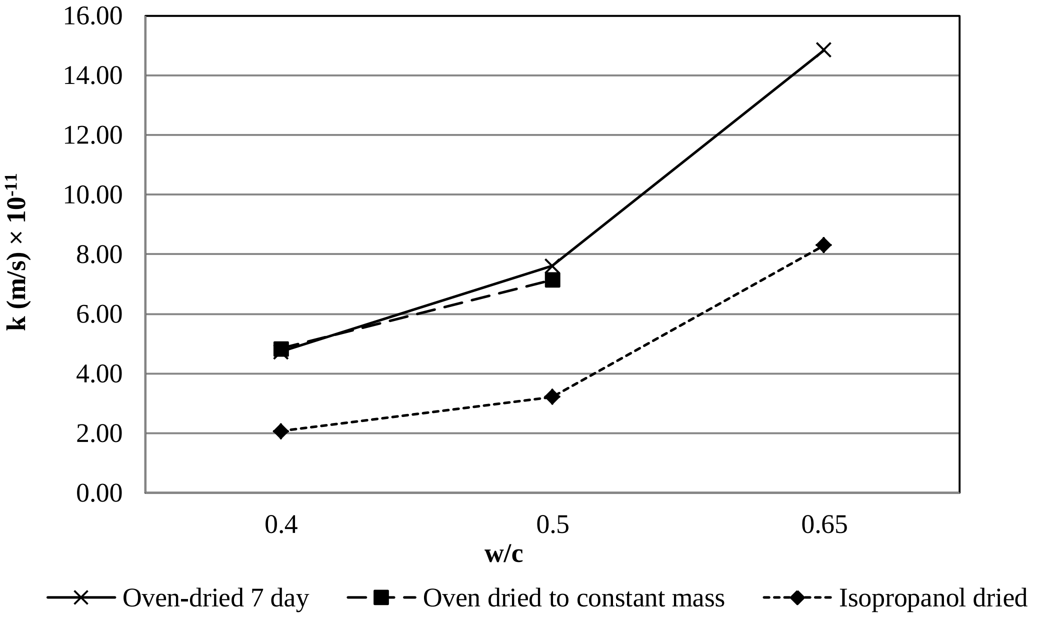

**Figure 4.8: Permeability coefficient results for CEMI 52.5N/GGBS specimens**

Figure 4.8 shows that there are differences in the k-values obtained when using the different drying options for the GGBS blended binders. The oven drying results were similar to each other for the w/c ratios of 0.4 and 0.5, indicating that prolonged drying in the oven has little effect on these mixes. The oven dried to constant mass result for the 0.65 w/c ratio was omitted as previously mentioned, due to it being a suspected outlier. Although permeability is known to generally be variable, the standard deviation for this mix was particularly high (shown in Table 4.4). The mean k-value for this mix was also much higher than any of the other mixes tested. Furthermore, this result does not correlate with the moisture loss shown in Figure 4.2. The isopropanol-dried k-values were slightly lower than the oven-dried samples for all three w/c ratios possibly indicating that the microstructure was different as a result of drying.

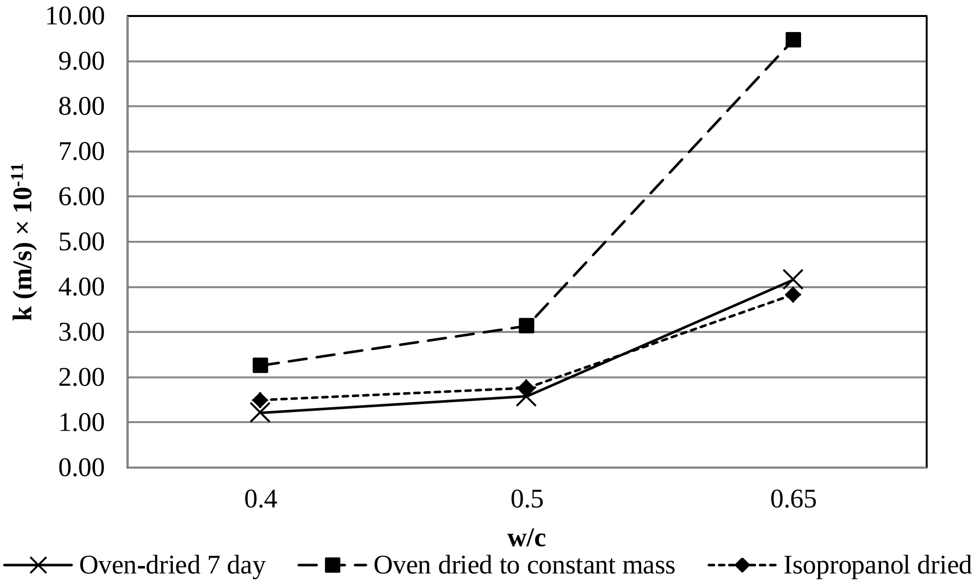

**Figure 4.9: Permeability coefficient results for CEMI 52.5N/FA specimens**

For the CEM I 52.5/FA binder, the isopropanol results more closely resembled the 7-day oven dried results. Both these drying methods show lower permeability than the method of oven drying to constant mass. The FA-containing mixes were generally less permeable than the other concrete mixes tested, as shown by permeability coefficients of the order of magnitude $10^{-11}$ for the FA mixes compared with $10^{-10}$ for the other binder types, indicating a denser pore structure.

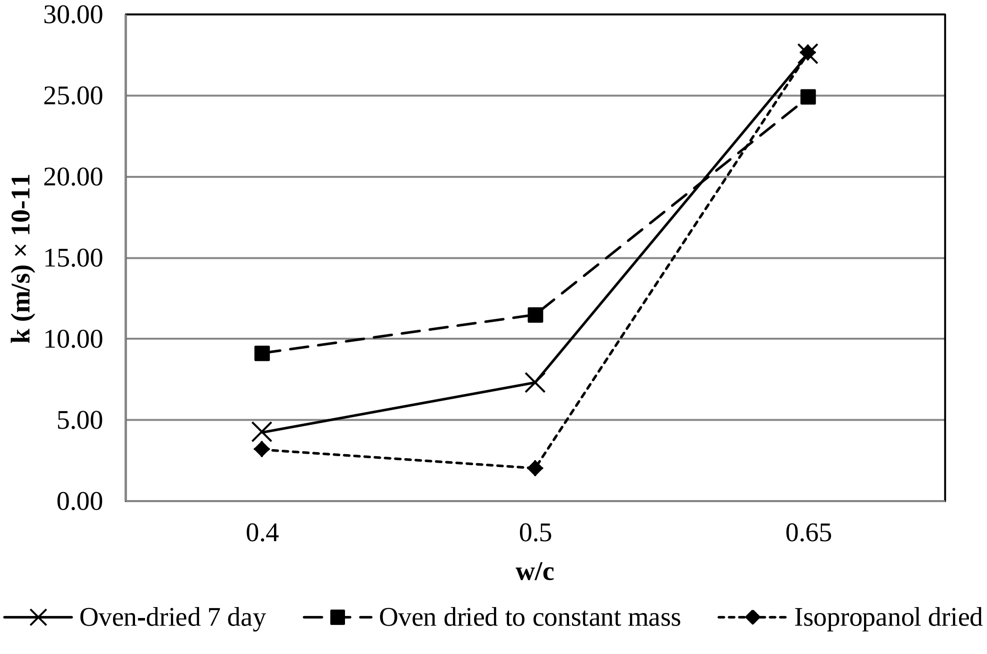

**Figure 4.10: Permeability coefficient results for CEM II (B) 42.5N specimens**

The results from the CEM II (B) 42.5N also show differences in the permeability coefficient, particularly at the w/c ratio of 0.5. There is a general increase in the permeability of the concrete as the w/c ratio increases. This shows that the test is sensitive to changes in w/c ratio for this binder.

In general, there were differences in permeability as a result of the drying method used. The oven-dried to constant mass generally resulted in a higher permeability. However, the results do not indicate whether the differences are statistically significant. The results were therefore analyzed statistically to determine if the drying methods do, statistically, produce different results. This is shown in Section 4.5.

### 4.4.2. Oxygen Permeability Index Test Results

The OPI test results for the four binder types are based on the negative logarithm of the k-values presented in the previous section. As a result, the coefficients of variation for the OPI results are expectedly low, as shown in Figure 4.11. Similar inferences can be drawn from the OPI results to those drawn from the results of the k-values. The OPI results generally decrease as w/c ratio increases due to the densification of the pore structure with decreasing w/c ratio.

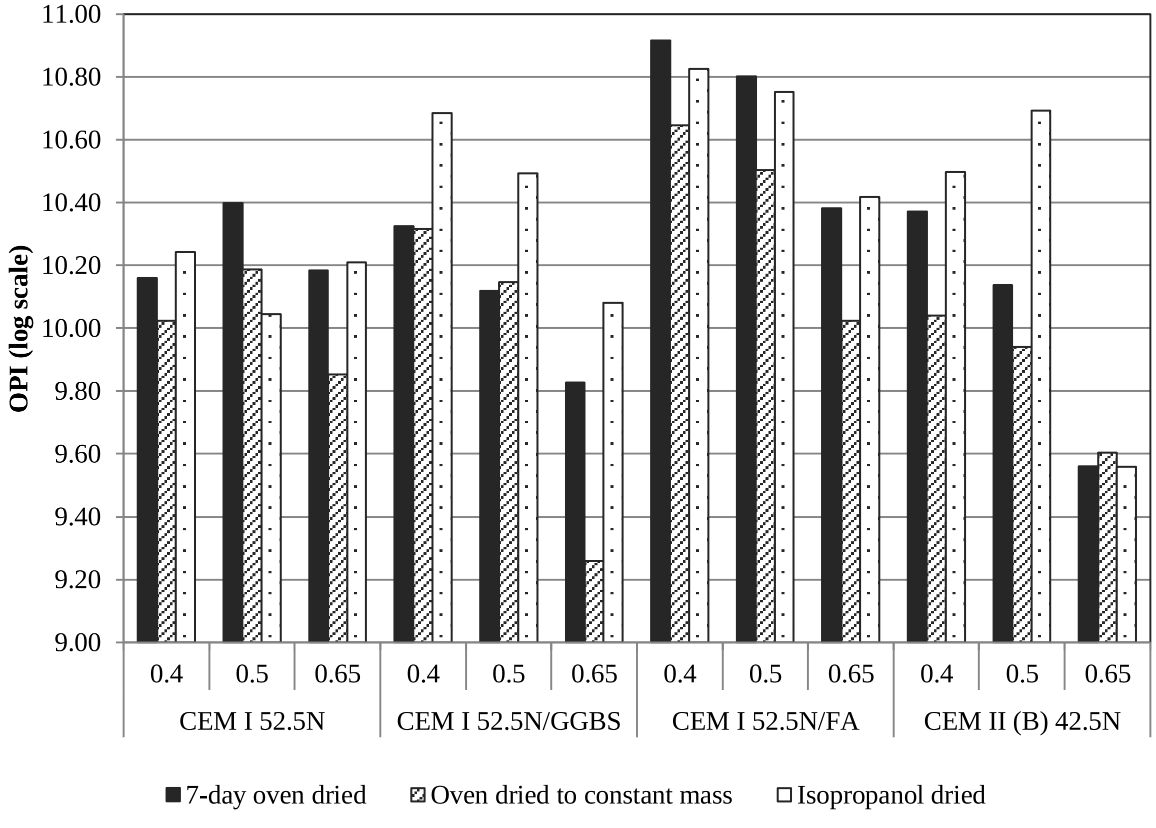

**Figure 4.11: Summary of the OPI results**

**Table 4.5: Variability of the OPI results**

| Binder | w/c | 7-day oven dried | | | Oven dried to constant mass | | | Isopropanol dried | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | OPI | SDEV | CoV (%) | OPI | SDEV | CoV (%) | OPI | SDEV | CoV (%) |
| CEM I 52.5N | 0.4 | 10.16 | 0.20 | 1.93 | 10.02 | 0.16 | 1.62 | 10.24 | 0.20 | 1.99 |
| | 0.5 | 10.40 | 0.08 | 0.77 | 10.19 | 0.14 | 1.36 | 10.04 | 0.13 | 1.26 |
| | 0.65 | 10.18 | 0.13 | 1.29 | 9.85 | 0.21 | 2.10 | 10.21 | 0.14 | 1.37 |
| CEM I 52.5N/ GGBS | 0.4 | 10.33 | 0.25 | 2.38 | 10.32 | 0.19 | 1.84 | 10.68 | 0.24 | 2.29 |
| | 0.5 | 10.12 | 0.18 | 1.74 | 10.15 | 0.20 | 2.00 | 10.49 | 0.18 | 1.67 |
| | 0.65 | 9.83 | 0.15 | 1.55 | 9.26 | 0.22 | 2.36 | 10.08 | 0.21 | 2.12 |
| CEM I 52.5N/ FA | 0.4 | 10.92 | 0.13 | 1.15 | 10.65 | 0.16 | 1.48 | 10.83 | 0.03 | 0.27 |
| | 0.5 | 10.80 | 0.12 | 1.08 | 10.50 | 0.08 | 0.72 | 10.75 | 0.17 | 1.58 |
| | 0.65 | 10.38 | 0.18 | 1.70 | 10.02 | 0.25 | 2.46 | 10.42 | 0.17 | 1.65 |
| CEM II (B) 42.5N | 0.4 | 10.37 | 0.21 | 2.07 | 10.04 | 0.28 | 2.81 | 10.50 | 0.35 | 3.32 |
| | 0.5 | 10.14 | 0.25 | 2.49 | 9.94 | 0.21 | 2.08 | 10.69 | 0.10 | 0.95 |
| | 0.65 | 9.56 | 0.20 | 2.05 | 9.60 | 0.27 | 2.78 | 9.56 | 0.17 | 1.83 |

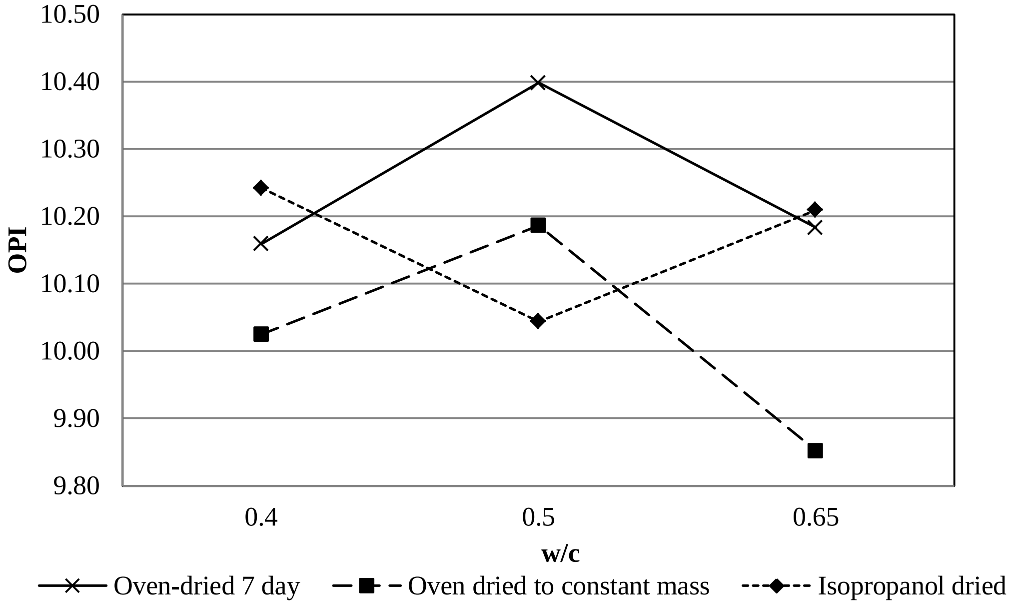

**Figure 4.12: OPI results for CEM I 52.5N specimens**

The OPI results for the CEMI52.5N binder, shown in Figure 4.12, are interesting in that they do not follow the expected trend of decreasing with an increase in w/c ratio. Both the oven drying methods had OPI results which increased from w/c ratio 0.4 to w/c ratio 0.5 and then decreased for the w/c ratio 0.65. The isopropanol dried specimens showed the opposite results. As with the k-values the same explanation can be offered that full saturation with isopropanol did not occur for the 0.5 w/c ratio mix so there was likely moisture in the specimens blocking the passage of oxygen.

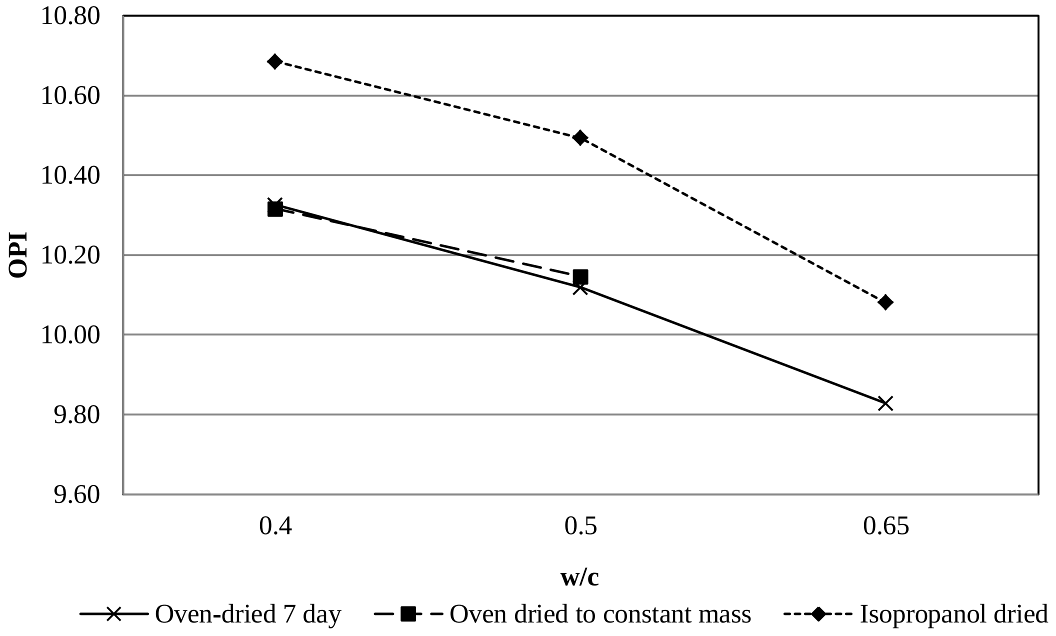

**Figure 4.13: OPI results for CEM I 52.5N/GGBS specimens**

There is a close match between the OPI results from the two oven drying in Figure 4.13 for the GGBS-blended mixes at w/c ratios of 0.4 and 0.5. As with the k-value results, the OPI for the oven dried to constant mass method at the 0.65 w/c ratio is omitted as it was considered an outlier. The isopropanol-dried samples show higher OPI values at all w/c ratios indicating lower permeability.

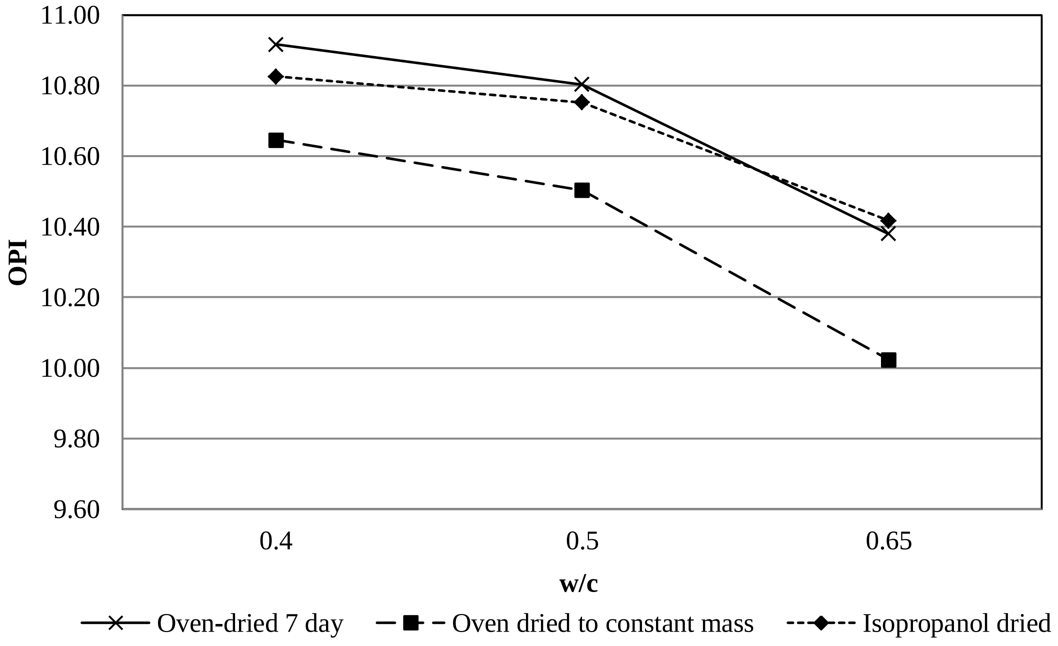

**Figure 4.14: OPI results for CEM I 52.5N/FA specimens**

The OPI results for the FA-extended binder are shown in Figure 4.14. The 7 day oven dried specimens closely match the isopropanol-dried results. The oven-dried to constant mass samples all have lower OPI results. The results show that the OPI is sensitive to changes in w/c ratio for this binder. The OPI for the FA-extended mixes are also consistently higher than for the other binders indicating that using the pozzolanic FA as an extender results in concrete with a more refined pore structure.

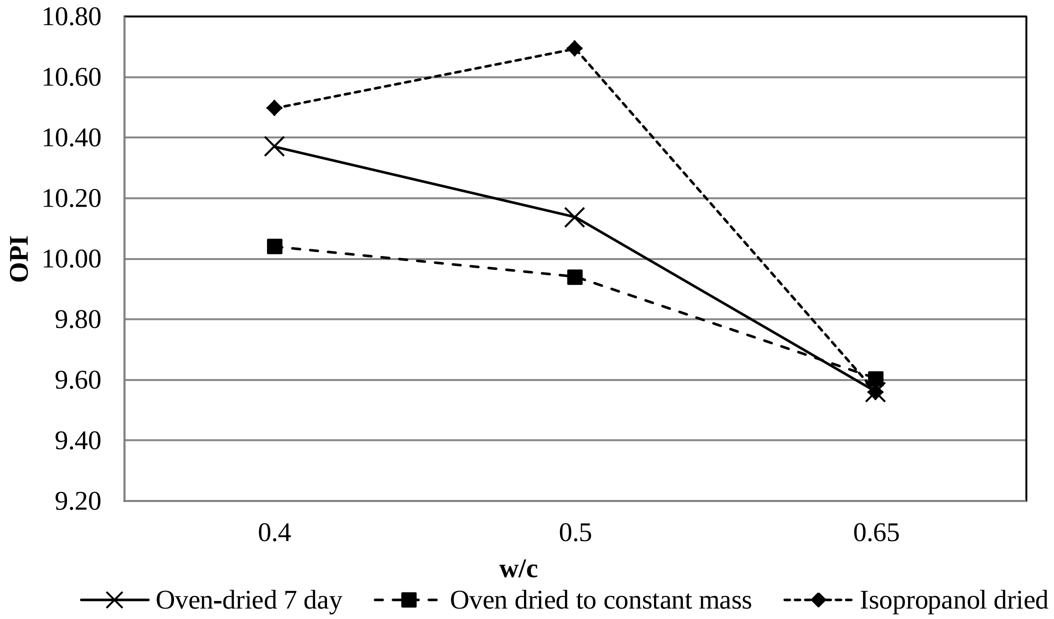

**Figure 4.15: OPI results for CEM II (B) 42.5N specimens**

The differences in OPI between the drying methods are, again, similar in pattern to the k-values. While there were differences between the results from the various mixes, it still remained to be investigated if these differences were statistically significant. Clearly anomalies occur in the results from time to time which may be of concern, but these are difficult to explain considering the OPI values are mean values of 4 specimens. Concrete is a variable material.

### 4.4.3. Water Sorptivity Results

Once the specimens were tested in the OPI chambers, they were removed and prepared for sorptivity testing as per the 2010 UCT DI Manual. This involved sealing the sides of the specimens with packaging tape to ensure that only the bottom face would be in contact with the liquid when tested and then either conducting the sorptivity test immediately after OPI testing, or re-drying for 24 hours in the event that specimens remained in the OPI chamber for an extended period.

The sorptivity generally increased with increasing w/c ratio explained by the larger quantity of capillary pores able to take up liquid at the higher w/c ratios. The CEM I 52.5N showed less surprising sorptivity results compared to the OPI results, in the sense that there were fewer fluctuations between w/c ratios.

The sorptivity results are shown in Figures 4.16 to 4.20 for the different binders and w/c ratios. As with the OPI test results, there appeared to be differences between the results obtained from the three drying methods.

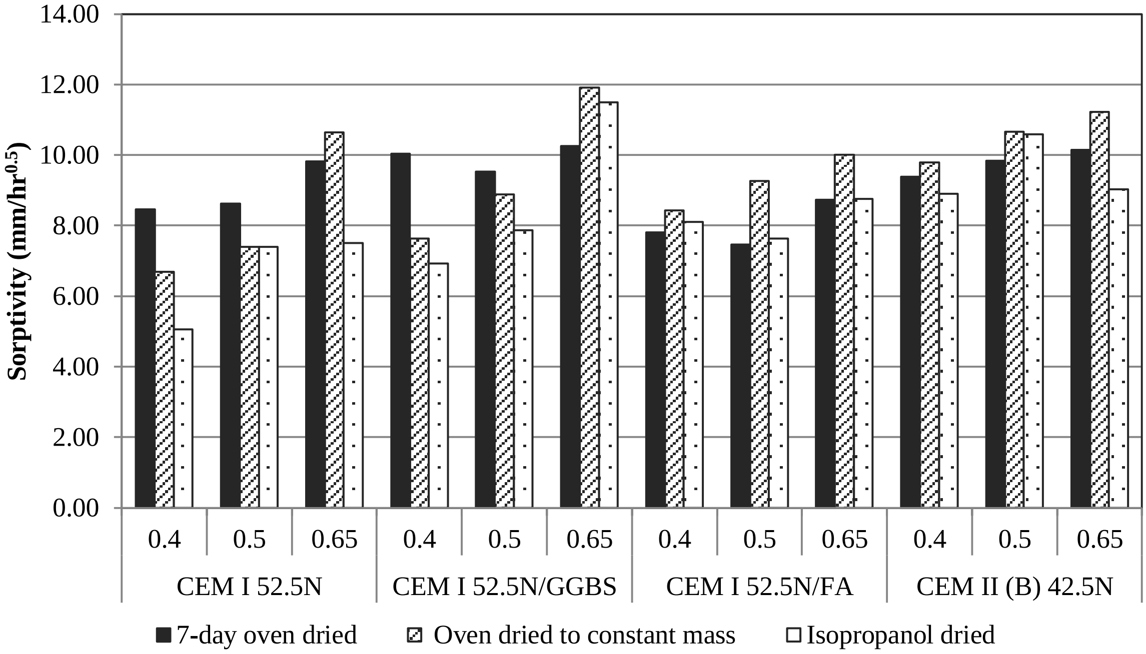

**Figure 4.16: Summary of the sorptivity results**

**Table 4.6: Variability of sorptivity results**

| **Binder** | **w/c** | **7-day oven dried** | | | **Oven dried to constant mass** | | | **Isopropanol dried** | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | WSI (mm/hr$^{0.5}$) | SDEV | CoV (%) | WSI (mm/hr$^{0.5}$) | SDEV | CoV (%) | WSI (mm/hr$^{0.5}$) | SDEV | CoV (%) |
| CEM I 52.5N | 0.4 | 8.46 | 0.92 | 9.92 | 6.69 | 0.66 | 9.92 | 5.06 | 0.43 | 8.54 |
| | 0.5 | 8.63 | 0.88 | 10.24 | 7.40 | 0.96 | 12.95 | 7.40 | 0.96 | 12.95 |
| | 0.65 | 9.83 | 1.06 | 10.82 | 10.64 | 0.92 | 8.61 | 7.51 | 0.35 | 4.65 |
| CEM I 52.5N/ GGBS | 0.4 | 10.04 | 1.80 | 17.93 | 7.63 | 0.31 | 4.08 | 6.93 | 0.40 | 5.74 |
| | 0.5 | 9.54 | 2.30 | 24.08 | 8.89 | 0.67 | 7.49 | 7.88 | 0.55 | 7.05 |
| | 0.65 | 10.25 | 0.43 | 4.21 | 11.92 | 1.27 | 10.68 | 11.49 | 0.61 | 5.28 |
| CEM I 52.5N/ FA | 0.4 | 7.81 | 1.13 | 14.48 | 8.44 | 1.20 | 14.22 | 8.10 | 0.68 | 8.37 |
| | 0.5 | 7.47 | 1.12 | 15.01 | 9.27 | 0.76 | 8.21 | 7.63 | 0.65 | 8.55 |
| | 0.65 | 8.73 | 1.09 | 12.47 | 10.01 | 0.73 | 7.32 | 8.76 | 1.07 | 12.26 |
| CEM II (B) 42.5N | 0.4 | 9.38 | 1.00 | 10.65 | 9.79 | 1.20 | 12.21 | 8.90 | 0.97 | 10.89 |
| | 0.5 | 9.85 | 0.88 | 8.93 | 10.67 | 1.88 | 17.59 | 10.59 | 1.16 | 10.98 |
| | 0.65 | 10.14 | 1.04 | 10.23 | 11.22 | 1.22 | 10.91 | 9.03 | 0.71 | 7.87 |

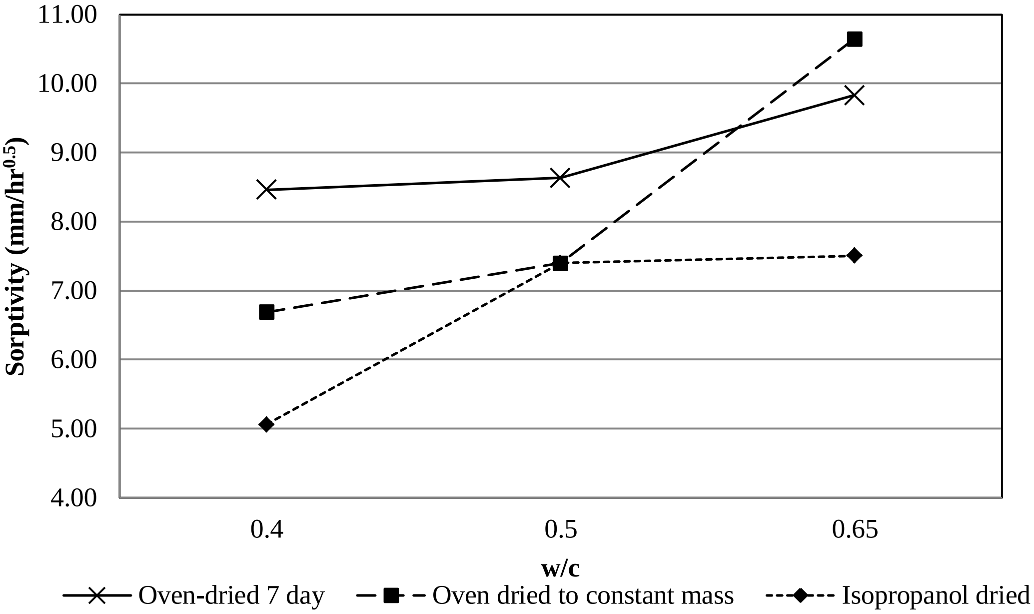

**Figure 4.17: Sorptivity results for the CEM I 52.5N specimens**

Figure 4.17 shows that the 7-day oven dried sorptivity was less sensitive to the change in w/c ratio than their oven dried to constant mass counterparts for the CEM I 52.5N binder. This is illustrated by the wider range of results for the three w/c ratios for the oven dried to constant mass specimens. The sorptivities for the isopropanol specimens changed very slightly from 7.40 to 7.51mm/hr$^{0.5}$ for the w/c ratios of 0.5 and 0.65 respectively. This is surprising because there was more mass lost from the 0.65 w/c ratio specimens compared with the 0.5 w/c (see Figure 4.2) so one would expected that the sorptivity would be greater for the higher w/c ratio. There appeared to be a larger difference in results obtained from the three drying methods at the w/c ratio of 0.4. This suggests that the lower w/c ratios viz. the higher quality concrete made with CEM I 52.5N is more sensitive with regard to sorptivity to the type and duration of drying method used. Generally it would be expected that the sorptivity for the 7-day oven dried specimens would be lower than the constant mass oven dried specimens. The case here is the opposite.

The 7-day oven dried sorptivity was less sensitive to the oven dried to constant mass and isopropanol-dried counterparts for the GGBS-blended mixes shown in Figure 4.18. Surprisingly, the sorptivity decreased from 10.04 to 9.54 mm/hr$^{0.5}$. These sorptivity values are quite high, bordering on ‘poor’ in the classification suggested by Alexander et al. (2001), especially for the w/c ratio of 0.4. It would normally be expected for this to be lower. The oven dried to constant mass results seem to follow the trend of the isopropanol dried results reasonably well, albeit they are higher for all w/c ratios. The expected trend is followed, that the lower w/c ratios have lower sorptivities.

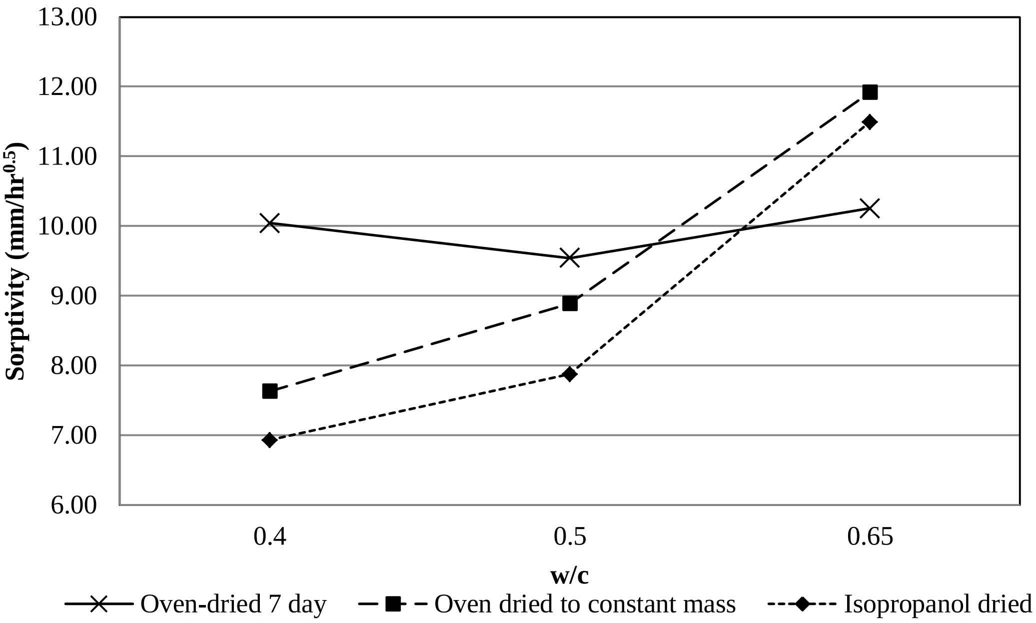

**Figure 4.18: Sorptivity results for the CEM I 52.5N/GGBS specimens**

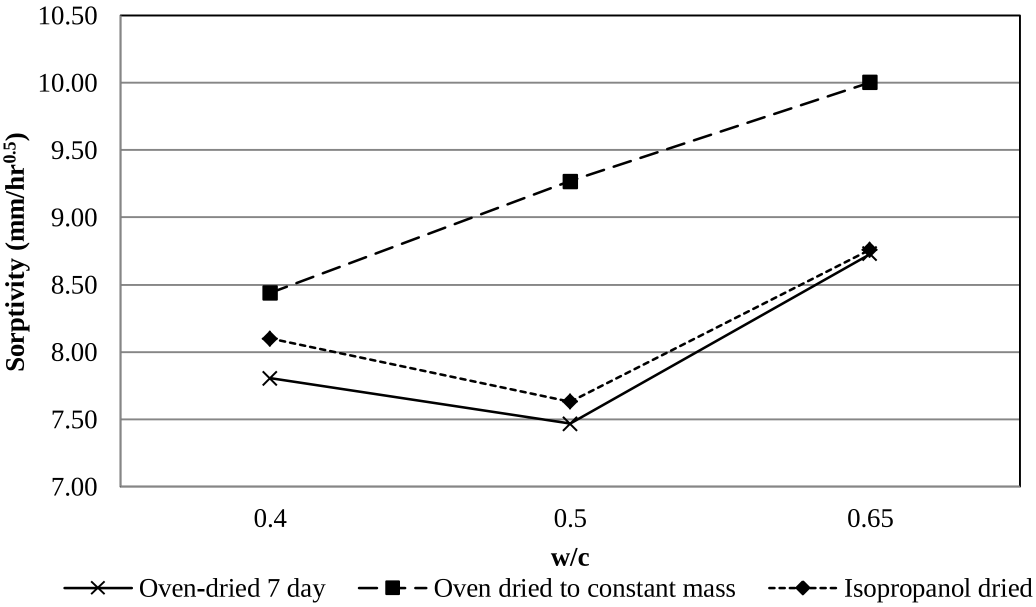

**Figure 4.19: Sorptivity results for the CEM I 52.5N/FA specimens**

As with the other mixes, the sorptivity decreases for FA-blended mixes from the 0.4 w/c mix (7.81mm/hr$^{0.5}$) to the 0.5 w/c mix (7.47 mm/hr$^{0.5}$) and then increases again for the 0.65w/c mix (8.73 mm/hr$^{0.5}$) shown in Figure 4.19. However, unlike the other mixes, this is closely followed by the isopropanol-dried results. The oven dried to constant mass results show more sensitivity to changes in w/c ratio than the other drying methods, in addition to following the expected trend of increasing sorptivity as w/c ratio increases.

When comparing the two oven drying methods, the difference being the duration they were kept in the oven, it was noted that all the sorptivity results for mixes containing CEM I52.5N showed similar differences. For the 7-day dried results the sorptivity decreases and then increases for the w/c ratio 0.40-0.5-0.65, compared to a consistent increase in sorptivity with increasing w/c ratio for the oven dried to constant mass results. This may indicate that the 7-day drying duration is not adequate at the w/c ratio of 0.50, although it is difficult to explain why this would be the case. At the 0.4 w/c ratio the 7-day dried sorptivity results are higher than the oven dried to constant mass results. It would be expected, if drying had not completed in 7 days and there was more moisture in the 7-day dried specimens, that the sorptivity results should be lower.

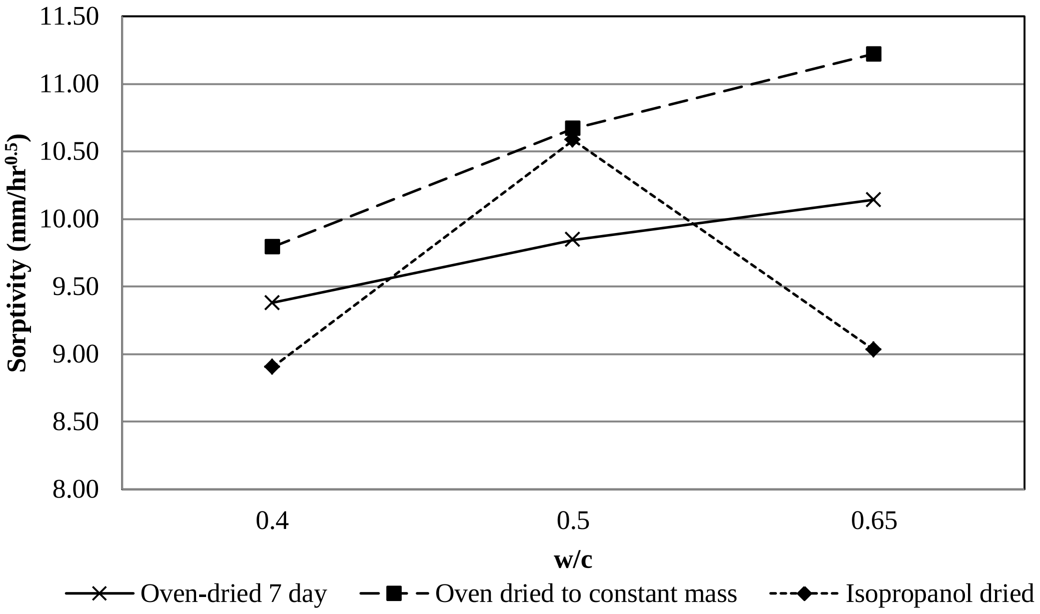

**Figure 4.20: Sorptivity results for the CEM II (B) 42.5 specimens**

The sorptivity results for the CEM II (B) 42.5N binder, illustrated in Figure 4.20, showed much less variation between the three drying methods than the other binder types. Both the 7 day oven dried and the oven dried to constant mass results increase with increasing w/c ratio. The constant mass results are slightly higher, possibly due to more moisture being lost in those specimens. The isopropanol-dried results show a decrease at the w/c ratio 0.65. From Figure 4.3 it can be seen that, unlike the other binders, the isopropanol-dried specimens at the 0.65 w/c ratio did not lose much more mass (and therefore moisture) than the 0.5 w/c ratio specimens. This could explain why a decrease in sorptivity was registered.

What is generally noteworthy for the sorptivity results is the general insensitivity to w/c ratio. This is likely a consequence of the curing regime where specimens are fully cured for 28 days. For poor curing, it would be expected that the sensitivity to w/c ratio would increase.

The general trends here indicate that the drying methods are not having a large influence on sorptivity. However, as with the previous tests, the statistical significance of the differences was investigated.

### 4.4.4. Chloride Conductivity Results

Four test specimens were preconditioned and dried for each of the three drying methods being investigated. They were then saturated with a 5M NaCl solution as stipulated in the 2010 DI manual. The saturated specimens were then tested for chloride conductivity. The trend shown in Figure 4.21 is that the conductivity increases with increasing w/c ratio, as is expected. The blended binders also perform better, having lower conductivities than the other binders. Figures 4.22 to 4.25 show the results for the various binder types used in this study.

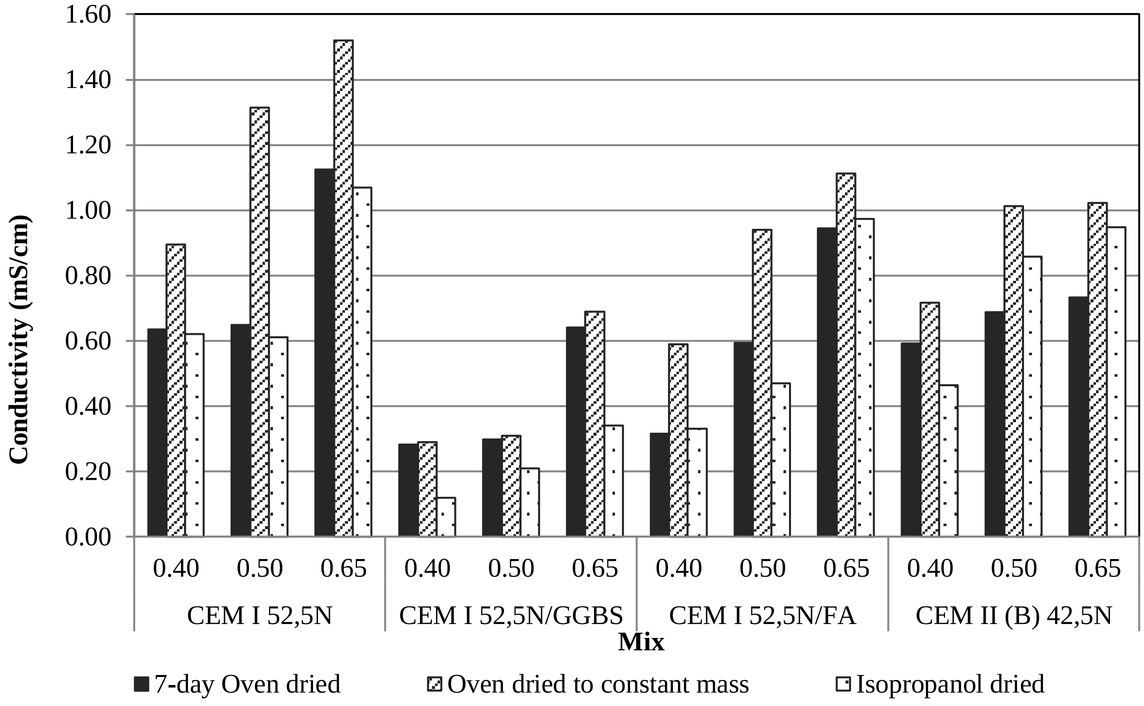

**Figure 4.21: Summary of the chloride conductivity results**

**Table 4.7: Variability of the chloride conductivity test results**

| Binder | w/c | 7-day oven dried | | | Oven dried to constant mass | | | Isopropanol dried | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | CCI (mS/cm) | SDEV | CoV (%) | CCI (mS/cm) | SDEV | CoV (%) | CCI (mS/cm) | SDEV | CoV (%) |
| CEM I 52.5N | 0.4 | 0.63 | 0.06 | 8.97 | 0.90 | 0.04 | 5.02 | 0.74 | 0.04 | 4.83 |
| | 0.5 | 0.65 | 0.08 | 11.57 | 1.31 | 0.14 | 10.31 | 0.86 | 0.13 | 14.99 |
| | 0.65 | 1.13 | 0.08 | 7.41 | 1.52 | 0.08 | 5.60 | 1.64 | 0.25 | 15.06 |
| CEM I 52.5N/ GGBS | 0.4 | 0.28 | 0.04 | 12.62 | 0.29 | 0.03 | 12.06 | 0.34 | 0.04 | 10.61 |
| | 0.5 | 0.30 | 0.03 | 8.47 | 0.31 | 0.01 | 4.49 | 0.49 | 0.03 | 5.83 |
| | 0.65 | 0.64 | 0.02 | 3.21 | 0.69 | 0.03 | 4.36 | 1.09 | 0.02 | 1.45 |

**Table 4.7: Variability of the chloride conductivity test results (continued)**

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| CEM I 52.5N/ FA | 0.4 | 0.32 | 0.03 | 9.46 | 0.59 | 0.06 | 10.24 | 0.59 | 0.05 | 8.43 |
| | 0.5 | 0.59 | 0.06 | 10.48 | 0.94 | 0.08 | 8.77 | 0.70 | 0.08 | 11.10 |
| | 0.65 | 0.94 | 0.12 | 12.51 | 1.11 | 0.04 | 3.19 | 1.03 | 0.13 | 12.71 |
| CEM II (B) 42.5N | 0.4 | 0.59 | 0.01 | 2.39 | 0.72 | 0.04 | 5.10 | 0.69 | 0.02 | 3.21 |
| | 0.5 | 0.69 | 0.04 | 6.27 | 1.01 | 0.05 | 4.73 | 0.79 | 0.02 | 2.96 |
| | 0.65 | 0.73 | 0.02 | 2.95 | 1.02 | 0.04 | 4.06 | 0.81 | 0.02 | 2.19 |

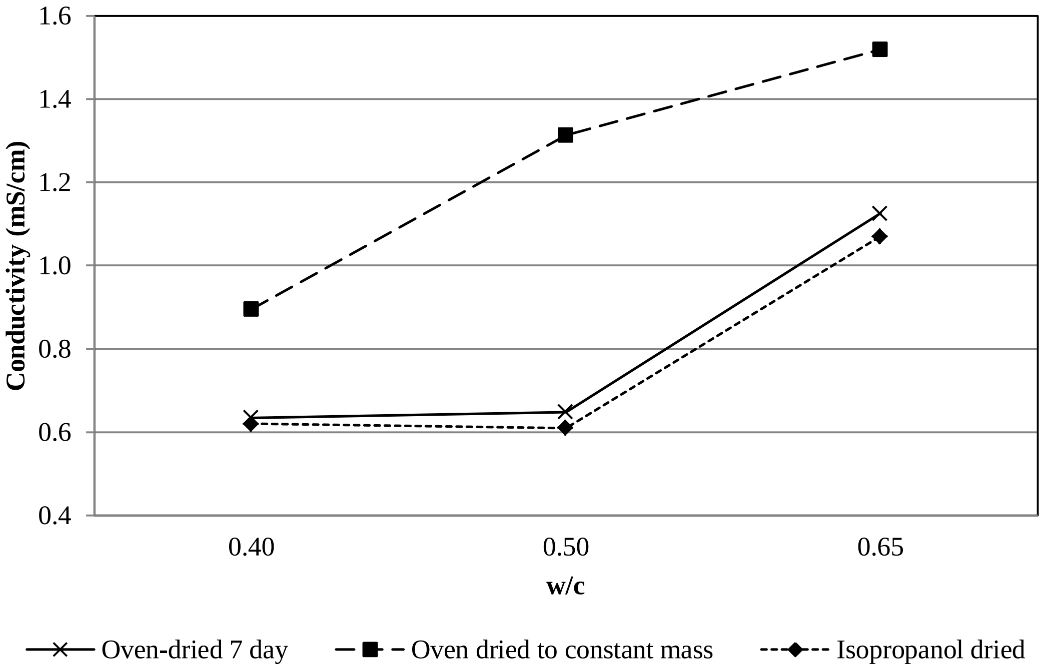

Figure 4.22: Conductivity results for the CEM I 52.5N specimens

The chloride conductivity increased with increasing w/c ratio as was expected. The CEM I 52.5N concrete showed similar conductivities for the 7-day drying method and the isopropanol dried specimens (see Figure 4.22). The specimens dried to constant mass exhibited a higher conductivity. There is a particularly large difference at the 0.5 w/c ratio. This effect could have to do with the specimens having more open pores available for transporting the chloride ions as a consequence of extended oven drying.

From Figure 4.23, it can be observed that the CEM I 52.5N/GGBS concrete generally has a low conductivity (< 0.8 mS/cm). In this case, the 7-day drying results more closely match the oven dried to constant mass results, with the isopropanol dried results showing a lower conductivity. The generally low conductivity is a result of the addition of GGBS which is known to be a good extender for chloride resistance due to the pore filler effect and good chloride binding abilities.

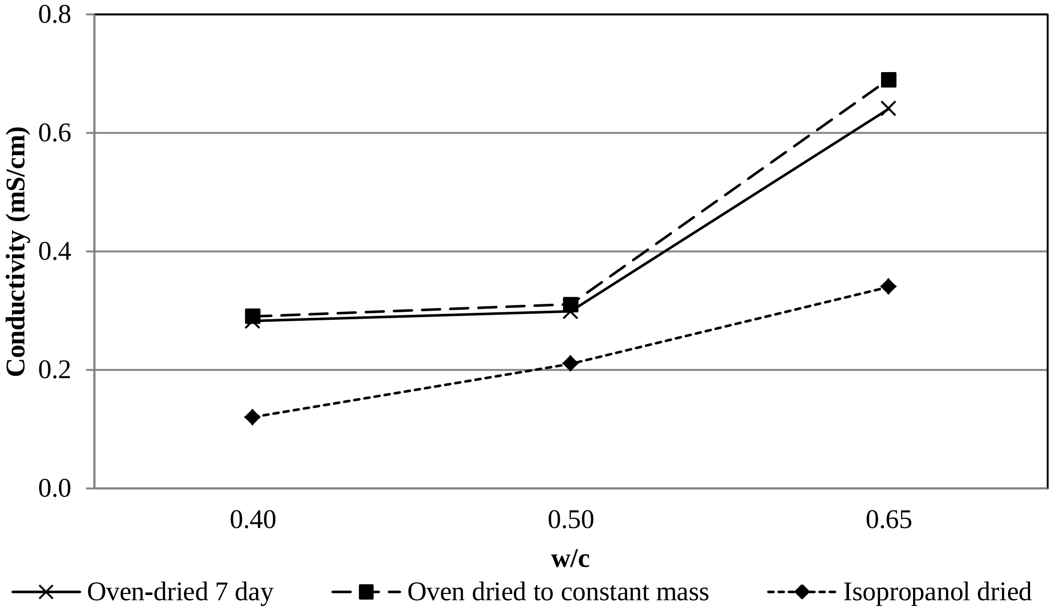

**Figure 4.23: Conductivity results for the CEM I 52.5N/GGBS specimens**

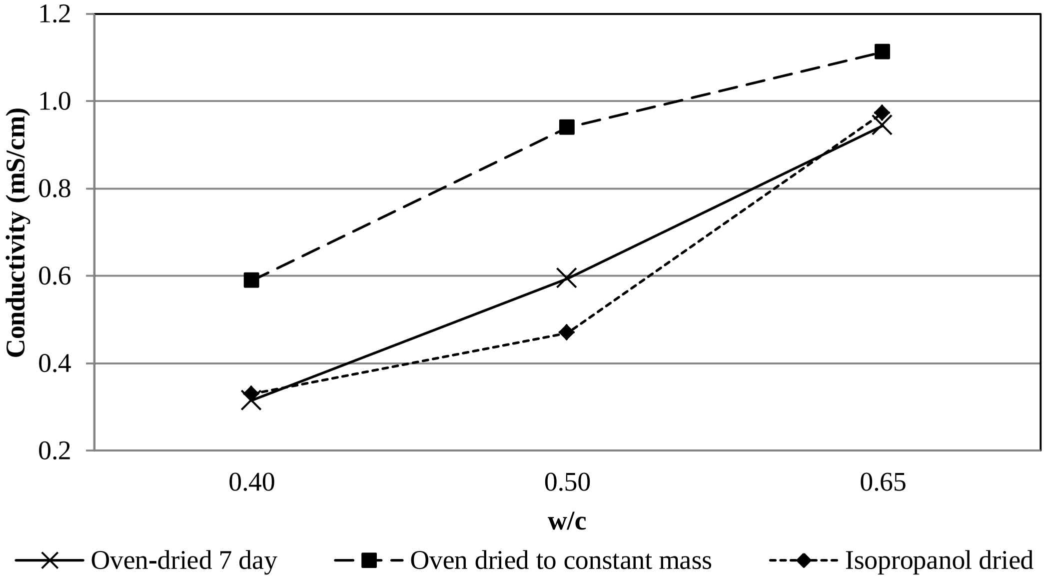

**Figure 4.24: Conductivity results for the CEM I 52.5N/FA specimens**

The conductivity results for the concrete containing FA, shown in Figure 4.24, showed trends similar to the CEM I 52.5N mix. The isopropanol-dried and 7-day drying regimes produced similar results while the oven dried to constant mass specimens exhibited higher conductivity values at all w/c ratios. A similar explanation could be put forward that the specimens dried to constant mass had more open pores thereby allowing more chloride ions through.

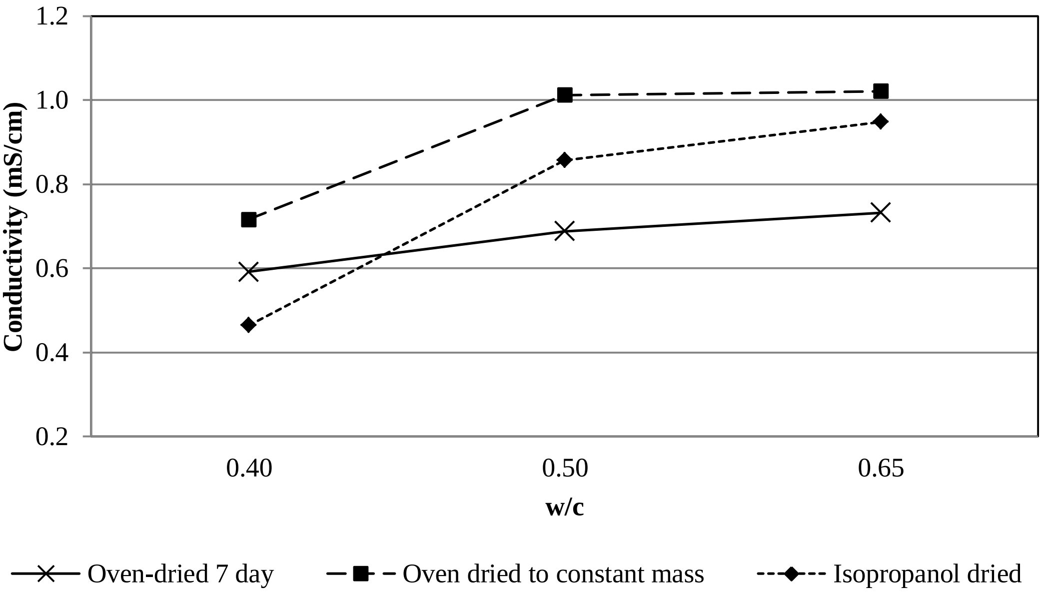

**Figure 4.25: Conductivity results for the CEM II (B) 42.5N specimens**

The conductivity results for the concrete made with the CEM II (B) 42.5N binder, shown in Figure 4.25, shows no discernible trends in the differences. Although the conductivities obtained from the fully dry specimens were higher than the other conductivities, the difference was not large. As expected, the increase in conductivity corresponded to an increase in w/c ratio. Compared with the CEM I 52.5N binder, the conductivity is lower. This could be partly due the CEM II (B) 42.5N containing slag.

As with the other DI results obtained, the results were difficult to interpret merely by observation. However, the oven dried to constant mass results are consistently higher than the other, therefore they were analyzed statistically find out the degree to which the drying methods are different.

## 4.5. Statistical Analysis of the DI Results

### 4.5.1. Effect of Drying Regime

In the previous section, it was shown that there was a difference in all the DI results depending on which drying method was used. These differences occurred to a varying degree for the different mixes. The results were therefore analyzed further to check if the differences were statistically significant.

A series of statistical tests were done using hypothesis testing. Firstly, all three drying methods (oven drying regime - including the oven drying to constant mass and oven drying for 7 days - and isopropanol drying regime) were compared to check if their means were equivalent at the 5% significance level. The null hypothesis was that the means of the three methods were equal (the three drying methods produce equivalent results); while the alternative hypothesis was that the means were not equal (the three drying methods do not produce equivalent results). The Kruskal-Wallis test was chosen as a suitable test for reasons outlined in Section 3.6.

The results of the Kruskal-Wallis test are shown in Table 4.8. It can be seen that the tests showed that some of the results were equivalent while others were not. There was no trend to help explain why this was the case. The results for the chloride conductivity test showed that all the tests rejected the null hypothesis. Therefore the three drying methods were not equivalent for producing similar chloride conductivity results. The isopropanol-dried samples produced different DI results compared to both oven drying methods.

**Table 4.8: Accept/Reject Null hypothesis that the DI results for the three drying methods are equivalent**

| **Binder** | **CEM I 52.5N** | | | **CEM I 52.5N/GGBS** | | | **CEM I 52.5N/FA** | | | **CEM II (B) 42.5N** | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **w/c** | **0.4** | **0.5** | **0.65** | **0.4** | **0.5** | **0.65** | **0.4** | **0.5** | **0.65** | **0.4** | **0.5** | **0.65** |
| OPI | ✓ | × | ✓ | ✓ | ✓ | × | × | × | ✓ | ✓ | × | ✓ |
| WSI | × | ✓ | × | × | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | × |
| CCI | × | × | × | × | × | × | × | × | × | × | × | × |

✓= Accept $H_0$
×= Reject $H_0$

The statistical results showed that in general, DI results from specimens dried using the isopropanol method and both oven drying methods are not conclusively equivalent. It was therefore decided that further statistical analysis was needed on the DI results obtained from the oven drying methods. This was in order to determine if the duration of time that the specimens are placed in the oven is important. The statistical tests were done in two stages. Firstly, a rank-sum test was performed on all the test results. This was because the permeability k-values are not normally distributed, so the use of a non-parametric method - which does not require that the test data be normally distributed - such as the rank-sum test is applicable. The results of the rank-sum test were verified using the t-test. This was only done to the WSI and CCI test results as they are known to follow a normal distribution.

### 4.5.2. Effect of Oven Drying Duration

The results shown in Section 4.4 showed some variation between the DI results for the different drying methods used. This section focuses on the statistical significance on the DI results of the duration that the specimens are placed in the oven. Two oven drying durations were used in this study – 7 days, and until the specimens, on average, reached a constant mass state. Section 3.6 details the calculation methods.

### OPI

Due to the log-normal nature of the distribution of the k-values, only the rank-sum test was conducted. It would be incorrect to perform the t-test which is based on the assumption that the data set is normally distributed. Table 4.9 shows the results of the rank-sum test. A critical value of U was obtained. The null hypothesis, that the two oven drying durations produce equivalent results, was rejected if the calculated U-statistic was less than 1.

The results show that the null hypothesis was accepted for most of the concrete mixes except the CEM I 52.5N/ GGBS 0.65 and the CEM I 52.5N/FA 0.40-0.50 mixes.

**Table 4.9: k-Value statistical analysis results for oven drying durations**

| **Binder** | **w/c** | **Rank-Sum Test** | | |
|---|---|---|---|---|
| | | **p-value** | **U*** | **Accept/Reject $H_0$** |
| CEM I 52.5N | 0.4 | 0.20 | 3 | Accept |
| | 0.5 | 0.06 | 1 | Accept |
| | 0.65 | 0.06 | 1 | Accept |
| CEM I 52.5N/GGBS | 0.4 | 1.00 | 8 | Accept |
| | 0.5 | 1.00 | 8 | Accept |
| | 0.65 | 0.03 | 0 | Reject |
| CEM I 52.5N/FA | 0.4 | 0.03 | 0 | Reject |
| | 0.5 | 0.03 | 0 | Reject |
| | 0.65 | 0.06 | 1 | Accept |
| CEM II (B) 42.5N | 0.4 | 0.20 | 3 | Accept |
| | 0.5 | 0.69 | 6 | Accept |
| | 0.65 | 0.69 | 6 | Accept |

**A U-value less than 1 is rejected*

Since OPI results are normally distributed, they can be statistically analyzed using the t-test. The rank-sum test was also performed and verified using the t-test, as shown in Table 4.10. In general, the rank-sum test results matched those of the k-values. However there were differences in some of the results where the null hypothesis was rejected by one test and accepted by the other. This is a consequence of the statistical test used. The rank-sum test is based on the rank of a particular determination from the combined sample of results, and due to the relatively small sample size, there is a higher likelihood of this test accepting the null hypothesis than the t-test. The only way the rank-sum test will reject the null hypothesis is if all the determinations are either higher or lower from one sample than the other i.e. there can be no overlap between the four results from the constant-mass and 7-day drying durations. The t-test on the other hand uses the actual value of the determination and the variances of the samples so it can take into account the size of the differences between determinations.

**Table 4.10: OPI statistical analysis results for oven drying durations**

| Binder | w/c | Rank-Sum Test | | | T-test | | |
|---|---|---|---|---|---|---|---|
| | | p-value | U | Accept/Reject $H_0$ | t | $t_{crit}$ | Accept/Reject $H_0$ |
| CEM I 52.5N | 0.4 | 0.20 | 3 | Accept | 1.21 | 2.45 | Accept |
| | 0.5 | 0.06 | 1 | Accept | 2.48 | 2.57 | Accept |
| | 0.65 | 0.061 | 1 | Accept | 2.58 | 2.57 | Reject |
| CEM I 52.5N/ GGBS | 0.4 | 1.00 | 8 | Accept | 1.50 | 2.45 | Accept |
| | 0.5 | 1.00 | 8 | Accept | -0.30 | 2.45 | Accept |
| | 0.65 | 0.03 | 0 | Reject | 4.14 | 2.57 | Reject |
| CEM I 52.5N/ FA | 0.4 | 0.03 | 0 | Reject | 2.59 | 2.45 | Reject |
| | 0.5 | 0.03 | 0 | Reject | 4.41 | 2.57 | Reject |
| | 0.65 | 0.06 | 1 | Accept | 2.13 | 2.57 | Accept |
| CEM II (B) 42.5N | 0.4 | 0.20 | 3 | Accept | 1.67 | 2.45 | Accept |
| | 0.5 | 0.69 | 6 | Accept | 1.27 | 2.45 | Accept |
| | 0.65 | 0.69 | 6 | Accept | -0.44 | 2.45 | Accept |

The t-test rejected more tests showing that differences exist in the OPI as a result of drying durations for many mixes. However, there was no specific trend that was observed. The CEM I 52.5N and GGBS-blended concretes were rejected at the 0.65 w/c ratio. The reverse was seen for the mixes containing FA, where the 0.4 and 0.5 w/c ratio mixes were accepted and the higher 0.65 w/c ratio mix was rejected. This showed that differences exist in the OPI as a result of drying durations for these mixes.

### WSI

Table 4.11 shows the statistical test results conducted on the WSI results. The WSI appears to be less sensitive to duration of oven drying compared with the OPI results. The rank-sum test only rejected the CEM I 52.5N mix at the 0.4 w/c ratio. The t-test also rejected this mix. However it also rejected the 0.5 w/c ratio mix for CEM I 52.5N /FA. There was no observable trend to help identify whether or not certain mixes were rejected. The CEM II (B) 42.5N mix was accepted at all w/c ratios resulting in the conclusion that the two drying methods produced equivalent WSI results for these mixes.

**Table 4.11: WSI statistical analysis results for oven drying durations**

| **Binder** | **w/c** | **Rank-Sum Test** | | | **t-test** | | |
|---|---|---|---|---|---|---|---|
| | | **p-value** | **U** | **Accept/Reject $H_0$** | **t** | **$t_{crit}$** | **Accept/Reject $H_0$** |
| CEM I 52.5N | 0.4 | 0.03 | 0 | Reject | 3.14 | 2.57 | Reject |
| | 0.5 | 0.11 | 2 | Accept | 1.89 | 2.45 | Accept |
| | 0.65 | 0.49 | 5 | Accept | -1.16 | 2.45 | Accept |
| CEM I 52.5N/GGBS | 0.4 | 0.20 | 3 | Accept | 2.64 | 3.18 | Accept |
| | 0.5 | 0.69 | 6 | Accept | 0.54 | 2.78 | Accept |
| | 0.65 | 0.20 | 3 | Accept | -2.48 | 2.78 | Accept |
| CEM I 52.5N/ FA | 0.4 | 0.48 | 5 | Accept | -0.77 | 2.45 | Accept |
| | 0.5 | 0.11 | 2 | Accept | -2.62 | 2.57 | Reject |
| | 0.65 | 0.20 | 3 | Accept | -1.95 | 2.57 | Accept |
| CEM II (B) 42.5N | 0.4 | 0.69 | 5 | Accept | -0.53 | 2.45 | Accept |
| | 0.5 | 0.89 | 6 | Accept | -0.80 | 2.78 | Accept |
| | 0.65 | 0.20 | 5 | Accept | -1.35 | 2.45 | Accept |

### CCI

The CCI test results were similar for both the rank-sum test and the t-test. These are shown in Table 4.12. The CEM I 52.5N and CEM II (B) 42.5N binders were rejected at all w/c ratios indicating that these binders in particular are relatively sensitive to the oven drying duration.

The GGBS blended mixes, on the other hand, were all accepted. The CCI results were very low, as is expected with GGBS mixes, so a tentative conclusion is that the CCI test is more insensitive to the duration of drying used for good quality concretes. A possible reason for this could be different levels of chloride saturation at the different drying times. For these very chloride-resistant mixes with very low conductivities, small differences in results are accentuated. Practically, however, they are very resistant to chloride penetration so the effect of drying would not be very significant, i.e. highly chloride resistant binders are still shown up as such by the test regardless of the moisture condition.

The FA mixes were all rejected, except for the 0.65 w/c ratio.

The CCI test appears to be more sensitive to the duration of drying than the other DI tests as more mixes were rejected overall.

**Table 4.12: CCI statistical analysis results for oven drying durations**

| Binder | w/c | Rank-Sum Test | | | t-test | | |
|---|---|---|---|---|---|---|---|
| | | p-value | U | Accept/Reject $H_0$ | t | $t_{crit}$ | Accept/Reject $H_0$ |
| CEM I 52.5N | 0.4 | 0.03 | 0 | Reject | -7.18 | 2.45 | Reject |
| | 0.5 | 0.03 | 0 | Reject | -8.58 | 2.57 | Reject |
| | 0.65 | 0.03 | 0 | Reject | -6.57 | 2.45 | Reject |
| CEM I 52.5N/ GGBS | 0.4 | 0.80 | 7 | Accept | -0.15 | 2.45 | Accept |
| | 0.5 | 0.57 | 5 | Accept | -1.10 | 2.57 | Accept |
| | 0.65 | 0.86 | 1 | Accept | -2.44 | 2.57 | Accept |
| CEM I 52.5N/ FA | 0.4 | 0.03 | 0 | Reject | -8.16 | 2.78 | Reject |
| | 0.5 | 0.03 | 0 | Reject | -6.71 | 2.45 | Reject |
| | 0.65 | 0.11 | 2 | Accept | -2.73 | 2.78 | Accept |
| CEM II (B) 42.5N | 0.4 | 0.03 | 0 | Reject | -6.38 | 2.78 | Reject |
| | 0.5 | 0.03 | 0 | Reject | -10.05 | 2.45 | Reject |
| | 0.65 | 0.03 | 0 | Reject | -12.37 | 2.57 | Reject |

### 4.5.3. General Comments

The statistical analysis of the drying methods showed that, for the most part, there is an impact on the DI results when the isopropanol and oven drying methods are used. The isopropanol method was used in this work to examine its feasibility for use as an alternative method. The differences in DI results, in addition to the differences experienced in moisture loss, between the isopropanol dried specimens and their oven dried counterparts supports the continued use of the simpler oven drying method in the DI procedure.

Further statistical analysis on the DI results from the specimens dried for 7 days and to constant mass revealed that the duration of drying does have an influence on the DI test results for certain mixes. This is especially true for the CCI test results, which showed the most differences between the drying durations. In general, all the mixes were affected apart from the concretes containing GGBS, which is known to contribute to a comparatively high chloride resistance. Despite their differences, the CCI results are still very low when the specimens are dried to constant mass. This result has practical implications for the execution of the DI tests, in the sense that although the drying duration is an important factor, increasing it can have a significant impact on the efficiency of the tests. The time taken for the tests to run needs to be relatively short to minimise delays in projects, for which delays could have substantial financial implications. Therefore, it does not seem feasible to have to wait until specimens are dried to a constant mass state. A compromise should be incorporated.

## 4.6. Chloride Saturation Tests

To determine whether specimens used in the chloride conductivity test were fully saturated samples underwent the standard saturation procedure and were then split in two and sprayed with silver nitrate solution on the internal face. A white precipitate indicated the presence of chloride ions. A brown colour occurred when there were no chlorides present due to the silver nitrate reacting with the hydroxide ions in the concrete. Examples specimens which have been sprayed with silver nitrate solution are shown in Figures 4.26 and 4.27, showing incomplete and complete chloride penetration respectively.

**Figure 4.26: Example of incomplete saturation for CEM I 52.5N/GGBS 0.4 w/c ratio specimen**

**Figure 4.27: Example of complete saturation for CEM I 52.5N 0.5 w/c ratio specimen**

The colorimetric technique provides a semi-quantitative method to assess the degree to which the specimen is penetrated with chloride solution. To provide a quantitative description, the unsaturated area as a percentage of the total cross section area was estimated using the images from photographs taken during testing. The results are shown in Table 4.13 and the trends can be seen in Figure 4.28. The specimens tested underwent the same isopropanol and oven drying regimes (both to constant mass) used in this study.

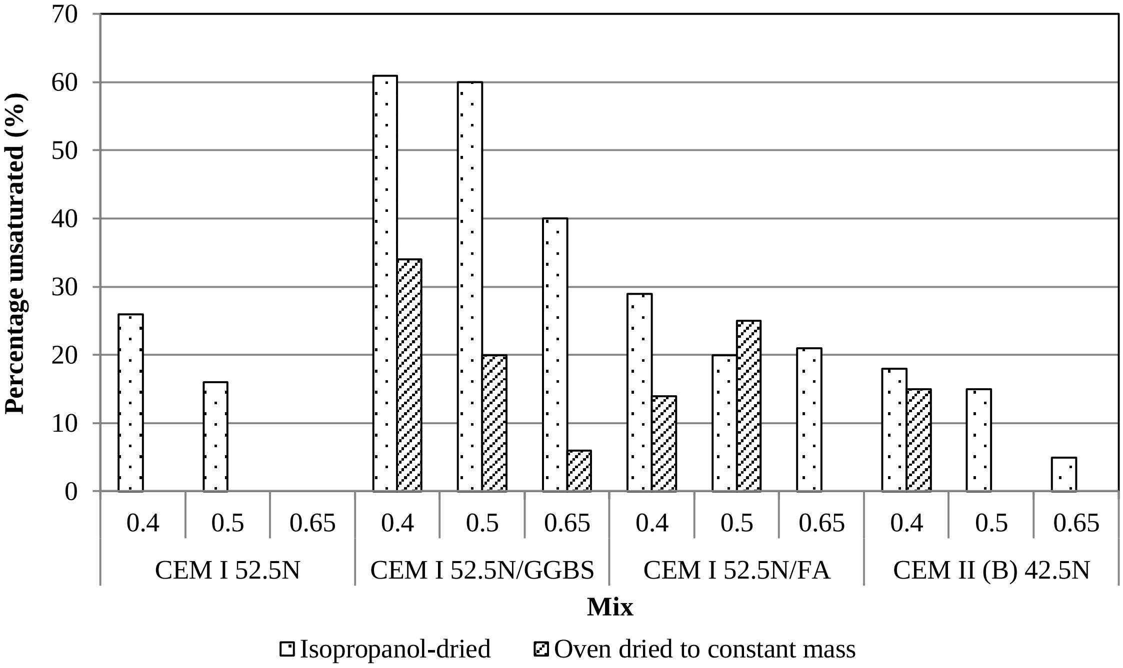

**Figure 4.28: Percentage of "unsaturated" cross-section**

The CEMI52.5N mixes showed good saturation for the oven drying regime. Only the isopropanol-dried specimens for w/c ratios of 0.4 and 0.5 showed unsaturated areas. This meant that the saturation procedure for theses mixes under the current regime is adequate. The CEM I 52.5N/GGBS showed poor saturation. When the specimens were split open they were completely dry towards the centre. This means that the saturation time is not long enough for the solution to penetrate to the centre of the specimens or the concrete is simply too dense for this to occur in any meaningful time period. This could also explain why GGBS extended concretes typically show low chloride conductivity results. There are not enough mobile chloride ions within the pore structure that can be transported. On the other hand, it does show the inherent chloride resistance of the GGBS extended mixes. The CEM I 52.5N/FA mixes showed some degree of unsaturation in the centre for the 0.4 and 0.5 w/c ratios. The CEM II (B) 42.5N mixes show that only 15% of the centre face was unsaturated for the oven-dried specimens.

**Table 4.13: Percentage of cross-section unsaturated**

| Binder | w/c | Drying Regime | Percentage of sample area “unsaturated” (%) |
|---|---|---|---|
| CEM I 52.5N | 0.4 | Isopropanol | 26 |
| | | Oven | 0 |
| | 0.5 | Isopropanol | 16 |
| | | Oven | 0 |
| | 0.65 | Isopropanol | 0 |
| | | Oven | 0 |
| CEM I 52.5N/GGBS | 0.4 | Isopropanol | 61 |
| | | Oven | 34 |
| | 0.5 | Isopropanol | 60 |
| | | Oven | 20 |
| | 0.65 | Isopropanol | 40 |
| | | Oven | 6 |
| CEM I 52.5N/FA | 0.4 | Isopropanol | 29 |
| | | Oven | 14 |
| | 0.5 | Isopropanol | 20 |
| | | Oven | 25 |
| | 0.65 | Isopropanol | 21 |
| | | Oven | 0 |
| CEM II (B) 42.5N | 0.4 | Isopropanol | 18 |
| | | Oven | 15 |
| | 0.5 | Isopropanol | 15 |
| | | Oven | 0 |
| | 0.65 | Isopropanol | 5 |
| | | Oven | 0 |

The saturation condition of the specimens undergoing chloride conductivity testing is important because to have comparable results between different specimens they need to be uniformly saturated or else there may exist unused “pathways” which, if saturated, would allow more chloride ions easier passage. Chloride ions may still migrate, however they would have to travel around the unsaturated areas. Thus non-uniform saturation may not invalidate the test, however it allows for better interpretation if specimens are in a uniform condition before undergoing chloride conductivity testing.

In general, the oven drying method resulted in better saturation. This could be a consequence of a drier specimen having more open pores which can be filled with the saturating solution. The oven dried specimens were shown to lose more mass than the isopropanol dried specimens in Section 4.3. Another possibility is that microstructural damage as a result of the oven drying may have occurred which would also contribute to the increased ability of the oven dried specimens to be fully saturated.

## 4.7. Saturation for Water Sorptivity Tests

The question was posed whether it was necessary to soak the specimens for the duration of 18 hours upon completion of the vacuum saturation step of the water sorptivity test to achieve a fully saturated state. This time had no scientific backing and was presumably motivated for convenience to the test operator. The previous section showed that the chloride conductivity saturation procedure did not fully saturate some specimens with NaCl so the 18 hour soaking time was investigated when the saturating liquid was $Ca(OH)_2$.

The 7-day oven dried specimens that underwent sorptivity testing in Section 4.4.3 were soaked for the “standard” 18 hours after vacuum saturation and then left to soak for an additional 24 hours thereafter. The mass gain over the period is shown in Table 4.14.

**Table 4.14: Mass gain after an additional 24 hour saturation in $Ca(OH)_2$**

| Binder | w/c | Average Mass (g) | | | Percentage mass gain (%) |
|---|---|---|---|---|---|
| | | After vacuum saturation | After additional 24 hours saturation | Gain | |
| CEM I 52.5N | 0.4 | 255.15 | 255.15 | 0.00 | 0.00 |
| | 0.5 | 264.30 | 264.32 | 0.02 | 0.01 |
| | 0.65 | 266.03 | 266.03 | 0.01 | 0.00 |
| CEM I 52.5N/ GGBS | 0.4 | 263.86 | 263.89 | 0.02 | 0.01 |
| | 0.5 | 272.59 | 272.64 | 0.05 | 0.02 |
| | 0.65 | 283.68 | 283.72 | 0.04 | 0.01 |
| CEM I 52.5N/ FA | 0.4 | 302.12 | 302.14 | 0.02 | 0.00 |
| | 0.5 | 284.70 | 284.75 | 0.05 | 0.02 |
| | 0.65 | 286.42 | 286.45 | 0.03 | 0.01 |
| CEM II (B) 42.5N | 0.4 | 289.64 | 289.72 | 0.08 | 0.03 |
| | 0.5 | 287.71 | 287.72 | 0.01 | 0.00 |
| | 0.65 | 293.58 | 293.58 | 0.01 | 0.00 |

The mass gain is negligible for all mixes. It can therefore be concluded that the current saturation time is adequate for the mixes tested in this work. Further tests to determine if the soaking time could be shortened were unfortunately not carried out. This could have a practical implication and make the test more time efficient if the saturation time can be shortened.

## 4.8. Use of Different Chloride Conductivity Cells

The DI test manual provides a schematic diagram for the chloride conductivity test and can be interpreted in different ways provided the schematic is followed. The test was developed at UCT in 1996 and has been in use since its inception. A new design has been put forward which incorporated functional features that are supposed to make the test more accurate, robust and easy to use. The inclusion of telescopic luggin capillaries ensure that the potential difference is measured exactly across the specimen faces as opposed to those used in the UCT rig, which are fixed in place and do not measure the potential difference directly across the specimen. The space between the end of the luggin and the specimen face is difficult to measure due to variable specimen thicknesses and the fact that the test rig is opaque, so one cannot be sure of the exact distance that the luggin is away from the specimen face once the rig is assembled. The New rig has been described in more detail in Section 2.10.3.

One of the main practical issues with the CCI test is that it is a messy test to perform, resulting from spillage of the NaCl solution when filling and refilling the cells. This is largely unavoidable because the apparatus needs to be disassembled and reassembled, and topped up with solution, before each specimen is tested.

Tests were conducted using the UCT rig and the New Rig to determine the difference in conductivity results, if any. Specimens were prepared according to the 2010 DI manual and then tested in both rigs. The same specimens were used in both rigs. The order they were tested was alternated; the first specimen was tested in the UCT rig and then, immediately after, in the New rig; and then the order reversed for the next specimen i.e. test in the New Rig then in the UCT rig, and so on. The results are shown in Table 4.15. The results are displayed for the individual binders, showing the variability, in Figures 4.29 to 4.32.

**Table 4.15: Chloride Conductivity Results for the UCT rig and the New Rig**

| Binder | w/c | UCT Rig | New Rig | Percentage Difference |
|---|---|---|---|---|
| CEMI 52.5N | 0.40 | 0.63 | 0.74 | 17% |
| | 0.50 | 0.65 | 0.86 | 32% |
| | 0.65 | 1.13 | 1.64 | 46% |
| CEMI 52.5N/GGBS | 0.40 | 0.28 | 0.34 | 22% |
| | 0.50 | 0.30 | 0.49 | 66% |
| | 0.65 | 0.64 | 1.09 | 69% |

**Table 4.15: Chloride Conductivity Results for the UCT rig and the New Rig (continued)**

| Binder | w/c | UCT Rig | New Rig | Percentage Difference |
|---|---|---|---|---|
| CEMI 52.5N/FA | 0.40 | 0.32 | 0.59 | 88% |
| | 0.50 | 0.59 | 0.70 | 17% |
| | 0.65 | 0.94 | 1.03 | 9% |
| CEM II (B) 42.5N | 0.40 | 0.59 | 0.69 | 17% |
| | 0.50 | 0.69 | 0.79 | 14% |
| | 0.65 | 0.73 | 0.81 | 10% |

The difference between the results obtained from both rigs are shown in Table 4.15. The percentage difference in conductivities for the CEM I 52.5N binder increases from 17% to 46% as the w/c ratio increases from 0.4 to 065. The New rig therefore appears to produce results which are higher than the UCT rig, with differences that become more pronounced as the w/c ratio increases for this binder, as shown in Figure 4.29.

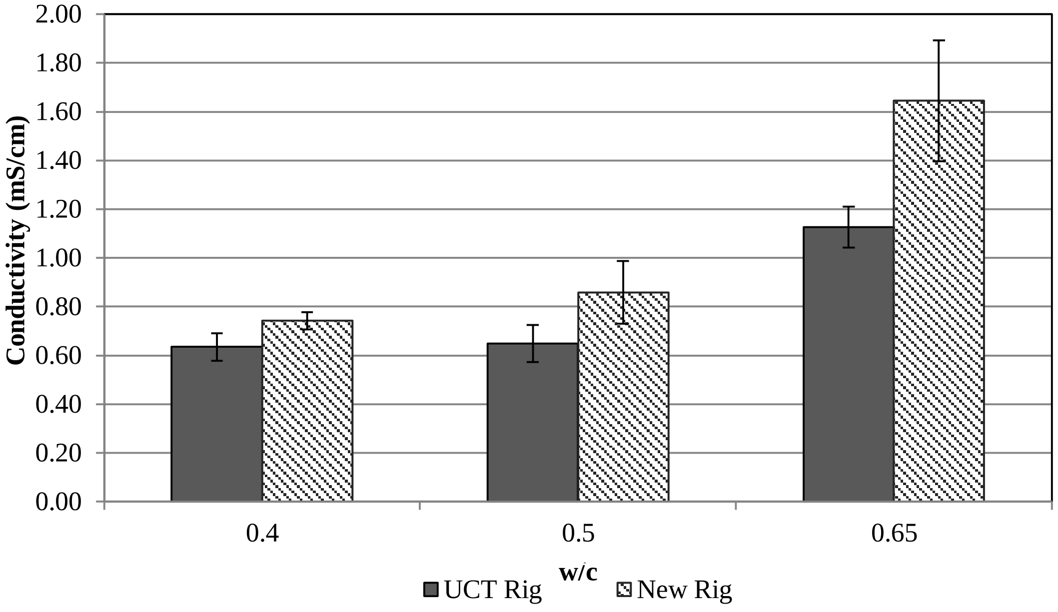

**Figure 4.29: CCI results for CEM I 52.5N**

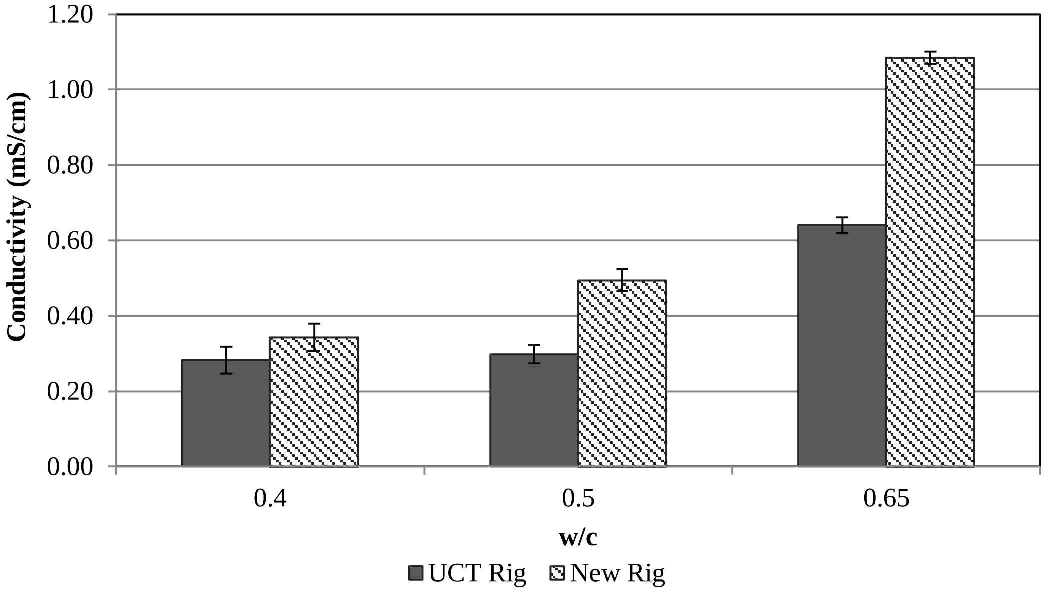

**Figure 4.30: CCI results for CEM I 52.5N/GGBS**

As with the chloride conductivity results obtained for CEM I52.5N, there is a difference between the results obtained from the two rigs for the GGBS-blended binder. The large difference of 66% and 69% can be attributed to the low conductivities. Even a small change in conductivity will register a large percentage difference when the conductivities are low. GGBS is known to have good binding abilities therefore the low chloride conductivities are expected.

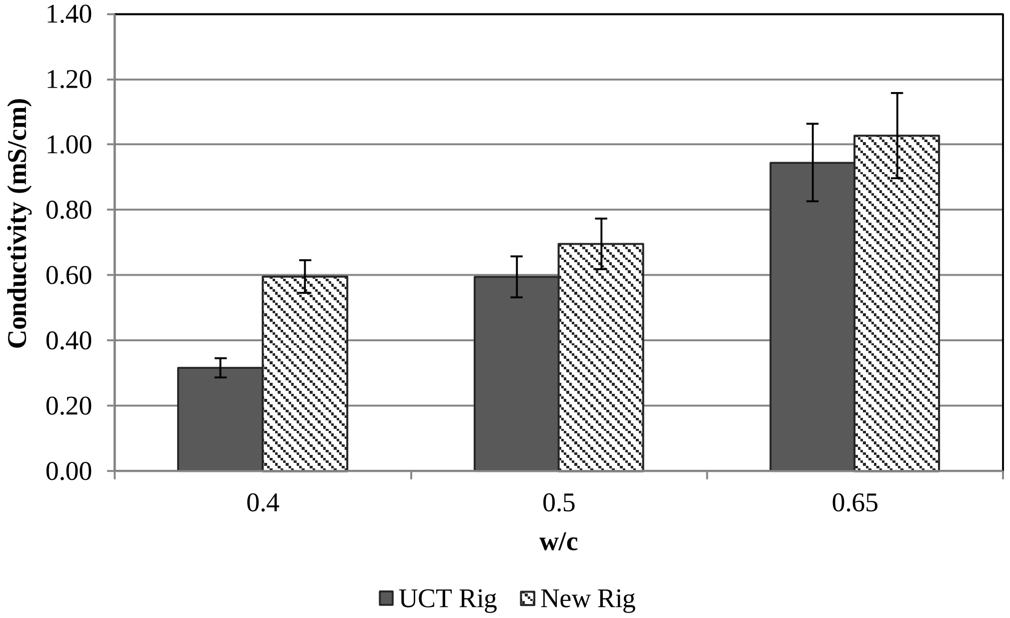

**Figure 4.31: CCI results for CEM I 52.5N/FA**

The percentage difference decreased as the w/c ratio increased for the FA-blended binder. The 0.4 w/c ratio mix shows a large difference of 88%, and is not consistent with the relatively lower differences for this w/c ratio for other binders. It may represent an anomaly.

The 9 % difference for the 0.65 w/c ratio is small indicating a low difference for this w/c ratio.

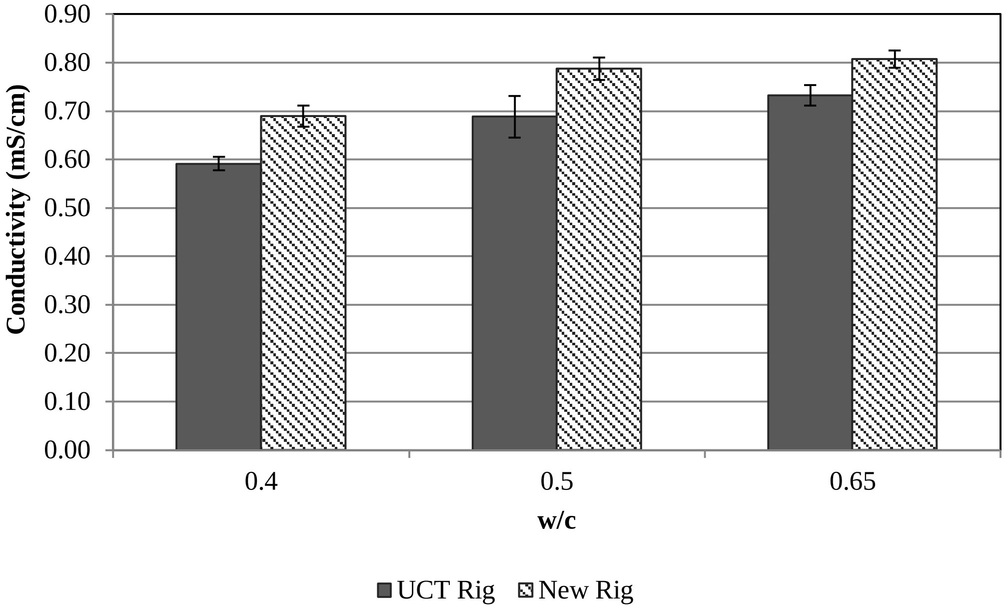

**Figure 4.32: CCI results for CEM II (B) 42.5N**

As with the other binders, there were differences in results obtained with the two rigs for the CEM II (B) 42.5N binder. However, the differences were smaller compared to those from other binders.

The chloride conductivity results for the two rigs were consistently different. The New Rig always produced results which were higher than the UCT rig. The difference between the results however was variable, nor was there a trend noticed for the different concretes tested. The conductivity results were therefore analyzed statistically to determine if these differences were statistically significant at the 95% confidence level. As with the previous statistical tests used in this study, the rank sum test was performed on the conductivity results and then verified with the t-test. This is shown in Table 4.16. The null hypothesis, $H_0$, is that the means of the results for the two rigs are equivalent. Section 3.6 described the statistical method in more detail.

The results of the statistical analysis show that, for the majority of the mixes, there do exist differences in the chloride conductivities obtained from the two rigs. The t-test rejected more tests than the rank-sum test, due to the way that the rank-sum test calculations are performed. The rank-sum test did not exhibit any trend for whether the null hypothesis was accepted or rejected. From these results it cannot be concluded that the rigs produce similar results. These results imply that it is very important that a consistent rig design and manufacture is used, and that different designs will lead to different results.

**Table 4.16: Statistical analysis results of differences between the New Rig and UCT rig**

| Binder | w/c | Rank-Sum Test |  |  | T-test |  |  |
|---|---|---|---|---|---|---|---|
|  |  | p-value | U* | Accept/Reject $H_0$ | t | $t_{crit}$ | Accept/Reject $H_0$ |
| CEM I 52.5N | 0.4 | 0.06 | 1 | Accept | -3.19 | 2.57 | Reject |
|  | 0.5 | 0.06 | 1 | Accept | -2.80 | 2.57 | Reject |
|  | 0.65 | 0.03 | 0 | Reject | -3.97 | 2.78 | Reject |
| CEM I 52.5N/GGBS | 0.4 | 0.06 | 1 | Accept | -2.41 | 2.45 | Accept |
|  | 0.5 | 0.03 | 0 | Reject | -10.20 | 2.45 | Reject |
|  | 0.65 | 0.03 | 0 | Reject | -34.33 | 2.45 | Reject |
| CEM I 52.5N/FA | 0.4 | 0.03 | 0 | Reject | -9.57 | 2.57 | Reject |
|  | 0.5 | 0.11 | 2 | Accept | -2.03 | 2.45 | Accept |
|  | 0.65 | 0.34 | 4 | Accept | -0.94 | 2.45 | Accept |
| CEM II (B) 42.5N | 0.4 | 0.03 | 0 | Reject | -7.47 | 2.57 | Reject |
|  | 0.5 | 0.03 | 0 | Reject | -4.05 | 2.57 | Reject |
|  | 0.65 | 0.03 | 0 | Reject | -5.33 | 2.45 | Reject |

**$H_0$ rejected when a U-value less than 1 was obtained.*

These results can have implications for compliance testing. The New Rig which produces higher results may, depending on the concrete being tested, produce higher conductivity readings which may be over a required design threshold, while the UCT rig may be below it. As a result, there will be uncertainty regarding which results are more appropriate to use. Ultimately this discrepancy may have financial implications on projects if they are used as the criteria for acceptance or rejection of the concrete as one test rig may reject concrete which is of adequate quality while another may accept poor-quality concrete.

The possible reasons for the differences in the two rigs are discussed below.

### 4.8.1. Possible Reasons for Differences Between Cells

The New Rig consistently produced higher conductivity results for all concrete mixes tested. To determine the factors which may be responsible for the differences it is necessary to look at the differences between the two rigs.

- **The presence of the telescopic luggin capillaries in the new rig**

The calculations provided here aim to determine whether a change in the luggin distance from the specimen face accounts for the large differences in measured conductivities between the rigs. There are potential losses that occur in the electrodes, the electrode-solution interface, in the solution, the solution-specimen interface and through the specimen itself as illustrated in Figure 4.33. The equation to calculate the conductivity is shown in Equation 4.1.

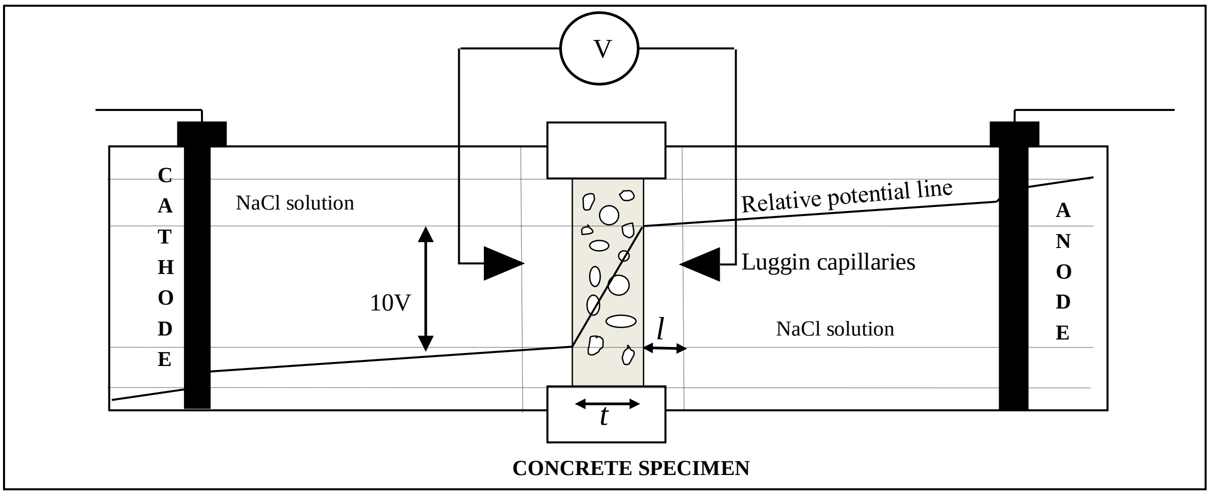

**Figure 4.33: Representation of the Chloride Conductivity Cell showing potential drops**

The total resistance in the system can be thought of as comprising the resistivity in the solution and the resistance of the specimen. Conductivity is the inverse of resistance. Streicher (1997) gives the conductivity of the 5M NaCl solution used in the CCI test as 213.7 mS/cm, therefore its resistance is 0.047 Ωm. Equation 4.2 states Ohms Law which is used to derive a relationship between the applied potential difference (*V*), the current (*i*), the resistivity (*ρ*), cross sectional area (*A*), and distance from the face of the specimen (*l*) in Equations 4.3 and 4.4.

$$\sigma = \frac{it}{VA} \tag{4.1}$$

$$V = iR \tag{4.2}$$

Where $R$ is the total resistance across the luggin capillaries given by $R_{\mathrm{NaCl}}+R_{\mathrm{concrete}}$

$$V = i\left(R_{NaCl} + R_{concrete}\right) = i\left(\left(\rho\frac{l}{A}\right)_{NaCl} + \left(\rho\frac{l}{A}\right)_{concrete}\right) \tag{4.3}$$

$$10 = i\left(\left(0.047\frac{2l}{A_{NaCl}}\right) + \left(\rho_{concrete}\frac{t}{A_{concrete}}\right)\right) \tag{4.4}$$

If the voltage across the area of measurement (i.e. across the distance $t$+2$l$ in Figure 4.33) is 10V and the current, $i$, is constant in the system it can be seen that an increase in the luggin capillary distance from the specimen face should register an increase in the conductivity. However, the total contribution from the solution is of the order of $10^{-2}$ so the effect of the length, $l$, on the total resistance is small. Hence, while the telescopic luggin capillaries in the new rig is an advanced design feature, the large difference in conductivities obtained from the New and UCT rigs cannot be attributed this feature.

- **Fully filling the cells in the new rig**

The New rig makes use of bleed holes to ensure that the cells are full with solution. Furthermore, these holes allow entrapped air to be removed by tapping or lightly shaking the rig thereby causing air bubbles to rise to the top and be released. The UCT rig does not have this feature and once the solution is poured in the half-cell there is no way to be certain that the cell is completely full, or that there is no air present in the system. If there is air in the system this could potentially block the migration of chloride ions causing a decrease in the current registered and as a result, a lower conductivity.

- **Differences in electrode material**

The New rig makes use of steel-steel electrodes. The UCT rig is based on the first design and makes use of steel-carbon electrodes. The change from steel to carbon occurred about 10 years ago on the basis that it made no difference to the operation of the rig. The rapid chloride permeability test (ASTM C 1202), another migration test, also makes use of stainless steel electrodes. Although the type of electrode should not affect the conductivity readings, it does represent a difference in the two CCI rigs. The materials used in the CCI rigs, as far as possible, should be standard.

#### General Comments

It is unclear what is occurring within the two cells that could explain the difference in results. It would be preferable if the rig was transparent so that the solution could be visible and the operator could check whether or not the cell was correctly filled. Perspex® could be used as it is easily workable. The drawback of using Perspex® is that over time the material would become hazy and lose its transparent property due to salt deposits rendering the rig commercially unattractive. However, this could be used initially for research purposes to answer some of the questions that have arisen.

### 4.8.2. The Need for Calibration of Chloride Conductivity Rigs

During the process of testing the two rigs it became apparent that there is a need for calibration of different rigs. It is simple to use a non-conductive “blank” disc, such as PVC, to check the condition of the cells and rubber collars to check for leaks. In this case, the ammeter should measure a zero current in the system. However, there is currently no way of testing different cells to check if they produce similar conductivity results. For this reason it was decided to experiment with different materials to determine their potential to be used as calibration discs for chloride conductivity tests.

Different materials were brainstormed to for their potential use. It was decided to try out the following materials, chosen for their availability. Three materials were used in this study:

- Meranti teak hardwood
- CEM I 52.5N Mortar Sample of w/c ratio 0.65
- SMEG ceramic disc

The conductivities of the materials are shown in Table 4.17 and all fall outside the typical range of conductivities expected for concrete. The meranti teak hardwood proved difficult to saturate with NaCl. It would simply float to the top of the solution. Furthermore this material was thought to be fairly heterogeneous in its pore structure. The CEM I 52.5N mortar sample showed significant bleeding at the time of casting probably due to an inadequate volume of fine aggregate used in the mix so the conductivity results obtained may not be representative of the actual material. The ceramic disc was thought to provide the most promise as it is readily available for purchase and manufactured commercially so it would typically be of uniform porosity even though the thickness of the disc (18.87 mm) lay outside the recommended thickness of 30 ± 2 mm. Upon coring the ceramic disc it was noticed that the material was made up of different layers - a smooth surface layer and a slightly darker layer sandwiched in between the top and bottom cream-coloured layers indicating perhaps a composite material.

**Table 4.17: Possible materials for us as calibration specimens**

| **Calibration Disc** | **Current (mA)** | **Conductivity (mS/cm)** |
|---|---|---|
| Meranti teak | 400 | 3.21 |
| CEM I 52.5N mortar | 695 | 5.49 |
| Ceramic Disc | 730 | 3.93 |

Due to its initial promise, it was decided to test the ceramic disc over an eight week period to determine its stability in chlorides over prolonged periods and its ultimate suitability for use as a calibration disc. The disc was therefore kept soaking in a 5M NaCl solution and tested weekly for chloride conductivity. The results are shown in Table 4.18.

Week 1 was regarded as a “setting in period” because the conductivity increased from week 1 and then largely remained approximately constant thereafter. The mean chloride conductivity achieved from testing between week 2 and week 8 was 4.44 mS/cm with a standard deviation of 0.10 mS/cm. This resulted in a calculated coefficient of variation of 2.2%. The low coefficient of variation indicates a low variation between the results for the seven weeks. There is a general increase in the conductivity of the ceramic disc from week 1-3, after which it fluctuated between 4.30 and 4.57 mS/cm. This is shown in Figure 4.34.

**Table 4.18: Chloride conductivity test results for ceramic calibration disc**

| Week | Current (mA) | Chloride Conductivity (mS/cm) | Mean Conductivity (mS/cm) | Standard Deviation (mS/cm) | CoV (%) |
|---|---|---|---|---|---|
| 1 | 730 | 3.93 | "setting in" period | | |
| 2 | 810 | 4.36 | 4.44 | 0.10 | 2.2 |
| 3 | 831 | 4.47 | | | |
| 4 | 799 | 4.30 | | | |
| 5 | 850 | 4.57 | | | |
| 6 | 810 | 4.36 | | | |
| 7 | 830 | 4.47 | | | |
| 8 | 840 | 4.52 | | | |

The saturated mass of the ceramic disc also fluctuated during the testing period, indicating a change in the porosity of the specimen, possibly due to external factors such as fluctuations in ambient temperature. Figure 4.35 shows the changes in conductivity and changes in porosity relative to the results from week 2. A change in conductivity coincides with a change in porosity. A lower porosity, given by a lower saturated mass, means that the volume of NaCl solution in the specimen has decreased. This would have an effect of decreasing the conductivity of the specimen. The changes in terms of percentages are shown in Figure 4.36, where it can be seen that the percentage change in the porosity does not produce a similar percentage change in the conductivity. Therefore, while the porosity may have an effect on the conductivity, it is unclear exactly how this is related so it is difficult to predict the expected change in conductivity from the change in porosity.

The physical state of the SMEG disc was important as it is hoped that a calibration disc should be stable is the chloride solution. There were no visible signs of deterioration of the disc over the testing period. Furthermore, the saturated-mass and current readings for weeks 2 and 6 were the same (178.4 g and 810 mA respectively), indicating that the structure of the ceramic disc was preserved over that period. This can be expected since the ceramic is designed to withstand very high temperatures so similarly, it may perform well in a 'harsh' environment such as being placed in NaCl solution. Based on the results of these tests it appears that there is promise in using a ceramic disc such as the SMEG disc for calibration purposes. The problem however lies in the high conductivity calculated compared to typical concrete values which tend to be less than 2.5 mS/cm (Alexander et al., 2001).

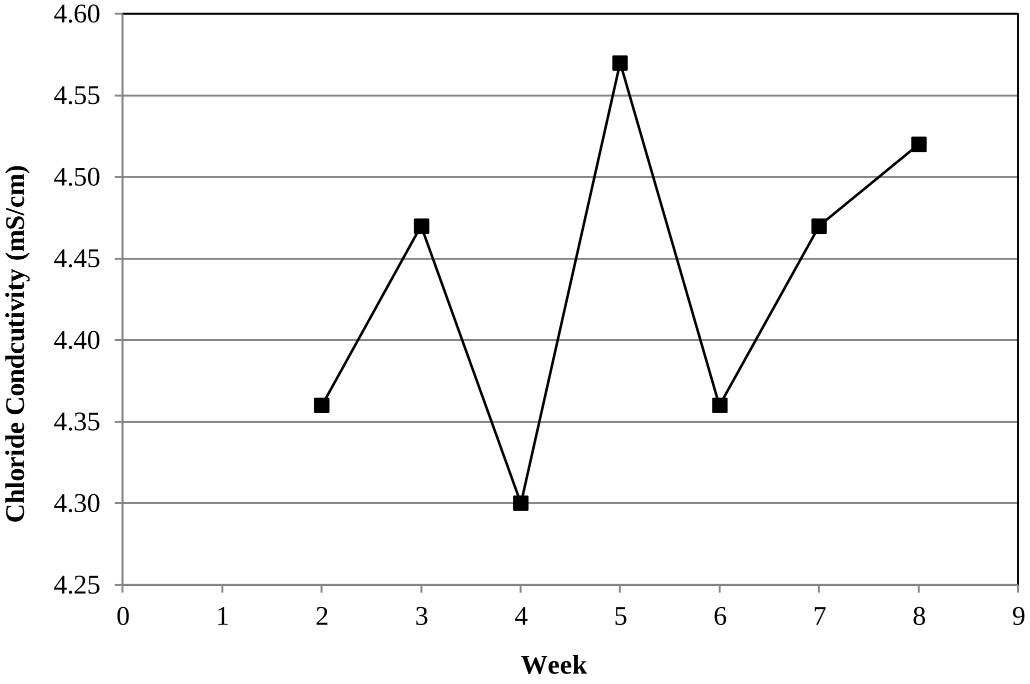

**Figure 4.34: Chloride conductivity of the SMEG disc**

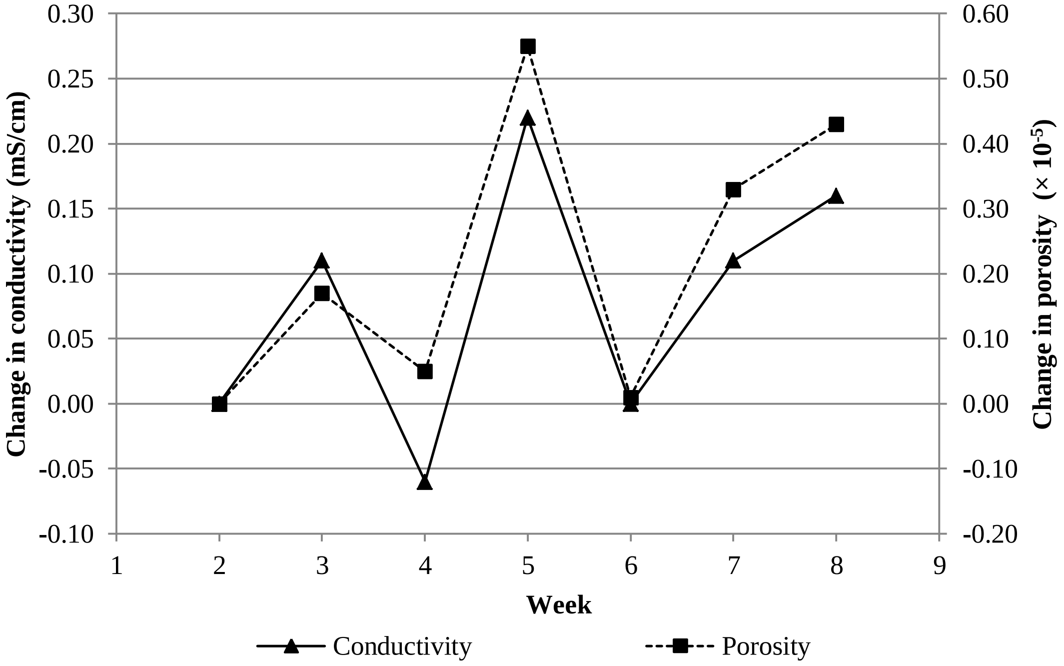

**Figure 4.35: Changes in conductivity and porosity of the SMEG disc using week-2 as a baseline**

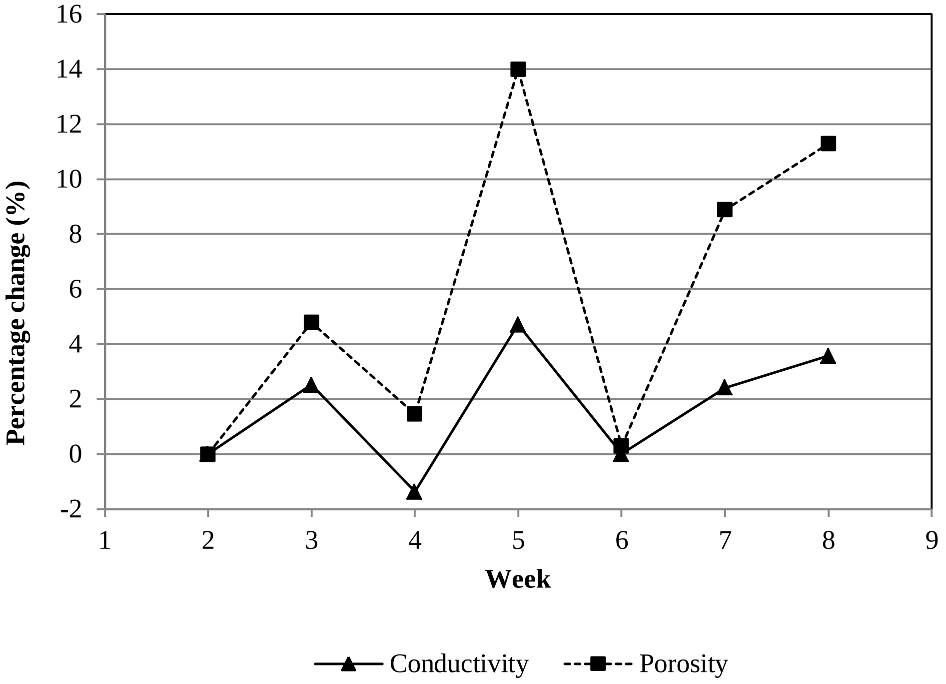

**Figure 4.36: Percentage changes in conductivity and porosity of the SMEG disc using week 2 as a baseline**

While testing the calibration disc a distinct pungent smell of chlorine gas was observed. Attempts were made to prove the nature of this gas using the litmus paper test. Typical tests would be done using blue litmus paper which turns red and then white in the presence of chlorine gas. There was only red litmus paper available for use in the laboratory. However, the damp red litmus paper did not turn bleached indicating the lack of a significant amount of chlorine gas evolution. Nevertheless, the smell could be disturbing. The reason for the evolution of a chlorine gas odour could be as a result of the high current in the system.

Due to the presence of $OH^-$ and $Cl^-$ ions in solution, either oxygen or chlorine gas may be discharged at the anode depending on the concentration of the solution. In the reactivity series, $OH^-$ ions lie below $Cl^-$ ions and are therefore oxidized more easily to form oxygen gas. Thus, in a dilute NaCl solution, oxygen would be produced at the anode. However, due to the NaCl solution used in the CCI test being concentrated, there is a higher concentration of $Cl^-$ ions available so they are able to be preferentially oxidised to chlorine gas at the anode. The higher than usual current experienced when using the ceramic calibration disc would mean a faster rate of oxidation at the anode resulting in more $Cl_2$ gas being released. Such high currents have not been experienced so this problem has not been observed before. A reduction in the volume of chlorine produced at the anode could be achieved by applying a 5 V potential difference across the specimen instead of 10 V to reduce the induced current.

## 4.9. General Discussion of Non-Experimental Aspects

There were other topics that did not fall within the experimental scope of work but were worth reviewing. This section provides a general discussion of these aspects for the three DI tests.

### 4.9.1. OPI Test

**Validity of the OPI Calculation Method**

The current method of calculating the OPI is to take the negative logarithm of the mean of the k values from four specimens. The arithmetic mean is used in this formula. The arithmetic mean as a measure of central tendency is based on the assumption of normality. However, permeability (k) values tend not to follow a normal distribution. Instead, they are log-normally distributed, meaning that the logarithm of the permeability follows a normal distribution. As a result, it is fundamentally incorrect to take the arithmetic mean of the k-values in the calculation of OPI. The geometric mean, on the other hand, can be used as a measure of central tendency for log-normally distributed data (Neves et al., 2013). This is equivalent to taking the mean of the log of the averages. Equation 4.5 shows that the geometric mean is equivalent to raising the power of the base ($a$) to the arithmetic mean of the logarithm. In terms of the OPI calculation, this translates into taking the negative average of the logs of individual k-values instead of the negative log of the average k-values, as shown in Equation 4.6.

$$\bar{k}_{geometric} = \left( \prod_{i=1}^{n} k_i \right)^{1/n} = a^{\left( \frac{1}{n} \sum_{i=1}^{n} \log_a k_i \right)} \tag{4.5}$$

For the case of four specimens, $n = 4$:

$$\bar{k}_{geometric} = \left( \prod_{i=1}^{4} k_i \right)^{1/4} = \sqrt[4]{k_1 k_2 k_3 k_4}$$

Taking the logarithm of both sides:

$$\log_{10}\left( \sqrt[4]{k_1 k_2 k_3 k_4} \right) = \frac{1}{4} \log_{10}\left( k_1 k_2 k_3 k_4 \right)$$

Using the property of logs and taking the negative of the terms, we obtain:

$$-\log_{10}\left( \sqrt[4]{k_1 k_2 k_3 k_4} \right) = -\left( \frac{\log_{10} k_1 + \log_{10} k_2 + \log_{10} k_3 + \log_{10} k_4}{4} \right) \tag{4.6}$$

The effect on the OPI when using the geometric mean instead of the arithmetic mean is shown in Table 4.19. The permeability results from the twelve mixes that were tested in this work were used in the calculations. The OPI was calculated using the following 3 equations (where $OPI_1$ is the traditional method and $OPI_2$ and $OPI_3$ use the geometric mean):

$$OPI_1 = -\log\left(\frac{k_1 + k_2 + k_3 + k_4}{4}\right) \tag{4.7}$$

$$OPI_2 = -\log\left(\sqrt[4]{k_1 k_2 k_3 k_4}\right) \tag{4.8}$$

$$OPI_3 = -\left(\frac{\log_{10} k_1 + \log_{10} k_2 + \log_{10} k_3 + \log_{10} k_4}{4}\right) \tag{4.9}$$

**Table 4.19: OPI using three different calculation methods**

| **Binder** | **w/c** | **Arithmetic Mean k (m/s) x $10^{-11}$** | **Geometric Mean k (m/s) x $10^{-11}$** | **$OPI_1$** | **$OPI_2$** | **$OPI_3$** |
|---|---|---|---|---|---|---|
| CEM I 52.5N | 0.4 | 6.93 | 6.47 | 10.16 | 10.20 | 10.20 |
| | 0.5 | 4.00 | 3.95 | 10.40 | 10.40 | 10.40 |
| | 0.65 | 6.56 | 6.32 | 10.18 | 10.20 | 10.20 |
| CEM I 52.5N/GGBS | 0.4 | 4.72 | 4.24 | 10.33 | 10.37 | 10.37 |
| | 0.5 | 7.61 | 7.21 | 10.12 | 10.14 | 10.14 |
| | 0.65 | 14.9 | 14.3 | 9.83 | 9.85 | 9.85 |
| CEM I 52.5N/FA | 0.4 | 1.21 | 1.18 | 10.92 | 10.93 | 10.93 |
| | 0.5 | 1.58 | 1.53 | 10.80 | 10.81 | 10.81 |
| | 0.65 | 4.16 | 3.94 | 10.38 | 10.40 | 10.40 |
| CEM II (B) 42.5N | 0.4 | 4.26 | 3.88 | 10.37 | 10.41 | 10.41 |
| | 0.5 | 7.30 | 6.47 | 10.14 | 10.19 | 10.19 |
| | 0.65 | 27.60 | 25.40 | 9.56 | 9.59 | 9.59 |

The results in Table 4.19 are significant in that they illustrate that using the geometric mean of the k-values ($OPI_2$ and $OPI_3$) is different to the traditional method, even if minimally in most cases. The $OPI_2$ and $OPI_3$ methods have a scientific basis. It is therefore proposed to change the OPI calculation to the following, taking the average of the log of the individual k values:

$$OPI = -\left(\frac{\log_{10} k_1 + \log_{10} k_2 + \log_{10} k_3 + \log_{10} k_4}{4}\right) \tag{4.10}$$

### Position of Inlet/Outlet Pipe

The OPI rigs have an inlet pipe through which the oxygen gas is introduced to the chamber. There is also an outlet pipe controlled by a valve which is used to remove the gas. Opening and closing the valves of the inlet/outlet pipes allows control of the pressure inside the chamber. Before the OPI test commences any gas that is in the chamber (air) needs to be purged with the pure oxygen. The density of pure oxygen is 1.331 kg/m$^3$ at 20 °C, while the density of air is 1.205 kg/m$^3$ at 20 °C. It stands to reason that the position of the inlet pipe will play a role in determining whether or not the air inside the chamber is all removed. If it is at the top then the oxygen introduced from the top will push down the 'impure' air and allow it to all be removed. If it is at the bottom then there will still be some 'impure' air remaining in the chamber above the oxygen.

### The Inclusion of Additional Volume at Top of Collar

The volume of the OPI chambers is 5 L ± 5%. Each OPI chamber may have a different volume due to tolerances in its manufacture. The rubber collar sits on top of the chamber and the specimen is placed within it. Depending on the orientation of the collar, there will be an additional volume which the oxygen will have to occupy before it reaches the concrete specimen. Although it is small, this volume should be included in the calculations to be more accurate.

The orientation of the collar also may play a role in the variability of results. Each collar has a lip on one end against which the specimen is pushed. If the lip is placed face-down to the chamber then the surface area of the concrete face that is in contact with the oxygen is reduced. A decrease in surface area will result in an increase in the calculated permeability coefficient. Furthermore, if the specimen is placed in the collar with the lip face down, the specimen may be pushed up to the top of the collar when the high pressure is applied, thereby increasing the volume of the 'chamber'.

### The Need for Proper Calibration and Zero Pressure Reading

The electronic pressure transducers constantly monitor the pressure in the OPI cylinders. They are supposed to be on a zero reading before the oxygen is introduced to a pressure of 100 ±5 kPa. If the pressure in the cylinder is not on 0 kPa to begin with then the initial 100 kPa value is misleading as the initial pressure will not be 100 kPa. Furthermore, the initial pressure has a tolerance of 5 kPa either side of 100 kPa, so it can range between 95 – 105 kPa. The calculation of z (Equation 4.11) makes use of the ratio of the initial pressure ($P_0$) to the pressure at time $t$ ($P_t$) is, and is ultimately used in the calculation of the permeability coefficient. The effect of different initial pressures on the permeability coefficient is not highly significant if the pressure decay is linear, as the ratio $P_0/P_t$ would not significantly change.

$$\mathrm{z} = \frac{\sum[\ln(P_0/P_t)]^2}{\sum[\ln(P_0/P_t)t]} \tag{4.11}$$

To demonstrate the effect of this on the k value, three calculations are performed, with the initial pressure at 95 kPa, 100 kPa and 105 kPa. All the pressure readings from the OPI test for one specimen from the CEM I 52.5N 0.5 w/c ratio mix used in this study were offset to have the initial pressure as 95 kPa, 100 kPa and 105 kPa and then the k-values using these pressures were calculated.

**Table 4.20: Example of having different initial pressures**

| Initial Pressure $P_0$ (kPa) | k (m/s) x $10^{-10}$ | OPI |
|---|---|---|
| 95 | 1.37 | 9.86 |
| 100 | 1.28 | 9.89 |
| 105 | 1.21 | 9.92 |

As can be seen from Table 4.20, there is a difference in the permeability coefficient calculated which will result in a variable OPI value depending on what the initial pressure is. While this difference is small, it can be avoided by eliminated the tolerance to only commence testing when the pressure is at 100 kPa. However this will result in a very stringent test, therefore some degree of tolerance should be included. A reduced tolerance of 100 ± 2 kPa is suggested as a compromise.

### 4.9.2. Water Sorptivity Test

#### Time to Test After OPI

The 2010 DI manual states that specimens may be tested for sorptivity immediately after they are taken out of the OPI rigs. It is assumed that the moisture condition of the specimens has not changed in that time. The OPI test has a maximum duration of 6 hours. Practically, it becomes unfeasible to control the timing if, for example, specimens are placed in the OPI rigs at 3p.m. This means that the OPI test will be complete by 9.p.m. It is unlikely that a laboratory test operator will be willing to commence sorptivity testing at that time. It is more likely that the test specimens will remain in the OPI rigs overnight. Therefore, in that time there may be moisture gain. The current manual does not state whether these specimens can be used without undergoing some form of additional drying before commencing sorptivity testing. Additional drying by placing the specimens in the 50 °C oven overnight should be introduced.

#### The Number of Specimens Tested at a Time

The sorptivity test takes 25 minutes to complete. As many specimens may be tested in this time as the user is able to. During this work, the author was able to test eight specimens simultaneously. It was difficult to test more than eight as the timing for recording the mass began to overlap. If there are many specimens to be tested then this test can be quite lengthy. A possible system may be incorporated which automatically records the mass gain. However, it may be difficult and expensive to have equipment that could measure the mas gain for individual samples.

### 4.9.3. Chloride Conductivity Test

**The Condition of the Electrodes**

The chloride conductivity test over time will corrode the electrodes. Currently there is no description of what constitutes electrodes which are damaged and reliance is given on the user judgement. Although the luggin capillaries ensure that the potential difference across the specimen is recorded independent of the potential loss across the electrodes, the metal electrodes dissolve and can be deposited on the specimen thereby affecting results (Streicher, 1997).

**The Condition of the Salt Solution**

The procedure is described in the 2010 manual on how to prepare the NaCl solution. A large batch may be prepared and stored to save the user time by not having to prepare the solution each time a test is done. However, experience has shown that the solution over time becomes brown in colour due to impurities. Thus it should be made clear that the solution should be checked to be of adequate quality before the test is conducted. The solution purity will have an effect on its conductivity and as a result it will also impact the concrete chloride conductivity results.

**General Usability of the Rig**

In testing the New CCI rig against the UCT version the New rig was, in the author’s experience, less user friendly to use than the UCT version; having more spillage and taking more time to set up. The UCT rig itself is not immune from this criticism. When there are many test specimens the testing time can be lengthy and tedious. Each time, the specimen needs to be placed in the central section leads need to be connected and disconnected. Obtaining readings is almost instantaneous as the voltage is increased to 10 V. A redesign of the rig should be considered to address its user-friendliness. One possible conceptual design has been included in Appendix F.

## 4.10. Critique of the DI Test Procedures

There have been new method statements for durability testing drawn up in 2013 for application as South African National Standards based on the OPI and CCI tests. At the time of writing these were in the process of being reviewed for approval. Until these are approved, the UCT method statement will continue to be used. This section presents a critique of the current method statements and where appropriate, presents possible modifications reflecting the conclusions from experimental work.

The version in use for this work was the 2010 DI Manual and can be found in Appendix D.

The following changes are suggested:

**Part 1: Standard Procedure for Preparation of Test Specimens**

This section was generally clear and simple to follow. In this study the preparation was done according to Clause 3. Preparation of specimens from cubes.

a) Concrete cubes shall be cast according to the relevant specification, and with minimum dimensions of 100 mm.

- This statement makes reference to a minimum dimension. It does not say how many cores may be obtained from each cube. Only one core can be obtained from the 100 mm cube when drilled in the centre. Previous studies have shown that one can obtain two cores when using a cube dimension of 150 mm (Govender, 2010). The number of obtainable cores is obviously limited by the dimensions of the cube. However, laboratory tests, have not been performed on cores obtained from cubes other than 100 mm or 150 mm. Therefore it is suggested to re-word the clause as the following:

*Concrete cubes shall be cast according to the relevant specification, and with dimensions of 100 mm, from which 1 core may be obtained; or 150 mm, from which 2 cores may be obtained.*

c) Coring of the cubes must take place at 28 ± 3 days after casting, unless otherwise required by project specifications.

- The leeway of 3 days either side of 28 day curing period will have an influence on the results. Although not tested in this dissertation, it is known that the curing period affects the OPI test results (Ballim, 1994; Govender, 2010). Ideally, the curing period should be 28 days with a tolerance of only a few hours. This, however, may be impractical. Therefore the effect of the six day tolerance could form a case for further study.

**Part 2: Standard Procedure for oxygen permeability Test**

Generally the OPI method statements are well explained. Several changes were made from the 2009 version to the 2010 version which made the method statements more refined. The content is generally easy to follow.

3 b) Remove the specimens from the oven and immediately place them in the desiccator.

- From the drying tests performed in this study, this point should be stressed so that the specimens are not kept in the oven longer than 7 days. The inclusion of the following:

*Remove the specimens from the oven and immediately place them in the desiccator. Specimens should not remain in the oven for longer than the abovementioned duration and should be kept in the desiccator until testing is required or for a maximum of one week, whichever occurs first.*

5. Calculations

- The link is given to obtain in a free copy of the spreadsheet from www.cnci.org.za. The Cement and Concrete Institute (CnCI) has since closed and only registered users may obtain it through this channel. Although new users may still register, it is not certain that this channel will be available for the foreseeable future. An alternative link should be provided.

d) The coefficient of permeability is calculated for each of the test specimens…

- For reasons described in Section 4.9.1, the OPI calculation method should be changed to

$$OPI = -\left(\frac{\log_{10} k_1 + \log_{10} k_2 + \log_{10} k_3 + \log_{10} k_4}{4}\right) \tag{4.12}$$

**Part 3: Standard Procedure for Water Sorptivity Test**

3 b) Remove the specimens from the oven and immediately place them in the desiccator.

- From the drying tests performed in this study, this point should be stressed so the at the specimens are not kept in the oven longer. The inclusion of the following:

*Remove the specimens from the oven and immediately place them in the desiccator. Specimens should not remain in the oven for longer than the abovementioned duration and should be kept in the desiccator until testing is required or for a maximum of one week, whichever occurs first.*

d) Ten layers of absorbent paper towel…

- Common paper towel may be purchased as 1-ply or 2-ply etc. It may be worth explaining the thickness of the paper towels. Non-standard conditions may be set up if one laboratory used 1-ply and others use a different thickness for example. The purpose of the paper towel is to be saturated with the $Ca(OH)_2$ solution so the test face of the concrete specimens makes good contact with the solution. Thus it does not matter how many layers are used, as long as the solution is adequately poured on the towels.

- The images for the test including the saturation tanks should be clearer.

**Part 4: Standard Procedure for Chloride Conductivity Test**

4 b) Remove the specimens from the oven and immediately place them in the desiccator.

- From the drying tests performed in this study, this point should be stressed so the at the specimens are not kept in the oven longer. The inclusion of the following:

*Remove the specimens from the oven and immediately place them in the desiccator. Specimens should not remain in the oven for longer than the abovementioned duration and should be kept in the desiccator until testing is required or for a maximum of one week, whichever occurs first.*

h) Adjust the DC power supply until the voltage applied across the specimen is approximately 10 Volts.
*Note: The preferred specimen test voltage is 10 V, but 5 V may be used for very permeable specimens.*

- Although the voltage should not have an effect on the conductivity calculations, a deviation from the 10 V standard potential difference may introduce a source of variability. It is unclear the impact on results that the 5 V potential difference will have.

6.c) The porosity as a percentage is determined by…:

- There is an error in the spelling of "percentage". This should be corrected.
- The purpose of using the chloride conductivity test for porosity calculations is not clear. Typically the WSI test would be used for porosity calculations in DI tests.

## 4.11. Conclusions

This chapter presented and discussed the results of the experimental work that was performed in the laboratory. A general discussion also highlighted the need for some areas of improvement with the test manual and the general test set up. The following sections summarise and provide conclusions from this chapter.

### 4.11.1. Compressive Strength

Compressive strength tests were performed as a measure of quality control of the mixes. The compressive strength generally followed the expected trends and showed that the mixes cast were of good consistency to perform the other tests required in this study.

### 4.11.2. Drying Tests

#### Drying Regime

The drying of specimens required in the DI tests was investigated. Two regimes were considered – oven drying and solvent replacement using isopropanol. The results showed that drying in an oven and drying with isopropanol do not result in similar amounts of moisture removal when the specimens were dried to constant mass. The isopropanol drying regime resulted in a lower overall mass loss in all the specimens compared to the oven drying method, possibly due to incomplete moisture removal. The oven drying method removed the majority of moisture within 7 days. This means that the current specified 7-day drying duration is therefore adequate.

Statistical analysis of the DI results for the isopropanol and oven drying methods also showed differences. The isopropanol method was used as an alternative drying method in this study. However it is not a feasible alternative to be used in practice and gave inconclusive evidence of complete moisture removal.

#### Oven Drying Duration

The effect of prolonged drying in the oven was investigated by comparing the DI results from specimens dried for 7 days and specimens dried to constant mass. Drying the specimens for 7 days and until constant mass resulted in statistically significant different DI results for some of the mixes tested. These differences were more pronounced in the CCI test results than in the OPI and WSI test results, which showed that there was no difference for certain mixes investigated.  The differences are most sensibly attributed to the increase in moisture loss; however it is not known if a change in microstructure also occurred as a result of prolonged oven drying which would also affect the DI results. It is therefore important to control the duration for which the specimens are dried so that they are in a similar state before they undergo the relevant DI test.  It is impractical to wait for the specimens to dry fully before performing the DI tests, which in some cases took up to 17 days.  An alternative solution should be considered, possibly removing the specimens after 7 days drying and placing them in a desiccator with a desiccant at a maximum 60% relative humidity for a maximum of one week until the DI test is performed to preserve their moisture state.

### 4.11.3. NaCl Saturation for CCI Test

The saturation of specimens with NaCl is an important step in the CCI test. Inadequate saturation will result in a more difficult passage of chloride ions through the concrete specimen. The saturation tests were performed by splitting the ‘saturated’ specimens in the centre and applying silver nitrate to indicate the presence of chloride ions. Results showed that many specimens, especially those made with GGBS, did not achieve a fully saturated state and were therefore not fully penetrated with chloride ions. This is important for the conductivity value as the non-uniform saturation of different mixes represents a source of variability in the test. The actual quantity of chlorides penetrating the centre of the specimen is unknown and should be investigated, since this will also affect the conductivity value.

The practical implications of the incomplete saturation of, mainly, low w/c slag mixes are also important. Generally, the CCI values for these mixes are already very low (typically <0.4 mS/cm, often as low as 0.2 mS/cm), meaning that these concretes are highly impenetrable by chlorides. Thus, it is unrealistic to expect them to be fully penetrated during the pre-saturation phase of the test. Nevertheless, the results can still be used for classifying concretes in terms of their chloride resistance, and for other purposes such as modelling, provided the limitations are borne in mind.

### 4.11.4. Adequacy of $Ca(OH)_2$ Saturation for WSI Test

The WSI requires specimens to be saturated with $Ca(OH)_2$ before determining the sorptivity and porosity of the specimen. According to the current procedure, this requires soaking in the solution for a period of 18 hours after the 4 hour vacuum saturation step. Additional 24 hour soaking was performed to determine if the 18 hour soaking period is adequate for fully saturating the specimens. It was concluded that the 18 hour soaking duration after the vacuum saturation of $Ca(OH)_2$ is adequate for producing fully saturated specimens. It may also be possible to shorten this duration to increase the tolerance of the test, however this was not investigated.

### 4.11.5. The Use of Two Different CCI Rigs

Two chloride conductivity rigs were tested to determine if there is a difference in results depending on the choice of rig selected. This involved testing specimens from different mixes in the UCT rig and a New Rig to determine if the conductivities from both the rigs were the same. It was shown that the New Rig consistently produced higher results, but to a variable degree. The type of rig used therefore has an influence on the conductivity results obtained.

The need for means of calibrating different conductivity rigs was investigated and discussed. This could take the form of a calibration disc made from a material which would be stable in the sodium chloride solution over time to produce consistent conductivity results. A ceramic disc manufactured by SMEG was investigated and showed good promise, by consistently producing similar conductivity results over a 10 week period. The drawback of this disc was the high average conductivity it showed of 4.4 mS/cm.

### 4.11.6. Calculation of OPI

The present method of taking the logarithm of average of four k-values was questioned in terms of its scientific basis. It was shown that the type of statistical mean used has an effect on the OPI, with the geometric mean considered to be more accurate and sound than the arithmetic mean. This is because of the log-normal distribution of permeability values. As a result, it was decided that the average of the logarithm of individual k-values should be used instead of the present calculation method.

### 4.11.7. General OPI Issues

The case was presented for the position of the inlet and outlet ports on OPI rigs to be standardised with the inlet port placed towards the top of the rig to ensure removal of all the existing air inside the chamber.

The additional volume due to the position of the rubber collar in which the test specimen is placed should be included as part of the volume of the chamber to increase the accuracy of the permeability calculations. Furthermore, the orientation of the rubber collar should be standardised to have the lip of the collar placed face-up so that it does not reduce the surface area of the contact between the oxygen and the specimen face.

The electronic pressure transducers used to measure the pressure inside the chambers should be properly zeroed before each test as this will impact on the results. The initial pressure tolerance of 5 kPa either side of 100 kPa also represents a source of variability as it affects the slope for linear regression which is used in the calculation of the permeability coefficient. The tolerance should therefore be reduced to 100 ± 2 kPa.

### 4.11.8. General WSI Issues

The WSI test was generally considered to be a simple test with the main issues considered to be the time taken after OPI testing and the number of specimens tested at one time. If specimens have undergone a change in their moisture condition then there may be a need to perform additional drying before performing the WSI test. This can be done by placing the specimens back in the 50 °C oven overnight and then commencing testing the next day.

The number of specimens tested should be limited to eight, as this was found to be the maximum number to comfortably perform the WSI test.

### 4.11.9. General CCI Issues

The condition of the electrodes was discussed and the need for adequate description of what constitutes damaged electrodes was highlighted. The purity of the salt solution used in the test was also highlighted and should be checked at regular intervals before conducting the

CCI test. In general, the usability of the CCI test was questioned and it was suggested that a new rig be designed which is more user-friendly without compromising accuracy.

#### 4.11.10. The DI Procedures

The DI procedures were reviewed and were considered to be generally simple to follow. They have undergone many revisions from the time the tests were first produced. However some alterations were proposed regarding the changing of wording for certain points.

# 5. CONCLUSIONS

## 5.1. Introduction

Over the past two decades, three DI tests, the oxygen permeability index (OPI), water sorptivity (WSI) and chloride conductivity (CCI) test have been developed in South Africa and refined to a stage where they are being used in industry and are in the process of being introduced as National Standards for the evaluation of concrete durability. They have a sound theoretical basis and are able to measure the transport processes of permeation, absorption and diffusion which are related to deterioration mechanisms in concrete. They are sensitive to changes in a number of intrinsic concrete properties. However, there are still some concerns surrounding the tests such as the variability of results and the general usability of the tests. The tests themselves need to be accurate and reliable if they are to be used with confidence in industry, especially since their results may form the basis for quality control compliance which may have financial implications. This study aimed to provide a critique of the current tests and, if possible, propose solutions where issues were identified. The main objectives evaluated in this work were to:

- Investigate the effect of the preconditioning methods currently used in the DI tests on the results for a range of w/c ratios and binders. Specifically, these included:
    - The oven drying preconditioning method, and a comparison of this with an alternative method involving the use of isopropanol as drying agent
    - The pre-saturation of specimens with NaCl solution before undergoing chloride conductivity testing
    - The saturation time required for the water sorptivity test
- Investigate the effect on chloride conductivity results when a different test rig is used
- Refine the Standard Test Method Statements as appropriate

This chapter shows how the above objectives were addressed and presents the main conclusions. Recommendations for further work are also proposed.

## 5.2. Investigation of the Preconditioning Methods

The preconditioning stage of the DI tests is an important step required to bring the specimens to a reference moisture state from which the relevant DI measurement can be taken. The presence of moisture in specimens effectively presents a barrier to the passage of oxygen for the OPI test and dilutes the NaCl solution used to saturate the specimens for the CCI test. Thus, it is critical that sufficient moisture be removed from the specimens before testing is performed. In order to investigate this, the specimens were exposed to different drying regimes involving the use of the present oven drying procedure ( 50 °C for 7 days) as well as a solvent replacement technique using isopropanol. The effect of prolonged 50 °C oven exposure, where specimens were dried to constant mass, was also investigated and compared to the current 7-day oven drying method. The specimens were exposed to the DI tests to determine the differences, if any, as a result of drying.

### 5.2.1. The Effect of Specimen Drying Regime and Duration

The type of drying method used in the preconditioning stage was found to have an effect on the DI results. The isopropanol drying technique would be difficult to implement in practice and therefore it is not suggested as an alternative method to the oven drying method. It resulted in less moisture loss, as measured by the mass loss over time, in all the mixes tested. It is therefore recommended that the current oven drying method, whereby specimens are placed in the oven at 50 °C, remains in place.

The duration of drying at 50 °C was found to be important. The results discussed in Chapter 4 showed that specimens dried to constant mass had different DI results to specimens removed from the oven after 7 days. It is interesting that only between 0.5 - 0.8 % of the total specimen mass was lost after 7 days, indicating that the majority of moisture is removed within the 7-day duration. However, the additional moisture in the pores did affect the DI results for many mixes. The OPI and WSI tests were shown to have a varying degree of sensitivity to the drying duration for certain mixes. The CCI test, on the other hand, was shown to be highly sensitive to the changes in drying duration for most of the mixes tested. Therefore, it is important to control the drying time to ensure that the moisture state of the specimens is similar to other specimens undergoing testing. A compromise between drying to constant mass and drying for 7 days should be achieved as it is impractical to wait for the specimens to completely dry, which took up to 17 days in some cases. It is therefore recommended to dry the specimens for the 7-day period at 50 °C and then remove them and place them in a desiccator, containing anhydrous silica gel and with the relative humidity at a maximum of 60%, for the remainder of the time required until they are tested. However this time should not exceed 1 week. This should be done for the OPI, WSI and CCI tests.

Of particular interest was the concrete tested which contained CEM I 52.5N as the binder. These mixes behaved differently compared to the expected behaviour, especially when exposed to the OPI test. It would be expected for the OPI results to follow a trend of the values becoming less (more permeable) as the w/c ratio increases due to the pore structure at higher w/c ratios become increasingly less dense. This was not the case, as seen by the fluctuating OPI results at different w/c ratios. This was attributed to the finely ground nature of the binder which enabled a more tightly packed concrete paste resulting in a relative insensitivity to changes in w/c ratio. These results highlight the need for OPI results obtained when CEMI 52.5N is used to be interpreted with care.

While this study did show that the DI results were affected by the drying method and duration, it was unclear whether this was caused purely due to differences in moisture condition or as a result of an alteration of the concrete microstructure. For this reason further work should be carried out to determine the nature of the concrete microstructure undergoing different drying durations.

### 5.2.2. The Specimen Saturation Condition for CCI

An important step in the CCI test is the pre-saturation of the test specimens with NaCl. This is the means by which chloride ions are introduced into the test specimen so that a more uniform pore solution between different mixes can be obtained. Concrete mixes based on plain PC, as well as blended mixes with higher w/c, can generally be penetrated by the chloride solution during the saturation phase of the test. However, blended cement concretes at lower w/c, and particularly slag blended concretes, are highly resistant to chloride penetration due to their dense microstructure and chloride binding capacity. In these cases, it is difficult to fully saturate the specimens with the chloride solution. Clearly this is not ideal. However, provided the test is carried out in a consistent and standard fashion, this test anomaly does not invalidate the test *per se*, since a chloride conductivity value can still be measured. These values are generally very low, indicating the excellent chloride resistance. Saturating for longer periods of time is also not practical due to the importance of a rapid test for quality control purposes. At this stage, this anomaly will need to be accommodated provided the limitations are understood.

### 5.2.3. The Specimen Saturation Duration for WSI

The WSI test requires that the test specimens be saturated with $Ca(OH)_2$ solution towards the end of the test. This involves a vacuum saturation step for 4 hours, followed by 18 hours soaking in the $Ca(OH)_2$ solution. There was concern whether specimens were exposed to the solution long enough to produce fully saturated specimens. It was determined that the current time employed is in fact adequate to fully saturate the specimens. This issue may be investigated further to determine if shorter soaking durations can be used. This would speed up the process and increase the tolerance of the sorptivity test.

## 5.3. The Effect of Using Two Different CCI Rigs

The current manual provides a schematic diagram for the chloride conductivity test. This is open to interpretation by different manufacturers, provided that the schematic is followed. The UCT rig has been in use for many years in the UCT laboratory and is the version that was initially developed. Another version of the rig, called the New Rig, was tested in this work. It had the same fundamental design as the UCT rig, but also had features such as telescopic luggin capillaries and bleed holes which proposed a more accurate conductivity reading. The conductivity tests performed using both rigs on twelve mixes made with different binders at different w/c ratios showed that the New rig consistently produced higher results than the UCT rig. Possible reasons for the differences were due to the New rig being able to be more fully filled with solution, and to a lesser extent the inclusion of the telescopic luggin capillaries which allowed the potential difference to be measured exactly across the specimen faces. The differences in results were, however, not consistent indicating that it was not a systematic error. This is significant in that it highlights an area of concern regarding the use of different CCI rigs.

Different equipment needs to provide comparable, accurate and reliable results if they are to be compared with limits for compliance. The results showed that there is a need for calibration of different chloride conductivity rigs. Until this issue can be adequately addressed, the UCT rig should be the ‘standard’ rig that is used, as this has been in use for many years and operators should be more experienced in using it.

### 5.3.1. Calibration of CCI Test

The previous section showed that there was a need for a calibration system to be implemented to ensure that different CCI rigs give comparable conductivity results. In this study, it was decided that a calibration disc be used which should be made from a material that is stable in a 5M NaCl solution over time. Different materials were assessed for use as a calibration disc in CCI tests including a meranti teak hardwood, a CEM I 52.5N mortar and a ceramic disc manufactured by SMEG. The ceramic disc showed good potential to be used in this manner. Initial tests that were conducted with the UCT rig showed that the ceramic disc gave stable results in a NaCl solution over a 10 week testing duration. It is recommended to continue using the disc and extend the testing to other conductivity rigs. The calibration disc should be prepared in the same way as the concrete specimens i.e. after coring and cutting, it should be placed in the 50 °C oven for 7 days and then vacuum saturated with 5M NaCl solution. Following this the disc should be kept in a 5M NaCl solution at all times when not being tested. The quality of the solution, in terms of its concentration purity, should be checked regularly and, if necessary, the solution should be changed to maintain the 5M concentration. In this way the disc will always be saturated with the required concentration of NaCl solution, ready to be tested when needed.

### 5.3.2. The Practicality of the CCI

The New Rig proposed many improvements over the UCT version but in actual use it proved to be inferior. In general, the CCI test is a messy test with a high likelihood of spillage occurring. In the experience of the author, the New Rig was messier than the UCT version. Furthermore both tests, at times, took a long time to set up. Each time a different specimen is to be tested, the rigs need to be dismantled, reassembled and connected with the leads. The test itself, where the voltage on the power supply is increased to obtain a 10 V across the specimen, takes only a few seconds.

Another issue with the CCI test is the filling of the cells with solution. As it stands, there is no way of ensuring that the cells are full when conducting the test. This is an area where the test can be improved. It would be ideal if the test operator could visually observe the solution inside the cells to determine whether they are full or not.

Due to the above issues, it is recommended that the CCI rig be redesigned to be more efficient in this regard. A conceptual design for a different set up is shown in Appendix F.

### 5.4. Non-Experimental Investigation of DI tests

Certain aspects surrounding all three DI tests were encountered in this work which were not tested experimentally but were investigated and discussed. In terms of the OPI, this included the need to change the present calculation method; it is proposed rather to take the negative average of the logarithms of the individual permeability coefficient values to make the calculation more statistically sound (see Equation 4.8). The inclusion of the additional volume in the rubber collar should also be included in calculations. Other aspects include the standardisation of the OPI rig design to have the oxygen inlet port close to the top and the outlet port close to the bottom of the chamber so that the denser oxygen can drive out the air. It was also highlighted that the pressure transducers need to be correctly calibrated and zeroed to be reliable. It was suggested that the initial pressure tolerance be reduced to 100 ± 2 kPa.

The WSI test was discussed and it was recommended that the testing of specimens directly after OPI testing should involve some additional overnight drying in the 50 °C oven to remove any moisture that may have been introduced during, and after, the OPI testing period.

Regarding the CCI test, the NaCl solution used should be monitored regularly to ensure that the 5M concentration is maintained. In addition, it is important to have properly maintained equipment to ensure reliability of the measurements obtained. A clear definition is required of what constitutes damaged electrodes in the CCI test.

### 5.5. Refining the Method Statements

The DI procedures outlined in the 2010 DI manual were used in this study. This was critiqued and where necessary changes to the wording were made. As previously mentioned, the test procedures are in the process of being formulated as National Standards. Unfortunately, these documents could not be reviewed because of confidentiality purposes. Until these are released, the current manual will be used. Several changes were proposed to reflect the work performed in this study. These include limiting the drying duration to 7 days and the change of the OPI calculation method mentioned in the preceding sections.

### 5.6. Recommendations for Further Work

As is the case with most research, many new questions arise after some have been answered. This work focussed on many areas in order to provide a general idea of whether or not suspected problem areas were, in reality, actual problems. A number of areas for further work were identified that went beyond the scope of this dissertation.

The issue of specimen dying was shown to have an effect on DI results. A limitation of this work is that the sample size for each test was relatively small (4 test specimens per test). It is suggested that further work examines this aspect in more detail by investigating specific binders and utilising many more repeat tests and larger sample sizes to provide a more conclusive result.

The oven drying method has been focussed on substantially in this work. This method remains the most feasible option for moisture removal, however, more work needs to be conducted to determine its effect on the microstructure of the concrete. Tests involving the use of Scanning Electron Microscopy (SEM) or Mercury Intrusion Porosity (MIP) could be conducted on specimens undergoing different drying durations in the oven. This would potentially answer the question of whether the difference in DI results shown in in this study was due to better removal of moisture at longer drying durations, or due to increased porosity as a result of a combined effect of moisture removal and micro-cracking.

The isopropanol solvent replacement method was investigated as an alternative drying regime, but was found to give much different results for the DI tests. It was thought that the specimens may not have had all the water removed by this method and would therefore have pores which were at least partially saturated with water and block the passage of oxygen in the case of the OPI. To test if all the moisture had in fact been removed, the specimens could be placed back in the oven at 50 °C to determine if further mass loss occurs.

It was shown that the vacuum saturation and subsequent 18 hour soaking duration was adequate to provide fully saturated test specimens in the case of the WSI. The possibility of shortening the soaking duration should be investigated. If the results show that shorter soaking durations can be used, then an improvement in the efficiency of the test can be made. This will increase the leniency in the tolerances of the test.

The chloride conductivity rig should be looked at from a practical perspective and a redesign could be produced to make it more efficient to use. A possible starting point could be the conceptual design shown in Appendix F.

An outcome of this dissertation was the promise of the ceramic calibration disc. More work should be done specifically to focus on this material and possible alternative ceramic materials to find a durable material that can be used as a calibration disc. This will go far to ensure that different CCI rigs produce comparable results.

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# APPENDIX A: CONCRETE MIX DETAILS

**Table A.1: Mix Specifications for CEM I 52.5N w/c ratio 0.4 Mix**

| **Material** | **Type** | **Relative Density** | **Mass/m3 (kg/m$^3$)** | **Absolute Volume (l)** | **Mass/20 L (kg)** |
|---|---|---|---|---|---|
| Water (Superplasticiser added if necessary) | | 1 | 170 | 170 | 3.4 |
| Binder | CEM I 52.5N | 3.14 | 425 | 135 | 8.5 |
| Extender | - | - | - | - | |
| Coarse Aggregate | Crushed Greywacke (19mm) | 2.7 | 1020 | 378 | 20.4 |
| Fine Aggregate | Philippi Dune Sand (50%) | 2.67 | 420 | 158 | 8.4 |
| | Klipheuwel Sand (50%) | 2.65 | 420 | 158 | 8.4 |
| | | Σ (SUM) | **2410** | **999** | **49.1** |

| Water:binder Ratio | 0.4 |
|---|---|

**Table A.2: Mix Specifications for CEM I 52.5N w/c ratio 0.5 Mix**

| **Material** | **Type** | **Relative Density** | **Mass/m3 (kg/m$^3$)** | **Absolute Volume (l)** | **Mass/20 L (kg)** |
|---|---|---|---|---|---|
| Water (Superplasticiser added if necessary) | | 1 | 170 | 170 | 3.4 |
| Binder | CEM I 52.5N | 3.14 | 340 | 108 | 6.8 |
| Extender | - | - | - | - | |
| Coarse Aggregate | Crushed Greywacke (19mm) | 2.7 | 1030 | 381 | 20.6 |
| Fine Aggregate | Philippi Dune Sand (50%) | 2.67 | 455 | 170 | 9.1 |
| | Klipheuwel Sand (50%) | 2.65 | 455 | 170 | 9.1 |
| | | Σ (SUM) | **2410** | **999** | **49** |

| Water:binder Ratio | 0.5 |
|---|---|

**Table A.3: Mix Specifications for CEM I 52.5N w/c ratio 0.65 Mix**

| Material | Type | Relative Density | Mass/m3 ($kg/m^3$) | Absolute Volume (l) | Mass/20 L (kg) |
|---|---|---|---|---|---|
| Water (Superplasticiser added if necessary) | | 1 | 170 | 170 | 3.4 |
| Binder | CEM I 52.5N | 3.14 | 262 | 83 | 5.24 |
| Extender | - | - | - | - | |
| Coarse Aggregate | Crushed Greywacke (19mm) | 2.7 | 1150 | 426 | 23 |
| Fine Aggregate | Philippi Dune Sand (50%) | 2.67 | 425 | 160 | 8.5 |
| | Klipheuwel Sand (50%) | 2.65 | 425 | 160 | 8.5 |
| | | Σ (SUM) | **2410** | **999** | **48.64** |

| Water:binder Ratio | 0.65 |
|---|---|

**Table A.4: Mix Specifications for CEM I 52.5N/GGBS w/c ratio 0.4 Mix**

| Material | Type | Relative Density | Volume ($kg/m^3$) | Absolute Volume (l) | Mass/20 L (kg) |
|---|---|---|---|---|---|
| Water (Superplasticiser added if necessary) | | 1 | 170 | 170 | 3.4 |
| Binder | CEM I 52.5N | 3.14 | 212 | 67 | 4.24 |
| Extender | GGBS | 2.9 | 212 | 73 | 4.24 |
| Coarse Aggregate | Crushed Greywacke (19mm) | 2.7 | 1020 | 378 | 20.4 |
| Fine Aggregate | Philippi Dune Sand (50%) | 2.67 | 420 | 158 | 8.4 |
| | Klipheuwel Sand (50%) | 2.65 | 420 | 158 | 8.4 |
| | | Σ (SUM) | **2410** | **1004** | **49.08** |

| Water:binder Ratio | 0.40 |
|---|---|

**Table A.5: Mix Specifications for CEM I 52.5N/GGBS w/c ratio 0.5 Mix**

| Material | Type | Relative Density | Volume (kg/m$^3$) | Absolute Volume (l) | Mass/20 L (kg) |
|---|---|---|---|---|---|
| Water (Superplasticiser added if necessary) | | 1 | 170 | 170 | 3.4 |
| Binder | CEM I 52.5N | 3.14 | 170 | 54 | 3.4 |
| Extender | GGBS | 2.9 | 170 | 58 | 3.4 |
| Coarse Aggregate | Crushed Greywacke (19mm) | 2.7 | 1030 | 381 | 20.6 |
| Fine Aggregate | Philippi Dune Sand (50%) | 2.67 | 455 | 170 | 9.1 |
| | Klipheuwel Sand (50%) | 2.65 | 455 | 170 | 9.1 |
| | | Σ (SUM) | **2410** | **1003** | **49** |
| Water:binder Ratio | 0.5 | | | | |

**Table A.6: Mix Specifications for CEM I 52.5N/GGBS w/c ratio 0.65 Mix**

| Material | Type | Relative Density | Volume (kg/m$^3$) | Absolute Volume (l) | Mass/20 L (kg) |
|---|---|---|---|---|---|
| Water (Superplasticiser added if necessary) | | 1 | 170 | 170 | 3.4 |
| Binder | CEM I 52.5N | 3.14 | 131 | 42 | 2.62 |
| Extender | GGBS | 2.9 | 131 | 45 | 2.62 |
| Coarse Aggregate | Crushed Greywacke (19mm) | 2.7 | 1150 | 425 | 23 |
| Fine Aggregate | Philippi Dune Sand (50%) | 2.67 | 425 | 159 | 8.5 |
| | Klipheuwel Sand (50%) | 2.65 | 425 | 159 | 8.5 |
| | | Σ (SUM) | **2410** | **1000** | **48.64** |
| Water:binder Ratio | 0.65 | | | | |

**Table A.7: Mix Specifications for CEM I 52.5N/FA w/c ratio 0.4 Mix**

| Material | Type | Relative Density | Volume (kg/m³) | Absolute Volume (l) | Mass/20 L (kg) |
|---|---|---|---|---|---|
| Water (Superplasticiser added if necessary) | | 1 | 170 | 170 | 3.4 |
| Binder | CEM I 52.5N | 3.14 | 297 | 94 | 5.94 |
| Extender | FA | 2.9 | 128 | 45 | 2.56 |
| Coarse Aggregate | Crushed Greywacke (19mm) | 2.7 | 1020 | 377 | 20.4 |
| Fine Aggregate | Philippi Dune Sand (50%) | 2.67 | 420 | 157 | 8.4 |
| | Klipheuwel Sand (50%) | 2.65 | 420 | 157 | 8.4 |
| | | Σ (SUM) | **2410** | **1000** | |
| Water:binder Ratio | 0.4 | | | | |

**Table A.8: Mix Specifications for CEM I 52.5N/FA w/c ratio 0.5 Mix**

| Material | Type | Relative Density | Volume (kg/m³) | Absolute Volume (l) | Mass/20 L (kg) |
|---|---|---|---|---|---|
| Water (Superplasticiser added if necessary) | | 1 | 170 | 170 | 3.4 |
| Binder | CEM I 52.5N | 3.14 | 238 | 75 | 4.76 |
| Extender | FA | 2.9 | 102 | 35 | 2.04 |
| Coarse Aggregate | Crushed Greywacke (19mm) | 2.7 | 1030 | 381 | 20.6 |
| | Philippi Dune Sand (50%) | 2.67 | 455 | 170 | 9.1 |
| Fine Aggregate | | | 455 | | 9.1 |
| | Klipheuwel Sand (50%) | 2.65 | 455 | 170 | 9.1 |
| | | Σ (SUM) | **2410** | **1001** | |
| Water:binder Ratio | 0.5 | | | | |

**Table A.9: Mix Specifications for CEM I 52.5N/FA w/c ratio 0.65 Mix**

| Material | Type | Relative Density | Volume (kg/m$^3$) | Absolute Volume (l) | Mass/20 L (kg) |
|---|---|---|---|---|---|
| Water (Superplasticiser added if necessary) | | 1 | 170 | 170 | 3.4 |
| Binder | CEM I 52.5N | 3.14 | 183 | 58 | 3.66 |
| Extender | FA | 2.9 | 78 | 27 | 1.56 |
| Coarse Aggregate | Crushed Greywacke (19mm) | 2.7 | 1150 | 426 | 23 |
| Fine Aggregate | Philippi Dune Sand (50%) | 2.67 | 425 | 160 | 8.5 |
| | Klipheuwel Sand (50%) | 2.65 | 425 | 160 | 8.5 |
| | | **Σ** (SUM) | **2410** | **1001** | |
| Water:binder Ratio | 0.65 | | | | |

**Table A.10: Mix Specifications for CEM II B-M (L-S) 42.5N w/c ratio 0.4 Mix**

| Material | Type | Relative Density | Volume (kg/m$^3$) | Absolute Volume (l) | Mass/20 L (kg) |
|---|---|---|---|---|---|
| Water (Superplasticiser added if necessary) | | 1 | 170 | 170 | 3.4 |
| Binder | CEM II B-M (L-S) 42,5N | 3.05 | 425 | 139 | 8.5 |
| Extender | - | - | - | - | |
| Coarse Aggregate | Crushed Greywacke (19mm) | 2.7 | 1020 | 377 | 20.4 |
| Fine Aggregate | Philippi Dune Sand (50%) | 2.67 | 420 | 157 | 8.4 |
| | Klipheuwel Sand (50%) | 2.65 | 420 | 157 | 8.4 |
| | | **Σ** (SUM) | **2410** | **1000** | |
| Water:binder Ratio | 0.4 | | | | |

**Table A.11: Mix Specifications for CEM II B-M (L-S) 42.5N w/c ratio 0.5 Mix**

| Material | Type | Relative Density | Volume (kg/m$^3$) | Absolute Volume (l) | Mass/20 L (kg) |
|---|---|---|---|---|---|
| Water (Superplasticiser added if necessary) | | 1 | 170 | 170 | 3.4 |
| Binder | CEM II B-M (L-S) 42,5N | 3.05 | 340 | 111 | 6.8 |
| Extender | - | - | - | - | |
| Coarse Aggregate | Crushed Greywacke (19mm) | 2.7 | 1030 | 381 | 20.6 |
| Fine Aggregate | Philippi Dune Sand (50%) | 2.67 | 455 | 170 | 9.1 |
| | Klipheuwel Sand (50%) | 2.65 | 455 | 170 | 9.1 |
| | | Σ (SUM) | **2410** | **1002** | |

| Water:binder Ratio | 0.50 |
|---|---|

**Table A.12: Mix Specifications for CEM II B-M (L-S) 42.5N w/c ratio 0.65 Mix**

| Material | Type | Relative Density | Volume (kg/m$^3$) | Absolute Volume (l) | Mass/20 L (kg) |
|---|---|---|---|---|---|
| Water (Superplasticiser added if necessary) | | 1 | 170 | 170 | 3.4 |
| Binder | CEM II B-M (L-S) 42,5N | 3.05 | 262 | 86 | 5.24 |
| Extender | - | - | - | - | |
| Coarse Aggregate | Crushed Greywacke (19mm) | 2.7 | 1150 | 426 | 23 |
| Fine Aggregate | Philippi Dune Sand (50%) | 2.67 | 425 | 159 | 8.5 |
| | Klipheuwel Sand (50%) | 2.65 | 425 | 159 | 8.5 |
| | | Σ (SUM) | **2410** | **1000** | |

| Water:binder Ratio | 0.65 |
|---|---|

# SIEVE ANALYSIS

Uct Concrete Laboratory

Aggregate sample: New klipheuwel sand

1000g

Test No: 1

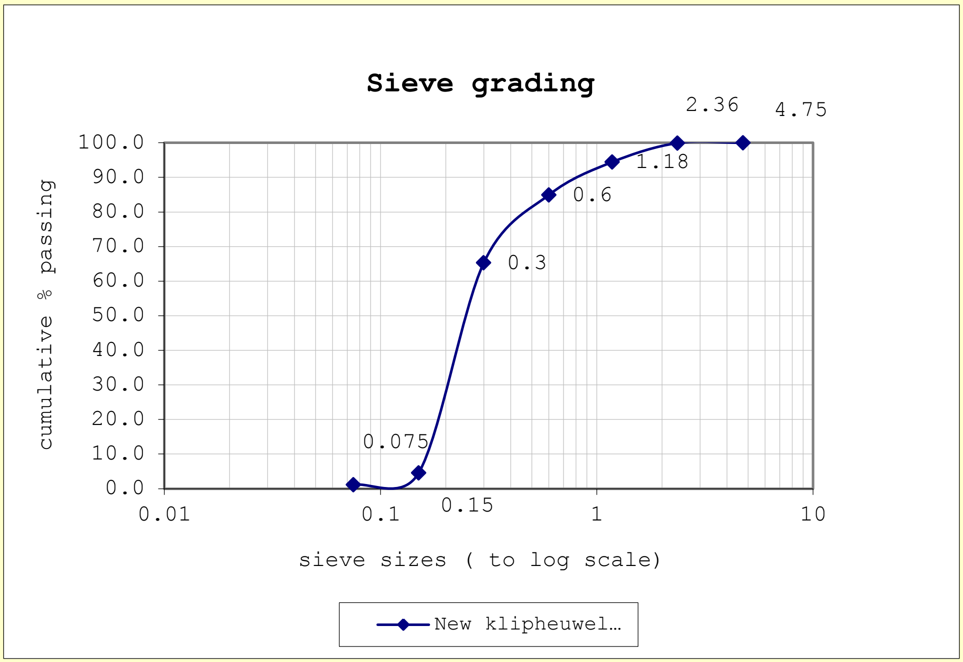
Date: 06/2012

Tested by: cmay

**Finess Modulus:** 1.5

| Sieve opening (mm) | MASS Sieve (g) | MASS Sieve + aggr. (g) | MASS retained (g) | Mass retained % | cum. Retained (%) | cum. % passing (%) |
|---|---|---|---|---|---|---|
| 4.75 | 437.6 | 437.6 | 0.0 | 0.0 | 0.0 | 100.0 |
| 2.36 | 413.0 | 413.7 | 0.7 | 0.1 | 0.1 | 99.9 |
| 1.18 | 548.4 | 603.1 | 54.7 | 5.5 | 5.5 | 94.5 |
| 0.6 | 503.3 | 598.4 | 95.1 | 9.5 | 15.1 | 84.9 |
| 0.3 | 490.5 | 686.4 | 195.9 | 19.6 | 34.7 | 65.3 |
| 0.15 | 473.4 | 1080.5 | 607.1 | 60.8 | 95.4 | 4.6 |
| 0.075 | 261.2 | 295.8 | 34.6 | 3.5 | 98.9 | 1.1 |
| PAN | 457.2 | 468.2 | 11.0 | 1.1 | | |
| | | | | | | |
| | | | | | | |

| | |
|---|---|
| sum mass | 999.1 |



# SIEVE ANALYSIS

Uct Concrete Laboratory

Aggregate sample:
DUNE SAND
1000g

Test No: 2
Date: 13.04.2013
Tested by: cmay

**Finess Modulus:** 2.6
**Loose Bulk Density ($kg/m^3$)** 1600
**Compacted Bulk Density ($kg/m^3$)** 1720

| Sieve opening (mm) | MASS Sieve (g) | MASS Sieve + aggr. (g) | MASS retained (g) | Mass retained % | cum. Retained (%) | cum. % passing (%) |
|---|---|---|---|---|---|---|
| 4.75 | 480.0 | 480.0 | 0.0 | 0.0 | 0.0 | 100.0 |
| 2.36 | 415.0 | 415.0 | 0.0 | 0.0 | 0.0 | 100.0 |
| 1.18 | 350.0 | 380.0 | 30.0 | 3.0 | 3.0 | 97.0 |
| 0.6 | 505.0 | 1170.0 | 665.0 | 66.8 | 69.8 | 30.2 |
| 0.3 | 280.0 | 475.0 | 195.0 | 19.6 | 89.4 | 10.6 |
| 0.15 | 520.0 | 615.0 | 95.0 | 9.5 | 99.0 | 1.0 |
| 0.075 | 260.0 | 270.0 | 10.0 | 1.0 | 100.0 | 0.0 |
| PAN | 495.0 | 495.0 | 0.0 | 0.0 | | |
| | | | | | | |
| | | | | | | |
| | | sum mass | 995.0 | | | |

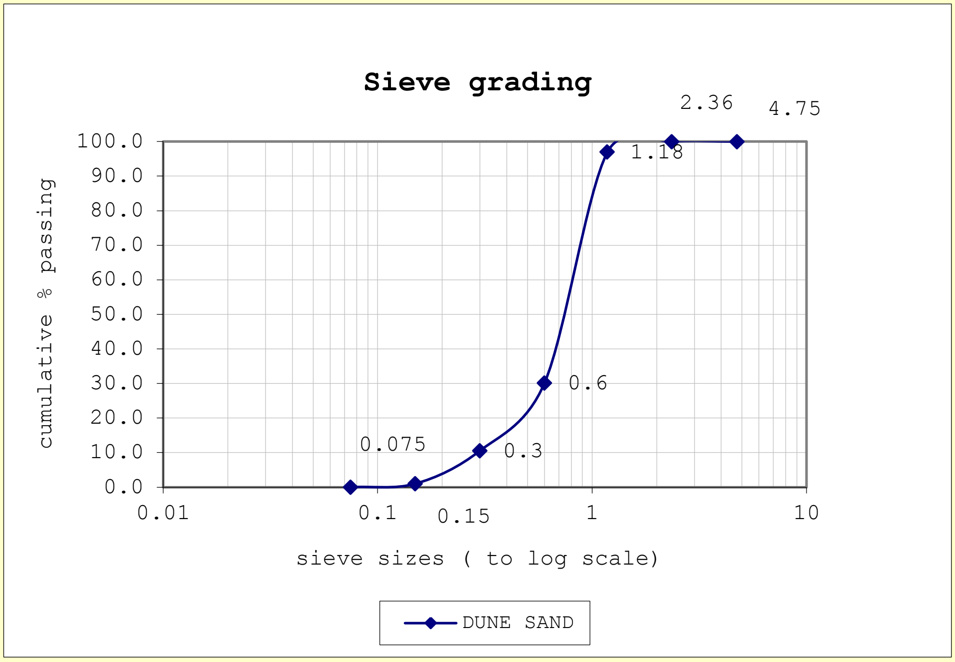


**Table A.13: Mix Slump and Superplasticizer Values**

| Casting Date | MIX | Binder Type (w/c) | Super-plasticizer added to 20L mix (ml) | Slump (mm) |
|---|---|---|---|---|
| 18-02-13 | 1 | CEM I 52,5N (0.4) | 45 | 80 |
| 20-02-13 | 2 | CEM I 52,5N (0.5) | 30 | 80 |
| 25-02-13 | 3 | CEM I 52,5N (0.65) | 0 | 85 |
| 27-02-13 | 4 | CEM I 52,5N/GGBS (0.4) | 45 | 85 |
| 04-03-13 | 5 | CEM I 52,5N/GGBS (0.5) | 35 | 80 |
| 07-03-13 | 6 | CEM I 52,5N/GGBS (0.65 | 0 | 80 |
| 18-03-13 | 7 | CEM I 52,5N/FA (0.4) | 35 | 95 |
| 25-03-13 | 8 | CEM I 52,5N/FA (0.5) | 20 | 86 |
| 06-04-13 | 9 | CEM I 52,5N/FA (0.65 | 0 | 118 |
| 19-04-13 | 10 | CEM II(B) 42,5N (0.4) | 40 | 85 |
| 20-04-13 | 11 | CEM II(B) 42,5N (0.4) | 30 | 90 |
| 21-04-13 | 12 | CEM II(B) 42,5N (0.4) | 0 | 95 |

# APPENDIX B: DI TEST RESULTS

**Table B.1: k-Values (m/s) for each specimen from the OPI test**

| **Binder** | **w/c** | **7-day Oven Dried** | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | **1** | **2** | **3** | **4** | **Mean** | **S.D** | **CoV(%)** |
| CEMI 52.5N | 0.4 | 4.5E-11 | 5.4E-11 | 5.5E-11 | 1.2E-10 | 6.9E-11 | 3.6E-11 | 5.3E+01 |
| | 0.5 | 3.5E-11 | 3.3E-11 | 4.2E-11 | 5.0E-11 | 4.0E-11 | 7.6E-12 | 1.9E+01 |
| | 0.65 | 9.8E-11 | 5.7E-11 | 5.7E-11 | 5.0E-11 | 6.6E-11 | 2.2E-11 | 3.4E+01 |
| CEMI 52.5N/GGBS | 0.4 | 2.0E-11 | 7.3E-11 | 3.7E-11 | 5.8E-11 | 4.7E-11 | 2.3E-11 | 4.9E+01 |
| | 0.5 | 7.6E-11 | 9.7E-11 | 4.0E-11 | 9.1E-11 | 7.6E-11 | 2.6E-11 | 3.4E+01 |
| | 0.65 | 1.7E-10 | 8.4E-11 | 1.7E-10 | 1.7E-10 | 1.5E-10 | 4.3E-11 | 2.9E+01 |
| CEMI 52.5N/FA | 0.4 | 1.4E-11 | 1.4E-11 | 7.8E-12 | 1.2E-11 | 1.2E-11 | 3.1E-12 | 2.6E+01 |
| | 0.5 | 1.1E-11 | 1.7E-11 | 2.1E-11 | 1.4E-11 | 1.6E-11 | 4.1E-12 | 2.6E+01 |
| | 0.65 | 5.6E-11 | 4.4E-11 | 4.4E-11 | 2.2E-11 | 4.2E-11 | 1.4E-11 | 3.4E+01 |
| CEMII (B) 42.5N | 0.4 | 7.2E-11 | 2.4E-11 | 2.9E-11 | 4.6E-11 | 4.3E-11 | 2.2E-11 | 5.1E+01 |
| | 0.5 | 3.4E-11 | 1.1E-10 | 1.0E-10 | 4.6E-11 | 7.3E-11 | 3.9E-11 | 5.3E+01 |
| | 0.65 | 1.8E-10 | 1.9E-10 | 2.7E-10 | 4.7E-10 | 2.8E-10 | 1.4E-10 | 5.0E+01 |
| | | **Oven Dried to Constant Mass** | | | | | | |
| CEMI 52.5N | 0.4 | 1.2E-10 | 7.7E-11 | 5.7E-11 | 1.3E-10 | 9.4E-11 | 3.3E-11 | 3.5E+01 |
| | 0.5 | 5.2E-11 | 7.5E-11 | 4.5E-11 | 8.9E-11 | 6.5E-11 | 2.0E-11 | 3.1E+01 |
| | 0.65 | 1.7E-10 | 6.6E-11 | 4.5E-11 | 1.4E-10 | 1.4E-10 | 5.7E-11 | 3.9E+01 |
| CEMI 52.5N/GGBS | 0.4 | 3.4E-11 | 4.4E-11 | 4.5E-11 | 3.2E-11 | 4.8E-11 | 6.4E-12 | 5.0E+01 |
| | 0.5 | 4.0E-11 | 1.2E-10 | 4.5E-11 | 7.7E-11 | 7.1E-11 | 3.6E-11 | 4.8E+01 |
| | 0.65 | 6.6E-10 | 6.9E-10 | 4.5E-11 | 2.4E-10 | 5.5E-10 | 3.2E-10 | 3.8E+01 |
| CEMI 52.5N/FA | 0.4 | 1.5E-11 | 1.8E-11 | 4.5E-11 | 2.4E-11 | 2.3E-11 | 1.3E-11 | 3.7E+01 |
| | 0.5 | 3.1E-11 | 3.6E-11 | 4.5E-11 | 3.5E-11 | 3.1E-11 | 5.9E-12 | 1.6E+01 |
| | 0.65 | 5.3E-11 | 8.2E-11 | 4.5E-11 | 5.9E-11 | 9.5E-11 | 1.6E-11 | 6.5E+01 |
| CEMII (B) 42.5N | 0.4 | 4.5E-11 | 5.2E-11 | 1.9E-10 | 7.8E-11 | 9.1E-11 | 6.7E-11 | 7.4E+01 |
| | 0.5 | 2.1E-10 | 8.0E-11 | 8.9E-11 | 7.9E-11 | 1.1E-10 | 6.5E-11 | 5.7E+01 |
| | 0.65 | 4.8E-10 | 1.2E-10 | 1.5E-10 | 2.5E-10 | 2.5E-10 | 1.6E-10 | 6.5E+01 |
| | | **Isopropanol Dried** | | | | | | |
| CEMI 52.5N | 0.4 | 5.2E-11 | 8.9E-11 | 2.9E-11 | 5.9E-11 | 5.7E-11 | 2.5E-11 | 4.3E+01 |
| | 0.5 | 9.8E-11 | 1.1E-10 | 5.7E-11 | 9.7E-11 | 9.0E-11 | 2.3E-11 | 2.5E+01 |
| | 0.65 | 4.6E-11 | 6.0E-11 | 9.3E-11 | 4.8E-11 | 6.2E-11 | 2.2E-11 | 3.5E+01 |
| CEMI 52.5N/GGBS | 0.4 | 1.5E-11 | 4.2E-11 | 1.3E-11 | 1.3E-11 | 2.1E-11 | 1.4E-11 | 6.8E+01 |
| | 0.5 | 2.1E-11 | 4.3E-11 | 4.3E-11 | 2.1E-11 | 3.2E-11 | 1.2E-11 | 3.9E+01 |
| | 0.65 | 4.7E-11 | 6.9E-11 | 1.5E-10 | 6.6E-11 | 8.3E-11 | 4.6E-11 | 5.5E+01 |
| CEMI 52.5N/FA | 0.4 | 1.6E-11 | 1.4E-11 | 1.4E-11 | 1.5E-11 | 1.5E-11 | 1.0E-12 | 6.8E+00 |
| | 0.5 | 1.2E-11 | 1.7E-11 | 2.8E-11 | 1.3E-11 | 1.8E-11 | 7.5E-12 | 4.3E+01 |
| | 0.65 | 5.8E-11 | 2.6E-11 | 4.3E-11 | 2.6E-11 | 3.8E-11 | 1.5E-11 | 4.0E+01 |
| CEMII (B) 42.5N | 0.4 | 4.4E-11 | 5.7E-11 | 1.3E-11 | 1.3E-11 | 3.2E-11 | 2.3E-11 | 7.1E+01 |
| | 0.5 | 1.6E-11 | 1.8E-11 | 2.0E-11 | 2.7E-11 | 2.0E-11 | 5.0E-12 | 2.5E+01 |
| | 0.65 | 2.2E-10 | 4.7E-10 | 2.3E-10 | 1.9E-10 | 2.8E-10 | 1.3E-10 | 4.6E+01 |

**Table B.2: OPI results for each specimen**

| Binder | w/c | 7-day Oven Dried | | | | |
|---|---|---|---|---|---|---|
| | | **1** | **2** | **3** | **4** | **Mean** |
| CEMI 52.5N | 0.4 | 10.35 | 10.27 | 10.26 | 9.91 | 10.16 |
| | 0.5 | 10.46 | 10.48 | 10.38 | 10.30 | 10.40 |
| | 0.65 | 10.01 | 10.24 | 10.25 | 10.30 | 10.18 |
| CEMI 52.5N/GGBS | 0.4 | 10.69 | 10.13 | 10.43 | 10.23 | 10.33 |
| | 0.5 | 10.12 | 10.02 | 10.40 | 10.04 | 10.12 |
| | 0.65 | 9.76 | 10.07 | 9.77 | 9.77 | 9.83 |
| CEMI 52.5N/FA | 0.4 | 10.84 | 10.84 | 11.11 | 10.93 | 10.92 |
| | 0.5 | 10.96 | 10.76 | 10.69 | 10.85 | 10.80 |
| | 0.65 | 10.25 | 10.36 | 10.35 | 10.66 | 10.38 |
| CEMII (B) 42.5N | 0.4 | 10.14 | 10.62 | 10.54 | 10.34 | 10.37 |
| | 0.5 | 10.47 | 9.96 | 9.98 | 10.34 | 10.14 |
| | 0.65 | 9.75 | 9.73 | 9.57 | 9.33 | 9.56 |
| | | **Oven Dried to Constant Mass** | | | | |
| CEMI 52.5N | 0.4 | 9.93 | 9.93 | 9.93 | 9.93 | 9.93 |
| | 0.5 | 10.29 | 10.12 | 10.35 | 10.05 | 10.19 |
| | 0.65 | 9.78 | 10.18 | 9.71 | 9.87 | 9.85 |
| CEMI 52.5N/GGBS | 0.4 | 10.47 | 10.36 | 10.08 | 10.49 | 10.32 |
| | 0.5 | 10.39 | 9.93 | 10.29 | 10.12 | 10.15 |
| | 0.65 | 9.18 | 9.16 | 9.22 | 9.62 | 9.26 |
| CEMI 52.5N/FA | 0.4 | 10.84 | 10.74 | 10.47 | 10.63 | 10.65 |
| | 0.5 | 10.51 | 10.44 | 10.61 | 10.46 | 10.50 |
| | 0.65 | 10.27 | 10.09 | 9.73 | 10.23 | 10.02 |
| CEMII (B) 42.5N | 0.4 | 10.35 | 10.29 | 9.72 | 10.11 | 10.04 |
| | 0.5 | 9.67 | 10.10 | 10.05 | 10.10 | 9.94 |
| | 0.65 | 9.32 | 9.91 | 9.84 | 9.60 | 9.60 |
| | | **Isopropanol Dried** | | | | |
| CEMI 52.5N | 0.4 | 10.28 | 10.05 | 10.54 | 10.23 | 10.24 |
| | 0.5 | 10.01 | 9.96 | 10.24 | 10.01 | 10.04 |
| | 0.65 | 10.33 | 10.22 | 10.03 | 10.32 | 10.21 |
| CEMI 52.5N/GGBS | 0.4 | 10.81 | 10.38 | 10.90 | 10.88 | 10.68 |
| | 0.5 | 10.67 | 10.37 | 10.37 | 10.67 | 10.49 |
| | 0.65 | 10.33 | 10.16 | 9.82 | 10.18 | 10.08 |
| CEMI 52.5N/FA | 0.4 | 10.79 | 10.86 | 10.84 | 10.81 | 10.83 |
| | 0.5 | 10.93 | 10.76 | 10.55 | 10.88 | 10.75 |
| | 0.65 | 10.24 | 10.58 | 10.36 | 10.59 | 10.42 |
| CEMII (B) 42.5N | 0.4 | 10.35 | 10.24 | 10.89 | 10.90 | 10.50 |
| | 0.5 | 10.80 | 10.75 | 10.70 | 10.56 | 10.69 |
| | 0.65 | 9.67 | 9.33 | 9.64 | 9.72 | 9.56 |

The standard deviation and CoV for the OPI are based on the k-values in Table B.1.

**Table B.3: Sorptivity (mm/hr$^{0.5}$) results for each specimen**

| Binder | w/c | 7-day Oven Dried | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | 1 | 2 | 3 | 4 | Mean | S.D | CoV (%) |
| CEMI 52.5N | 0.4 | 8.84 | 9.55 | 7.47 | 7.99 | 8.46 | 0.92 | 9.92 |
| | 0.5 | 9.16 | 9.28 | 7.35 | 8.73 | 8.63 | 0.88 | 10.24 |
| | 0.65 | 9.97 | 10.60 | 8.29 | 10.46 | 9.83 | 1.06 | 10.82 |
| CEMI 52.5N/GGBS | 0.4 | 11.14 | 7.35 | 10.85 | 10.82 | 10.04 | 1.80 | 17.93 |
| | 0.5 | 6.48 | 11.73 | 9.17 | 10.78 | 9.54 | 2.30 | 24.08 |
| | 0.65 | 10.15 | 9.69 | 10.65 | 10.53 | 10.25 | 0.43 | 4.21 |
| CEMI 52.5N/FA | 0.4 | 6.74 | 7.22 | 7.92 | 9.34 | 7.81 | 1.13 | 14.48 |
| | 0.5 | 8.55 | 6.70 | 6.32 | 8.30 | 7.47 | 1.12 | 15.01 |
| | 0.65 | 9.18 | 8.34 | 9.96 | 7.43 | 8.73 | 1.09 | 12.47 |
| CEMII (B) 42.5N | 0.4 | 10.77 | 8.73 | 9.44 | 8.58 | 9.38 | 1.00 | 10.65 |
| | 0.5 | 10.38 | 10.03 | 8.55 | 10.42 | 9.85 | 0.88 | 8.93 |
| | 0.65 | 9.81 | 9.99 | 11.60 | 9.17 | 10.14 | 1.04 | 10.23 |
| | | Oven Dried to Constant Mass | | | | | | |
| CEMI 52.5N | 0.4 | 6.95 | 7.14 | 5.70 | 6.96 | 6.69 | 0.66 | 9.92 |
| | 0.5 | 8.81 | 6.69 | 6.97 | 7.12 | 7.40 | 0.96 | 12.95 |
| | 0.65 | 11.61 | 11.09 | 9.50 | 10.38 | 10.64 | 0.92 | 8.61 |
| CEMI 52.5N/GGBS | 0.4 | 7.92 | 7.29 | 7.87 | 7.45 | 7.63 | 0.31 | 4.08 |
| | 0.5 | 8.14 | 8.53 | 9.55 | 9.35 | 8.89 | 0.67 | 7.49 |
| | 0.65 | 12.68 | 12.84 | 12.10 | 10.07 | 11.92 | 1.27 | 10.68 |
| CEMI 52.5N/FA | 0.4 | 7.64 | 9.47 | 9.47 | 7.19 | 8.44 | 1.20 | 14.22 |
| | 0.5 | 9.60 | 10.06 | 9.14 | 8.28 | 9.27 | 0.76 | 8.21 |
| | 0.65 | 11.03 | 9.82 | 9.89 | 9.28 | 10.01 | 0.73 | 7.32 |
| CEMII (B) 42.5N | 0.4 | 10.93 | 8.14 | 10.33 | 9.78 | 9.79 | 1.20 | 12.21 |
| | 0.5 | 13.34 | 8.99 | 9.94 | 10.41 | 10.67 | 1.88 | 17.59 |
| | 0.65 | 11.04 | 10.73 | 10.14 | 12.97 | 11.22 | 1.22 | 10.91 |
| | | Isopropanol Dried | | | | | | |
| CEMI 52.5N | 0.4 | 4.79 | 5.70 | 4.79 | 4.98 | 5.06 | 0.43 | 8.54 |
| | 0.5 | 8.81 | 6.69 | 6.97 | 7.12 | 7.40 | 0.96 | 12.95 |
| | 0.65 | 7.14 | 7.39 | 7.97 | 7.52 | 7.51 | 0.35 | 4.65 |
| CEMI 52.5N/GGBS | 0.4 | 6.90 | 7.48 | 6.79 | 6.54 | 6.93 | 0.40 | 5.74 |
| | 0.5 | 7.56 | 8.67 | 7.82 | 7.45 | 7.88 | 0.55 | 7.05 |
| | 0.65 | 10.91 | 11.03 | 11.95 | 12.08 | 11.49 | 0.61 | 5.28 |
| CEMI 52.5N/FA | 0.4 | 7.96 | 7.81 | 9.08 | 7.54 | 8.10 | 0.68 | 8.37 |
| | 0.5 | 7.62 | 6.85 | 8.45 | 7.61 | 7.63 | 0.65 | 8.55 |
| | 0.65 | 10.08 | 7.50 | 8.48 | 8.99 | 8.76 | 1.07 | 12.26 |
| CEMII (B) 42.5N | 0.4 | 9.29 | 9.88 | 8.86 | 7.59 | 8.90 | 0.97 | 10.89 |
| | 0.5 | 9.57 | 11.64 | 11.55 | 9.59 | 10.59 | 1.16 | 10.98 |
| | 0.65 | 9.80 | 9.11 | 8.08 | 9.12 | 9.03 | 0.71 | 7.87 |

**Table B.4: Chloride conductivity (mS/cm) results for each specimen**

| **Binder** | **w/c** | **7-day Oven Dried** | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | **1** | **2** | **3** | **4** | **Mean** | **S.D** | **CoV (%)** |
| CEMI 52.5N | 0.4 | 0.76 | 0.58 | 0.62 | 0.62 | 0.65 | 0.08 | 11.94 |
| | 0.5 | 0.74 | 0.68 | 0.59 | 0.58 | 0.65 | 0.08 | 11.57 |
| | 0.65 | 1.05 | 1.13 | 1.09 | 1.24 | 1.13 | 0.08 | 7.41 |
| CEMI 52.5N/GGBS | 0.4 | 0.28 | 0.32 | 0.24 | 0.29 | 0.28 | 0.04 | 12.62 |
| | 0.5 | 0.29 | 0.33 | 0.27 | 0.31 | 0.30 | 0.03 | 8.47 |
| | 0.65 | 0.64 | 0.62 | 0.67 | 0.64 | 0.64 | 0.02 | 3.21 |
| CEMI 52.5N/FA | 0.4 | 0.31 | 0.28 | 0.33 | 0.35 | 0.32 | 0.03 | 9.46 |
| | 0.5 | 0.57 | 0.54 | 0.58 | 0.68 | 0.59 | 0.06 | 10.48 |
| | 0.65 | 0.90 | 0.93 | 0.84 | 1.11 | 0.94 | 0.12 | 12.51 |
| CEMII (B) 42.5N | 0.4 | 0.57 | 0.59 | 0.61 | 0.59 | 0.59 | 0.01 | 2.39 |
| | 0.5 | 0.73 | 0.72 | 0.65 | 0.65 | 0.69 | 0.04 | 6.27 |
| | 0.65 | 0.72 | 0.74 | 0.71 | 0.76 | 0.73 | 0.02 | 2.95 |
| | | **Oven Dried to Constant Mass** | | | | | | |
| CEMI 52.5N | 0.4 | 0.91 | 0.85 | 0.95 | 0.88 | 0.90 | 0.04 | 5.02 |
| | 0.5 | 1.47 | 1.35 | 1.14 | 1.29 | 1.31 | 0.14 | 10.31 |
| | 0.65 | 1.50 | 1.47 | 1.64 | 1.46 | 1.52 | 0.08 | 5.60 |
| CEMI 52.5N/GGBS | 0.4 | 0.33 | 0.29 | 0.29 | 0.24 | 0.29 | 0.03 | 12.06 |
| | 0.5 | 0.30 | 0.33 | 0.30 | 0.32 | 0.31 | 0.01 | 4.49 |
| | 0.65 | 0.66 | 0.73 | 0.68 | 0.67 | 0.69 | 0.03 | 4.36 |
| CEMI 52.5N/FA | 0.4 | 0.66 | 0.56 | 0.53 | 0.63 | 0.59 | 0.06 | 10.24 |
| | 0.5 | 0.98 | 1.01 | 0.82 | 0.95 | 0.94 | 0.08 | 8.77 |
| | 0.65 | 1.12 | 1.08 | 1.16 | 1.10 | 1.11 | 0.04 | 3.19 |
| CEMII (B) 42.5N | 0.4 | 0.69 | 0.68 | 0.75 | 0.75 | 0.72 | 0.04 | 5.10 |
| | 0.5 | 1.02 | 0.96 | 1.07 | 1.00 | 1.01 | 0.05 | 4.73 |
| | 0.65 | 0.97 | 1.03 | 1.02 | 1.07 | 1.02 | 0.04 | 4.06 |
| | | **Isopropanol Dried** | | | | | | |
| CEMI 52.5N | 0.4 | 0.70 | 0.57 | 0.59 | 0.61 | 0.62 | 0.06 | 9.38 |
| | 0.5 | 0.56 | 0.59 | 0.63 | 0.66 | 0.61 | 0.04 | 7.28 |
| | 0.65 | 1.06 | 1.15 | 1.05 | 1.01 | 1.07 | 0.06 | 5.45 |
| CEMI 52.5N/GGBS | 0.4 | 0.12 | 0.12 | 0.11 | 0.11 | 0.12 | 0.01 | 4.57 |
| | 0.5 | 0.21 | 0.19 | 0.21 | 0.22 | 0.21 | 0.01 | 4.24 |
| | 0.65 | 0.31 | 0.33 | 0.37 | 0.34 | 0.34 | 0.02 | 7.21 |
| CEMI 52.5N/FA | 0.4 | 0.30 | 0.33 | 0.37 | 0.34 | 0.33 | 0.03 | 8.18 |
| | 0.5 | 0.49 | 0.49 | 0.46 | 0.44 | 0.47 | 0.02 | 4.49 |
| | 0.65 | 0.98 | 1.02 | 0.93 | 0.97 | 0.97 | 0.04 | 3.66 |
| CEMII (B) 42.5N | 0.4 | 0.48 | 0.45 | 0.47 | 0.45 | 0.46 | 0.02 | 3.51 |
| | 0.5 | 0.81 | 0.82 | 0.93 | 0.87 | 0.86 | 0.06 | 6.44 |
| | 0.65 | 0.95 | 0.99 | 0.91 | 0.95 | 0.95 | 0.03 | 3.35 |

**Table B.5: Chloride conductivity (mS/cm) results for each specimen for UCT Rig and New Rig**

| **Binder** | **w/c** | **UCT Rig** | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | **1** | **2** | **3** | **4** | **Mean** | **S.D** | **CoV (%)** |
| CEMI 52.5N | 0.4 | 0.76 | 0.58 | 0.62 | 0.62 | 0.65 | 0.08 | 11.94 |
| | 0.5 | 0.74 | 0.68 | 0.59 | 0.58 | 0.65 | 0.08 | 11.57 |
| | 0.65 | 1.05 | 1.13 | 1.09 | 1.24 | 1.13 | 0.08 | 7.41 |
| CEMI 52.5N/GGBS | 0.4 | 0.28 | 0.32 | 0.24 | 0.29 | 0.28 | 0.04 | 12.62 |
| | 0.5 | 0.29 | 0.33 | 0.27 | 0.31 | 0.30 | 0.03 | 8.47 |
| | 0.65 | 0.64 | 0.62 | 0.67 | 0.64 | 0.64 | 0.02 | 3.21 |
| CEMI 52.5N/FA | 0.4 | 0.31 | 0.28 | 0.33 | 0.35 | 0.32 | 0.03 | 9.46 |
| | 0.5 | 0.57 | 0.54 | 0.58 | 0.68 | 0.59 | 0.06 | 10.48 |
| | 0.65 | 0.90 | 0.93 | 0.84 | 1.11 | 0.94 | 0.12 | 12.51 |
| CEMII (B) 42.5N | 0.4 | 0.57 | 0.59 | 0.61 | 0.59 | 0.59 | 0.01 | 2.39 |
| | 0.5 | 0.73 | 0.72 | 0.65 | 0.65 | 0.69 | 0.04 | 6.27 |
| | 0.65 | 0.72 | 0.74 | 0.71 | 0.76 | 0.73 | 0.02 | 2.95 |
| | | **New Rig** | | | | | | |
| CEMI 52.5N | 0.4 | 0.76 | 0.70 | 0.73 | 0.78 | 0.74 | 0.04 | 4.83 |
| | 0.5 | 0.86 | 1.03 | 0.82 | 0.72 | 0.86 | 0.13 | 14.99 |
| | 0.65 | 1.31 | 1.72 | 1.64 | 1.90 | 1.64 | 0.25 | 15.06 |
| CEMI 52.5N/GGBS | 0.4 | 0.34 | 0.38 | 0.29 | 0.36 | 0.34 | 0.04 | 10.61 |
| | 0.5 | 0.46 | 0.53 | 0.49 | 0.50 | 0.49 | 0.03 | 5.83 |
| | 0.65 | 1.08 | 1.07 | 1.10 | 1.09 | 1.09 | 0.02 | 1.45 |
| CEMI 52.5N/FA | 0.4 | 0.52 | 0.61 | 0.64 | 0.60 | 0.59 | 0.05 | 8.43 |
| | 0.5 | 0.78 | 0.60 | 0.67 | 0.73 | 0.70 | 0.08 | 11.10 |
| | 0.65 | 0.99 | 1.01 | 0.90 | 1.21 | 1.03 | 0.13 | 12.71 |
| CEMII (B) 42.5N | 0.4 | 0.68 | 0.71 | 0.71 | 0.67 | 0.69 | 0.02 | 3.21 |
| | 0.5 | 0.80 | 0.81 | 0.77 | 0.76 | 0.79 | 0.02 | 2.96 |
| | 0.65 | 0.79 | 0.82 | 0.80 | 0.83 | 0.81 | 0.02 | 2.19 |

# APPENDIX C: CHLORIDE SATURATION PHOTOS

Mix 2 Oven-dried

Mix 1 Isopropanol-dried

Mix 3 Oven-dried

Mix 1 Oven-dried

Mix 2 Isopropanol-dried


Mix 5 Oven-dried


Mix 6 Isopropanol-dried


Mix 4 Isopropanol-dried


Mix 6 Oven-dried


Mix 4 Oven-dried


Mix 5 Isopropanol-dried

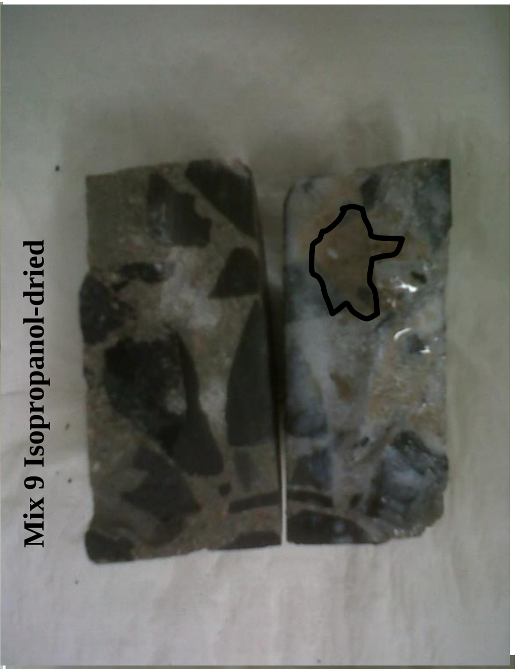
Mix 7 Oven-dried

Mix 7 Isopropanol-dried

Mix 8 Oven-dried

Mix 8 Isopropanol-dried

Mix 9 Oven-dried



Mix 9 Isopropanol-dried


Mix 11 Oven-dried


Mix 12 Isopropanol-dried


Mix 10 Isopropanol-dried


Mix 12 Oven-dried


Mix 10 Oven-dried


Mix 11 Isopropanol-dried

# APPENDIX D: 2010 DI Test Manual

The following pages contain the 2010 version on the Durability Index Manual that was used in this study. The pages are numbered from as they are in the manual, but make up pages 179 -207 of this document.

# CONCRETE DURABILITY INDEX TESTING

## PART 1: STANDARD PROCEDURE FOR PREPARATION OF TEST SPECIMENS

### SCOPE

This test method describes how to cut and prepare test specimens to perform the concrete durability index tests as originally described by Alexander, Ballim and Mackechnie (1).

Where durability index results are required for concrete mix acceptance and mix design purposes, representative samples can be prepared from concrete cubes, cast in the laboratory. Where durability index results are required for quality control purposes on site, representative samples shall be taken from the structure itself.

This test method shall not be used for concrete with a maximum nominal aggregate size exceeding 26.5 mm.

### 1. TEST SPECIMENS

a) The test specimens considered in this method statement are circular discs prepared by coring and cutting concrete cubes in the laboratory, or by taking cores from concrete elements on site.

b) Each test specimen shall consist of a 70 ± 2 mm diameter, 30 ± 2 mm thick concrete disc.

### 2. APPARATUS

a) A water-cooled diamond tipped core barrel with a nominal inner diameter of 70 mm, attached to a suitable coring drill.

b) A holding device in which cubes can be clamped firmly and securely to ensure they remain in position while coring takes place.

c) A water-cooled moveable bed diamond saw.

### 3. PREPARATION OF SPECIMENS FROM CUBES

a) Concrete cubes shall be cast according to the relevant specification, and with minimum dimensions of 100 mm.

b) The cubes shall be cured according to standard or project specifications. The duration and method of curing, and concrete age at time of test must be reported.

c) Coring of the cubes must take place at 28 ± 3 days after casting, unless otherwise required by project specifications.

d) Direction of coring must be perpendicular to the casting direction.

e) Clamp the cube firmly into the holding device and place the core barrel perpendicular to and in the centre of the concrete face to be cored (with a tolerance of 2 mm in any direction).

f) Core the entire way through the cube, ensuring that when the far side is reached and the core breaks off, the extent of the rough zone created is not greater than 5 mm from the end of the core. This may require slowing the speed of travel of the core drill as it approaches the far side. The sides of the core shall be parallel and within 5° of perpendicular to the face.

g) Cut the first 5 mm from the cored face of the core and discard. Cut the required thickness (30 ± 2 mm) of the test specimen from the core.

h) Mark the test specimens with the correct reference number on the original interior face.

i) Where a specimen is damaged during the coring and cutting process, e.g. where aggregate spalls from the surfaces to be tested, this specimen shall not be used for testing.

j) The durability index test procedure shall be started immediately after cutting.

## 5. PREPARATION OF SPECIMENS FROM SITE ELEMENTS

*Note: This section only describes the procedure of preparation of test specimens from site concrete elements. The project specifications should indicate frequency and number of cores per exposed surface area of concrete elements.*

a) Coring must take place between 28 and 35 days after casting, unless otherwise required by project specifications.

b) Place the core barrel perpendicular to the surface of the concrete, and secure it so that it cannot move.

c) Core to a depth of 80-100 mm. The sides of the core must be parallel and within 5° of perpendicular to the face.

d) Break off the core from the concrete face with a hammer and chisel, ensuring the surface 35 mm is undamaged.

e) Mark each core and remove to the laboratory in sealed plastic bags.

f) Cut the first 5 mm from the exposed face of the core and discard. Cut the required thickness (30 ± 2 mm) of the test specimen from the core.

g) Mark the test specimens with the correct reference number on the originally interior face.

h) Where a specimen is damaged during this process, e.g. where aggregate spalls from the surfaces to be tested, such a specimen shall not be used for testing.

i) Cores from site elements must be protected from conditions of adverse drying and damage on site, and during transport to a laboratory. These conditions may include, *inter alia*, high drying temperatures and/or very low humidities, rough handling and impact, etc. It is good practice to wrap samples in plastic-wrap and transport them in a container that protects them from shock, damage, and high temperatures

j) Between coring and cutting, the specimens shall be kept at ambient conditions in the laboratory for a maximum of 3 days. The durability index test procedure shall be started immediately after cutting.

## 6. REFERENCES

(1) Alexander MG, Ballim Y, Mackechnie JM, ‘ Concrete durability index testing manual’ Research Monograph No. 4, Departments of Civil Engineering University of Cape Town and University of the Witwatersrand, March 1999

(2) Gouws S, ‘Durability Index Approach – Method Statements.’ Document submitted to the Durability Index Test Method working group (under the auspices of the C&CI Technical Committee), University of the Witwatersrand, 14 August 2003.

(3) Gouws SM, ‘Durability Index Approach – Progress Report 1: Method Statements Summary document of major amendments to Durability Index Test Methods as agreed upon by Durability Index Test Method working group (Under the auspices of the C&CI Technical Committee, University of the Witwatersrand, 19 August 2003.)

(4) Gouws SM, ‘Durability Index Approach – Progress Report 2: Method Statements. Summary document of major amendments to Durability Index Test Methods as agreed upon by Durability Index Test Method Working Group (Under the auspices of the C&CI Technical Committee, University of the Witwatersrand, 7 October 2003.)

## 7. REVISIONS

| **Revision** | **Description** | **Date** |
|---|---|---|
| A | For approval | 8 April 2002 |
| B | Indicated with subscript B | 15 May 2002 |
| C | K. Stanish – remove marked with strike through, added in bold, comments in italics | 13 Feb 2003 |
| C1 | S. Gouws – suggested changes in red, *comments/questions in red italics, see also reference 2* | 16 July 2003 |
| D | For approval after Durability Index Test Method meeting on 19 August 2003, see also reference 3 | 19 August 2003 |
| E | For approval after Durability Index Test Method meeting on 22 September 2003, see also reference 4 | 22 September 2003 |
| F | Editorial improvements and clarifications - MGA | April-July 2004 |
| G | KS – Clarification and illustration | May 2005 |
| H | MGA – Editorial corrections | Jan/Oct 2007 |
| I | MGA – Editorial and other corrections | Sep-Oct 2008 |
| J | All MGA<br>Cl. 5 c) Core to depth of 80-100 mm<br>Cl. 5 i) new clause added, to cover transport of cores | Feb 2009 |

|  |  |  |
|---|---|---|
|  | from site<br>Cl. 5 j) original Cl 5 i) |  |
| K | SG as agreed with MGA<br>2.d, 3 and 5 – The option of using the facing machine was omitted | May 2010 |

# CONCRETE DURABILITY INDEX TESTING

# PART 2: STANDARD PROCEDURE FOR OXYGEN PERMEABILITY TEST

## SCOPE

This test method sets out the procedure for determining the oxygen permeability index as originally described by Alexander, Ballim and Mackechnie(1). The method described herein supersedes the 1999 version in ref. 1.

The test is suitable for the evaluation of materials and mix proportions for design purposes, and for research and development. The test can also be used for quality control of concrete on site. It is not recommended that this test be performed before 28 days after casting. Specimen age may have a significant effect on the test results, depending on the type of concrete and the curing procedure.

The oven drying procedure has been selected to result in the minimum degree of micro-structural alteration of the concrete specimens, while still giving minimal uniform moisture content. Research has shown, however, that significant amounts of micro-structural damage may occur for some high quality concrete,(2) notably high strength concrete incorporating silica fume. Thus care should be taken in interpreting the test results from these concretes.

This test method shall not be used for concrete with a maximum nominal aggregate size exceeding 26.5 mm.

## 1. APPARATUS

a) An oven capable of maintaining a temperature of 50 ± 2ºC.

*Note: Most lab ovens are of the forced draft, ventilated type. If, however, the oven being used is of the closed (unventilated) type, then the relative humidity inside the oven must be maintained by the inclusion of trays of saturated calcium chloride solution. The trays should provide a total exposed area of at least 1 $m^2$ per 1 $m^3$ of volume of the oven and should contain sufficient solid calcium chloride to show above the surface of the solution throughout the test.*

b) Permeability test arrangement as shown in Figure 1. The permeability cell should have a volume of 5 L with a tolerance of ± 5%, and should be housed in a room where the temperature is controlled at 23 ± 2 ºC. The construction of the permeability cell shall be such that it does not expand or contract in the pressure range 0 to 120 kPa. The air-tightness of the equipment needs to be tested on a regular basis using impermeable test specimens. A 0 kPa drop in pressure from an initial chamber pressure of 100 kPa over a 24 hour period is required.

c) Compressible rubber collars with Shore Hardness 39A, as shown in Figure 2 for each cell, that allow a tight fit around the specimen to eliminate any leakage of oxygen, except through the pores of the specimen. The collars must be replaced when cracks and tears occur.

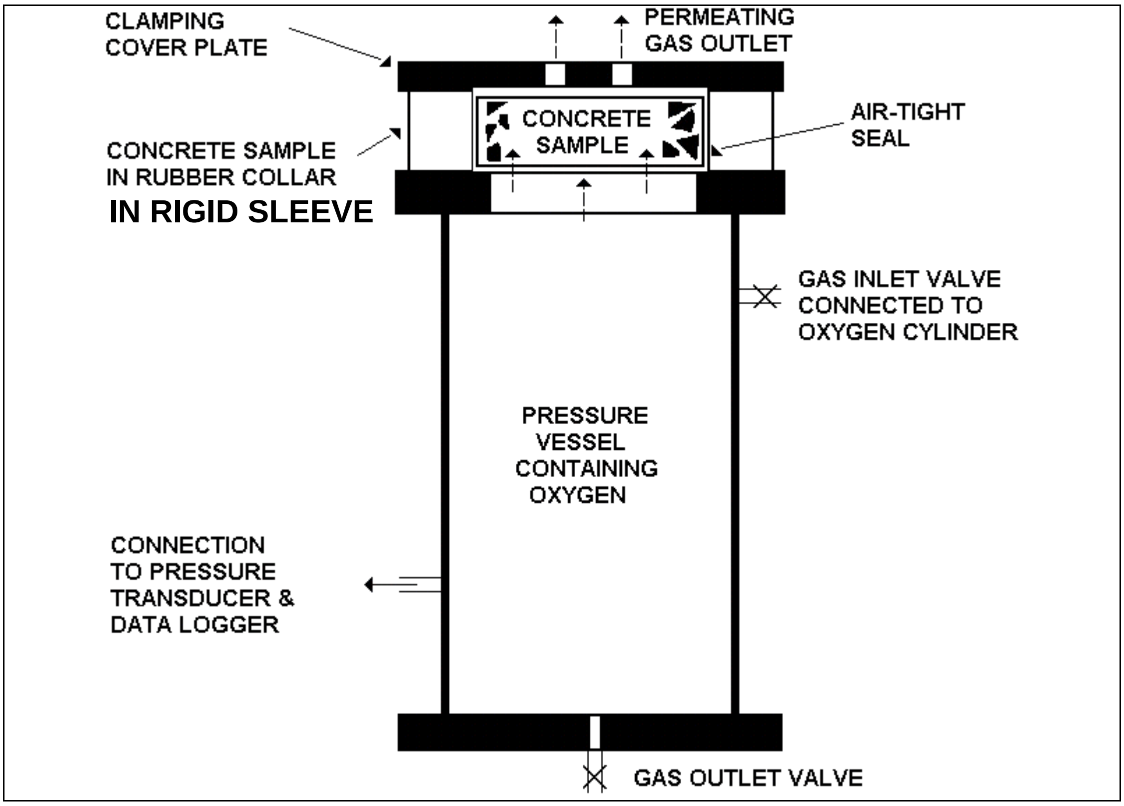


**Figure 1. Permeability Cell Arrangement.**

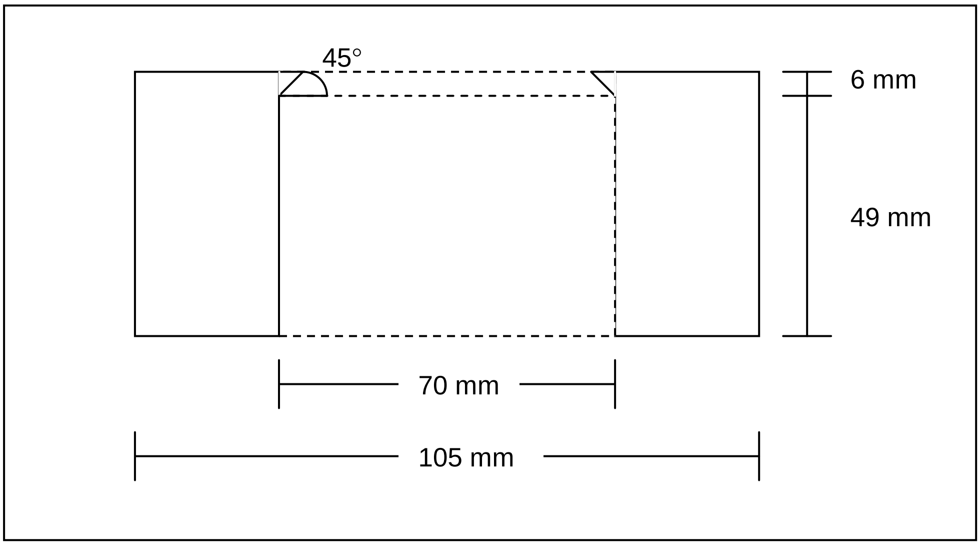


**Figure 2. Compressible Collar Cross-section.**

d) Gauges or pressure transducers, with an accuracy of at least 0.5 kPa.

e) Standard grade 99.8% oxygen supply and regulator capable of regulating pressure to 120 kPa.

f) Vernier calliper, capable of reading to 0.02 mm.

g) Desiccator, large enough to hold as many specimens as will be tested simultaneously, containing anhydrous silica gel as the desiccant, with the relative humidity controlled at a maximum of 60 %.

## 2. TEST SPECIMENS

a) Four test specimens are required per test. Each specimen shall consist of a 70 ± 2 mm diameter concrete disc with a thickness of 30 ± 2 mm cored and cut in accordance with Concrete Durability Index Testing, Part 1.

b) Mark the specimens of the same reference 1, 2, 3 and 4 on the inner face.

## 3. CONDITIONING OF SPECIMENS

a) Directly after cutting the specimens shall placed in the oven at 50 ± 2°C for 7 days ± 4 hours.

b) Remove the specimens from the oven and immediately place them in the desiccator.

## 4. TESTING OF SPECIMENS

a) Cool the specimens to 23 ± 2 °C in the desiccator. This cooling period should last for no less than 2 hours and no longer than 4 hours.

b) Measure to the nearest 0.02 mm the thickness and diameter of each specimen with the vernier calliper at 4 points equally spaced around the perimeter of the specimen, and record. Determine the average of the four readings and record to the nearest 0.02 mm.

c) Place the specimen in the compressible collar within the rigid sleeve with the test face (outer face) at the bottom. No gaps should be visible between the sides of the test specimen and the collar. The specimens shall be placed so that the outer face rests against the lip of the collar, see Figure 3.

**Figure 3. Proper Positioning of Specimen in Collar.**

d) Place the sample, collar and rigid sleeve on top of the test chamber so that it covers the hole. Place the solid ring (the solid ring is optional depending on the equipment arrangement) on top of the collar, once again ensuring that no gaps are visible between the collar and the sleeve. Place the coverplate on top of the solid ring, see Figure 4.

e) Partially tighten the top screw on the coverplate to ensure that it is centred. On the underside of the coverplate is a groove. The edges to solid ring (or the collar if a ring is not present) should be within the groove. If this is not the case, adjust the location of the bottom portion. Once the specimen has been centred, tighten the apparatus – first finger tight and then one and a half revolutions with a spanner.

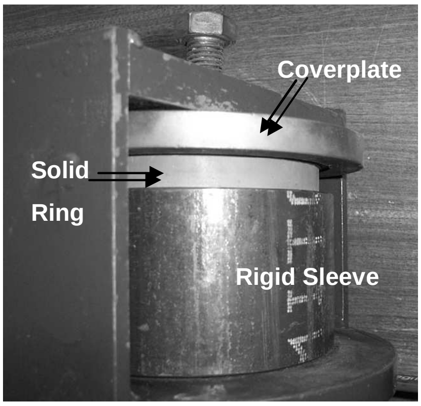


**Figure 4. Permeameter Set Up.**

f) Start each test within 30 min after removing the specimen from the desiccator.

g) Open the oxygen inlet and outlet valves of the permeability cells. Open the valve of the oxygen supply tank to between 100 and 120 kPa, and allow oxygen to flow through the permeameter for 5 seconds. This will purge the test chamber of gases other than oxygen.

h) Close the oxygen outlet valve of the permeability cells.

i) When the pressure is above 100 kPa on the gauge of the permeability cell, close the inlet valve.

j) Tap the gauge to ensure a correct reading. Adjust the pressure in the cell to 100 ± 5 kPa by opening the outlet valve slightly. Record the initial time, $t_0$, to the nearest minute and initial exact pressure, $P_0$, to the nearest 0.5 kPa. This initial pressure shall be within 5 kPa of 100 kPa. Use $t_0$ and $P_0$ as such in the calculations.

k) After 5 minutes tap the gauge and record the time and pressure reading. If the pressure drops too quickly (> 5 kPa per minute), there may be a leak present. In such a case, release the pressure in the chamber, check that the sample fits tightly in the collar, and restart the test immediately, starting at paragraph (g).

l) Subsequent readings should be taken with sufficient frequency that the pressure has dropped 5 ± 1 kPa between readings. For each reading, note the time to the nearest minute and pressure in the test cell to the nearest 0.5 kPa.

m) The test may be terminated when the pressure has dropped to 50 ± 2.5 kPa or after 6 hours ± 15 min, whichever occurs first. A minimum of 8 readings is required.

*Note: It is possible to automate the readings. In this case, pressure readings shall be recorded on the computer at 15 minute intervals until the pressure drops to 50 ± 5 kPa or up to 6 hours ± 15 min, whichever occurs first. All the data points so generated shall be used in the calculation.*

n) The same specimens that were used in the oxygen permeability test can also be used in the water sorptivity test. For details of the procedure, please refer to Part 3: ‘Standard Procedure for Water Sorptivity Test’.

## 5. CALCULATIONS

**NOTE: A standard spreadsheet has been developed to perform the calculations described below, and it is strongly recommended that this spreadsheet be utilized. A copy of this spreadsheet is a free download from www.cnci.org.za in the 'Structural Concrete' Section.**

a) Determine the best fit line using linear regression of $\ln(P_o/P_t)$ against t, forcing the regression line through the (0,0) point where:

$P_o$ = initial pressure at start of test (at time $t_o$) to the nearest 0.5 kPa

$P_t$ = subsequent readings in pressure to the nearest 0.5 kPa at times t, measured from $t_o$

t = time in seconds, recorded to the nearest minute.

b) The coefficient of correlation should be greater than 0.99. Where the correlation is less than 0.99 a retest should be done on the same specimen. If the subsequent test of the specimen also has a correlation coefficient of less than 0.99, this specimen should be discarded and another test specimen prepared.

*Note: Every reading recorded as described in section 4 shall be used in the regression analysis. No data points shall be excluded in the determination of the correlation coefficient. No additional manipulation or exclusion of data points is allowed in order to improve the correlation coefficient. If the correlation coefficient is less than 0.99, the sample shall be retested.*

*Note: The slope of the linear regression line forced through the (0,0) point can be calculated from the equation:*

$$z = \frac{\sum[\ln(P_0/P_t)]^2}{\sum[\ln(P_0/P_t)t]}$$

*where*

$z$ *is the slope of the linear regression*

$P_0$, $P_t$, *t are defined as in 5 a).*

*The* $r^2$ *value can be calculated from the equation:*

$$r^2 = 1 - \frac{\sum[t_i - t_{p,i}]^2}{\sum t_i^2 - \left(\sum t_{p,i}\right)^2 \big/ n}$$

*where*

$t_i$ *is the time at any given pressure reading*

$t_{p,i}$ *is the predicted time at the same pressure reading (based on the linear regression)*

$n$ *is the number of data points being considered*

*The value of $t_{p,i}$ can be calculated from*

$t_{p,i}=ln(P_0/P_t)/z$

*where $P_0$, $P_t$ and z is as defined above.*

*The ‘slope’ and ‘rsq’ functions available in Excel CANNOT be used, as they do not force the line through the zero point.*

c) The D’arcy coefficient of permeability is given by:

$$k = \frac{\omega V g d z}{R A \theta}$$

k = coefficient of permeability of test specimen (m/s)
ω = molecular mass of oxygen = 0.032 kg/mol
V = volume of oxygen under pressure in permeameter ($m^3$) recorded to the nearest 0.01 litre or 0.00001 $m^3$. The volume of the pressure cell includes the volume of the opening in the top plate and the rubber collar annulus below the sample. The volume shall be determined by dimensional measurement, accurate to the nearest mm, or by the volume of water contained at 23 ± 2°C.
g = acceleration due to gravity (9.81 $m/s^2$)
R = universal gas constant = (8.313 Nm/K mol)
d = average specimen thickness (m) to the nearest 0.02 mm
A is the cross sectional area of the specimen, in square meters
θ = absolute temperature (K)
z = slope of the line determined in the regression analysis

d) The coefficient of permeability is calculated for each of the test specimens. The oxygen permeability index (OPI) is given as the negative log of the average of the coefficients of permeability of the specimens, which for four specimens is:

$$OPI = -\log_{10} [\tfrac{1}{4} (k_1+k_2+k_3+k_4)]$$

## 6. REPORTING

Report the following:

a) Each individual test determination of a specimen (i.e. k value) to three decimal places.

b) The oxygen permeability index to two decimal places.

c) Identification number of specimens.

d) Description of specimen. This is particularly important in this test since the test is stated to be indicative of macro-structural problems. Specifically mention whether there are visible cracks, honeycombing defects or visible bleed paths present.

The following shall be reported if known

e) Source of the specimen.

f) Location of specimen within cube, core or member

g) Identification mark of each specimen

h) Type of concrete, including binder type, water/cement ratio and other relevant data supplied with the specimen.

i) Curing history.

j) Unusual specimen preparation e.g. removal of surface treatment.

k) Unusual features such as cracks, voids, excessively chipped edges, etc.

l) Test operator.

m) Age of concrete at time of testing.

## 7. REFERENCES

(1) Alexander MG, Ballim Y, Mackechnie JM, ' Concrete durability index testing manual' Research Monograph No. 4, Departments of Civil Engineering, University of Cape Town and University of the Witwatersrand, March 1999

(2) Mackechnie JR and Alexander MG, 'Practical considerations for rapid chloride conductivity testing.' Proceedings of the Second International RILEM Workshop on Testing and Modelling the Chloride Ingress Into Concrete, C. Andrade and J. Kropp, ed., 2000

(3) Gouws SM, 'Durability index approach – method statements.' Document submitted to the Durability Index Test Method working group (Under the auspices of the C&CI Technical Committee), University of the Witwatersrand, 14 August 2003.

(4) Gouws SM, 'Durability Index Approach – Progress Report 1: Method Statements' Summary document of major amendments to Durability Index Test Methods as agreed upon by Durability Index Test Method working group (Under the auspices of the C&CI Technical Committee, University of the Witwatersrand, 19 August 2003.

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## 8. REVISIONS

| **Revision** | **Description** | **Date** |
|---|---|---|
| A | For approval | 8 April 2002 |
| B | Indicated with subscript B | 15 May 2002 |
| C | K. Stanish – remove marked with strike through, added in bold, comments in italics | 14 Feb 2003 |
| C1 | S. Gouws - suggested changes in red, *questions and* | 21 July 2003 |

| | | |
|---|---|---|
| | *remarks in italics, see also reference 3* | |
| D | For approval after Durability Index Test Method meeting on 19 August 2003, see also reference 4 | 19 August 2003 |
| E | For approval after Durability Index Test Method meeting on 22 September 2003, see also ref. 5 | 22 September 2003 |
| F | Editorial improvements and clarifications - MGA | April-July 2004 |
| G | KS – Clarification and illustration | April/May 2005 |
| H | MGA - Definition of $r^2$ terms; addition of cell volume; Editorial Corrections | Jan/Oct 2007 |
| I | MGA – Editorial and other corrections | Sep-Oct 2008 |
| J | All MGA<br>Cl. 5 b): addition of definition of 'z'.<br>Cl. 5 c): insert 'to the nearest 0.02 mm' at end of definition of 'd'<br>Cl. 6 a) replace 'significant figures' with 'decimal places' | 25 Feb. 2009 |
| K | SG as confirmed with MGA<br>Cl. 1 b): added 'constructed not to expand at 100 to 120 kPa.<br>Cl. 1g): removed the note 'not required if relative humidity in the laboratory is below 60%' Now only allowed to cool in desiccators.<br>Cl. 3: added ' Remove the specimens from the oven and immediately place them in the desiccators.<br>Cl. 4 a): Removed the allowance of cooling in the laboratory, cooling may now only take place in the desiccators.<br>Cl. 4 g): Added 'open the valve of the oxygen supply tank to between 100 and 120 kPa'. This is done so as not to open the tank to maximum pressure so as to protect the transducers.<br>Cl. 5: Changed from UCT to C&CI's website.<br>Cl. 5 b): Added the calculation of $t_{p,i}$<br>Cl. 5 c): Changed $\omega$ to 0.032 kg/mol instead of 32 g/mol and added that the temperature of the water must be 23 ± 2ºC. | May 2010 |

# CONCRETE DURABILITY INDEX TESTING

## PART 3: STANDARD PROCEDURE FOR WATER SORPTIVITY TEST

### SCOPE

This test method sets out the procedure for determining the water sorptivity index as originally described by Alexander, Ballim and Mackechnie (1). The method described herein supersedes the 1999 version in ref. 1.

The test is suitable for the evaluation of materials and mix proportions for design purposes, and for research and development. The test can also be used for quality control of concrete on site. It is not recommended that this test be performed before 28 days after casting. Specimen age may have a significant effect on the test results, depending on the type of concrete and the curing procedure. Care should be taken in interpreting the results of this test when it is used on surface treated concretes, or on concrete that has been exposed to environmental influences such as carbonation or marine salts.

The oven drying procedure has been selected to result in the minimum degree of micro-structural alteration of the concrete specimens, while still giving minimal uniform moisture content. Research has shown, however, that significant amounts of micro-structural damage may occur for high quality concrete.(2) Thus care should be taken in interpreting the test results from these concretes.

This test method shall not be used for concrete with a maximum nominal aggregate size exceeding 26.5 mm.

Specific commercial products are mentioned for illustration purposes only. This is not intended to be an endorsement of any particular product or company.

### 1. APPARATUS

a) An oven capable of maintaining a temperature of 50 ± 2ºC.

*Note: Most lab ovens are of the forced draft, ventilated type. If, however, the oven being used is of the closed (unventilated) type, then the relative humidity inside the oven must be maintained by the inclusion of trays of saturated calcium chloride solution. The trays should provide a total exposed area of at least 1 $m^2$ per 1 $m^3$ of volume of the oven and should contain sufficient solid calcium chloride to show above the surface of the solution throughout the test.*

b) Vacuum saturation facility as shown in Figure 1.

c) Plastic or stainless steel tray 20 mm deep and large enough to hold as many specimens as will be tested simultaneously.

d) Ten layers of absorbent paper towel. Alternatively 2 rollers or 4 pins can be used to support the specimens tested.

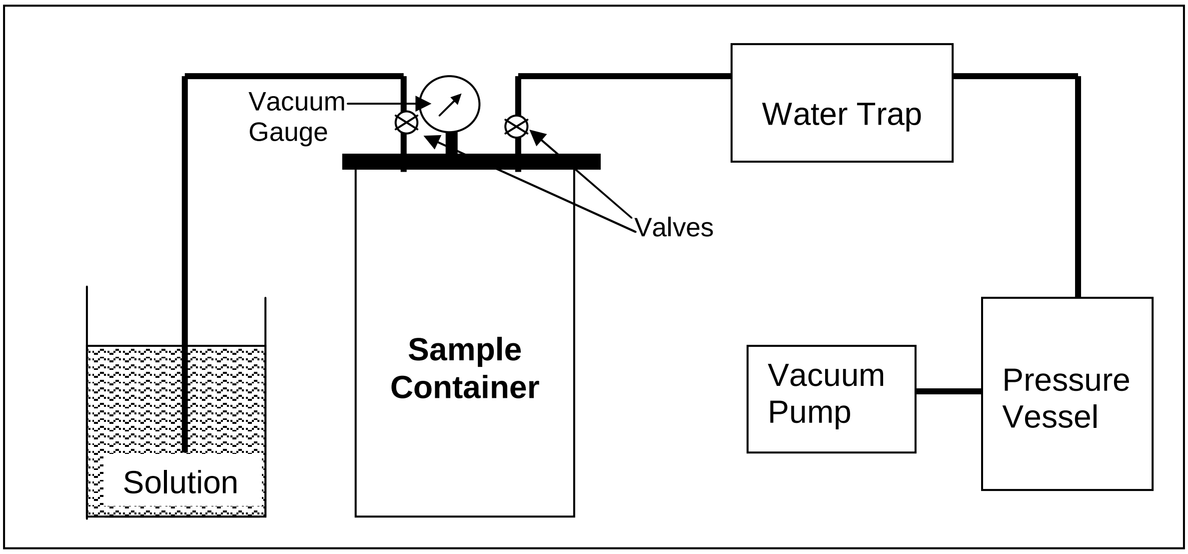

**Figure 1. Vacuum Saturation Facility.**

e) Vernier calliper, capable of reading to 0,02 mm.

f) Measuring scale with accuracy to 0.01 g.

g) A solution of tap water saturated with calcium hydroxide. (5 grams of $Ca(OH)_2$ per 1 litre of water), maintained at 23 ± 2℃.

h) One or more stopwatches as required.

i) Sealant to provide a watertight seal around the vertical curved edges of the specimens without blocking any part of the test face whatsoever. Any of the following can be used:

   - Packaging tape

k) Desiccator, large enough to hold as many specimens as will be tested simultaneously, containing anhydrous silica gel as the desiccant, with the relative humidity controlled at a maximum of 60 %.

## 2. TEST SPECIMENS

a) Four test specimens are required per test. Each specimen shall consist of a 70 ± 2 mm diameter concrete disc with a thickness of 30 ± 2 mm, cored and cut in accordance with Concrete Durability Index Testing, Part 1. It is permitted to use specimens that have previously been used in the oxygen permeability test, provided they have not been exposed to moisture. Refer to Part 2: ‘Standard Procedure for Oxygen Permeability Test’.

b) Mark the specimens of the same reference 1, 2, 3 and 4 on the inner face.

## 3. CONDITIONING OF SPECIMENS

a) If using new specimens, directly after cutting, the specimens shall be placed in the oven at 50 ± 2°C for 7 days ± 4 hrs.

b) Remove the specimens from the oven and seal the vertical curved sides of the specimens using any of the recommended products as per the product specifications.

c) If the specimens have been previously tested in the oxygen permeability cells, they shall be tested immediately upon removal. No additional drying is necessary provided the specimens have not got wet or have had an opportunity to absorb moisture from the atmosphere. Alternatively, specimens may be placed back in the 50 ºC oven overnight prior to cooling in the dessicator as per part 2, if they cannot be tested immediately after the OPI test.

## 4. TESTING OF SPECIMENS

a) The water sorptivity test shall be conducted in a room in which the temperature is controlled at 23 ± 2ºC.

b) If the specimens are taped and have not been previously tested in the oxygen permeability test, cool the specimens to 23 ± 2 °C in the desiccator. This cooling period should last for no less than 2 hours and no longer than 4 hours.

b) Measure to the nearest 0.02 mm the thickness and diameter of each specimen with the vernier calliper at 4 points equally spaced around the perimeter of the specimen, and record. Determine the average of the four readings and record to the nearest 0.02 mm.

c) Place the 10 layers of paper towel on the tray. Alternatively, place the rollers/pins in position to support the specimen in the solution.

**Figure 2a. Test Set Up Using Paper Towels.**

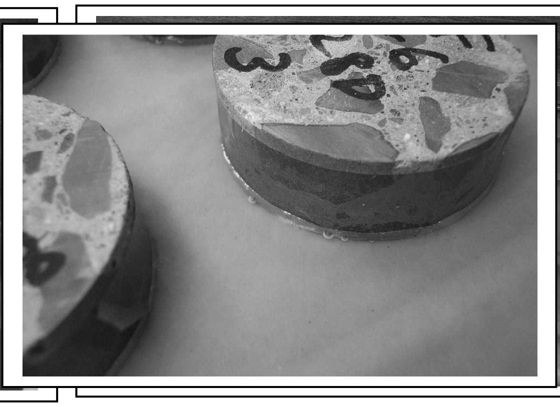

**Figure 3. Proper Solution Depth.**

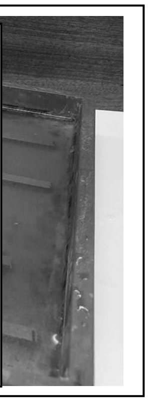

**Figure 2b. Test Set Up Using Supports.**

d) Pour calcium hydroxide solution into the tray. If used, the paper towel should be saturated and water is visible on the top surface. All air bubbles shall be removed by smoothing the paper pad towards the edges, see Figure 2a. If rollers or pins are used, they should be arranged as to support the specimens and the calcium hydroxide solution should be above the top of the support, Figure 2b. The final water level should be such that it will be slightly above the bottom edge of the specimen and a maximum 2 mm up the side of the specimen as shown in Figure 3. Dampen an additional piece of paper towel for use in removing the excess water from the specimens (in paragraph (g)) and keep next to the tray to be used during the test.

e) Within 30 min after removing the specimen from the desiccator, or oxygen permeability cell for taped specimens, determine the mass of the specimen to an accuracy of 0.01 g and record as the dry mass. This mass must be determined after the method used to seal the sides of the specimens (either epoxy or tape) has been applied.

f) Immediately place the specimen with the test face (outer face or originally exposed face) on the wet paper pad/pins/rollers and start the stopwatch, at time $t_0$..

g) Weigh the specimen at 3, 5, 7, 9, 12, 16, 20 and 25 minutes, after patting it once on the damp piece of absorbent paper. The specimen should appear saturated surface dry (SSD) on the exposed face at the time the mass is determined, i.e. it should look damp, but not have free water on the test face. During removal of a specimen from the tray for weighing, care must be taken to prevent dripping from one specimen onto the top of another.

h) Record the mass of the specimen to the nearest 0.01 g within 10 s of removal of the specimen from the tray and wiping excess water. Replace the specimens each time with the test face on the wet paper or rollers/pins if used. The stopwatch shall not be stopped during the weighing procedure.

i) Within a maximum of 1 day after weighing of the specimen is completed, place the specimen in the vacuum saturation tank. The tape must be left in place. The specimens shall be arranged so as to maximize their exposed surface area. This is typically done be standing the individual specimens upon their curved edges, rather than the flat side, as in Figure 4. Seal the lid with petroleum jelly and close it.

j) Evacuate the tank to between -75 and -80 kPa and maintain the specimens under vacuum of between -75 and -80 kPa for 3 hours ± 15 min. The pressure must not be allowed to rise above -75 kPa during this period.

k) After 3 hours ± 15 min isolate the tank and allow calcium hydroxide saturated water to flow into the chamber until the water level is approximately 40 mm above the top of the specimens. Air shall not be allowed to enter the vacuum chamber during this procedure.

**Figure 4. Specimens arranged for saturation.**

l) Re-establish the vacuum between -75 and -80 kPa. This shall be maintained for 1 hour ± 15 minutes. At no point during this time period shall the vacuum be permitted to rise above -75 kPa.

m) After 1 hour ± 15 min, release the vacuum and allow air to enter. Allow the specimens to soak for a further 18 ± 1 hours.

n) After 18 ± 1 hr soaking, remove the specimens from the solution, dry the surface to a SSD condition with a paper towel, and immediately weigh to an accuracy of 0.01 g. Record this as the vacuum saturated mass $M_{sv}$ of the specimen.

## 5. CALCULATIONS

**NOTE: A standard spreadsheet has been developed to perform the calculations described below, and it is strongly recommended that this spreadsheet be utilized. A copy of this spreadsheet is a free download from www.cnci.org.za in the 'Structural Concrete' Section.**

a) Determine the porosity (n) of each specimen, as a percentage, by applying the following formula:

$$n = \frac{M_{sv} - M_{s0}}{Ad\rho_w} \times 100$$

where

$M_{sv}$ is the vacuum saturated mass of the specimen to the nearest 0.01g, in grams

$M_{s0}$ is the mass of the specimen at time $t_0$ (start of the test) to the nearest 0.01g, in grams

A is the cross-sectional area of the specimen to the nearest 0,02 $mm^2$, in millimeter squared

d is the average specimen thickness to the nearest 0,02 mm, in millimeters

$\rho_w$ is the density of water, $10^{-3}$g/$mm^3$, in grams per millimeter cubed

b) Determine and plot the mass gain ($M_{wt}$) versus the square root of time, by applying the following formula:

$$M_{wt} = F\sqrt{t}$$

where

F is the slope of the best fit line from plotting $M_{wt}$ against $\sqrt{t}$, in gram per squareroot of hour

t is the time in hours after a specimen was first exposed to water on its lower face, to the nearest 0.001 h, in hours

Use the following equation to determine the mass gain ($M_{wt}$):

$$M_{wt} = M_{st} - M_{s0}$$

where

$M_{st}$ is the mass to the nearest 0,01g of the specimen at particular time t, in grams

$M_{s0}$ is the mass to the nearest 0.01g of the specimen at the initial time ($t_0$), in grams.

No allowance shall be made for the time taken to wipe and weigh each sample in the calculation.

Do not include the zero time reading in the data.

c) Determine the correlation coefficient, $r^2$ of the data:

$$r^2 = \left[ \frac{\sum \left(\sqrt{t_i} - T\right)\left(M_{wti} - \overline{M}_{wt}\right)}{\sqrt{\sum \left(\sqrt{t_i} - T\right)^2 \sum \left(M_{wti} - \overline{M}_{wt}\right)^2}} \right]^2$$

where

$M_{wti}$ is the mass gain as calculated in 7.2 at any given time, in grams

$t_i$ is the time corresponding to the mass gain $M_{wti}$, in hours

and

$$\overline{M}_{wt} = \frac{\sum M_{wti}}{n}$$

and

$$T = \frac{\sum \sqrt{ti}}{n}$$

where

n is the number of data points.

Do not include the zero time reading in the data.

d) If the coefficient of correlation is less than 0,98, discard the last (25 min) value from the analysis, and re-determine the correlation coefficient, adjusting the value of n, the number of data points, in the calculation as is relevant.

e) If the coefficient of correlation is still less than 0,98, discard the next value (i.e. 20 min) from the analysis, and re-determine the correlation coefficient, adjusting the value of n, the number of data points, in the calculation as is relevant.

f) Repeat the procedure until a coefficient of above 0,98 is achieved, or there are less than 5 data points remaining.

g) If a correlation coefficient of 0,98 cannot be obtained with a set of five or more values, regard the specimen as unsuitable for the determination of the sorptivity. However, record the range of data able to give a correlation coefficient of above 0,98.

h) Using the values obtained from procedures contained in c) to f) (inclusive), determine the slope of the line of best fit (F) by linear regression analysis:

$$F = \frac{\sum\left(\sqrt{t_i} - T\right)\left(M_{wti} - \overline{M}_{wt}\right)}{\sum\left(\sqrt{t_i} - T\right)^2}$$

where

$M_{wti}$ is the mass gain as calculated in 7.2 at any given time, in grams

$t_i$ is the time corresponding to the mass gain reading $M_{wti}$, in hours

and

$$\overline{M}_{wt} = \frac{\sum M_{wti}}{n}$$

and

$$T = \frac{\sum\sqrt{ti}}{n}$$

where

n is the number of data points.

Do not include the zero time reading in the data.

i) The water sorptivity of the specimen (S) is given by:

$$\mathrm{S} = \frac{\mathrm{Fd}}{\mathrm{M_{sv}} - \mathrm{M_{s0}}}$$

where

*F* is the slope of the best fit line (5 g) ), in grams per squareroot of the hour

$d$ is the average specimen thickness to the nearest 0,02 mm, in mm

$M_{sv}$ is the vacuum saturated mass to the nearest 0,01 g of the specimen, in g

$M_{s0}$ is the mass to the nearest 0,01 g of the specimen at the initial time ($t_0$), in g

j) The procedure 5c) through 5g) is carried out separately for each specimen. The sorptivity index is given as the average of the water sorptivity of at least three valid individual test determinations.(3)

*Note: A simple way to calculate the slope of the regression line is by entering the data in a Microsoft Excel range and use the function SLOPE {data range of* $M_{wt}$*; data range* $\sqrt{t}$*}. The correlation coefficient can be obtained by using the RSQ {data range* $M_{wt}$*; data range of* $\sqrt{t}$*} function.*

## 6. REPORTING

Report the following:

a) Identification number of specimen.

b) Description of specimen.

c) The effective porosity of each specimen to the nearest 2 decimal places.

d) The water sorptivity of each individual specimen.

e) The water sorptivity index to the nearest 1 decimal place.

f) The range of data used in the calculations.

The following shall be reported if known

g) Source of the specimen.

h) Location of specimen within cube, core or member

i) Identification mark of each specimen

j) Type of concrete, including binder type, water/cement ratio and other relevant data supplied with the specimen.

k) Curing history.

l) Unusual specimen preparation e.g. removal of surface treatment.

m) Unusual features such as cracks, voids, excessively chipped edges, etc.

n) Test operator.

o) Age of concrete at time of testing.

## 7. REFERENCES

(1) Alexander M.G., Ballim Y., Mackechnie J.M., ‘Concrete durability index testing manual’ Research Monograph no. 4, Departments of Civil Engineering, University of Cape Town and University of the Witwatersrand, March 1999

(2) Mackechnie JR and Alexander MG, ‘Practical considerations for rapid chloride conductivity testing.’ Proceedings of the Second International Workshop on Testing and Modelling the Chloride Ingress Into Concrete, C. Andrade and J. Kropp, ed., 2000

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## 8. REVISIONS

| Revision | Description | Date |
|---|---|---|
| A | For approval | 8 April 2002 |
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| F | Editorial improvements and clarifications - MGA | April-July 2004 |
| G | KS – Clarification and illustration | April 2005 |
| H | MGA – Editorial corrections | Jan/Oct 2007 |
| I | MGA – Editorial and other corrections | Sep-Oct 2008 |
| J | All MGA | 25 Feb 2009 |

| | | |
|---|---|---|
| | Cl. 1 e)  Accuracy of Vernier inserted<br>Cl. 5 h)  Added definitions of *F, d, M*sv, and $M_{s0}$.<br>Cl. 6 c)  Changes accuracy to 2 decimal places. | |
| K | SG with MG<br>Cl.1 i), 3 b), 4 i): Removed all references to epoxy paint<br>Cl. 1 k): Must use desiccator for cooling<br>Cl. 3 a): Added ‘if using new specimens’<br>Cl. 3 b): Added ‘remove the specimens from the oven’<br>Cl. 3 c): Added ‘oven at 50 ºC’ and ‘as per part 2’<br>Cl. 4 b): Must use desiccator for cooling<br>Cl. 4 e): Moved ‘at time $t_0$’ to the end of 4 f)<br>Cl. 5 a): Changed from UCT’s website to C&CI’s website<br>Cl. 5 c): Added ‘use only relevant number of data points for n’ | May 2010 |

# CONCRETE DURABILITY INDEX TESTING

## PART 4: STANDARD PROCEDURE FOR CHLORIDE CONDUCTIVITY TEST

### SCOPE

This test method sets out the procedure for determining the chloride conductivity index as originally described by Alexander, Ballim and Mackechnie (1). The method described herein supersedes the 1999 version in ref. 1.

The test is suitable for the evaluation of materials and mix proportions for design purposes and for research and development. The test can also be used for quality control of concrete on site. Specimen age may have a significant effect on the test results, depending on the type of concrete and the curing procedure. Care should be taken in interpreting the results of this test when it is used on surface treated concretes. In general, the test should not be performed on concrete that has been exposed to environmental influences such as carbonation or marine salts.

The oven drying procedure has been selected to result in the minimum degree of micro-structural alteration of the concrete specimens, while still resulting in minimal uniform moisture content. Research has shown, however, that significant amounts of micro-structural damage may occur for some high quality concrete.(2) Thus care should be taken in interpreting the test results from these concretes.

Since the test results are a function of the electrical resistance of the specimen, the presence of reinforcing steel or other embedded electrically conductive material may have a significant effect.

This test method shall not be used for concrete with a maximum nominal aggregate size exceeding 26.5 mm.

Care should be taken when performing this test, since the chloride solution used is hazardous to the skin. It is recommended that gloves be used.

### 1. APPARATUS

a) An oven capable of maintaining a temperature of 50 ± 2ºC.

*Note: Most lab ovens are of the forced draft, ventilated type. If, however, the oven being used is of the closed (unventilated) type, then the relative humidity inside the oven must be maintained by the inclusion of trays of saturated calcium chloride solution. The trays should provide a total exposed area of at least 1 $m^2$ per 1 $m^3$ of volume of the oven and should contain sufficient solid calcium chloride to show above the surface of the solution throughout the test.*

b) Vacuum saturation facility as shown in Figure 1.

c) Conduction cell as shown in Figure 2 with anode and cathode parts permanently marked on the outside of the cell. The flexible collars need to be replaced when cracks or tears occur.

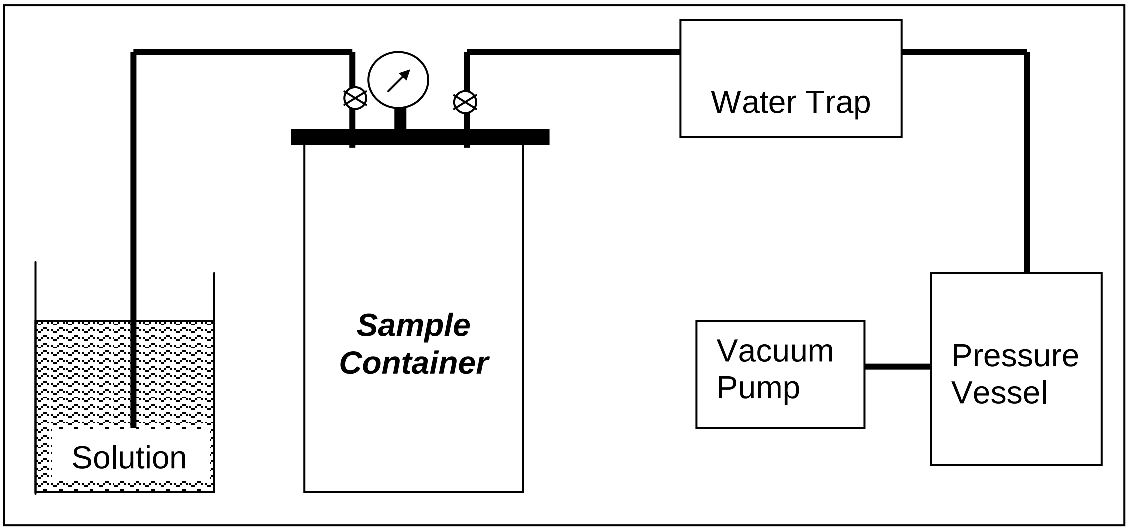


**Figure 1. Vacuum Saturation Apparatus.**

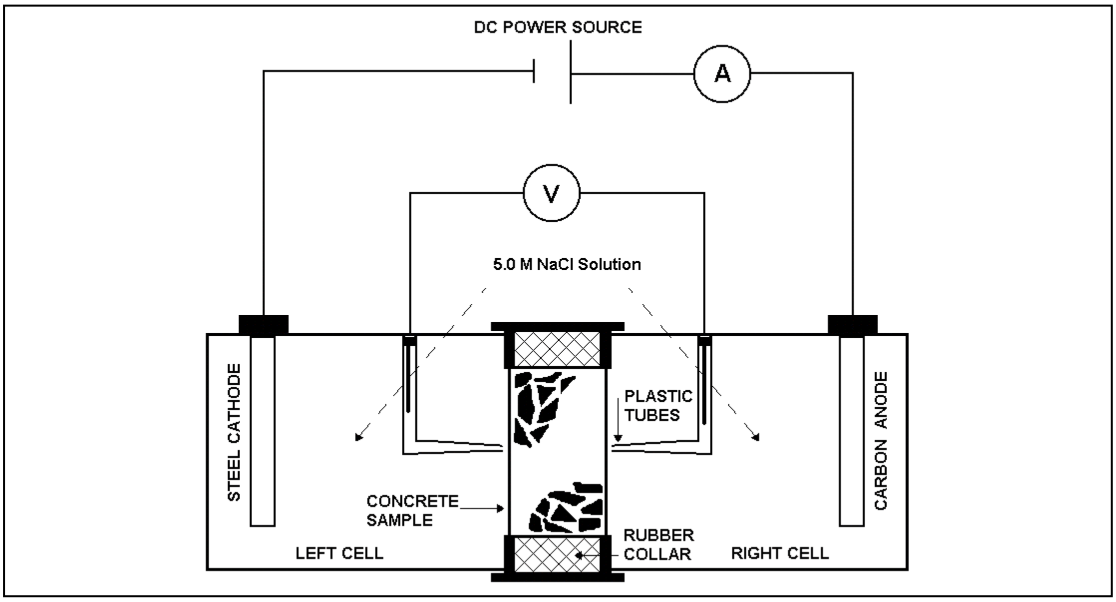


**Figure 2. Chloride Conductivity Test Apparatus.**

d) DC power supply 0-12 Volt, 0-1 Ampere stabilised.

e) Digital voltmeter and ammeter (two multi-meters), 4 digits, 0-20 V range, 0-300 mA, rated accuracy 0.1 %.

f) Electrical cables and plugs.

g) Weighing scale capable of determining mass to 0.01 g.

h) 10 litre container with a lid.

i) CP grade NaCl (99% purity).

j) Desiccator, large enough to hold as many specimens as will be tested simultaneously, containing anhydrous silica gel as the desiccant, with the relative humidity controlled at a maximum of 60 %.

*Note: Due to the highly corrosive solutions used during the test, all equipment must be cleaned thoroughly with warm soapy water after each use. The copper electrodes and banana plugs need to be cleaned with sandpaper or an acidic solution. Replacement electrical connections are necessary from time to time. To protect electrical test equipment, it is advisable to place such equipment on a shelf above the bench on which the conductivity cell is placed.*

## 2. CHEMICAL SOLUTIONS

a) 5.0 M sodium chloride solution: Add 2.93 kg of NaCl (salt) to the 10-litre container. Add potable water to the 10-litre mark. Stir occasionally for 1 day or until all the salt has dissolved. Seal the lid of the container. The solution must be maintained at 23 ±2℃.

## 3. TEST SPECIMENS

a) Four test specimens are required per test. The test specimen shall consist of a 70 ± 2 mm diameter concrete disc with a thickness of 30 ± 2 mm cored and cut in accordance with Concrete Durability Index Testing, Part 1.

b) Mark the specimens of the same reference 1, 2, 3 and 4 on the inner face.

## 4. CONDITIONING OF SPECIMENS

a) Directly after cutting, the specimens shall be placed in the oven at 50 ± 2℃ for seven days ± 4 hours.

b) Remove the specimens from the oven and immediately place them in the desiccator (maintained at (23 ± 2) ℃) for cool ing. Allow the specimens to cool for a minimum of 2 h and a maximum of 4 h. Remove the specimens from the desiccator and commence the testing in accordance with the procedure outlined in 5. within 30 min.

c) Measure to the nearest 0.02 mm the thickness and diameter of each specimen with the vernier calliper at 4 points equally spaced around the perimeter of the specimen, and record. Determine the average of the four readings, and record to the nearest 0.02 mm. Determine the dry mass of the specimens to the nearest 0.01 g.

d) Place the specimens in the vacuum saturation tank. The specimens shall be arranged so as to maximize their exposed surface area. This is typically done be standing the individual specimens upon their curved edges, rather than the flat side, as in Figure 3. Seal the lid with petroleum jelly and close it.

e) Evacuate the tank to between -75 and -80 kPa and maintain the specimens under vacuum of between -75 and -80 kPa for 3 hours ± 15 min. The pressure must not be allowed to rise above -75 kPa during this period.

f) After 3 hours ± 15 min isolate the tank and allow the salt solution to enter the vacuum tank

**Figure 3. Specimen Arrangement in Vacuum Chamber.**

without releasing the vacuum to cover all the specimens to a depth of approximately 40 mm. Air shall not be allowed to enter the vacuum chamber above the top of the specimens during this procedure.

g) Re-establish the vacuum between -75 and -80 kPa. This shall be maintained for 1 hour ± 15 minutes. At no point during this time period shall the vacuum be permitted to rise above -75 kPa.

h) After 1 hour ± 15 min, release the vacuum and allow air to enter. Allow the specimens to soak for a further 18 ± 1 hours.

i) After 18 ± 1 hours soaking, remove the specimens from the solution, dry the surface with a paper towel to a SSD condition and immediately weigh to an accuracy of 0.01 g. Record this as the vacuum saturated mass $M_S$ of the specimen. The test procedure as described in Section 5 shall commence immediately after this weighing.

## 5. TESTING OF SPECIMENS

a) The chloride conductivity test must be conducted in a room in which the temperature is controlled at 23 ± 2 °C.

b) Unscrew the connecting points and fill the luggin capillaries (small plastic tubes shown in Figure 4) of the chloride cell and both chambers of the cell with the 5.0 M NaCl solution.

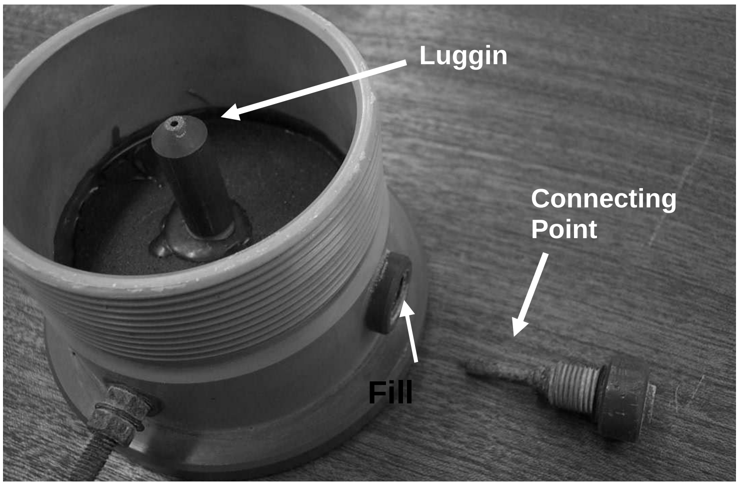


**Figure 4. Luggin and Connecting Point of Chloride Conductivity Cells.**

c) With the flexible collar in the rigid ring central portion of the cells, place a concrete sample within the flexible collar. The concrete sample must be placed so that one face is against the plastic lip of the rigid ring, as in Figure 5.

d) Screw the central portion of the cell into the cathode section of the cell. The cathode section has a solid

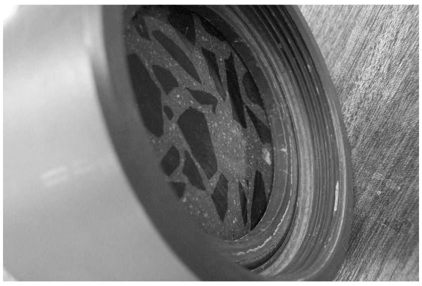


**Figure 5. Properly placed specimen in collar.**

plastic lip that must press against and compress the flexible collar. The correct alignment is shown in Figure 6. Screw the combined sections into the anode part of the cell. Tighten both parts sufficiently to ensure sealing of the sample and that no leaks occur.

e) Place the conduction cell horizontally.

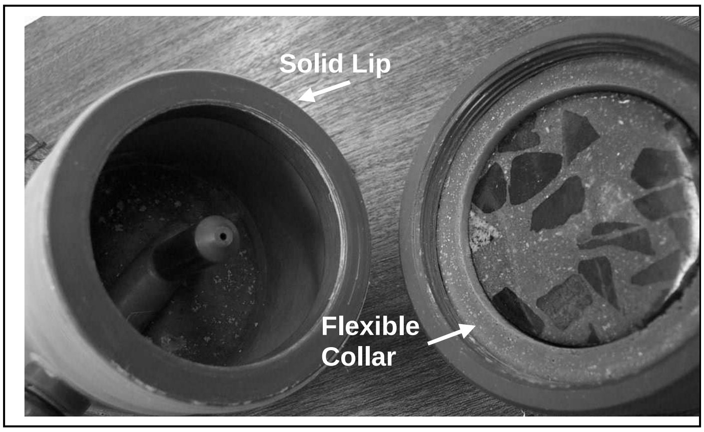


**Figure 6. Alignment of cells for proper sealing.**

g) Connect the ammeter and voltmeter as shown in Figure 2.

h) Adjust the DC power supply until the voltage applied <u>across the specimen</u> is approximately 10 Volts.

*Note: The preferred specimen test voltage is 10 V, but 5 V may be used for very permeable specimens.*

i) Simultaneously record the current and voltage readings.

j) Testing should be completed within 15 minutes of removing a specimen from the NaCl solution.

k) Specimens can be re-tested within 30 minutes of the first test, but should be discarded after that and cannot be re-tested. Specimens should be kept in the NaCl solution during this time.

## 6. CALCULATIONS

a) For each test specimen, individually calculate the chloride conductivity using the equation:

$$\sigma = \frac{id}{VA}$$

where:

σ = chloride conductivity of the specimen (mS/cm)
i = electric current (mA)
V = voltage difference (V)
d = average thickness of specimen (cm)
A = cross-sectional area of the specimen (cm$^2$)

b) The chloride conductivity index is given as the average of the chloride conductivity of at least three valid individual test determinations.

c) The porosity as a oercentage is determined by:

$$n = \frac{M_S - M_D}{Ad\rho_S} x100$$

where:

$M_S$ = the vacuum saturated mass of the specimen determined in section 4(h) to the nearest 0.01 g.
$M_D$ = mass of the dry specimen determined in section 4(b) to the nearest 0.01 g.
A = cross-sectional area of the specimen to the nearest 0.02 $mm^2$.
d = average specimen thickness to the nearest 0.02 mm.
$\rho_S$ = density of salt solution = $1.19 \times 10^{-3}$ $g/mm^3$.

*Note: It has been found that the porosity determined from the chloride conductivity test is normally lower than that determined in the sorptivity test.*

## 7. REPORTING

Report the following:

a) Identification number of specimen.

b) Description of specimen.

c) The chloride conductivity of each individual specimen.

d) The chloride conductivity index to the nearest 2 decimal places.

e) The porosity of the individual test specimens.

The following shall be reported if known

f) Source of the specimen.

g Location of specimen within cube, core or member

h) Identification mark of each specimen

i) Type of concrete, including binder type, water/cement ratio and other relevant data supplied with the specimen.

j) Curing history.

k) Unusual specimen preparation e.g. removal of surface treatment.

l) Unusual features such as cracks, voids, excessively chipped edges, etc.

m) Test operator.

n) Age of concrete at time of testing.

## 8. References

(1) Alexander MG, Ballim Y, Mackechnie JM, ‘ Concrete durability index testing manual’ Research Monograph no. 4, Departments of Civil Engineering, University of Cape Town and University of the Witwatersrand, March 1999

(2) Mackechnie JR and Alexander MG, ‘Practical considerations for rapid chloride conductivity testing.’ Proceedings of the Second International Workshop on Testing and Modelling the Chloride Ingress Into Concrete, C. Andrade and J. Kropp, ed., 2000.

(3) Gouws SM, ‘Durability index approach – method statements.’ Document submitted to the Durability Index Test Method working group

(Under the auspices of the C&CI Technical Committee), University of the Witwatersrand, 14 August 2003.

(4) Gouws SM, ‘Durability Index Approach – Progress Report 1: Method Statements’ Summary document of major amendments to Durability Index Test Methods as agreed upon by Durability Index Test Method working group (Under the auspices of the C&CI Technical Committee, University of the Witwatersrand, 19 August 2003.

(5) Gouws SM, ‘Durability Index Approach – Progress Report 2: Method Statements’ Summary document of major amendments to Durability Index Test Methods as agreed upon by Durability Index Test Method working group (Under the auspices of the C&CI Technical Committee, University of the Witwatersrand, 7 October 2003.

## 13. REVISIONS

| **Revision** | **Description** | **Date** |
|---|---|---|
| A | For approval | 8 April 2002 |
| B | Indicated with subscript B | 15 May 2002 |
| C | K. Stanish – remove marked with strike through, added in bold, comments in italics | 19 Feb 2003 |
| C1 | S. Gouws – suggested changes in red, *questions and remarks in italics, see also reference 3* | 16 July 2003 |
| D | Skipped | |
| E | For approval after Durability Index Test Method meeting 22 September 2003, see also reference 5. | 22 September 2003 |
| F | Editorial improvements and clarifications - MGA | April-July 2004 |
| G | KS – Clarification and illustration | April/May 2005 |
| H | MGA – Editorial corrections | Jan/Oct 2007 |
| I | MGA – Editorial and other corrections | Sep-Oct 2008 |
| J | All MGA<br>New Cl 4 b) inserted; other clause numbers in Cl. 4. changed accordingly<br>Cl. 6 a): ‘t’ changed to ‘d’ for specimen thickness; ditto Cl. 6 c) | 25 Feb 2009 |
| K | SG with MGA<br>Cl. 1 j): Must cool in desiccators only<br>Cl. 2 a): Changed ‘tap’ to ‘potable’<br>Cl. 6 c): change t to d in the calculation to be consistent | May 2010 |

# APPENDIX E: STATISTICAL TABLES

**Table E.1: Student Distribution Table (From Walpole et al., 2012)**

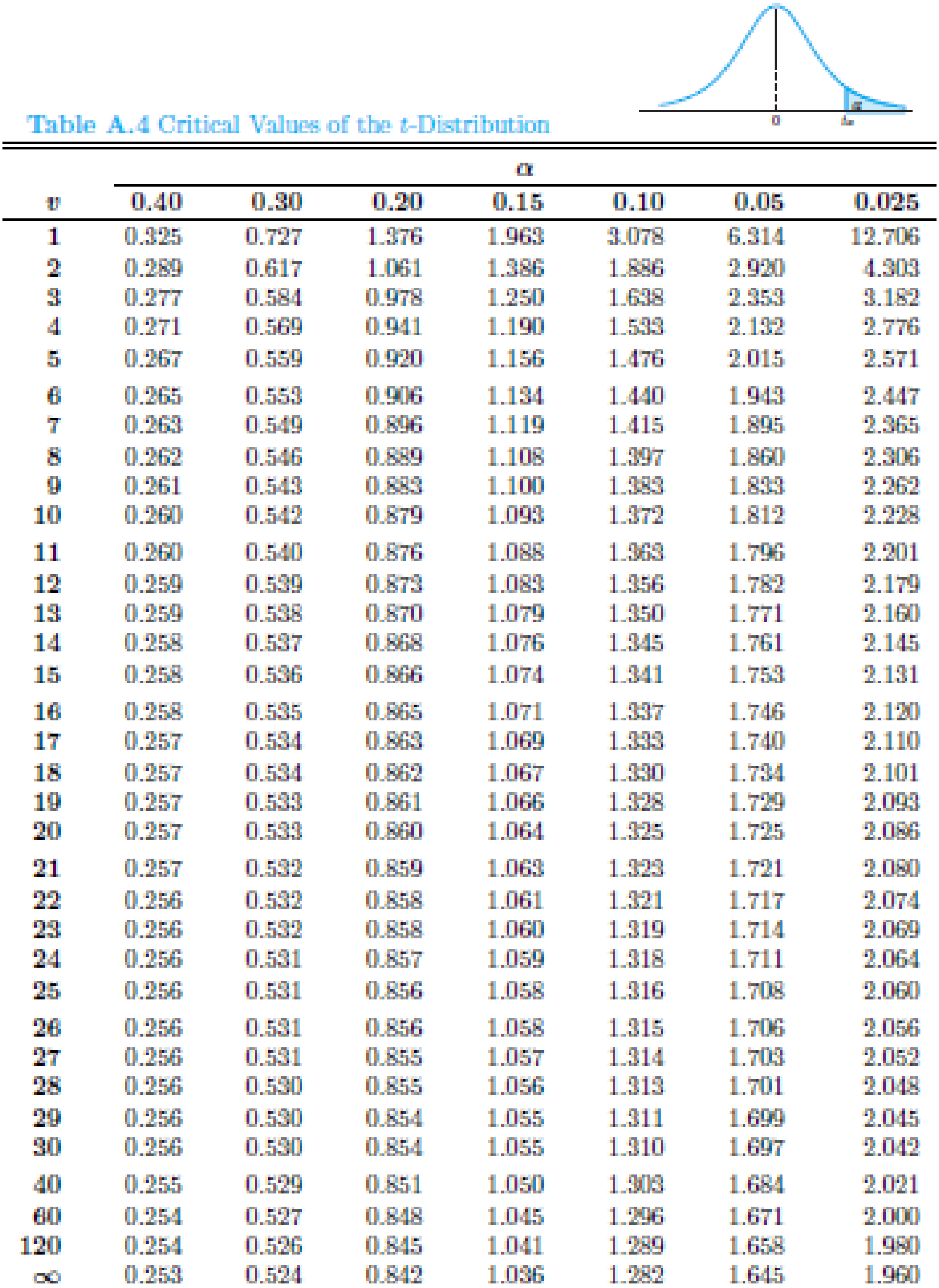


Table A.4 Critical Values of the $t$-Distribution

| | $\alpha$ | | | | | | |
|---|---|---|---|---|---|---|---|
| $v$ | 0.40 | 0.30 | 0.20 | 0.15 | 0.10 | 0.05 | 0.025 |
| 1 | 0.325 | 0.727 | 1.376 | 1.963 | 3.078 | 6.314 | 12.706 |
| 2 | 0.289 | 0.617 | 1.061 | 1.386 | 1.886 | 2.920 | 4.303 |
| 3 | 0.277 | 0.584 | 0.978 | 1.250 | 1.638 | 2.353 | 3.182 |
| 4 | 0.271 | 0.569 | 0.941 | 1.190 | 1.533 | 2.132 | 2.776 |
| 5 | 0.267 | 0.559 | 0.920 | 1.156 | 1.476 | 2.015 | 2.571 |
| 6 | 0.265 | 0.553 | 0.906 | 1.134 | 1.440 | 1.943 | 2.447 |
| 7 | 0.263 | 0.549 | 0.896 | 1.119 | 1.415 | 1.895 | 2.365 |
| 8 | 0.262 | 0.546 | 0.889 | 1.108 | 1.397 | 1.860 | 2.306 |
| 9 | 0.261 | 0.543 | 0.883 | 1.100 | 1.383 | 1.833 | 2.262 |
| 10 | 0.260 | 0.542 | 0.879 | 1.093 | 1.372 | 1.812 | 2.228 |
| 11 | 0.260 | 0.540 | 0.876 | 1.088 | 1.363 | 1.796 | 2.201 |
| 12 | 0.259 | 0.539 | 0.873 | 1.083 | 1.356 | 1.782 | 2.179 |
| 13 | 0.259 | 0.538 | 0.870 | 1.079 | 1.350 | 1.771 | 2.160 |
| 14 | 0.258 | 0.537 | 0.868 | 1.076 | 1.345 | 1.761 | 2.145 |
| 15 | 0.258 | 0.536 | 0.866 | 1.074 | 1.341 | 1.753 | 2.131 |
| 16 | 0.258 | 0.535 | 0.865 | 1.071 | 1.337 | 1.746 | 2.120 |
| 17 | 0.257 | 0.534 | 0.863 | 1.069 | 1.333 | 1.740 | 2.110 |
| 18 | 0.257 | 0.534 | 0.862 | 1.067 | 1.330 | 1.734 | 2.101 |
| 19 | 0.257 | 0.533 | 0.861 | 1.066 | 1.328 | 1.729 | 2.093 |
| 20 | 0.257 | 0.533 | 0.860 | 1.064 | 1.325 | 1.725 | 2.086 |
| 21 | 0.257 | 0.532 | 0.859 | 1.063 | 1.323 | 1.721 | 2.080 |
| 22 | 0.256 | 0.532 | 0.858 | 1.061 | 1.321 | 1.717 | 2.074 |
| 23 | 0.256 | 0.532 | 0.858 | 1.060 | 1.319 | 1.714 | 2.069 |
| 24 | 0.256 | 0.531 | 0.857 | 1.059 | 1.318 | 1.711 | 2.064 |
| 25 | 0.256 | 0.531 | 0.856 | 1.058 | 1.316 | 1.708 | 2.060 |
| 26 | 0.256 | 0.531 | 0.856 | 1.058 | 1.315 | 1.706 | 2.056 |
| 27 | 0.256 | 0.531 | 0.855 | 1.057 | 1.314 | 1.703 | 2.052 |
| 28 | 0.256 | 0.530 | 0.855 | 1.056 | 1.313 | 1.701 | 2.048 |
| 29 | 0.256 | 0.530 | 0.854 | 1.055 | 1.311 | 1.699 | 2.045 |
| 30 | 0.256 | 0.530 | 0.854 | 1.055 | 1.310 | 1.697 | 2.042 |
| 40 | 0.255 | 0.529 | 0.851 | 1.050 | 1.303 | 1.684 | 2.021 |
| 60 | 0.254 | 0.527 | 0.848 | 1.045 | 1.296 | 1.671 | 2.000 |
| 120 | 0.254 | 0.526 | 0.845 | 1.041 | 1.289 | 1.658 | 1.980 |
| $\infty$ | 0.253 | 0.524 | 0.842 | 1.036 | 1.282 | 1.645 | 1.960 |

**Table E.2: Critical values for the Kruskal Wallis test (From Walpole et al., 2012)**

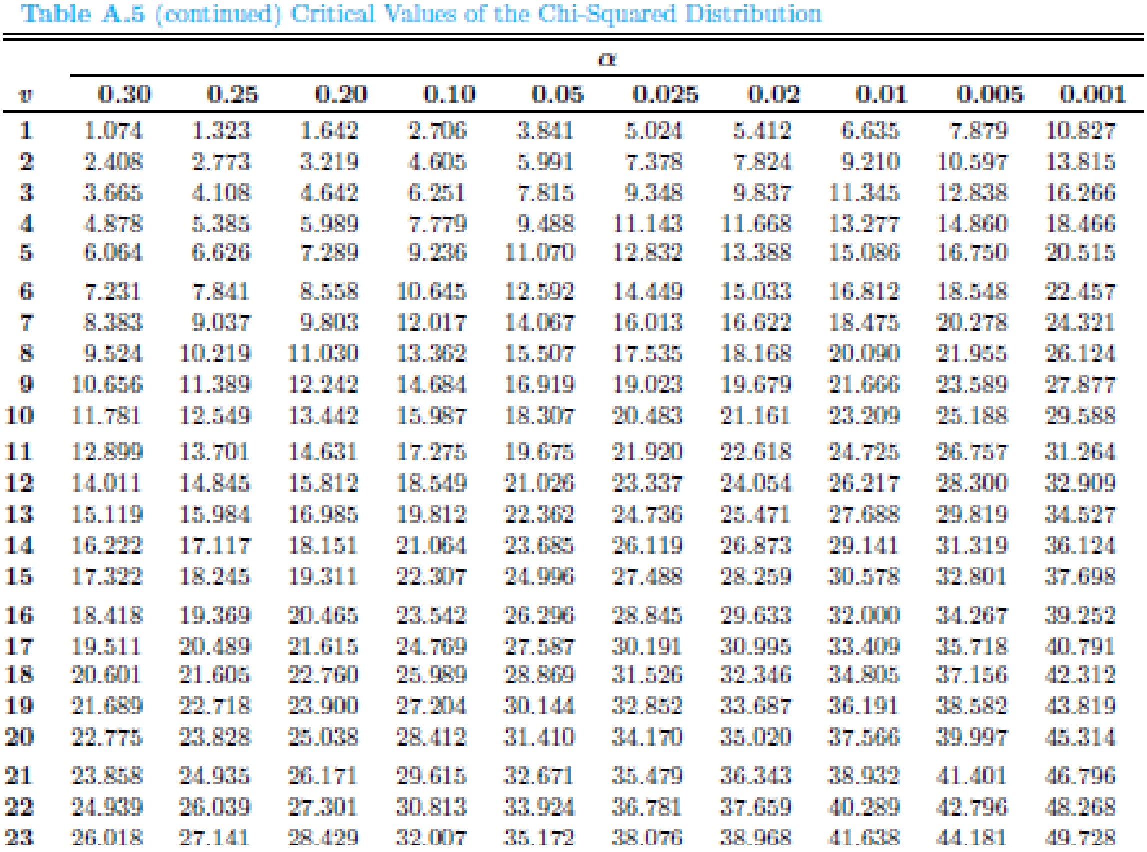
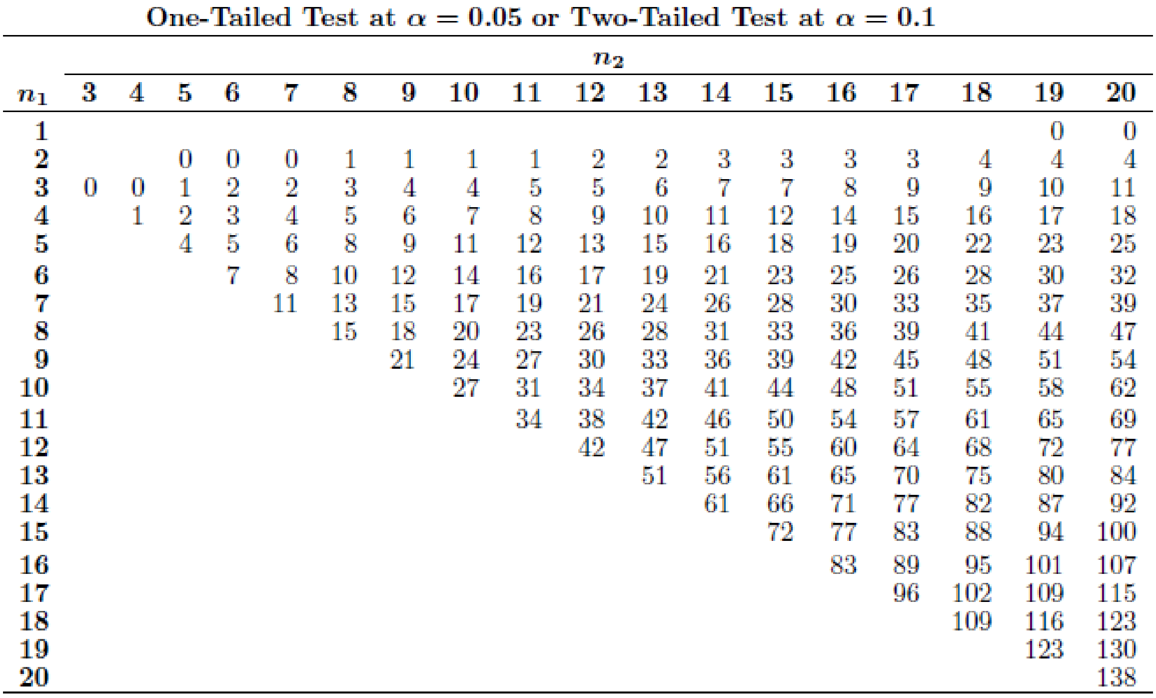


Table A.5 (continued) Critical Values of the Chi-Squared Distribution

| | $\alpha$ | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| $v$ | 0.30 | 0.25 | 0.20 | 0.10 | 0.05 | 0.025 | 0.02 | 0.01 | 0.005 | 0.001 |
| **1** | 1.074 | 1.323 | 1.642 | 2.706 | 3.841 | 5.024 | 5.412 | 6.635 | 7.879 | 10.827 |
| **2** | 2.408 | 2.773 | 3.219 | 4.605 | 5.991 | 7.378 | 7.824 | 9.210 | 10.597 | 13.815 |
| **3** | 3.665 | 4.108 | 4.642 | 6.251 | 7.815 | 9.348 | 9.837 | 11.345 | 12.838 | 16.266 |
| **4** | 4.878 | 5.385 | 5.989 | 7.779 | 9.488 | 11.143 | 11.668 | 13.277 | 14.860 | 18.466 |
| **5** | 6.064 | 6.626 | 7.289 | 9.236 | 11.070 | 12.832 | 13.388 | 15.086 | 16.750 | 20.515 |
| **6** | 7.231 | 7.841 | 8.558 | 10.645 | 12.592 | 14.449 | 15.033 | 16.812 | 18.548 | 22.457 |
| **7** | 8.383 | 9.037 | 9.803 | 12.017 | 14.067 | 16.013 | 16.622 | 18.475 | 20.278 | 24.321 |
| **8** | 9.524 | 10.219 | 11.030 | 13.362 | 15.507 | 17.535 | 18.168 | 20.090 | 21.955 | 26.124 |
| **9** | 10.656 | 11.389 | 12.242 | 14.684 | 16.919 | 19.023 | 19.679 | 21.666 | 23.589 | 27.877 |
| **10** | 11.781 | 12.549 | 13.442 | 15.987 | 18.307 | 20.483 | 21.161 | 23.209 | 25.188 | 29.588 |
| **11** | 12.899 | 13.701 | 14.631 | 17.275 | 19.675 | 21.920 | 22.618 | 24.725 | 26.757 | 31.264 |
| **12** | 14.011 | 14.845 | 15.812 | 18.549 | 21.026 | 23.337 | 24.054 | 26.217 | 28.300 | 32.909 |
| **13** | 15.119 | 15.984 | 16.985 | 19.812 | 22.362 | 24.736 | 25.471 | 27.688 | 29.819 | 34.527 |
| **14** | 16.222 | 17.117 | 18.151 | 21.064 | 23.685 | 26.119 | 26.873 | 29.141 | 31.319 | 36.124 |
| **15** | 17.322 | 18.245 | 19.311 | 22.307 | 24.996 | 27.488 | 28.259 | 30.578 | 32.801 | 37.698 |
| **16** | 18.418 | 19.369 | 20.465 | 23.542 | 26.296 | 28.845 | 29.633 | 32.000 | 34.267 | 39.252 |
| **17** | 19.511 | 20.489 | 21.615 | 24.769 | 27.587 | 30.191 | 30.995 | 33.409 | 35.718 | 40.791 |
| **18** | 20.601 | 21.605 | 22.760 | 25.989 | 28.869 | 31.526 | 32.346 | 34.805 | 37.156 | 42.312 |
| **19** | 21.689 | 22.718 | 23.900 | 27.204 | 30.144 | 32.852 | 33.687 | 36.191 | 38.582 | 43.819 |
| **20** | 22.775 | 23.828 | 25.038 | 28.412 | 31.410 | 34.170 | 35.020 | 37.566 | 39.997 | 45.314 |
| **21** | 23.858 | 24.935 | 26.171 | 29.615 | 32.671 | 35.479 | 36.343 | 38.932 | 41.401 | 46.796 |
| **22** | 24.939 | 26.039 | 27.301 | 30.813 | 33.924 | 36.781 | 37.659 | 40.289 | 42.796 | 48.268 |
| **23** | 26.018 | 27.141 | 28.429 | 32.007 | 35.172 | 38.076 | 38.968 | 41.638 | 44.181 | 49.728 |

**Table E.3 Critical values for the Wilcoxon Rank-Sum Test (From Walpole et al., 2012)**



One-Tailed Test at $\alpha = 0.05$ or Two-Tailed Test at $\alpha = 0.1$

| | $n_2$ | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| $n_1$ | **3** | **4** | **5** | **6** | **7** | **8** | **9** | **10** | **11** | **12** | **13** | **14** | **15** | **16** | **17** | **18** | **19** | **20** |
| **1** | | | | | | | | | | | | | | | | | 0 | 0 |
| **2** | | | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 2 | 2 | 3 | 3 | 3 | 3 | 4 | 4 | 4 |
| **3** | 0 | 0 | 1 | 2 | 2 | 3 | 4 | 4 | 5 | 5 | 6 | 7 | 7 | 8 | 9 | 9 | 10 | 11 |
| **4** | | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 14 | 15 | 16 | 17 | 18 |
| **5** | | | 4 | 5 | 6 | 8 | 9 | 11 | 12 | 13 | 15 | 16 | 18 | 19 | 20 | 22 | 23 | 25 |
| **6** | | | | 7 | 8 | 10 | 12 | 14 | 16 | 17 | 19 | 21 | 23 | 25 | 26 | 28 | 30 | 32 |
| **7** | | | | | 11 | 13 | 15 | 17 | 19 | 21 | 24 | 26 | 28 | 30 | 33 | 35 | 37 | 39 |
| **8** | | | | | | 15 | 18 | 20 | 23 | 26 | 28 | 31 | 33 | 36 | 39 | 41 | 44 | 47 |
| **9** | | | | | | | 21 | 24 | 27 | 30 | 33 | 36 | 39 | 42 | 45 | 48 | 51 | 54 |
| **10** | | | | | | | | 27 | 31 | 34 | 37 | 41 | 44 | 48 | 51 | 55 | 58 | 62 |
| **11** | | | | | | | | | 34 | 38 | 42 | 46 | 50 | 54 | 57 | 61 | 65 | 69 |
| **12** | | | | | | | | | | 42 | 47 | 51 | 55 | 60 | 64 | 68 | 72 | 77 |
| **13** | | | | | | | | | | | 51 | 56 | 61 | 65 | 70 | 75 | 80 | 84 |
| **14** | | | | | | | | | | | | 61 | 66 | 71 | 77 | 82 | 87 | 92 |
| **15** | | | | | | | | | | | | | 72 | 77 | 83 | 88 | 94 | 100 |
| **16** | | | | | | | | | | | | | | 83 | 89 | 95 | 101 | 107 |
| **17** | | | | | | | | | | | | | | | 96 | 102 | 109 | 115 |
| **18** | | | | | | | | | | | | | | | | 109 | 116 | 123 |
| **19** | | | | | | | | | | | | | | | | | 123 | 130 |
| **20** | | | | | | | | | | | | | | | | | | 138 |

# APPENDIX F: PROPOSED CONCEPTUAL DESIGN FOR CHLORIDE CONDCUTIVITY RIG

It was concluded that the current chloride conductivity rig is found wanting in many areas. The test is messy and the opaque nature of the material makes it difficult to determine if the cells are full with solution.in addition, the luggin capillaries do not ensure that the potential difference reading is taken exactly across the specimen faces. A redesign of rig is therefore warranted. One concept has been put forward in this Appendix.

The proposed design moves away from the current version, which requires the two sections of the cell to be screwed onto a central section which houses the test specimen. The new design proposes that the cell be open to the atmosphere so that the operator can see what is happening inside the cell. This will also result in there being no entrapped air once the solution is filled. The specimen is placed in a central section which can be inserted, like a cartridge, into a compartment in the centre of the cell. The specimen is sealed in the central section by means of a sealing plate which, like the current rig, pushes against the rubber collar and seals it. This is achieved by tightening the four screws shown in Figure F2. The central section itself is sealed in the cell by means of tightening the wing-nuts on the ends of the cell. Solution is then poured in the cell up to a level to cover the test specimen. The luggin capillaries have supports which rest on the threaded rods shown in Figure F.1 that hold the two cell compartments together. The luggins can be moved so that they touch the face of the specimen, ensuring that the voltage reading is taken exactly across the specimen.

To speed up the testing process, it can be possible to use several central sections which the specimens can be placed in and sealed. They can each then be tested, one-by-one, in the same cell without having to remove the solution. This can be done by simply loosening the wing-nuts to remove one central section and then inserting another one and tightening the wing-nuts again. Unlike the current test, the leads will not need to be removed to replace the specimens each time. This should make the whole process quicker.

The concept for the test rig is shown in Figure F.1.

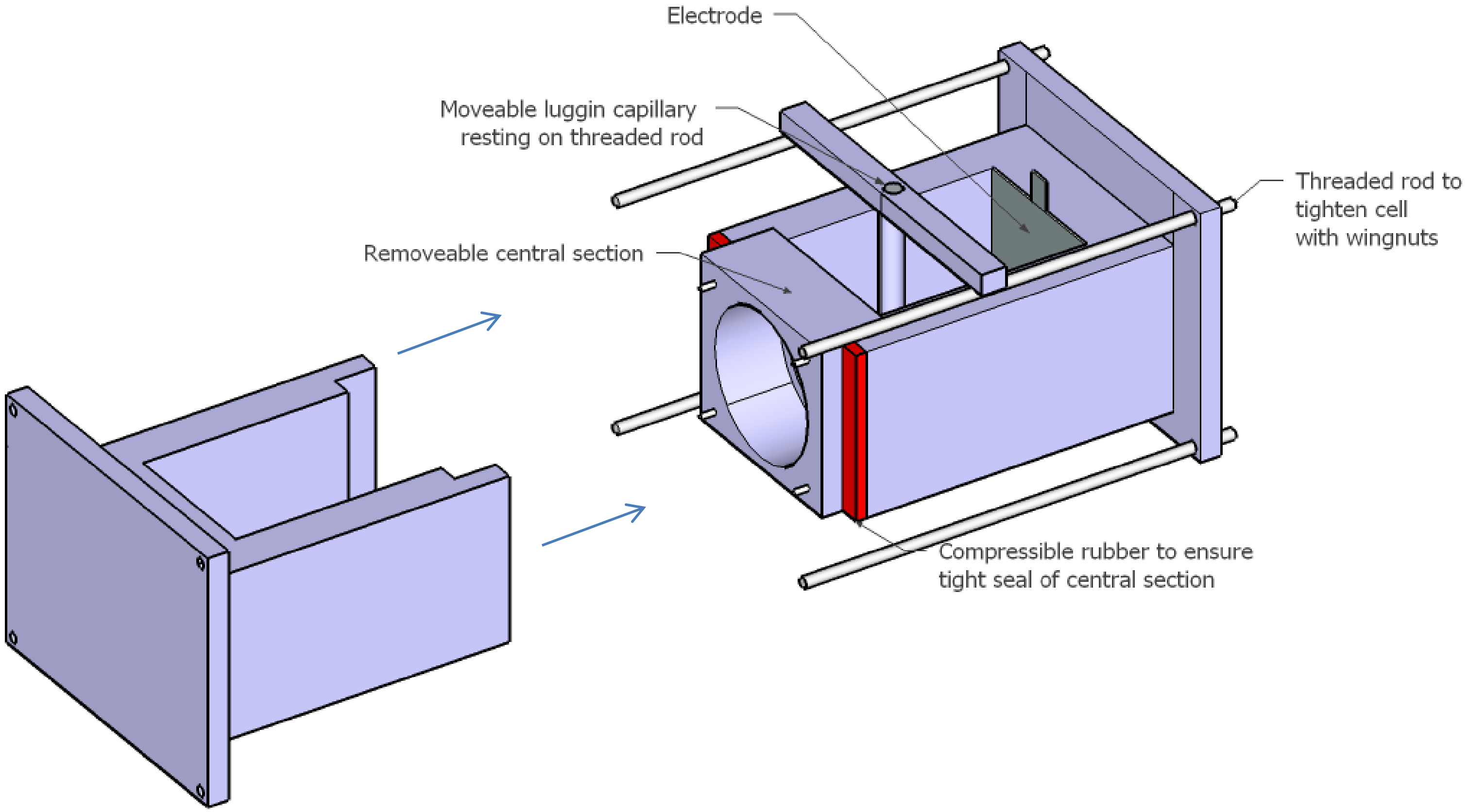


Figure F.1: Concept for chloride conductivity rig

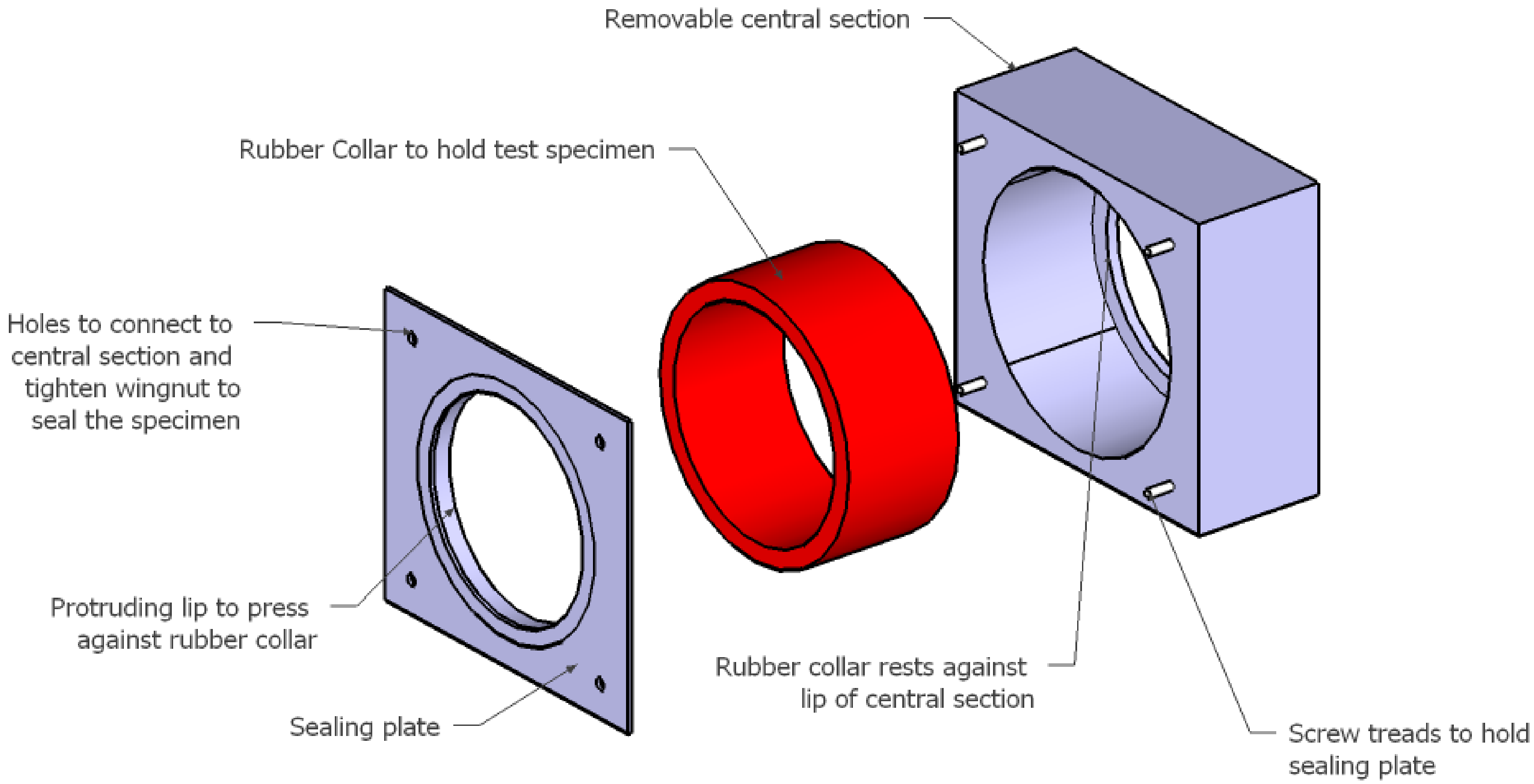


**Figure F.2: Central section detail for proposed chloride conductivity rig**