DOCTOR OF PHILOSOPHY

Influence of near surface moisture gradients in concrete on 'Autoclal' permeation measurements

Nolan, Éanna A.

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Influence of near surface moisture gradients in concrete on 'Autoclam' permeation measurements

by

Éanna A. Nolan

Thesis submitted to
The Queen's University of Belfast

for

The degree of Doctor of Philosophy

Faculty of Engineering
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Abstract

In this thesis the effect of moisture in the surface layers of concrete on the permeation properties measured using the 'Autoclam' is investigated. A literature survey indicated two main ways of removing the influence of moisture on these tests - to assess the moisture condition of the concrete and allow for it, or physically to alter the moisture state of the concrete prior to permeation testing.

A series of laboratory tests was undertaken to investigate the influence of different moisture conditions in the concrete on 'Autoclam' air permeability and sorptivity measurements. An analysis of the results indicated a unique relationship between the air permeability and the relative humidity recorded in a 10 mm deep drilled hole. A linear relationship was found between the sorptivity and the relative humidity.

A theoretical study of the effect of temperature on air permeability and sorptivity was undertaken and was confirmed by laboratory tests. In addition, an 18 month investigation was carried out on exposed samples of concrete to study the influence of changes in moisture conditions on permeation properties. Significant differences were found between the permeation results obtained for the laboratory and in in-situ concrete. Permeation measurements in exposed concrete with a relative humidity greater than 80% were shown to be ineffective in determining the quality of the concrete. On the basis of the in-situ results a procedure has been developed which renders Autoclam permeation test results largely unaffected by the influence of environmental factors in concrete with a relative humidity less than 80%. Thus, Autoclam permeation results obtained from in-situ tests, which were previously considered to be unreliable, can be corrected.

A parallel investigation into the feasibility of preconditioning in-situ concrete prior to permeation tests was also carried out. The application of hot air was found to be more efficient than other methods as a means of removing moisture from concrete. Overall, the use of preconditioning concrete prior to insitu permeation tests was found to be impracticable.
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1. Introduction

Reinforced concrete has been used in various forms since 1824 when Joseph Aspdin invented Portland cement. The way in which the general public have viewed concrete can be judged by use of the word 'concrete', which when used as an adjective in the English language, has come to mean 'existing in physical form, definite and/or positive' (Hawkins, 1990). Therefore, people in general have viewed concrete, and by implication reinforced concrete, as a positive and good invention which is beneficial. The absolute confidence with which the public once viewed concrete has changed significantly in recent decades as durability problems have become more common in reinforced concrete.

The response of the Civil Engineering research industry to the durability failures of reinforced concrete (Hime (1994), Montague, (1990), Murty et al, (1994) and Mehta (1994)) has been a large increase in interest in the areas of service life prediction, mechanisms of deterioration, the development of new materials, concrete additions etc. Most codes of practice now include some recognition that reinforced concrete must be designed for durability as well as mechanical strength, although the scientific basis for the clauses is often superficial. What has been noticeable, from its absence, is the adoption of an agreed method of inferring the durability of a concrete from tests, either insitu or laboratory based.
Without a method of predicting the likely durability of a concrete structure there is no way of assuring a client that a new structure will remain durable for a specified minimum period of time or that an existing structure is unlikely to require refurbishment within a specified period of time. This lack of information means that reinforced concrete as a structural material has an unknown total lifetime cost (including maintenance) and the quality factor included is a subject of uncertainty. Apart from the problems associated with promoting a product with an unknown life-span, the uncertainty that surrounds reinforced concrete durability is an indictment of an industry which has used this product for almost a century.

1.1 Durability of reinforced concrete

A durable reinforced concrete has been defined as 'one that is designed and constructed to protect the embedded metal from corrosion and to perform satisfactorily in the working environment for the life-time of the structure' BS 8110 part 1, (1989). As the definition implies, different structures are subjected to different environments and therefore to different environmental loadings. This uncertainty is compounded by the possibility of bad concrete practice which may occur on-site. The result of such bad practice is often a reduced ability of the hardened concrete to resist environmental attack. These factors together make a persuasive case for checking the suitability of the concrete which is actually in the structure and not testing cubes or cylinders in a laboratory.
1.1.2 The critical role of near surface concrete in durability

The above definition of reinforced concrete durability draws attention to the fact that, in a durable structure, the concrete will protect the embedded reinforcing steel from aggressive elements in the immediate environment of the structure (figure 1.01). The compaction process on-site causes the entrapped air and excess water from the mix to migrate both upwards and outwards towards the formwork. This results in the formation of a near surface concrete (also known as covercrete or cover concrete) which is more porous and permeable than the bulk concrete (figure 1.02). The ability of this near surface concrete to exclude harmful substances will often determine the durability of a reinforced concrete member.

1.1.3 Permeation properties of the cover concrete

The permeation properties of the concrete allow the ingress of harmful substances through the near surface concrete to the reinforcing steel. Once the aggressive substances have reached the steel in significant quantities a corrosion process will occur, given an adequate supply of oxygen and water. The formation of iron oxide (rust) on the steel generally causes cracking of the concrete and eventually spalling of the near surface concrete. Therefore, the permeation properties of the near surface concrete can be related to the likely durability of a reinforced concrete member.

1.1.4 Permeation testing on-site

The idea of using in-situ permeation tests to determine the ability of the near surface concrete to protect the reinforcing steel from environmental attack has been well documented in literature. A testament to the acceptance of the importance of insitu permeation testing
can be gauged from the number of different permeation tests which exist (detailed in chapter 2). The rationale for using permeation tests in-situ rather than in a laboratory is that the best indication of the true physical properties of a cover concrete can only be obtained from an 'undisturbed' sample of the concrete. The laboratory tests invariably use a cored sample which may have been microstructurally damaged by the sampling process.

The 'Autoclam' permeation system has been developed in the Department of Civil Engineering at the Queen's University of Belfast, to measure the air permeability and sorptivity of near surface concrete. The unit is designed for use on site and uses a metal ring which is bonded to the concrete surface to ensure ease and completeness of seal. The 'Autoclam' permeation system is commercially available and is currently in use world-wide.

1.1.5 The need to remove the influence of environmental parameters from in-situ permeation results

In-situ permeation tests are influenced not only by the capillary pore structure of the near surface concrete but also by:

(i) the moisture condition of the concrete.
(ii) the temperature at which the test is carried out.

Both of these factors, which are uncontrollable on-site, have the ability to mask the 'true' permeation properties of a concrete and to give an incorrect impression of the ability of the concrete to exclude harmful substances. The permeation properties of a concrete, uncorrected for these environmental effects, give a measurement of the durability of a concrete at the time of test alone. This data is not useful as the environmental factors are constantly fluctuating and there is no guarantee that these measurements are representative of the average permeation properties which will exist over the service life of the structure.
The removal of the influence of both moisture and temperature from insitu permeation measurements allows the categorisation of the concrete material itself. Then, with a knowledge of the material properties of the near surface concrete and an estimate of the environmental load to which a structure will be subjected over its life span, it should be possible to predict the likely durability of the structure.

1.2 Research objectives

The main objectives of the work reported in this thesis are to:
(i) investigate the relationship between air permeability, sorptivity and the moisture condition of concrete.
(ii) measure and allow for the influence of environmental factors on in-situ permeation measurements carried out on exposed concrete.
(iii) investigate the use of preconditioning methods to remove the influence of moisture from permeation tests.

1.3 Outline of the thesis

This thesis deals with the measurement of permeation properties of in-situ concrete with the 'Autoclam' permeation system.

Chapter 2 reviews the published material on the influence of the properties of concrete and its moisture condition on the permeation properties of concrete.

Chapter 3 details the procedures and test methods which were used in the experimental investigations. Preliminary investigations into the measurement of relative humidity in concrete are also presented.

Chapter 4 details an investigation into the influence of moisture on the permeation properties of concrete. A laboratory investigation into the
influence of moisture in concrete on Autoclam air permeability and sorptivity measurements is presented and analysed. The use of resistance measurements between embedded electrodes in the concrete to measure moisture movement is examined and compared with the use of relative humidity, as a method of measuring free moisture in capillary pores.

Chapter 5 examines the influence of temperature on the Autoclam permeation properties and details the results of an 18 month investigation into the fluctuating moisture and permeation properties of concrete kept outdoors.

Chapter 6 examines the feasibility of using a method of preconditioning concrete to remove the influence of moisture prior to the permeation tests. The effect of four different methods of preconditioning concrete was examined, with the movement of moisture through the concrete during the preconditioning period being monitored. The use of Autoclam air permeability and relative humidity tests after a period of preconditioning were used to identify the success of each method.

Finally, chapter 7 summarises the main conclusions derived from the investigations and presents recommendations for further work.
Concrete Surface

Ingress of aggressive substances, \( \text{O}_2, \text{H}_2\text{O} \) etc.

Cover concrete

Spalling allows increased ingress of aggressive substances and hence increased corrosion

Reinforcing steel
Rust causes cracking of cover concrete. Greater ingress of aggressive substances. Rust expansion causes spalling

Figure 1.01 - Corrosion of reinforcement in concrete

Zone of much poorer quality concrete

Zone of generally uniform higher quality concrete (Heartcrete)

Zone of poorer quality concrete (Covercrete)

Cross section of a structural member (Sitecrete) or (Realcrete)

Figure 1.02 - Effect of water mobility and lack of curing on the quality of site concrete (Dewar, 1985)
2. Moisture conditions and permeation properties of concrete

2.0 Introduction

This chapter details the background to the research work which was undertaken for this thesis. Variables in concrete manufacture and constituents which influence the permeation properties are examined and definitions of transport mechanisms and relevant test methods are reviewed. Finally the influence of moisture on the measured permeation properties of concrete is examined. The purpose of the review is to present the scope of current research, test methods which are used and to highlight the significance of the studies presented in this thesis.

2.1 The relevance of permeation properties to durability

2.1.1 Durability failures in reinforced concrete

'Concrete was once thought to be maintenance free. The experience of the last 20 years [1970’s and 80’s] have shown this not to be the case. While some cases of durability problems have been noted before 1970 (Hime, 1994), general concern about reinforced concrete durability has grown significantly in the recent past. In the UK £4 M repair works have recently been carried out to a twenty year old, ten lane motorway bridge in Glasgow, which originally cost £6.5 M (Montague, 1990). World-wide, durability failures have occurred in prestige projects such as tunnel
schemes in Hong Kong, Abu Dhabi and Suez (Murty et al, 1994). Mehta (1994) reports that in 1987 some 253,000 bridges in the USA were identified as having durability problems. The economic consequences of durability failures of concrete structures are immense, and recent studies have indicated that up to 40% of the original construction costs are spent on repair and maintenance of concrete structures (Gerwick, 1994).

Why such an apparent decrease in the durability of reinforced concrete structures should have occurred has been attributed to a change in the material characteristic of concrete. This change in concrete properties has been studied by Murty et al (1994), and Ben-Bassat (1990) with reference to changes in cement properties. The C₃S content of cement has increased considerably in the past forty years, along with an increase in fineness of the cement in the same period. These changes have helped to speed up construction. However they also have other less desirable effects such as increased heat of hydration, increased drying shrinkage and increased water / cement (w/c) ratios. Typically, a concrete mix of given strength can be produced with modern cement with a much greater w/c ratio than that required to achieve a similar strength forty years ago. An increase in the w/c ratio leads to an increase in the free water content of the concrete mix, an increased volume of capillary pores in the hardened concrete and hence greater permeability. The ingress of aggressive substances the concrete cover is primarily through the capillary pore system therefore an increase in permeability causes a decrease in durability.

2.1.2 Durability failure mechanisms in reinforced concrete

The long-term durability of a reinforced concrete structure is governed by the interrelationship of the environment, the microstructure of the cement paste and the fracture strength of the concrete. A survey of
literature (Basheer et al, 1995) (figure 2.01) lists the frequency of references to the various mechanisms of deterioration in reinforced concrete. If this is taken as a measure of the concern about each of the deterioration mechanisms it can be seen that those mechanisms which are influenced or controlled by the permeability of the cover concrete account for over 75% of the references. These mechanisms include:

(i) Corrosion of reinforcement
(ii) Carbonation
(iii) Chloride ingress
(iv) Freeze-thaw
(v) Chemical / Sulphate attack

2.1.2.1 Corrosion of reinforcement

The corrosion of reinforcement in concrete is a final part of most durability failure mechanisms. However the process itself is controlled by the permeability of the cover concrete. Concrete normally provides a highly alkaline environment which deters corrosion. Should this alkalinity be lost the corrosion process, as described in figure 2.02, may commence. Some quantities of both oxygen and water may be present at the corrosion site at the initiation of corrosion. To sustain a corrosion reaction they must be transported through the concrete from the environment (Schiessl, 1988). Thus, the pore size, tortuosity and continuity of the capillary pore system in the concrete (which act as a conduit for oxygen and water) are of critical importance to the likelihood of sustained corrosion taking place. Permeability tests are influenced by the size, tortuosity and continuity of the capillary pore system of concrete. Thus the corrosion process of reinforcement steel in concrete is dependent on the alkalinity of the concrete and the permeability of the surrounding concrete. Rust production at the reinforcement can also crack the concrete,
increasing the permeability of the concrete and in turn increasing the rate of corrosion.

2.1.2.2 Carbonation

Carbonation can lead to the corrosion of steel reinforcement in concrete. Carbon dioxide, which forms roughly 0.03% by volume of the atmosphere, diffuses into the capillary pores of the concrete where it reacts with calcium hydroxide (Roberts, 1981, Roy, 1986, Beckett, 1983, Parrott, 1994). The reaction has the effect of lowering the pH of the concrete from 13 to below 11.5. This is significant as the reduction in alkalinity allows steel corrosion to occur. The speed of ingress of the carbonation front into the concrete is governed by the capillary pore structure of the cover concrete.

Roberts (1981) makes the general observation that in a well made concrete of low permeability, carbonation will take place very slowly over a period of years. The depth of carbonation in a concrete has been related to the air permeability of the concrete (Kropp 1995). Figg (1989) identified a linear relationship between air permeability (as measured by the Figg test) and depth of carbonation. Basheer (1991), in a series of site tests, showed a direct relationship between the air permeability of the concrete (as measured by the Autoclam) and the depth of carbonation found in road bridges in Northern Ireland.

2.1.2.3 Chloride attack on reinforced concrete

Chloride has been known, by experience, to cause metals to corrode as far back in history as 399 BC when Socrates is quoted as saying 'For this earth, and the stones, and the entire region which surrounds us, are spoilt and corroded, like the things in the sea which are corroded by the brine' (Hime, 1994). Therefore it should be of little surprise that reinforcing steel
embedded in concrete is likely to corrode when chloride is present in the surrounding concrete. The ingress of chloride ions to the depth of the reinforcing steel in concrete allows the initiation of the corrosion process.

Chloride ions can travel through the concrete in three distinct ways - absorption, permeability and diffusion (Maage, 1993). A typical absorption process may involve water which is laden with salts being absorbed into the concrete. This process is relatively rapid and can transport large quantities of chloride into the concrete, depending on the concentration of chloride ions in the water. The permeability process could typically be found at the base of a deep sea marine structure where sea water under pressure is forced into the pore system of the concrete. Thus, the ingress of significant quantities of chloride into concrete to the depth of the reinforcing steel is dependent on the capillary pore system in the concrete. Diffusion of chloride into concrete is a slow process taking place over a period of years and is described by Fick's second law.

2.1.2.4 Freeze-thaw deterioration

Freeze thaw deterioration of concrete can be described by a cycle of deterioration of concrete as illustrated in figure 2.03. The main mechanism of deterioration is caused by the freezing of water in concrete pores. The volume change associated with the phase change from water to ice is considered to be 9%. In reasonably large pores, as ice forms at one end of a pore, the expansion associated with phase change causes the remaining water in the pore to become pressurised (Mehta, 1994). These pressures are believed to be responsible for cracking the cement paste and/or aggregate which increases the permeability of the concrete and hence allows the corrosion of reinforcing steel. The use of mixes with w/c ratios sufficiently low for the paste to have only small capillaries and only little freezeable water is one method of producing frost resistant
concrete (Neville, 1981). This is illustrated by figure 2.04 which shows the relationship between freeze-thaw resistance of concrete and the water absorption of concrete. Hooton (1992) states that an investigation of dam structures in Canada showed that concrete with a low w/c ratio (and hence low permeability) had withstood 40 to 75 years exposure.

2.1.2.5 Chemical / Sulphate attack

Many different types of chemical attack are known to produce rapid loss of durability of concrete. Common in sulphate attacks is that the cement paste itself is weakened directly by sulphates in solution which attack one component of the calcium silicate hydrate (CSH). The resulting micro and macro cracking increases the permeability of the concrete which in turn allows more sulphate solution into the concrete. The capillary pore system of the cover concrete allows the ingress of sulphate solution and therefore the permeability of the concrete can be used as a measure of the likely resistance of a concrete to sulphate attack. Mehta (1994) states that '.. as a result of chemical attack only permeable concretes in a moist environment are vulnerable.' (Mehta's bold type). Neville (1981) also stresses the importance of the permeability of concrete in resisting sulphate and chemical attack by stating 'What is important to note is that in some cases the density and permeability of a concrete influence its durability to such a degree that they override the influence of type of cement used'.

2.1.3 Approaches to reinforced concrete life span estimation

Basheer et al (1996) outlines two approaches to estimating the life span of concrete structures - a macro-predictive model and a micro predictive model.
The macro predictive model shown in figure 2.05 was used in conjunction with Autoclam permeation measurements, and a knowledge of the exposure environment to give a prediction of the likely durability of concrete bridges in Northern Ireland. The model does not predict the rate of corrosion but rather gives a rating based on a classification of permeation measurements. A relatively similar model has been proposed by Dhir (1994) based on ISAT and Figg measurements. The main problem associated with these approaches to durability estimation is the fact that there is often a lack of long-term in-situ studies of durability against which to check the validity of the chosen classifications. With experience, classifications will become more refined and useful.

The micro predictive model outlined by Basheer et al. (1996) is presented in figure 2.06. The model is based on three interrelated models: a transport model, a deterioration model and a structural model. The transport model for chloride ingress is given as Fick's law which describes the diffusion of ions into concrete. Using finite element techniques the model predicts the chloride distributions in concrete for given conditions. The corrosion model gives an indication of the damage caused by rust formation on the reinforcing bar. The structural model considers the structural consequences of the deterioration of the concrete caused by rust formation. The micro predictive model considers each of the deterioration mechanisms in turn and so the interrelationship between them is not included in the output. It would be incorrect to suggest that the micro predictive model is a finished product or that it can be used with a degree of confidence, however it does offer a way of predicting the likely relative durability of a structure.
2.2 The properties of concrete which influence its permeation characteristics

2.2.1 The structure of concrete

The structure of a hardened concrete depends on the nature of the constituent materials, the volume ratio of constituents, the compaction of the concrete prior to setting, the ambient conditions during casting and the curing of the concrete after hardening. The structure of the cement paste evolves as the hydration of the compounds in cement proceeds. The hardened cement paste consists of the hydrates of the four main compounds (gel), water, unhydrated cement particles, calcium hydroxide (Ca(OH)$_2$ crystals), minor compounds and voids which were originally filled with water (Neville, 1981).

2.2.1.1 Cement paste structure

The calcium silicate hydrates (CSH gel) take the form of plate-like structures and diagrammatic representations of three models put forward to illustrate the structure of gel are shown in figure 2.07 (Oberholster, 1986). The Powers and Brunauer model consists of groups of CSH particles two or three layers thick which are randomly arranged and bonded by surface forces. The Feldman-Sereda model consists of single sheets of CSH which come together randomly to give interlayer spaces. The bonding between the CSH sheets is held to be somewhere between the force of bonding associated with Van der Waals forces and ionic covalent bonds. Solid to solid contact between the CSH is thought to form on drying and is disrupted on re-wetting. The Munich model (figure 2.07) is based on absorption measurements and was used to explain the behaviour of cement paste at different moisture contents.
Lea (1970) identifies two separate 3 dimensional networks which hold the CSH plate like structures together. A coagulation network based on weak Van der Waals forces and a crystalline network based on chemical valency bonds. The crystalline network, although strong, if broken will not re-form unless further crystallisation occurs, whereas the coagulation network can re-form its weak network readily.

Pores within the CSH gel vary in size from 1.5 and 2.0 nm in diameter (Neville, 1981), below 2.5 nm (Mindes, 1981). Oberholster, (1986) states that the interlayer spaces between the sub parallel CSH plates (as described in the Feldman-Sereda model) range in size between 0.5 and 2.5 nm and that both the interlayer spaces and gel pores both have narrow entrances. The size of these pores means that they are generally held not to contribute significantly to the permeability of a concrete. Water molecules held in the gel pore are only an order of magnitude smaller than the spaces they occupy in the CSH gel and hence they do not behave in the same manner as free water in capillary pores.

2.2.1.2 Capillary pores in concrete

More water than is needed to achieve full hydration is commonly added to concrete mixes in order to achieve reasonable workability. The excess water, when evaporated from the hardened concrete, leaves capillary spaces. However, the volume of cement gel and other hydration products is about 2.1 times that of the cement from which it is derived (Powers, 1959), therefore hydration products replace some of the water filled space. This growth of the CSH gel into the capillary space as hydration proceeds may cause loss of continuity in the larger capillary pores. Times to capillary discontinuity in cement paste are presented in table 2.01. The hydration process stops in smaller capillary pores due to lack of water or because the crystals have no room to develop (Powers...
1946, 1958). For this reason, the full discontinuity of the pore system, and hence the reduction to impermeability of concrete, does not occur.

The hydration process leaves a labyrinthine, interlinked, random system of pores of variable cross sectional area and shape. In theoretical work capillary pores are often taken to be cylindrical, however this is not an accurate description of their physical nature (Brunauer, 1967). The approximate diameter of capillary pores is in the order of 1300 nm (Neville, 1981). Oberholster (1986) estimates the size of capillary pores as being between 10 nm (small pores) to 15000 nm in diameter (larger pores) (Table 2.02).

2.2.2 Constituents of concrete

In concrete the main constituents are a binder, normally class 42.5 Portland cement (OPC), both fine and coarse aggregates and water. The constituent materials which make up concrete affect the properties of the hardened concrete.

2.2.2.1 Cement

The cement type most commonly used for general building and civil engineering works, is Class 42.5 Portland cement, commonly known as ordinary Portland cement (OPC). For a durable concrete a minimum cement content is commonly specified and BS 8110:part 1 gives minimum cement contents (Table 2.03). A selection of cement properties, taken from Corish (1994) is presented in this table 2.04. As can be seen from this table, the properties of cement have changed over the past 30 years. These changes have an influence on the structure of both the concrete mix and the hardened concrete as discussed in section 2.1.1. Corish (1994) reports that BS 12 (1991) gives no requirement for a cement producer to limit the fineness of the cement. Generally, finer cement particles result in a
quicker concrete set, an earlier strength development, and a smaller aggregate cement transition zone. Changes in the $C_3S/C_2S$ ratio in cement have been found to influence the properties of hardened concrete (Ben-Bassat, 1990).

The use of pozzollanic additions such as microsilica (silica fume), metakaolin, ground granulated blast furnace slag (GGBFS) and pulverised flyash (PFA) are becoming more popular ways of reducing the permeability of the cover concrete. A typical example of such a material is microsilica which consists of ultra fine microspheres of silicon dioxide. The silicon dioxide is a reactive material which, when added to a concrete mix forms a secondary crystalline structure which has been shown to reduce the permeability of the hardened concrete (Male, 1989). Similar reductions in permeation properties have been reported with the addition of metakaolin (Caldarone, 1994), GGBFS (Neville, 1981) and PFA. (Dhir, 1991) to concrete mixes.

2.2.2.2 Aggregates

Aggregates constitute roughly 75% of a hardened concrete and hence are important to the final overall properties of the concrete. The distribution of the sizes of aggregate is referred to as the aggregate grading. Very fine sands are often excluded from concrete as they have been held to increase the water requirement of a concrete mix and a large gap in the grading curve is thought to create handling and finishing problems. Research carried out by Banfill et al (1987) shows that the increase in water requirement is minor and that the sorptivity of the concrete is reduced by inclusion of very fine aggregate in a mix. The decrease in sorptivity can be explained by the filling effect of the fine aggregate.

The aggregate particles in a concrete are held not to contribute significantly to the measured permeability of a hardened concrete.
Generally the permeability of aggregates is so low that they are considered to be impermeable. The aggregate particles in a cement paste act as obstacles to the flow of water which tends to make capillary pore system more tortuous and hence reduces the permeability of the hardened concrete.

Around the aggregate particles, which are distributed throughout concrete, Ca(OH)$_2$ crystalline structures form. These are generally referred to as the transition zone. The aggregate attracts and holds water at its surface during mixing and compaction. The excess of water in this area leads to accelerated hydration, which in turn leads to the preferential formation of Ca(OH)$_2$ crystals. The large crystalline nature of the transition zone means that it has increased porosity and permeability when compared to the CSH matrix (Larbi, 1993). This zone is illustrated in figure 2.08 and is estimated to be in the order of 30 μm thick. Xie (1991) found that the transition zone between aggregate and the cement matrix decreased with particle size and that the thickness of the water layer in the transition zone decreases linearly with decreasing aggregate size.

The transition zone often does not contribute appreciably to the permeability of concrete as each zone is discrete and separate, however in concretes where the transition zones of the aggregates touch each other a system of large pores which extend throughout the concrete increases the concrete permeability significantly. Winslow et al (1994) describes this interlinking of the transition zones in concrete and reasons that the difference between the permeability of cement paste (low permeability) and concrete (can be much higher permeability) can be explained by the interlinked transition zones providing a continuous system of large pores which increase permeability.
2.2.2.3 Water

The quantity and quality of water used in a concrete mix affects the physical properties of the resultant concrete. The minimum quantity of water required to achieve full hydration of the cement binder in a concrete is generally expressed as being between 0.35 - 0.40 w/c (Neville, 1981). This means that concretes with a low w/c ratio and without admixtures will have poor workability and this may cause incomplete compaction and a porous and permeable structure. Alternatively, concrete mixes with large w/c ratios will have excess water which, when evaporated, leaves a porous and permeable hardened concrete. Table 2.03 gives maximum w/c ratios for different grades of concrete as specified by BS 8110 : part 1 (1989). The consequences of water in excess of that required for hydration have been discussed in greater detail in section 2.2.1.2.

2.2.3 Curing of concrete

Curing can be defined as the 'creation of an environment in which hydration reactions can proceed to help fulfil the aim of producing concrete of adequately low porosity' (Cather, 1994). Research into the hydration of cement particles has identified that, as the relative humidity in concrete drops below 100%, the hydration process is significantly reduced. Specifically, it was found that half of the full hydration occurs at 90 % internal relative humidity and that hydration stops in concrete with an internal RH of less than 80% (Parrott et al, 1986). In order to promote full hydration in the cover concrete, curing is applied to ensure that the concrete retains enough moisture to promote maximum hydration. While it is feasible to place laboratory test specimens in a water bath for 28 days it is rarely economic or practical to provide such efficient curing on site.
Conventional wisdom states that an efficient curing regime will promote a lower cover concrete permeability. The explanation for this is that the products of hydration fill the capillary pore system causing discontinuity in the pores and hence reducing permeability. Laboratory studies on small scale specimens have tended to conclude that the benefit of curing in reducing permeation properties is significant (Ho et al, 1989). A study using large scale test blocks which were subjected to commonly used site curing techniques shows a less significant reduction in covercrete permeation properties (Nolan et al, 1996). It was found that small scale specimens, which are generally used to assess the efficiency of curing, may not give a true indication of the effect of commonly used insitu curing methods in real structures.

2.3 Moisture in concrete

2.3.1 Moisture states in concrete

Parrott (1990) divides water in concrete into three main states

(i) Free water held in capillary pores.
(ii) Adsorbed water held by surface forces.
(iii) Chemically combined water.

The binding energy of the H$_2$O molecules can also be used to categorise the different water states in concrete (Englert and Wittmann, 1968), as given below:

(i) Water in the CSH is held with an energy equal to or less than that associated with the first layer of water adsorbed on the walls of a pore (monolayer water)
(ii) Interlayer water is a 'kind of link with pore water.'
(iii) Monolayer water absorbed on the surface of the CSH.
(iv) Water absorbed on top of the monolayer absorbed water.
(v) Water condensed in micropores (radius < 0.1 μm (10^{-7} m)).
(vi) Macropore water (radius > 0.1 μm).

It is difficult to categorise water in cement paste from physical measurements as the binding energy associated with each of the states overlaps significantly.

Harris, et al (1974) investigated the freedom of mobility of water in cement paste. They concluded that moisture could be divided up into free and bound water which could be identified by neutron scattering techniques. The free water is said to have the properties of bulk water and they found that this was approximately equal to the quantity of capillary water. The free water content was found to be consistently less than the quantity of water removed by drying at 100 °C. This indicates that both combined and free water is removed during 100°C drying and it illustrates the difficulty in quantifying the amount of moisture in the capillary pores.

2.3.2 Measurement of moisture in concrete

As stated previously, the measurement of moisture in concrete is often found to be difficult as the moisture states in concrete are difficult to distinguish. The following methods can be found in literature as methods of moisture measurement in concrete.

2.3.2.1 Internal relative humidity of concrete

'Relative humidity is perhaps the most familiar humidity parameter used, misused, confused and abused' (Penman, 1955). Relative humidity is a relative measure of the vapour pressure of the gas which is in contact with the water in the capillary pores compared to the pressure at which phase change from vapour to liquid occurs, as shown by the Kelvin equation [2.01] (Penner, 1965 and Morrow, 1970).
\[-\frac{2\gamma M \cos \vartheta}{rdRT} = \ln \frac{P}{P_o} = \ln \frac{RH}{100}\]  

Eqn. 2.01

where,

- $P_o$ is the partial vapour pressure
- $P$ is the equilibrium pressure,
- $P_o$ is the liquefaction pressure,
- $M$ is the molecular weight of water (amu.),
- $R$ is the gas constant (J/K.mol),
- $T$ is the absolute temperature (K),
- $r$ is the radius of the pore (mm),
- $\vartheta$ is the angle of contact between the pore water and the capillary pore wall (Deg)
- $d$ is the density of water (kg/m³),
- $\gamma$ is the interfacial tension between water and air (N/m),
- $RH$ is the relative humidity (%).

This equation has been verified by experiment down to a radius of 9 nm (Fisher et al 1981). Therefore, the measurement of internal RH is not a measure of the moisture content of concrete but rather an accurate indication of the radius of curvature of the air / water meniscus within concrete pores in a porous medium.

Relative humidity is influenced by several variables, as indicated by equation [2.02]. The temperature and salt content of the pore water have been shown to influence the RH. Those variables examined by Morrow (1970) are reported in table 2.05 which illustrates the comparative reductions in RH of water due to surface curvature, temperature reduction and equivalent salinity.

Instruments which measure RH are commonly based on either the dew-point principle, a hygroscopic material or wet and dry bulb thermometers. The principle of operation of the Dew-point instrument is
based on the lowering of the temperature of a plane surface until dew forms on the surface, which gives the dew-point temperature of the air. A separate measurement of the ambient temperature is also taken. Since all air holds water vapour, the warmer the air the greater the quantity of moisture that can be held. Using a psychometric chart or standard tables the quantity of vapour in a given volume of air at a given temperature can be calculated. The partial pressure and saturation pressure of the vapour can also be determined from this data since the quantity of vapour in air is another method of stating the quantity of water vapour in air. The dew point instrument physically measures the temperature at which dew forms on a plane surface by cooling the plane surface until dew droplets form (T\text{dew}) and the ambient temperature is measured, therefore the RH can be calculated.

The principle of an instrument based on a hygroscopic material is that a property of the chosen material acts in a predictable manner in the presence of different quantities of moisture vapour. An example of such material is hair (human or otherwise) which changes length and weight with different moisture conditions (Penman, 1955). Monfore (1963) gives details of a more modern version to this type of instrument based on capacitance of a chosen material. However the apparatus still suffers from a change in response time for different relative humidities and a tendency for the moisture content of the sensing element to increase if exposed to high humidities for long periods. These disadvantages necessitate a check on the calibration of the unit at regular intervals during use. A procedure of this type was described by Parrott (1991c).

Wet and dry bulb thermometers are also used to give a measure of relative humidity. Two identical thermometers are used - one with an exposed bulb and the other with a close fitting wick which is kept wet. Unless the air is saturated the moisture on the wet bulb will evaporate and
the energy required to cause evaporation manifests itself in that the wet bulb temperature decreases. Using a psychometric chart these temperatures can be used to give a measure of the relative humidity of the surrounding air (Giedt, 1971).

2.3.2.2 Electrical resistance of concrete

The electrical resistivity of concrete has been used to give an indication of moisture movement and moisture content in concrete. The resistivity of a concrete decreases with increasing moisture content because the resistivity of the pore fluid is much lower than that of the solid CSH, thus the moisture in the concrete is used to measure moisture content. The change in resistivity measurements, however, may be due in greater part to the loss of continuity of the fluid capillary pore water than the volume of capillary water (Parrott, 1990). Parrott (1990) further states that resistivity measurement will not give reliable estimates of moisture content without a detailed calibration for each concrete investigated. Should an accurate estimation of the gradients of moisture in concrete be required, then such a calibration may be feasible. Electrical resistance methods of moisture measurement are generally based upon,

\[ R = R_p \frac{L}{A} \]  \hspace{1cm} \text{Eqn. 2.02}

Where \( R \) = the resistance (Ohms), \( L \) is the length, \( A \) is the cross sectional area and \( R_p \) = the resistivity (Ohm.cm) (Monfore, 1968). The term resistivity is defined as the resistance between opposite faces of a unit cube of the material. Similarly conductance is defined as the reciprocal of resistivity.

Laboratory tests conducted by Bell et al (1963) showed that the electrical properties of concrete could be used to give an indication of the moisture content of the concrete. Other factors which were identified as...
influencing resistivity readings were, the electrical frequency at which the measurements were taken, the ionic content of the pore fluid and the moisture history of the concrete specimen under test (Bell et al, 1963). Parrott, (1990) states that the resistivity of a concrete increases with increasing cement hydration, decreasing w/c ratio and decreasing temperature.

Site moisture estimation tests based on the principle of resistance are not widely used since in-situ conditions such as a high resistivity surface layer, fluctuating temperature, the proximity of steel reinforcement and measurement close to edges of a member may produce inaccurate results (Bungey, 1996). The Wenner four probe apparatus works on the principle that a low frequency AC current is passed between the outer probes and the voltage drop between the inner two electrodes is measured. The apparent resistivity is calculated from,

\[
R_{\text{app}} = \frac{2 \pi s V}{I}
\]

Eqn. 2.03

Where \( R_{\text{app}} \) is the apparent resistivity,

\( s \) = the distance between probes, (normally 50 mm)

\( V \) = the voltage drop and

\( I \) = the current.

Whittington, et al (1981) details a simple model for the conduction of electrical current through concrete. Shown in figure 2.09, the model proposes three alternative flow paths for electrical current through concrete

(i) Through the cement paste.

(ii) Through the aggregate particles in contact with each other.

(iii) Through the aggregate and cement paste in series.

In practice it has been found that a high percentage of the current flows through the paste fraction of concrete. The electrical conductance is
described as having two components, through ionic conductance in capillary pores and also the electronic conductance through the CSH matrix including the bound water. The measurement of resistivity across embedded electrodes in concrete was used by Mc Carter et al (1995) to identify depths of water penetration into concrete. The idea of a resistance ratio was proposed as a way of monitoring change of resistance with time. The resistance ratio \((R_t/R_i)\) is the resistance measurement at any time \(t\) \((R_t)\) divided by the initial steady state resistance measurement \((R_i)\). Figure 2.10 shows the arrival of a water front at a pair of electrodes at a specific depth in concrete with typical resistance ratio graphs for each stage. As can be seen the arrival of the moisture at the level of the electrodes can be distinguished by identifying the steepest part of the resistance graph which has been found to correspond to the time at which moisture arrives at the electrode depth (McCarter et al, 1995).

2.3.3 Moisture movement in concrete

2.3.3.1 Sorption in concrete

Water absorption is a complex process which encompasses the following principles which are examined below:

(i) Surface Energy
(ii) Surface tension
(iii) Capillarity
(iv) Sorptivity of concrete

(i) **Surface Energy**:

Brown (1946) and Adam (1941) state that if it is considered that in a liquid the molecules exert a force of attraction on each other, then a molecule that is near to the surface of the liquid will be pulled inwards,
normal to the surface, towards the bulk of the liquid. To take a molecule
towards the surface from the bulk of the liquid requires extra energy to
overcome this force of attraction. Thus, since all systems tend towards a
state where there is a minimum of potential energy, it can be seen that the
surface of the liquid will tend to contract. Thus 'the spontaneous
contraction of a liquid surface' (Adam, 1941) can be used to explain the
phenomenon known as surface energy.

(ii) Surface tension:

Surface tension is a concept that was developed to model the
complex layer that separates two bulk phases. The original postulation
was that the mechanical properties of a surface layer were related to the
shape of its surface. An analogy of a stretched membrane was advanced to
illustrate the idea of 'tension of surfaces'. This theory has remained
largely unchanged and has become a widely used tool for explaining the
behaviour of interfacial layers.

When the smallest surface area of the liquid has been formed the
molecules at the surface still have more surface energy than those in the
bulk of the liquid and are still attracted inwards. Since there is no force
available to balance these forces, molecules tend to move inwards away
from the surface forming a concentration gradient with depth in the
liquid. When equilibrium is eventually reached the density at the surface
is lower than that in the bulk of the liquid. The volume of a liquid under
a pressure is dependent on its density, therefore, a lower density of the
surface layer compared to the bulk of the liquid can be associated with a
state of tension in the surface layer.

(iii) Capillarity:

When a small diameter tube is dipped into a body of water, the
water will rise in the tube to a level above that of the main body of water
(Figure 2.11). This is not an insignificant effect as discussed below. The
main factors which influence the height of rise of a liquid in a tube are the tube diameter and the physical properties of the liquid.

Shown in figure 2.12 is a representation of the liquid / solid / gas interface and the surface tension forces that are displayed in a capillary tube. The interface between the solid and the liquid is not similar to that of a liquid / gas interface primarily because the solid surface does not deform. Examining the forces on the point where solid, liquid and gas meet (A) shows how surface tension forces can be responsible for the rise of a liquid in a fine tube. The surface tension of the liquid acts tangentially to the surface of the liquid ($\sigma_{lg}$). If it is accepted that the gas/solid interface and the solid/liquid interface both have surface tensions then these can be represented by $\sigma_{sg}$ and $\sigma_{ls}$. Using equilibrium then it can be seen that:

$$\sigma_{gs} - \sigma_{ls} = \sigma_{lg} \cos \phi$$

Eqn. 2.04

Where,

$\sigma_{gs}$ is the surface tension at the solid / gas interface,

$\sigma_{ls}$ is the surface tension at the liquid / solid interface,

$\sigma_{lg}$ is the surface tension at the liquid / gas interface and

$\phi$ is the angle of contact between the liquid surface and the solid.

Two special cases where no equilibrium value of $\phi$ can be found are possible.

(i) The first case is where $\sigma_{gs} > \sigma_{ls} + \sigma_{lg}$

Eqn. 2.05

Here the point A will not attain equilibrium and the liquid gas surface becomes greater at the expense of the liquid solid interface. It can be said in this case that the liquid does not 'wet' the solid surface. The liquid could be expected to minimise its contact with the solid.

(ii) The second case is

when $\sigma_{ls} > \sigma_{gs} + \sigma_{lg}$

Eqn. 2.06

In this case the liquid solid interface becomes greater at the expense of the liquid gas surface. In this case the liquid is said to 'wet' the solid. This is
the mechanism that allows the water in a capillary tube to apparently defy the normal laws of hydrostatics and rise to a level greater than that of the reservoir level. In reality the capillary effect is driven by spreading of the liquid to cover the surface of the solid (which is the vertical sides of the capillary tube). The equilibrium condition shown in figure 2.12 is only reached when the rise of the liquid is stopped by the force of gravity acting on the raised water column.

The above example is useful in that it explains the basic mechanism that drives capillary action, however in reality other factors can be expected to influence the height of capillary rise.

(i) The roughness of the solid surface influences the angle of contact between the solid and the liquid.
(ii) The fact that capillary pores in concrete are not circular or of regular cross section.
(iii) The fact that capillary pores may be lined with adsorbed moisture.
(iv) Theoretical methods of describing water absorption

Washburn's equation

The Washburn equation (Washburn, 1921) has been used as the theoretical basis for describing the process of water absorption into a porous medium by many investigators. The theoretical work done by Washburn in the formulation of his equations is based on some significant assumptions, namely

(i) The capillary is of a constant circular cross section.
(ii) The capillary is horizontal or of small radius.
(iii) The capillary is straight.
(iv) The effect of air resistance can be neglected.

The assumptions which describe the physical properties of the ideal capillary are not truly representative of the capillary pore network in concrete where capillary pores are of variable cross section, are tortuous
are non circular and join and diverge with neighbouring pores in a random manner. The presence of air in concrete with a moisture gradient can prove to be an extremely significant as trapped pressurised air is thought to be responsible for reducing absorption very significantly.

The equation states that the rate of flow in a horizontal capillary due to capillary action is proportional to the radius of the capillary, to the cosine of the angle of contact, to the ratio of the surface tension to the viscosity of the liquid and inversely proportional to the length of capillary already filled by the liquid.

\[
\frac{dl}{dT} = \frac{r \gamma}{\eta 4l} \cos \phi
\]

Eqn.2.07

Where \( dl/dT \) is the rate of flow in a capillary,
\( r \) is the capillary radius,
\( l \) is the length of pore already filled with the liquid,
\( \eta \) is the viscosity of the liquid,
\( \phi \) is the angle of contact and
\( \gamma \) is the surface tension of the liquid.

Poiseuille Equation

The Poiseuille equation for viscous flow through a fine capillary tube is also used by investigators studying the absorption properties of concrete and cement paste.

\[
Q = \frac{\pi r^4 H}{8 \eta l}
\]

Eqn. 2.08

Where \( Q \) is the flow rate \((m^3/s)\),
\( H \) is the head of pressure in \((m)\),
\( r \) is the radius of the capillary \((m)\),
\( l \) is length of the capillary pore filled with water \((m)\) and
\( \eta \) is the viscosity \((Ns/m^2)\).
The head of pressure (H) is composed of 2 main constituent forces

(i) Capillary attraction pressure.
(ii) Applied head.

The capillary attraction forces are held to be the most significant component of the pressure head. Equation [2.09], also known as the Jouren equation, allows the calculation of the rise of a liquid in a capillary pore and can be used to illustrate the predominance of capillary forces over the applied head (Adam, 1941)(Levitt, 1971).

$$h = \frac{2\gamma\cos\phi}{gdr}$$

Eqn. 2.09

where $h$ is liquid rise in the capillary (m),

$r$ is the capillary radius (m),

$\phi$ is the angle of contact (Deg),

$\gamma$ is the surface tension (N/m),

$d$ is the density of the liquid (Kg/m$^3$) and

$g$ is the acceleration due to gravity (m/s$^2$).

Taking a capillary pore of 2μm radius, a water surface tension of 74 N/m, an angle of contact approaching 0 (Penner, 1965), a density of water of 1000 Kg/m$^3$, acceleration due to gravity as 9.81 m/s$^2$ gives a head of water of approximately 7.5 meters. Thus in comparison with pressures of an applied head of water of 200 mm it is possible to assume that the applied head is nominal. An analysis of theoretical work by Penner (1965), below suggests that at higher concrete moisture contents, the capillary force may become so low as to make the applied head of 200 mm significant. This conclusion seems logical and calls into question the validity of sorptivity measurements carried out on substantially saturated concrete.
(v) Sorptivity of concrete:

From the Poiseuille equation for viscous flow in a capillary [2.08], it may be assumed that the head of pressure \( H \) is constant if the pore radius remains constant, that \( I \) is unknown but proportional to \( v \), the viscosity \( \eta \) does not alter significantly with time. Therefore,

\[
\frac{dv}{dt} = \frac{b}{v}
\]

Eqn. 2.10

where \( b \) is a constant,

\( v \) is the volume of water in the capillary and

\( t \) is time

Levitt, (1971) integrated and substituted \( t \) for \( v \), which gives

\[
\frac{dv}{dt} = at^n
\]

Eqn. 2.11

where \( a \) is a constant

This was rewritten by (Gummerson et al. 1980) as,

\[
i = St^{-0.5}
\]

Eqn. 2.12

where \( n \) is a decay constant (taken as 0.5),

\( i \) is the cumulative absorbed volume per unit area.

\( S \) is the sorptivity, and

\( t \) is time.

The decay constant, \( n \) above, has been shown to vary between 0.3 and 0.7 (Levitt, 1971). Levitt (1971) further stated that \( n = 0.3 \) was thought to be common to concrete with a high cement content or with concrete made with a filler material and that \( n = 0.7 \) was associated with a capillary flushing action present mostly in single sized sand mortars. Hall (1981) showed in a series of absorption experiments on brick, that \( n = 0.5 \) as \( t \to 0 \) and that \( n \) increased with time of absorption for surface absorption tests with an infinite source, such as Autoclam and ISAT tests (C.f. section 2.4). He attributed the \( n \neq 0.5 \) values exclusively to the geometry of the test.
arrangement and noted that in one dimensional capillary rise experiments that \( n = 0.5 \) (figure 2.13).

Therefore the early absorption process can be represented as a \( \sqrt{\text{time}} \) relationship with absorption. Different researchers have all incorporated this into their descriptions of the absorption process. However they have used different aspects of the absorption process to relate it to. Depth / distance/ height of penetration, quantity of absorbed water per unit area, weight gain, and average permeability have all been used to obtain the sorptivity of concrete (Emerson, 1990). The most commonly used approach is to relate the quantity of absorbed water to \( \sqrt{\text{mins}} \) as described by Gummerson et al (1980). This is the basis of both the ISAT and Autoclam tests and thus the slope of the absorbed water versus \( \sqrt{\text{time}} \) graph is taken as the sorptivity or sorptivity index.

### 2.3.3.2 Drying of concrete

Drying is a multi stage process generally consisting of unsaturated flow within the porous solid, a liquid vapour phase change, vapour flow within the porous solid and a diffusive transfer of vapour from the surface of the solid to the environment (Hall et al, 1984). The third and fourth sub processes (evaporation) are influenced by heat, wind speed passing the drying surface and so the whole drying process can be influenced significantly by external factors as well as the material properties. Drying under constant external conditions is divided into two stages

(i) A constant drying period - The evaporation rate from a porous body is equal to the evaporation rate of a free water surface under similar conditions. This stage of the drying process is taken to be independent of the properties of the porous material and governed by vapour diffusion.
(ii) A falling drying rate period - The rate of evaporation is thought to be determined or limited by the rate of unsaturated flow of the liquid towards the drying surface. This stage of the drying process is governed by material properties and is in essence a reversal of the absorption process, hence the name desorption which has in the past been loosely applied to any drying process.

The effect of temperature, air flow and ambient RH on stage (i) drying are also examined by Hall et al (1984). The influence of temperature and wind speed on stage (i) drying was expressed as

\[
\ln\left(\frac{\bar{r}_1}{H}\right) = \ln A' - \frac{C}{T} + \frac{1}{2} \ln u.
\]

Eqn. 2.13

with \(A' = A/10^{0.5}\) and \(H = \) a humidity factor; \(H = (1 - h/100)\)

where \(\bar{r}_1/H\) = the average evaporation rate (g/m².h) normalised for the effects of humidity

\(A'\) = mainstream air flow velocity (m/s)

\(l\) = Length of porous medium in direction of air flow (m)

\(C\) = Rate of change in heat of vaporisation per Kelvin (5320 K for water)

\(T\) = temperature (K)

\(u^*\) = laminar air flow velocity (m/s)

\(h\) = relative humidity (%)  

The numerous assumptions and derivations can be found in the appendix of Hall, et al (1984) and Gummerson et al (1980). A graph of this relationship (for a specific specimen geometry) is shown in figure 2.14.

The effect of ambient relative humidity on stage (i) drying based on applying Ficks first law across a linearly changing boundary layer gives,

\[
\bar{r}_1 = \frac{M.D.e}{R.T} \frac{p_e H}{\partial}
\]

Eqn. 2.14

with \(H = (1 - h/100)\)
where \( r_1 \) = the drying rate,
\( M = \) molar mass of water,
\( D_v = \) the binary diffusion of vapour in air,
\( R = \) the universal gas constant,
\( T = \) temperature (K),
\( h = \) ambient relative humidity (%),
\( P_o = \) the saturated vapour pressure,
\( \partial = \) the thickness of the boundary layer at the drying surface.

Hedenblad (1993) states that the drying of concrete occurs under non steady state conditions and is described by Ficks second law of diffusion if little or no hydration occurs during the desorption process.

\[
\frac{\partial w_e}{\partial t} = -\frac{\partial (g)}{\partial x} = \frac{\partial (D_w \cdot \frac{\partial w_e}{\partial x})}{\partial x} \tag{Eqn. 2.15}
\]

where \( t = \) time,
\( w_e = \) evaporable moisture content mass by volume (kg/m³),
\( D_w = \) the moisture diffusivity (m²/s),
\( g = \) the density of flow rate (kg/m²s)
\( x \) is distance from the surface (m) and
\( t = \) time in seconds

Hedenblad (1993) reports work carried out by Pihlajavaara (1965) on the diffusivity of moisture in cement paste. Moisture diffusivity in cement mortar \( (D_w) \) was found to be a function of the time of drying and the drying climate. Figure 2.15 shows that below 60 % moisture content \( (u = 0.6) \) the diffusivity remained reasonably uniform whereas above 60 % moisture content the diffusivity increased with both time of drying and moisture content. If the evidence presented by Seligmann (1968) is considered, this lack of diffusivity below 60 % moisture content may be
due to water adsorbing to the sides of the pores rather than travelling through the pores.

Parrott (1991b) proposed a set of empirical equations in which the time taken for the internal relative humidity in concrete to reach half of its potential drop was predicted from ambient conditions in a laboratory. (Taken as 62% RH).

\[ t_{1/2} = 10j \text{d} \quad \text{For } t_{1/2} < 414 \text{ days drying} \quad \text{Eqn. 2.16} \]

\[ t_{1/2} = 3j \text{d} + 290 \quad \text{For } t_{1/2} \geq 414 \text{ days drying} \quad \text{Eqn. 2.17} \]

Where \( t_{1/2} \) is the time to achieve half the potential drop in relative humidity (79%),

\( j \) is a cement type factor (\( j=1 \) for OPC) and

\( \text{d} \) is the depth from the drying surface.

The plot of the data upon which [2.16] and [2.17] are based is shown in figure 2.16. As can be seen the relative humidity was found to drop more quickly the closer to the drying surface of the concrete which illustrates the importance of the drying path length. More surprisingly, the w/c ratio was not found to influence significantly the relative humidity change and Parrott surmised that the increase in the number and size of pores associated with the higher w/c concretes was offset by an increase in the quantity of moisture in the pores. No explanation for the bilinear nature of the time taken to achieve half the potential drop in relative humidity was forwarded, however it may be collaborative experimental evidence of the two stage drying process described by Hall, et al (1984).

2.3.3.3 Sorption / Desorption hysteresis

The difference in the absorption and desorption branches of isotherms can be explained by what is known as the 'ink-bottle' effect. In a
pore with a small entrance leading to a pore which is wider than the entrance, (shown to exist in very significant quantities in some cement pastes by Mikhail et al (1964)). the narrow entrance will fill at a relatively low vapour pressure and the main body of the pore will fill at a higher vapour pressure. During the drying process this pore will only begin to empty when the vapour pressure is reduced to the point where the meniscus in the neck of the pore becomes unstable (Brunauer et al (1967)). An example of a hysteresis loop in concrete is presented in figure 2.17. As shown, if the material is only partially dried out before rewetting occurs, or if drying commences before saturation is complete, then a curve such as that shown in figure 2.17 is followed (Nilsson, 1980).

2.3.3.4 Non-capillaric moisture movement in capillary pores

(i) Darcy's law

Darcy’s law has been used by most investigators to describe the steady state flow of a fluid through a porous media and is given as

\[ Q = K A \frac{dh}{dx} \]

Eqn. 2.18

where \( Q \) is the steady state volumetric flow rate (m\(^3\)/s),

\( K \) is the coefficient of permeability (m/s),

\( A \) is the sectional area of the porous sample (m\(^2\)) and

\( \frac{dh}{dx} \) is the hydraulic gradient across the sample in the direction of the flow.

The law is empirical and has well catalogued limitations and conditions (Scheidegger, 1963) among which is the requirement for the flow to be steady state and laminar, the fluid to be incompressible and that no other gradients should be responsible for the flow apart from the hydraulic gradient. Permeability (K) is defined as a measure of the resistance of the porous solid to flow. Nyame (1979) states that two main
deviations from the law can be expected to occur in cementicious materials.

(i) Molecular effects caused by high attraction forces between the fluid and the surfaces of the solid in small flow channels (Slip flow and molecular streaming).

(ii) Osmotic effects due to differences in calcium hydroxide concentrations in hardened cement paste for measurements made at low hydraulic gradients (<1000).

The concrete society report 31 (1988) states Darcy's law and gives a generalised version of the formula which applies to any fluid flowing in any direction through a porous medium, for viscous flow conditions.

\[ v = \frac{Q}{A} = -\frac{k}{\mu} \frac{\partial p}{\partial l} \]  
Eqn.2.19

Where \( v \) is the apparent velocity of the flow, 
\( Q \) is the flow rate, 
\( \mu \) is the viscosity, 
\( k \) is the intrinsic permeability and 
\( \partial p \) is pressure loss over the flow path of length \( \partial l \).

The intrinsic permeability (\( k \)), with units of area, is dependent on the material properties and independent of the properties of the fluid. The fluid characteristics can be identified by using, 

\[ \partial p = \rho \cdot g \cdot \partial h \]  
Eqn. 2.20

Where \( \partial h \) is the head loss over the flow length, 
\( \rho \) is the density of the fluid and 
\( g \) is acceleration due to gravity.

Comparing equations [2.18] and [2.20] a relationship between the permeability coefficient and the intrinsic permeability can be derived as,

\[ k = \frac{K \mu}{\rho g} \]  
Eqn 2.21
where $K$ is the coefficient of permeability and $k$ is the intrinsic permeability.

Using equation [2.21] it is possible to calculate the relationship between intrinsic permeability and the coefficient of permeability for different fluids at different temperatures. It is also theoretically possible to measure the coefficient of permeability with one fluid, calculate the intrinsic permeability and then calculate the coefficient of permeability for a different fluid. The equations [2.18] and [2.19] have been used as the basis of laboratory permeability tests, however, in-situ test methods commonly do not allow the determination of many of the variables associated with the equations.

(ii) Hvorslev's Equations

Figg (1989) used a non-steady state permeability equation, initially developed by geotechnical investigators (Hvorslev, 1951) to measure strata permeabilities in a homogeneous permeable rock, to relate Figg permeability test results (C.f. section 2.4) to intrinsic permeability. The geotechnical borehole arrangement which was used in the original geotechnical work is shown in figure 2.18. While the scale of the geotechnical test is considerably different to that of a Figg test, the similarities in the nature of the tests is undoubted. (Hvorslev, 1951) states that

$$ k = \frac{d^2 \ln \left( \frac{mL}{D} + \frac{\sqrt{1 + (mL/D)^2}}{2\pi} \right)}{8LT} $$ \hspace{1cm} \text{Eqn. 2.22} \tag{Variable Head}

$$ k = \frac{q \ln \left( \frac{mL}{D} + \frac{\sqrt{1 + (mL/D)^2}}{2\pi} \right)}{2\pi LH} $$ \hspace{1cm} \text{Eqn. 2.23} \tag{Constant Head}

where $m$ is a factor allowing for differences in horizontal and vertical permeability.
T is the basic time lag (secs) corresponding to \( H = 0.37 \) (In \( Ho/H = 1 \)),

\( q \) is the flow rate (ml/s),

L is the length of effective intake (cm)

d is the standpipe diameter (cm),

D is the intake diameter (cm),

H is the differential head (cm) and

\( Ho \) is the differential head at time 0 (cm).

The original equations (Hvorslev, 1951) are based on two different well situations - the variable head equation [2.22] is based on a test hole at the interface between an impermeable and permeable strata (shown in figure 2.18) while the constant head equation [2.23] is based on a bore hole in a uniform soil. The reason for the choice of equations is unclear as the test hole is similar for both Figg water and air tests. Furthermore the permeability coefficients obtained from these equations describe horizontal permeability of the soil, not the mean permeability (both horizontal and vertical) as is measured by the Figg tests. Hvorslev's equations are a mixture of theory and experimental observations and to use such equations, based on observations made in a different material and at a substantially larger scale without further investigation, appears inappropriate.

Figg (1989) assumed \( m \) to be unity, applied correction factors for the viscosity of air and water (assumed to be \( K_{air} = 6.5 \times 10^5 \) and \( K_{water} = 9.75 \times 10^6 \)) and entered the dimensions of his concrete test hole into the equations to give values of \( k(air) \) and \( k(water) \). This gives equations for intrinsic permeability based on both the Figg air and water measurements.

\[
\text{Air (} k \text{) } = \frac{5.14 \times 10^{-11} \text{ (m}^2\text{)}}{t_a} \quad \text{Eqn. 2.24}
\]

\[
\text{Water (} k \text{) } = \frac{1.18 \times 10^{-11} \text{ (m}^2\text{)}}{t_w} \quad \text{Eqn. 2.25}
\]
where $t_a$ is the time (secs) for the pressure change from 55 kPa to 50 Kpa below atmospheric pressure

$t_w$ is the time (secs) for 0.01 ml water absorption.

Figg (1989) claimed 'a reasonable correlation' between the intrinsic permeability values and the Figg permeability values.

(iii) **Stages of moisture transfer in concrete**

The moisture in concrete will travel in concrete from regions of greater moisture to lesser moisture. When capillary action is not present, moisture can move in the form of either vapour and/or fluid through the pores depending on the saturation of the pore. Figure 2.19 shows the stages in the transfer of moisture through an increasingly saturated capillary pore as proposed by Rose, (1965). Stage 1 represents the adsorbed layers of water attaching themselves to the sides of the capillary pores. Until at least a monolayer of water has adsorbed onto the pore walls no transfer of moisture through the pore will take place. Stage 2 is the transfer of moisture in vapour form through the pore (The vapour will behave as an ideal gas). Stage 3 shows the formation of menisci at the necks of the pore. The liquid in the menisci will act as a short cut for vapour transfer and the vapour path is reduced through the pore. A distillation process will occur at the menisci as vapour is turned into fluid on the up stream side of a meniscus and the fluid phase changes to vapour on the downstream side of the meniscus. Stage 4 exhibits not only liquid assisted transfer of moisture as described for stage 3 but also surface creep. Rose (1965) describes the process of creep as being the flow of water in a thin film and states that it will increase until liquid flow dominates. In stage 5 the liquid flow through the pore means that contribution of vapour diffusion to the transfer of moisture through the pore becomes negligible. The air void in the centre of the pore could be expected to be completely spherical as opposed to the air in stage 3 which will take its
form from the shape of the pore. Stage 6 shows saturation of the pore where all moisture transfer occurs by fluid flow.

The vapour diffusion as can be seen, only predominates where there is no direct, infinite (in terms of pore volume), supply of liquid water (as in capillary action) or where the overall pore system is relatively unsaturated (stages 1-4, figure 2.19). The liquid flow is generally described by Darcy's law [Eqn. 2.18] The process of vapour flow is described by Ficks first law of diffusion, (Concrete society, 1988)(Kropp, 1995) which is

$$q_v = D_{df}(\varepsilon)_v . \text{Grad} \rho$$  

Eqn. 2.26

where $q_v$ is the vector mass flux of vapour, $D_{df}(\varepsilon)_v$ is the molecular diffusion coefficient of water vapour in a porous material (vector direction $v$) and $\text{Grad} \rho$ is the gradient of water vapour density. Temperature is taken to be constant throughout.

2.3.3.5 The influence of ambient environmental conditions on the moisture state of an exposed concrete

The ambient conditions adjacent to the exposed face of concrete influence the moisture climate within the concrete. The moisture in the capillary pores of the concrete always strives to achieve equilibrium with the surrounding environment. Therefore, since the environmental conditions are constantly changing, it follows that the moisture conditions in concrete pores exposed to the environment are in a state of constant change. Parameters, such as ambient relative humidity and temperature influence the moisture conditions within concrete capillary pores. These parameters have been examined in relation to the processes of absorption and drying.
2.3.3.6 Gradients of moisture in exposed concrete

Moisture in concrete that is exposed to the environment is in a state of constant flux as the concrete attempts to achieve moisture equilibrium with its surroundings. The region of the concrete closest to the surface is able to achieve moisture equilibrium relatively quickly as it is in direct contact with its surroundings, whereas a region of the concrete at depth will take much longer to achieve equilibrium.

Non-linear moisture profiles in drying concrete were identified by Terrill et al (1986) in laboratory tests which simulated drying which could occur in a waterproofed bridge deck. The moisture profiles (figure 2.20) showed that during the drying process moisture was lost from the drying surface at an earlier age and faster rate than that farther away from the drying surface. Pihlajavaara (1976) reports a study which includes the monitoring of drying in columns over time. The findings are similar to those of Terrill et al (1986).

A similar but more specialised study of gradients in near surface concrete caused by drying was reported by Patel et al (1985). This study showed that concrete under drying for 163 days displayed gradients of porosity varying with distance from the drying surface. The regions nearest to the surface were less hydrated and more porous than those further away from the drying surface. Rankin (1993) identified in-situ moisture profiles in a young 100 mm thick concrete floor slab.

Bakker (1994) shows mathematically how the wetting and drying processes will influence the moisture profiles in a concrete which is regularly wetted and dried. Figure 2.21 shows a definite depth of influence of moisture fluctuation (shown as carbonation) which corresponds to the average length of the drying and wetting phases used in the calculation. Bakker proposes that for frequent wetting and drying that a maximum depth of drying is possible. Figure 2.22 shows a schematic of the limits of
moisture movement in concrete under one day wetting and 6 days drying climate.

2.3.4 Methods of removing moisture from concrete

Several methods of removing moisture from the capillary pores of the concrete were examined with a view to removing the moisture from the Autoclam test volume on-site and hence removing the influence of moisture from Autoclam measurements.

2.3.4.1 Vacuum application

The application of vacuum as a method of removing moisture has been used to precondition concrete prior to ISAT tests by Dhir et al (1993). The technique which was proposed consisted of the application of a vacuum through the ISAT test apparatus which had a quantity of silica indicator gel inside (Figure 2.23). The silica gel, which had been allowed to absorb moisture, was used as a method of indicating when moisture vapour had stopped being removed from the concrete i.e. when the gel indicated no moisture present. The time for the silica gel to change colour ranged from 1.5 to 6 hours of application of vacuum for various grades of concrete and different moisture histories. ISAT$_{10}$ values were compared for specimens of different initial moisture conditions using different periods of preconditioning. The findings indicated that the vacuum preconditioning method was not as effective in removing moisture as drying at 105° C and was slightly more effective at removing moisture than 2 days laboratory exposure. Vacuum application for twice the period deemed necessary to remove the moisture, gave results which were only slightly greater than those of standard applied vacuum technique. The depth of influence of the vacuum application was reported to be 20 mm
although no details of how this was ascertained were forwarded (Dhir, 1994).

The conclusions drawn from the test results were limited in that they did not claim that the capillary pore system was free from moisture and the only real benefit claimed was that the repeatability of the $\text{ISAT}_{10}$ test was improved compared to the air exposure for 2 days. The change in colour of the indicator gel, which was used as a measure of the moisture vapour exiting the concrete, does not necessarily mean that no moisture is being removed from the concrete but suggests that the initial moisture content of the gel itself was removed by the vacuum. It is difficult to envisage how the 'O' ring type seal used by the ISAT apparatus could promote the development of a bowl of influence in the concrete of a significant depth. The level of drying of the concrete was not reported and therefore the quantity of moisture removed and the depth of influence of the preconditioning test are unknown (Dhir, 1994).

2.3.4.2 Temperature increase

The increase in temperature of the concrete as a method of removing the influence of moisture in concrete on permeation measurements was reported by Basheer (1991). A camping gas heater was used to heat the surface of concrete prior to 'Autoclam' air and sorptivity tests. Heating for 20 minutes and allowing the concrete to cool for 1 hour in an ambient laboratory temperature ($20 \, ^\circ\text{C}$) was found to give comparable results to that of oven dried ($105 \, ^\circ\text{C}$) concrete for concretes conditioned at different relative humidities.
2.4 Measurement of the permeation properties of concrete

2.4.1 Introduction and definitions

As shown in section 2.1.2 the permeation properties of the near surface concrete are related to the transport mechanisms, which carry aggressive substances from the environment through the cover of concrete to attack the reinforcing steel. Therefore the permeation properties can be used as an indicator of the durability of concrete.

The test methods discussed below are all in-situ tests and laboratory based tests are not considered. The definition of an in-situ test adopted for this study is rigorous and does not include tests which can be carried out on specimens taken from in-situ concrete but tested in the laboratory. Cabrera and Lynsdale (1988) is an example of such a test. Since the thesis is primarily concerned with the air permeability and sorptivity of concrete, the examination of diffusion, and diffusion tests have been omitted, with the notable exceptions of the role of diffusion in the movement of moisture in capillary pores.

Given below are definitions of commonly used terms

(i) **Permeability** : The coefficient of permeability is a material characteristic describing the permeation of gasses or liquids through a porous material due to a pressure head (Kropp, 1995).

(ii) **Absorption** : is the process whereby the concrete takes in a fluid to fill spaces within the material (Concrete Society, 1988).

(iii) **Sorptivity** : is a quantitative measure of the speed of absorption of a liquid by a porous solid (Dolch, 1995).

(iv) **Adsorption** : is a process in which molecules adhere to the surface of the concrete (Concrete Society, 1988).

(v) **Diffusion** : The transfer of mass by random motion of free molecules or ions in the pore solution resulting in the net flow from
regions of higher concentration to regions of lower concentration of the diffusing substance (Kropp, 1995).

2.4.2 In-situ air permeability test methods

Several methods are currently available for measuring the air permeability of concrete on-site. Some of the main methods are listed below along with a statement of the principle of operation and some of the general features of each system. Also mentioned are the main perceived advantages and disadvantages of each method.

2.4.2.1 Figg Air Permeability Test

The original Figg air permeability apparatus (Figg, 1973) is shown in figure 2.24(a). The test is carried out in a drilled hole of diameter 5.5 mm and of 30 mm depth. The hole is cleared of dust and a foam disk is inserted at 20 mm from the surface. The hole is then filled with silicone material which is allowed to set. A hypodermic needle is inserted in to the cavity and is connected to a manometer and a vacuum generator. The vacuum in the system is reduced to 15 kN/m², and then the three way stopcock is switched to isolate the manometer and the cavity. The time taken for the pressure in the cavity to increase to 20 kN/m² is recorded. This time period is taken as a measure of the air permeability of the concrete.

Modified versions of the Figg air test have been developed by various researchers, most notably Cather, et al (1984). While all these methods use the same principle as the original Figg air permeability test, they differ in some important respects. The total volume of the test apparatus (this includes the volume of the tubing to the manometer and from the stopcock to the hole) is different for each method as full details of the original Figg apparatus were not widely disseminated. The
dimensions of the hole drilled in the concrete have also been altered by some of the investigators (Table 2.06).

The main advantages of this test are that it is well established and that the cost of carrying out a test is low (Basheer, 1993b). The fact that the air permeability is measured between 20 and 40 mm depth (Cather et al apparatus) means that the test will not be influenced by surface effects such as carbonation of the concrete surface. The test is not totally non-destructive as a small hole is left in the concrete, also the damage to the microstructure of the concrete caused by drilling may increase the air permeability of concrete. Although this method has been used extensively the results are rarely comparable, as discussed above. Variable moisture conditions in the concrete influence Figg air permeability results, as illustrated by an investigation by Dhir, Hewlett, Chan (1987) and discussed in a paper by Figg (1973). The test method described by Cather, Figg et al (1984) has become the most widely accepted of the range of Figg methods and a considerable body of experimental experience of the test has been accumulated (Bungey, 1996).

2.4.2.2 Autoclam air permeability test

The apparatus used consists of a control unit which is attached to the measurement apparatus. The measurement head is firstly secured to the base ring which is fixed to the surface of the concrete (Figure 2.25a). A pressure slightly in excess of 500 mBar is introduced to the apparatus by means of a syringe. The priming valve is then closed and the test is commenced by pressing a button on the control unit. The air pressure in the ring is monitored continuously and the first reading is taken when the pressure drops to 500 mB pressure. Readings of pressure are taken every minute thereafter for 15 minutes or until the pressure in the apparatus has dissipated to atmospheric pressure. Air is forced to flow through the
near surface concrete under the clam ring and so the rate at which the air pressure dissipates describes the permeability of the near surface concrete to air.

The Autoclam is commercially available and is easily used on-site. The results are quoted in terms of a permeability index and so are more easily compared to direct measures of permeability. The removal of bonded rings can cause surface damage to the concrete although this problem has recently been overcome with the development of a base ring which is secured using a vacuum. The controller unit allows the storage of data from 20 tests which can be downloaded into a computer for analysis. The influence of moisture on the Autoclam measurements is the subject of this thesis.

2.4.2.3 Torrent two chamber vacuum test

The Torrent two chamber apparatus (Torrent, 1992) is not widely used, however it has a distinctive feature - a 'guard ring', to ensure air flow from the sides of the ring are not measured, as shown in figure 2.26. The pressure in both the inner and outer chamber is lowered to the desired pressure by means of the vacuum generator. The vacuum is then isolated from the inner measurement chamber and the test begins. As the vacuum in the inner chamber decreases with time, the pressure in the outer chamber is constantly monitored and adjusted to be the same as the pressure of the inner chamber.

The main perceived advantage of this method is that it proports to remove the lateral ingress of air through the uppermost regions of the concrete which are sometimes held to produce large air permeability readings. The uniaxial nature of the permeability seems uncertain as for specimens of considerable thickness (in-situ concrete) it is unclear as to the origin of the air which degrades the vacuum in the instrument. Full
details of the method were not detailed in the original paper and it is also unclear as to whether the method is easily used on-site. The technique has not been used extensively. No published work on methods to remove the influence of moisture in the concrete on test readings have been widely disseminated. The apparatus has recently become commercially available.

2.4.2.4 Hansen et al air permeability test

As shown in figure 2.27 in the Hansen et al apparatus an 18 mm diameter hole is drilled at a shallow angle to the concrete surface and a pressure sensor is inserted into the drilled hole (Concrete Society, 1988). The pressure chamber is then pressurised by using pressurised gas and regulated by means of a valve.

The method is said to be suitable for use on-site, however it has not been extensively used. The test leaves an 18 mm diameter hole in the concrete as well as the fixing bolts necessary to secure the drilling jig. Drilling the concrete which is being tested may also increase the air permeability of the concrete. The main advantage of this test is that it gives an indication of the pressure gradient in the concrete by monitoring the pressure at depth as well as at the surface of the concrete. While the test does not conform to Darcys law, the measurement is based on the pressure gradient in the concrete. This should allow a more direct relationship to the permeability coefficient defined by Darcy since the Darcy equation has a similar pressure gradient term (Section 2.4.3.5).
2.4.3 In-situ water permeability test methods.

2.4.3.1 Autoclam water permeability test

The Autoclam water permeability test (similar to the sorptivity test set-up shown in figure 2.25b) is carried out by resetting the piston to the top of its travel and the apparatus is filled with water using a syringe. The pressure in the enclosed chamber is raised to just below 0.5 Bar (typically 495 to 499 mBar) and the test is started. The piston is advanced to maintain the pressure in the enclosed chamber at 500 mBar as water enters the concrete. The test is continued for 15 minutes or until the piston reaches the end of its travel. The travel of the piston is recorded at one minute intervals is then used to calculate the quantity of water entering the concrete. The slope of the cumulative water flow versus the square root of the time taken is plotted and the slope of this graph is taken as the Autoclam water permeability index.

The Autoclam water permeability test does not measure a steady state water permeability of concrete, rather it measures a water permeability index. As the cumulative flow of water is plotted against the square root of time this suggests that the test is in reality a pressurised sorptivity test. To obtain a more correct water permeability index the cumulative water flow should be plotted against time, and a steady state flow should be achieved (Basheer, 1991). The moisture condition of insitu concrete is known to influence the measurements and it is recommended that the concrete is fully saturated before testing. This test is not widely used.
2.4.3.2 Figg Water Permeability Test

The Figg water permeability test is carried out with an over pressure of only 100 mm head of water and so has been classified in this review as an absorption test and is discussed in section 2.3.4.

2.4.4 In-situ sorptivity test methods

2.4.4.1 Initial Surface Absorption Test (ISAT)

A cap with an area of at least 5000 mm$^2$ is sealed to the concrete surface and filled with water (figure 2.28) (BS 1881: Part 208, 1996). The amount of water which is absorbed by the concrete is monitored by means of a capillary tube at a height of 200 mm above the surface of the concrete. The duration of each test is normally either 10, 30 or 60 minutes and can be carried out in a laboratory or on-site. The results are quoted in terms of mL/m$^2$/s.

The apparatus used for this test can be simply manufactured and is inexpensive. This test has been used extensively by investigators in the past and is perhaps the most widely used method of measuring concrete sorptivity. Details of a modified apparatus to induce uniaxial sorption have been published (Price, Bamforth, 1993). In practice the test can be difficult to use on-site (Taylor, 1989) as there can be problems in achieving a good quality seal between the concrete and the test apparatus. Moisture has been found to influence the results of the apparatus.

2.4.4.2 Figg water permeability test

The Figg water permeability test is carried out with an over pressure of 100 mm head of water (which is a nominal pressure, when compared to the large forces associated with capillary suction) and so it may be classified as an absorption test. Indeed, some investigators (Dhir et
al 1987) refer to the test as the Figg water absorption test. The apparatus, shown in figure 2.24b is set up and the syringe is filled with distilled water. The three way stopcock is opened and water is introduced to the system displacing the air. One minute after first introducing the water the meniscus is marked on the capillary tube, the stopcock is closed, and the test is deemed to have begun. The time taken (in seconds) for the meniscus to travel 50 mm on the capillary tube is taken as a measure of the absorption by the concrete.

The Figg water test, as it is commonly known, has two main advantages. The apparatus is cheap to manufacture and it measures an average absorption value for the concrete between depths of 20 and 40 mm (modified apparatus). The fact that the average absorption over a depth of concrete is obtained can also be a disadvantage depending on the reason the test is being carried out (e.g. testing surface treatments). The test is not as widely used as the ISAT test and so little experience of its performance is available. The units of absorption given in this test are difficult to relate to quantities of water flow in a given time. Drilling the test hole will damage the structure of the concrete, and so will increase the measured absorption by the concrete. Dhir et al (1987) found that the use of a syringe to fill the test hole with water, gave an inconsistent pressure and introduced entrapped air which led to spurious results from some tests. That moisture in concrete has an effect on test results has been acknowledged (Figg, 1973) and so in-situ measurements must be used with caution.

2.4.4.3 Autoclam sorptivity test

The Autoclam unit is set up as shown in figure 2.24(b). The bleed valve at the top of the hydraulic cylinder is opened and water is introduced into the system through the lower priming valve. When the
system is filled with water both the bleed valve and the priming valve are closed and the test is started when the pressure is between 16 and 19 mBar. The pressure in the chamber is maintained at 20 mBar for the duration of the test by advancing the piston to compensate for the drop in pressure due to flow of water into the concrete. The travel of the piston is recorded at intervals of 1 minute for 15 minutes. From the results, the flow of water into the concrete is calculated. The cumulative water absorbed is plotted against the square root of time which results in a linear plot and the slope of the line is reported as the Sorptivity Index.

The advantages of this system is that it is accurate, easy to use on site and that it has been used fairly extensively in laboratory and in-situ work. A modified version of the base ring has been developed to give a uni-axial flow (Nolan, 1994b).

2.4.4.4 **Covercrete absorption test (CAT)**

Dhir, et al (1987) combined basic features of both the ISAT and the Figg water permeability test to produce the covercrete absorption test. A 13 mm diameter hole is drilled into the concrete to a depth of 50 mm and the apparatus, shown in figure 2.29, is set up. The apparatus basically consists of a two vertical tubes one leading to a reservoir and the other to a graduated capillary tube. A gasket seal at the surface of the concrete is fixed and de-ionised water is introduced through the water reservoir. When the system is filled with water and the water level in the reservoir is maintained at 200 mm above the surface of the concrete. The tap is used to shut off the reservoir supply 10 minutes after water is first introduced into the test hole and the travel of the meniscus over the following minute is measured. The advantages of this test are that it is reportedly easier to use on site than the ISAT and it is cheap to manufacture. The process which was adopted to fill the test hole reduces the risk of...
entrapped air compared to the Figg test (due to the water being introduced into the bottom of the hole and the outlet for air being at the top). The disadvantages are similar to those already outlined for the Figg water test and the fact that absorption flow is measured over just a one minute period is also a drawback. Blight et al (1995) reported the use of the CAT apparatus on site in less than favourable terms. The evacuation of air from the test hole prior to testing on a vertical surface required a modification of the apparatus and an application of pressure in excess of the test pressure to flush of air out of the system. The laboratory calibration tests confirmed the original verification work by Dhir et al (1987), however on-site test results from a range of existing structures showed little correlation with measured depths of carbonation, oxygen permeabilities and water sorptivity.

2.4.5 Discussion of test methods

The test methods outlined above have all been used to measure the permeation properties of concrete, and they all have advantages and disadvantages which are discussed in this section.

2.4.5.1 Air permeability measurement

The measurement of an air permeability characteristic of the cover concrete is carried out either in a hole drilled in the concrete (e.g. Figg test) or at the surface of the concrete (e.g. Autoclam). It is generally accepted that drilling a hole in concrete causes microcracking in the surrounding concrete which in turn promotes increased rates of air permeability. The amount of microcracking can be expected to vary with speed of drill, sharpness of drill bit and operator, therefore the reliability of site air permeability tests must be called into question. The hole based method of air permeability measurement does have an advantage in that it avoids...
the surface zone of the concrete and can justifiably claim to measure the permeability at over a given depth. Surface based techniques such as the Autoclam and the Torrent 2 chamber test do not disturb the concrete with no suspicion of increased permeability. These tests do however test the most permeable part of the concrete and therefore may not be representative of the full cover concrete depth. The depth of influence of the Autoclam apparatus has been determined to be 50 mm by finite element techniques (Arbaoui, 1988) and experimentally by Basheer (1991) as shown in figure 2.30. The Hansen et al apparatus combines the drilled hole and surface test and so combines the worst features of both types of air test.

2.4.5.2 Water permeability measurement

The lack of in-situ water permeability test methods is testament to the difficulties in measuring the water permeability characteristic of concrete in-situ. The Figg water test uses a similar head of water to absorption tests therefore it has been commonly used as a water absorption test. The Autoclam water permeability test uses a 0.5 Bar pressure. However it too has major drawbacks. As discussed in section 2.3.3.1 the water permeability characteristic, as presently obtained, is in reality a pressurised absorption characteristic. The need for saturation of the concrete prior to test is also difficult to obtain on in-situ vertical surfaces. Despite these drawbacks the test is not without its merits as illustrated in a field investigation (Polder et al, 1994) where saturated coastal concrete was found to have no sorptivity and the water permeability characteristic was measured successfully instead.
2.4.5.3 Sorptivity measurement

In-situ sorptivity test methods use either a drilled hole (Figg and CAT.) or are carried out on the concrete surface (ISAT, Autoclam). The damage done to the concrete by drilling a hole could influence measurements. The rationale of using a drilled hole for absorption measurements is not so clear as it is in the case of an air permeability test since the free water surface required for absorption invariably originates at the surface of the concrete. To measure the sorptivity of lower depths in concrete it would seem more correct to perform a surface sorptivity test for an extended period. The surface test method has been criticised by Hall (1980) who pointed out that the absorption rate obtained from a circular finite source (ISAT and Autoclam) is not independent of the size of the source. This criticism is of limited practical importance as both the Autoclarm and the ISAT have standard source dimensions. Both the Autoclam (Nolan, 1996b) and ISAT (Price et al, 1993) have a modified apparatus to obtain uni-axial absorption by providing a 'guard ring' arrangement.

The only major practical differences between the ISAT and Autoclam sorptivity test are the ease of use on-site as the ISAT apparatus is difficult to seal and the fact that the Autoclam measures the quantity of water absorbed every minute whereas the ISAT is commonly measured at 10, 30 and one hour after the start of the test. The automated nature of the Autoclam controller unit allows specialised tests of longer duration to be performed when necessary. Hour long sorptivity tests have been used to assess the sorptivity of treated and coated concrete (Basheer, 1994) and 24 hour sorptivity tests have been used to study the ingress of moisture into concrete (Nolan, 1994b).
2.4.5.4 Conclusion of discussion

It was decided that a main priority should be the use of surface tests since these gave the most accurate 'undisturbed' measurement of the permeation properties of the near surface concrete. Other requirements included ease of use on-site and a reasonably established test method. The Autoclaim permeation system fulfilled all of these requirements and therefore the Autoclaim air permeability and sorptivity tests were used in this series of investigations.

2.5. The influence of environmental conditions on the permeation properties of concrete

2.5.1 The influence of temperature on permeation properties

The ambient temperature of the concrete and the fluid at the time of test influence the permeation measurements. The following section identifies the work done in this field to date.

2.5.1.1 The influence of ambient temperature on air permeability

The ambient temperature of the concrete and the air used in the test on the air permeability was investigated by Bungey et al (1989). They concluded that since the viscosity of air does not change appreciably with increasing temperature then the air permeability of concrete does not change significantly. The hypothesis was reinforced by Figg air permeability test results carried out on concrete of different temperatures (Figure 2.31). The moisture content of the concrete did not seem to influence significantly the very small rate at which the air permeability increased with increasing temperature.
2.5.1.2 The influence of ambient temperature on absorption

The ambient temperature of the concrete and the test fluid on the absorption of water by concrete was investigated by Bungey et al (1989). Figure 2.32 shows the variation of ISAT results with temperature and, as can be seen, the quantity of moisture in the concrete at the time of test is an important variable. Temperature has a more pronounced effect on concrete with little moisture while the effect of temperature on saturated concrete is minimal. Bungey et al (1989) state that the change in the properties of water with changing temperature are responsible for the increase in absorption observed between 20 and 50 °C. The surface tension and viscosity of the absorbent fluid could be expected to change with temperature. This is similar to the correction factor proposed by Hall (1980) to allow for the normalisation of absorption rates at different temperatures

\[(\sigma \eta)^{0.5}\]  

Eqn. 2.27

where \(\sigma\) is the surface tension of water and \(\eta\) is the viscosity of water.

Bungey et al (1989) plots a correction factor based on the Poiseuille equation (Figure 2.33) with experimental ISAT results in the temperature range of 20 to 50° C. This can be seen to predict quite well the changes in ISAT measurements with temperature. Bungey also notes from this graph that the moisture content of concrete influences the change in ISAT results.

2.5.2 The influence of moisture on the permeation properties of concrete

Moisture in the capillary pores of the concrete influences the permeation measurements. This section identifies published work in this area.
2.5.2.1 The influence of moisture on air permeability

A concrete with little moisture in the capillary pore system will have more numerous, larger and more continuous air flow paths than a similar concrete with a substantially saturated capillary pore system. The fact that moisture in a porous solid such as concrete influences the gas permeability of the solid is well known, (Bungey et al (1989), Parrott (1990), Basheer (1991), Leeming, (1993), Kropp (1995)), however little work on quantifying this influence has been published.

Nagataki et al (1986), using a novel laboratory based apparatus for measuring air permeability, examined the influence of moisture content on air permeability. The air permeability of concrete was found to increase with the quantity of moisture evaporated from the surface of the concrete. It was also shown that an increase in the measured internal relative humidity of the concrete corresponded to a decrease in the air permeability coefficient.

Parrott (1991c) includes a graph of data taken from literature shown in (figure 2.34). This graph shows a 'plateau' in adjusted permeability indices between 40 % and 65 % R.H. where the air permeability of the concrete varies relatively little with a change in the relative humidity of the concrete. The explanation for this effect is that for values below 65 - 70 % RH. the concrete properties were measured, whereas above 65 - 70 % RH. moisture in the capillary pores influenced the air permeability. Parrott (1991c) also includes a study in which the relative humidity of concrete was observed over a period of 350 days and the permeability of the concrete was monitored simultaneously (figure 2.35). The investigation shows a clear but unquantified relationship between internal RH. and air permeability. For each increase in RH. the air permeability of the concrete was shown to decrease and as the concrete dried out the internal RH. decreased and the air permeability increased.
Parrott (1994b) presents another version of the RH. versus air permeability graph in which the relationship between RH. and air permeability is different for different concretes (figure 2.36). This graph differs from figure 2.34 in that the different relationships between RH and air permeability for concretes of different pore structure are recognised. The air permeability of concrete with larger w/c ratios (consequently larger more continuous pores) can be seen to vary significantly between RH range of approximately 85% to 100%. This reflects the dependence of air permeability on the larger pores in a concrete, as at 85% RH smaller pores are still saturated. The air permeability of concrete with a lower w/c ratio is shown to be influenced by pores of a smaller size since the effect of moisture on air permeability is distinguishable above 60% RH. The effect of w/c ratio on the air permeability in concrete in the RH range of 20 to 0% is more confused. Parrott (1994b) makes no reference to this section of the figure 2.36 and the data shows no consistent trend. RH values of 0 - 20% represent a concrete which is loosing moisture from the CSH structure and therefore microcracking could be expected to be prevalent. The breakdown of the concrete structure may explain the confused pattern of air permeability results at very low RH values.

Molin (1989), using the Figg air permeability index and a measurement of RH in the test hole, studied the relationship between RH. and air permeability in a series of different concretes. Figure 2.37 shows that the differences between concretes of different w/c ratio, aggregate size and curing could be distinguished irrespective of the moisture condition of the concrete. The graph does not extend much beyond the range 60 - 90% RH. and the spread of data is not shown, therefore it is somewhat unclear how useful these relationships are for practical application. This work fits very broadly into the relationship between RH. and air
permeability described by Parrot (1991c) and (1994b), although it covers a smaller range of RH.

### 2.5.2.2 The influence of moisture on sorptivity

The influence of moisture in a porous solid on the capillary rise can be examined using the Jouren Eqn. [2.09] and the Kelvin Equation [2.01]. Equating the terms \( r \) and \( d \) in both [2.09] and [2.01] (Penner, 1965) gives

\[
h = -\frac{RT}{Mg} \ln \frac{P}{P_o}
\]

Eqn. 2.28

Assuming an angle of contact (\( \vartheta \)) = 0.

Typical values for the variables in [2.28] are \( R = 8.31434 \) (J/K.Mol), \( T = \frac{P}{P_o} \) 293.15K (20 °C), \( M = 18.015 \) a.m.u., \( g = 9.81 \) m/s\(^2\), \( P_o = 0.49 \) (49% RH) gives a capillary rise (h) of 10.4 m. The same calculation with \( P_o = 0.99 \) (99% RH) gives a capillary rise of 0.139 m. Examining a fully saturated pore system (\( P_o = 1 \)), equation [2.28] gives no capillary rise. The theoretical relationship between capillary rise and the partial pressure of the vapour in a pore is illustrated graphically in figure 2.38. The graph shows capillary rise in the order of magnitudes generally experienced in concrete above approximately 40% RH. Below 40% RH, the internal pore structure of concrete could be expected to break down and hence the relationship may not hold. Above 90% RH, the capillary rise due to capillary suction is less than approximately 1.5 m, therefore, the over pressure of 200 mm head of water, used in many in-situ sorptivity tests, may become significant in substantially saturated concretes.
2.6 The need for further research

In recent years the number of premature reinforced concrete durability failures have been a matter of professional disquiet for engineers and a matter of financial disquiet for the owners of these structures. This literature review has identified that:

(i) The use of permeation properties as a guide to the likely durability of concrete has been validated in laboratory conditions by numerous test methods and instruments.

(ii) The transfer of this knowledge to real structures has been largely unsuccessful to-date, due to the influence of two main parameters which are uncontrollable on-site - moisture in the concrete and the ambient conditions at the time of test.

(iii) The need for further research seems clear, in that it has been stated by many investigators, there exists a requirement to make in-situ air permeability and sorptivity measurements independent of the conditions at the time of test and the moisture climate of the concrete.

Benefits which moisture and temperature independent in-situ permeation tests may provide include:

(i) The successful transfer of laboratory based knowledge of the 'Autoclam' instrument and its relationship to the transport properties of concrete, to real structures. This would potentially give an investigator a powerful tool with which to assess the likely service life of a reinforced concrete structure.

(ii) Allow the development of a reinforced concrete durability design based on the actual measured permeation properties of concrete structures and the ambient environmental conditions.
(iii) It may be possible to develop a quality control system for very young concrete on-site, based on in situ permeation tests. Such a quality control system could conceivably reduce the number of premature failures of reinforced concrete structures by allowing the identification of concrete which was of inadequate standard during construction.
Table 2.01 - Estimated time to capillary discontinuity in cement paste (Powers, 1958).

<table>
<thead>
<tr>
<th>w/c</th>
<th>Time to capillary discontinuity</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.4</td>
<td>3 days</td>
</tr>
<tr>
<td>0.45</td>
<td>7 days</td>
</tr>
<tr>
<td>0.5</td>
<td>14 days</td>
</tr>
<tr>
<td>0.6</td>
<td>6 months</td>
</tr>
<tr>
<td>0.7</td>
<td>1 year</td>
</tr>
<tr>
<td>&gt;0.7</td>
<td>Impossible</td>
</tr>
</tbody>
</table>

Table 2.02 - Classification of pore sizes in cement paste. Adapted from Oberholster (1986).

<table>
<thead>
<tr>
<th>Designation</th>
<th>Diameter</th>
<th>Description</th>
<th>Role of Water</th>
</tr>
</thead>
<tbody>
<tr>
<td>Macropores</td>
<td>15 - 1000 μm</td>
<td>Larger spherical voids</td>
<td>Behaves as bulk water</td>
</tr>
<tr>
<td>Capillary pores</td>
<td>0.05 - 15 μm</td>
<td>- Large Capillaries</td>
<td>Behaves as bulk water, moderate surface tension forces</td>
</tr>
<tr>
<td></td>
<td>10 - 50 nm</td>
<td>- Medium Capillaries, capillary cavities,</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>capillary cavities, inter gel-particle pores</td>
<td></td>
</tr>
<tr>
<td>Gel Pores</td>
<td>2.5 - 10 nm</td>
<td>- Small (gel) capillaries.</td>
<td>- Strong surface tension forces.</td>
</tr>
<tr>
<td></td>
<td>0.5 - 2.5 nm</td>
<td>- Inter crystallite pores.</td>
<td>- Strongly adsorbed water - no menisci.</td>
</tr>
<tr>
<td></td>
<td>&lt;0.5 nm</td>
<td>- Micropores ('interlayer')</td>
<td>- Structural water in bonding</td>
</tr>
<tr>
<td>Exposure Condition</td>
<td>Nominal Cover (mm)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>-------------------</td>
<td>--------------------</td>
<td>---</td>
<td></td>
</tr>
<tr>
<td></td>
<td>25</td>
<td>20</td>
<td>20*</td>
</tr>
<tr>
<td>Mild</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Moderate</td>
<td>-</td>
<td>35</td>
<td>30</td>
</tr>
<tr>
<td>Severe</td>
<td>-</td>
<td>-</td>
<td>40</td>
</tr>
<tr>
<td>Very severe</td>
<td>-</td>
<td>-</td>
<td>50+</td>
</tr>
<tr>
<td>Extreme</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Max. free w/c ratio</td>
<td>0.65</td>
<td>0.60</td>
<td>0.55</td>
</tr>
<tr>
<td>Min cement content (Kg/m³)</td>
<td>275</td>
<td>300</td>
<td>325</td>
</tr>
<tr>
<td>Lowest grade of concrete</td>
<td>C30</td>
<td>C35</td>
<td>C40</td>
</tr>
</tbody>
</table>

These covers may be reduced to 15 mm provided that the nominal maximum size of aggregate does not exceed 15 mm.

† Where concrete is subject to air freezing whilst wet, air-entrainment should be used.
<table>
<thead>
<tr>
<th>Year</th>
<th>C₃S (%)</th>
<th>C₂S (%)</th>
<th>C₃A (%)</th>
<th>Alkalies Na₂O equiv. (%)</th>
<th>Fineness (m²/kg)</th>
<th>Initial set (mins)</th>
<th>7 day strength N/mm²</th>
</tr>
</thead>
<tbody>
<tr>
<td>1960</td>
<td>47</td>
<td>23</td>
<td>11.5</td>
<td>(0.64)</td>
<td>343</td>
<td>176</td>
<td>24</td>
</tr>
<tr>
<td>1970</td>
<td>51</td>
<td>19</td>
<td>10.5</td>
<td>0.69</td>
<td>332</td>
<td>159</td>
<td>28</td>
</tr>
<tr>
<td>1975</td>
<td>52</td>
<td>19</td>
<td>10.0</td>
<td>0.72</td>
<td>332</td>
<td>161</td>
<td>30</td>
</tr>
<tr>
<td>1980</td>
<td>53</td>
<td>18</td>
<td>9.7</td>
<td>0.61</td>
<td>337</td>
<td>151</td>
<td>33</td>
</tr>
<tr>
<td>1985</td>
<td>54</td>
<td>18</td>
<td>9.5</td>
<td>0.65</td>
<td>368</td>
<td>122</td>
<td>35</td>
</tr>
<tr>
<td>1990</td>
<td>53</td>
<td>20</td>
<td>8.7</td>
<td>0.62</td>
<td>358</td>
<td>144</td>
<td>36</td>
</tr>
<tr>
<td>1992</td>
<td>55</td>
<td>18</td>
<td>8.5</td>
<td>0.62</td>
<td>355</td>
<td>135</td>
<td>34</td>
</tr>
</tbody>
</table>

Note: Figures in brackets denote estimated values
Table 2.05 - Comparative reductions in relative humidity of water due to surface curvature, temperature reduction and salinity (Morrow, 1970).

<table>
<thead>
<tr>
<th>Capillary Pressure (cm H₂O)</th>
<th>Surface Curvature (cm⁻¹)</th>
<th>Cylindrical capillary radius (cm)</th>
<th>Relative Humidity 100% @ 20°C (%)</th>
<th>Equivalent Temp. reduction below 20°C</th>
<th>Equivalent Salinity (g.mol NaCl/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>0</td>
<td>100</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>10</td>
<td>1.36 X 10²</td>
<td>1.47 X 10⁻²</td>
<td>99.999</td>
<td>1.6 X 10⁻⁴</td>
<td>3 X 10⁻⁴</td>
</tr>
<tr>
<td>10²</td>
<td>1.36 X 10³</td>
<td>1.47 X 10⁻³</td>
<td>99.99</td>
<td>1.6 X 10⁻³</td>
<td>3 X 10⁻³</td>
</tr>
<tr>
<td>10³</td>
<td>1.36 X 10⁴</td>
<td>1.47 X 10⁻⁴</td>
<td>99.92</td>
<td>1.3 X 10⁻²</td>
<td>2.4 X 10⁻²</td>
</tr>
<tr>
<td>10⁴</td>
<td>1.36 X 10⁵</td>
<td>1.47 X 10⁻⁵</td>
<td>99.27</td>
<td>1.2 X 10⁻¹</td>
<td>0.22</td>
</tr>
<tr>
<td>10⁵</td>
<td>1.36 X 10⁶</td>
<td>1.47 X 10⁻⁶</td>
<td>93.0</td>
<td>1.16</td>
<td>2.1</td>
</tr>
</tbody>
</table>

Table 2.06 - Variations in Figg air permeability test.

<table>
<thead>
<tr>
<th>Author</th>
<th>Year</th>
<th>Dia. of hole (mm)</th>
<th>Depth of hole (mm)</th>
<th>Depth of plug (mm)</th>
<th>Pressure range (below atmospheric - kPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Figg</td>
<td>1973</td>
<td>5.5</td>
<td>30</td>
<td>20</td>
<td>85 - 80</td>
</tr>
<tr>
<td>Philajavaara</td>
<td>1975</td>
<td>10.0</td>
<td>30</td>
<td>20</td>
<td>98.7 - 86.6</td>
</tr>
<tr>
<td>Cather</td>
<td>1984</td>
<td>10.0</td>
<td>40</td>
<td>20</td>
<td>55 - 50</td>
</tr>
<tr>
<td>Kasai</td>
<td>1984</td>
<td>5.0</td>
<td>40</td>
<td>10</td>
<td>85 - 80</td>
</tr>
<tr>
<td>Arup</td>
<td>1987</td>
<td>10.0</td>
<td>40</td>
<td>20</td>
<td>55 - 50</td>
</tr>
<tr>
<td>Dhir</td>
<td>1987</td>
<td>13.0</td>
<td>50</td>
<td>20</td>
<td>55 - 45</td>
</tr>
</tbody>
</table>
Mechanisms of Deterioration

- Cracking
- Abrasion
- AAR
- Chemical Attack
- Acid attack
- Alkali attack
- Leaching
- Salt attack
- Sulphate attack
- Corrosion
- Carbonation
- Chloride attack
- Freeze thaw

Heavy outlines denote mechanisms controlled and/or influenced by concrete permeability

Figure 2.01 - Reference to percentages assigned to the contribution of various mechanisms affecting durability. After Basheer, 1996.

Concrete Surface

Cathodic Reaction \(2H_2O + O_2 = 4e^- \rightarrow 4 OH^-\)

Anodic Reaction \(2 OH^- + Fe^{2+} \rightarrow Fe(OH)_2\) (Rust)

Figure 2.02 - Simplified corrosion process. After (Bungey, 1996)
Permeable concrete with little entrained air

Water absorbed from environment

Increased water absorbed from environment

Freeze-thaw action causes cracking

Increased permeability and sorptivity

Water from the environment freely available

Figure 2.03 - Cycle of deterioration due to freeze thaw action

Figure 2.04 - Relationship between absorption of concrete and the number of freeze thaw cycles required to cause a 2% reduction in weight of the specimens. Neville (1981)
Figure 2.05 - Macro prediction model for the deterioration of concrete. Basheer et al (1995)

- Moisture
- Ions
- Heat

- Corrosion
- Physical processes
- Chemical processes

- Dimensional changes
- Changes in strength
- Cracking

Figure 2.06 - Micro prediction model for the deterioration of concrete. Basheer et al (1995)
Figure 2.07 - Proposed models of C-S-H structure. after (Oberholster, 1986)
Figure 2.08 - Schematic representation of transition zone between aggregate and cement matrix. (Zie Ping, 1991) (Larbi, 1993)
Where

Ra = Resistivity of aggregate
Rp = Resistivity of cement paste

Figure 2.09 - Electrical current flow through concrete - diagrammatic representation. After Whittington, Mc Carter, Forde (1981)
Figure 2.10 - Change in electrical properties of concrete measured between embedded electrodes under an advancing water front
Figure 2.11 - Capillary rise in a glass tube

Figure 2.12 - Surface Tension in capillary action
Capillary rise absorption test

(a) One dimensional absorption into a semi-infinite medium.

Typical in-situ absorption test

Circular area of test common to ISAT and Autoclam tests

(b) Three dimensional absorption from a finite source into a semi infinite medium.

Figure 2.13 - Comparison of sorptivity test geometry. After (Hall 1981)
Effect of air flow velocity $u_*$ and temperature $T$ on the humidity corrected first stage drying rate $r_j/H$. The position of the family of lines has been fixed by an experimental point ($\Delta$). We emphasize that this point depends on specimen geometry but the relative position of the lines is independent of geometry (see text). The italic figures on the lines show drying rates and the figures at the upper ends of the lines indicate relative drying rates.

Figure 2.14 - Effect of air velocity and temperature on the humidity corrected stage (i) drying rate. Hall, Gummerson, Nixon (1984)
Figure 2.15 - Diffusivity as a function of moisture content of cement mortar. Hedenblad, (1993) reporting work by Pihlajavaara, (1965)
Figure 2.16 - Effect of depth on the time required for half of potential relative humidity drop. Parrott (1991b)

Figure 2.17 - Sorption isotherms in principle. After (Hedenblad, 1993)
Figure 2.18 - Geotechnical bore-hole method for determining rock strata permeability. after (Hvorslev, 1951)
Figure 2.19 - Moisture movement in capillary pores - stages 1-6
After Rose (1965)
Figure 2.20 - Humidity profiles in drying concrete. After Terrill (1986)
Figure 2.21 - Depth of carbonation front limit in exposed concrete. After Bakker (1994).

Figure 2.22 - Schematic diagram of area of changing moisture in exposed concrete. After Bakker (1994).
Figure 2.23 - Vacuum preconditioning technique (Dhir, 1993)
Figure 2.24 - Figg test apparatus for (a) air and (b) water permeability, after Figg (1973).
Test pressure
Air Permeability test: 0.5 bar

Test duration
15 minutes

Measurement
Air pressure in the test area is increased to 0.5 bar and the pressure decay is monitored at every minute for a total duration of 15 minutes.

Display of Pressure

(a) Method of operation for air permeability test

Test pressure
Sorptivity test: 0.02 bar
Water Perm. test: 0.5 bar

Test duration
15 or 60 minutes

Measurement
Piston is pushed down to keep the pressure constant and the travel of the piston is monitored to determine the flow into the concrete.

Display of time, pressure & piston travel

(b) Method of operation for sorptivity and water permeability test

Figure 2.25 - The Autoclam method of operation. After (Basheer, 1991)
Figure 2.26 - Torrent (1992) air permeability apparatus
Figure 2.27 - Apparatus by Hansen et al. to measure Air Permeability of concrete after (Concrete Society, 1988)
Graduated capillary tube

Reservoir

200 mm

Tap

Cap

clamp arrangement used to keep cap in position during test.

Cap is sealed by pushing into moulding clay

Bowl of water penetration

Concrete Specimen

Inlet

Outlet

Figure 2.28 - Initial Surface Absorption Test (I.S.A.T.) apparatus
Figure 2.29 - Covercrete Absorption Test apparatus (Dhir, et al (1989))
Figure 2.30 - Bowl of concrete influencing Autoclam test results

(a) Autoclam air permeability test

(b) Autoclam sorptivity test
Figure 2.31 - Figg air permeability variation with temperature
Bungey et al (1989)
Figure 2.32 - Variation of integrated ISAT measurements with temperature. Bungey et al (1989)

Figure 2.33 - ISAT temperature correction graph. Bungey et al (1989)
(a) - Effect of relative humidity upon permeation data from different investigators. Permeability values normalised by dividing by the permeability corresponding to 60 % RH. After Parrott (1991c).

(b) - Description of moisture condition in idealised capillary pore. Based on Kelvin equation.

Figure 2.34 - Effect of Relative humidity on permeation measurements
Figure 2.35 - Variations of relative humidity and permeability due to wetting and drying. Parrott (1991c)
Figure 2.36 - Air permeability, relative to that at 60% RH, versus RH
Parrott (1994b)
Figure 2.37 - Figg air permeability versus RH (Molin, 1989).
Temperature = 293.15K (20 Deg C)

$RH = 100.00 \times 10^{(-0.031490h)}$  $R^2 = 1.000$

Based on Equation

$$h = -\frac{RT}{Mg} \ln\frac{P}{Po}$$

Figure 2.38 - Theoretical capillary rise against relative humidity.
3. Experimental and test procedures

3.1 Introduction

A description of the procedures, the equipment and tests that were used, is given below. This chapter also includes an account of preliminary work done to verify some of the test methods. This chapter will provide the technical information necessary to reproduce accurately the experimental work described in this thesis.

3.2 Concrete manufacture

3.2.1 Selection of concrete mix

A concrete mix of proportion 1 cement binder : 1.65 fine aggregate : 3 coarse aggregate by weight was selected as suitable on the basis of experience gained in previous experimental work (Basheer, 1991). Three w/c ratios, 0.45, 0.55 and 0.65 w/c were selected to produce concrete of high, medium and poor quality, respectively. Additional water was added to the mix to allow for the absorption of water by the predried aggregates - 1.5 % for fine aggregate and 0.2 % for coarse aggregates. These figures were determined experimentally for the particular aggregates used in this experiment.
3.2.2 Constituents

All concrete manufactured used ordinary Portland cement (OPC) as a binder without any admixtures. The OPC was made by Blue Circle Plc., the chemical composition of which is given in Table 3.01.

The coarse aggregate was either 20 mm or 10 mm single size basalt aggregate, obtained from Craighill quarry, Ballyclare, Co. Antrim. The required quantities of the coarse aggregates were dried prior to use in a fan assisted drier at 40 °C for 48 hours.

The fine sand (Class M : BS. 812 : Pt 2 (1975)) was provided in a dry state by H. Corry, Craigavon, Co. Armagh and is sourced from Lough Neagh.

3.2.3 The manufacture of concrete

The mix procedure used in the investigation was largely based on the method outlined in B.S. 1881 : part 125 (1986). The concrete pan mixer used was a Cumflow G3-K01-831 Type 5122 with a capacity of 3 cubic feet (0.085 cubic meters). The procedure used for the manufacture of all concrete was as follows:

(i) The internal surface of the mixer was dampened slightly prior to the addition of the aggregates.

(ii) The coarse aggregate was added first and the fine aggregate was introduced directly afterwards.

(iii) The mixer was covered and the constituents were mixed for a period of 2 minutes.

(iv) Half of the total water content was then added and was mixed in for 1 minute.

(v) The mixer was covered, to prevent evaporation of water from the mix, and was allowed to stand for 15 minutes.
(vi) The total volume of cement required was added and mixed for 1 minute.
(vii) Finally, the second half of the water was added and the constituents were mixed for 2 minutes. The mix was then placed immediately.

3.2.4 Concrete compaction

Concrete was compacted either with a poker vibrator or by vibrating the mould on a vibrating table. The poker (25 mm diameter) was used in larger moulds where table compaction would be unsuitable or where site concrete reproduction was required. In the mould of section size 500 X 200 layers of 250 mm depth were vibrated in turn. The poker was always kept 50 mm away from the internal surface of the mould to avoid damage and undue interference with the surface zone of the concrete. Concrete was compacted in 2 layers and the times of vibration were different for the two layers in the mould with the upper layer requiring slightly longer vibration periods since it did not have any overburden pressure. In all cases, compaction was continued until there was no evidence of air rising to the surface of the concrete.

All concrete test cubes were vibrated using the table vibrator as were the smaller sized specimens. The periods of vibration for approximately 50 mm deep layers of concrete were found from experience to be 30 seconds for 0.65 w/c, 40 seconds for 0.55 w/c and 60 seconds for 0.45 w/c concrete. Cubes were compacted in two layers as were the concrete specimens that were typically 80 to 90 mm in total height. The vibration times for both layers were kept constant.

3.2.5 Quality control procedures

In addition to the normal care and attention to detail required to produce concrete of the same specification repeatedly, two specific quality
control procedures were adopted and adhered to rigidly. The strength of the concrete at 28 days and its workability at time of placing were measured and recorded so that the properties of different mixes could be shown to be as similar as possible. The results from these tests also ensured that gross errors in the manufacture of the concrete mix could be recognised.

Three 100 mm cubes to BS 1881 : pt 108 (1985) were produced for each mix of concrete produced. These cubes were kept in water at a temperature of 20 °C for 28 days and then tested for compressive strength to B.S. 1881 pt. 116 (1983).

The slump test, to B.S. 1881 : pt 102 (1983), was carried out on the freshly mixed concrete, prior to placing to facilitate comparison between similar mixes carried out at different times.

3.3 Test equipment and procedures

3.3.1 Permeation measurement

The measurement of concrete permeation properties was carried out using the 'Autoclam' permeability system (Basheer, 1991 and Montgomery, Adams, 1985) which is a commercially available unit developed in the Department of Civil Engineering, Queen's University, Belfast.

3.3.1.1 Permeation measurement system

The Autoclam permeability system (plate 3.1) consists of a measurement head and an electronic control unit. The measurement head has a chamber with a pressure transducer at the base, a movable piston and a stepping motor that is used to move the piston. The chamber, at the base of the measurement head, which is also in contact
with the concrete surface, may be primed through a valve and bled through a number of release valves. The unit is secured to a measurement ring which in turn can be either clamped or glued to the concrete surface.

When rings are clamped to the concrete surface, a soft rubber ring is used to provide the seal between the ring and the test surface (figure 3.01(a)). In the cases where bonding the ring to the surface is required, rings are glued to the surface by using a quick setting 'Isopon' material (Figure 3.01(b)). Prior to fixing a ring onto concrete it was necessary to prepare the surface of the concrete by removing dust and loose particles.

The Autoclam permeability system was used to carry out sorptivity and air permeability tests in this investigation. As separate procedures are used for these tests, they are described below.

3.3.1.2 Sorptivity test procedure

As shown in figure 3.02(a) the piston is reset to the top of its travel at the start of the test. The upper valve is opened and the chamber is filled through the priming valve. The upper valve is then closed and the pressure inside the chamber is brought up to a pressure just below 20 mBar (typically 16 - 18 mB). The priming valve is closed and the test is started. This priming operation generally takes less than 1 minute. As the piston moves downwards a pressure of 20 mBar is maintained inside the test chamber. The travel of the piston is displayed and recorded at minute intervals until the end of the 15 minute test. The test may end before 15 minutes if the piston has travelled its maximum permitted distance.

The recorded measurements are converted into volumes of water entering the concrete, by multiplying the piston travel by the area of the piston. The measurements taken between 5 and 15 minutes are used for determination of the sorptivity index as the initial readings are prone to be
influenced from ‘skin’ effects. These volumes are plotted against the square root of time to give a linear relationship. The slope of which is said to be the sorptivity index of the concrete. The data in table 3.02 is used to calculate the sorptivity index shown in figure 3.03(a).

3.3.1.3 Air permeability test procedure

The air permeability test (figure 3.02(b)) is started by resetting the piston to its lowest possible position with the upper bleed valve closed. The pressure of the air in the chamber is increased, by means of a syringe, until it is in excess of 500 mBar (typically 550 mBar). The priming valve is then closed and the test is started by pressing the appropriate button on the control unit. The test begins when the pressure in the isolated chamber falls below 500 mBar and air pressure readings are displayed and recorded at 1 minute intervals for the 15 minute duration of the test. Should the air pressure in the chamber fall to 0 mBar (atmospheric pressure) before the end of the 15 minutes then the test ends automatically.

The measurements of air pressure over the final 10 minutes of the test are used for determination of the air permeability index. Should the test end before 10 minutes then all available readings are used to obtain the index. The natural logarithms of the air pressures are calculated and plotted against the relevant times to give a linear relationship. The slope of this linear relationship is taken to be the air permeability index. In some cases the air pressure falls from 500 mBar to 0 mBar within the first minute, in these cases the air permeability is said to be in excess of 6.125 (This being the maximum measurable air permeability with the Autoclam.) A sample calculation of the air permeability index is shown in figure 3.03(a).
3.3.1.4  Autoclam checks and calibration

Before a series of Autoclam tests was started, a check was carried out to verify the air tightness of the unit. The test unit was fixed onto a solid metal plate and the air pressure was increased until the pressure was approximately 500 mBar. The priming valve was then closed and the pressure was monitored for 15 minutes. Since the metal plate was impermeable to the flow of air, the displayed reading should remain at the initial pressure. If the unit was shown to leak then it was not used for testing and was sent for repair.

At the start of testing on any given day and at regular intervals during testing the pressure transducer which is built into the measurement head of the unit was reset to atmospheric pressure. Care was taken to insure that the transducer was free from moisture and dust at the time of resetting to avoid any influence the accuracy of the calibration.

3.3.2  Relative Humidity measurement

3.3.2.1  Dew-point meter

A 'Protimeter chilled mirror probe' (plate 3.2) was used to measure relative humidity in the investigation. For the sample of air surrounding the probe tip the unit displays the temperature (°C), the dew-point temperature (°C), the relative humidity (%) and the weight of water per unit weight of air (g/Kg). (Protimeter, (1990), Dadachanji, F. (1992)).

The calibration of the probe was checked at the start of the investigation by measuring the known relative humidity over supersaturated solutions of salts, at a specific temperature as shown in figure 3.04. The salts used were lithium chloride (15% RH), potassium thiocyanate (47% RH), sodium chloride (75% RH) and copper sulphate (98% RH). The time taken for the probe to reach equilibrium was also
checked against the manufacturer's claim that the probe would reach a final reading after 20 minutes. Figure 3.05 shows the results of the calibration tests. The accuracy of the probe was duly verified. However, the time required for the probe reading to become stable was found to be between 30 and 40 minutes at 20 °C.

3.3.2.2 Establishment of a procedure for relative humidity measurements

The procedures described in sections 3.3.2.2 and 3.3.2.3 have been reached on the basis of preliminary studies carried out. Following work by Jaflus, (1992) the idea that a relative humidity measurement might require a response time for the relative humidity probe and/or an equilibrium time for a trapped atmosphere in concrete was investigated.

Test 1

Aim of test 1: To determine the time required to achieve thermal equilibrium and a stable relative humidity measurement in a drilled hole.

Description of test 1: Relative humidity measurements were taken in drilled holes of 10 and 30 mm depth without the shield device. The concrete specimens used were 0.65 w/c concrete that had an undetermined moisture gradient. The surface of the hole was covered with an impermeable plastic 'cling film', directly after drilling. The probe was then inserted into the drilled holes at periods of 0, 10 and 20 minutes after the hole was drilled. All probe readings were monitored until a stable measurement was achieved.

Test 1 Conclusions: The results of the tests are presented in figures 3.06(a) and (b). The idea that the heat from the drilled hole would influence the measurement of relative humidity was discounted as the heat generated from drilling was shown to dissipate quickly. Stable relative humidity measurements were generally found around 30
minutes. However, combined with the experience gained from the calibration tests described earlier, it was felt that some measurements took longer than 30 minutes to stabilise. From the findings of this investigation and experience, the following guideline for the measurement of relative humidity in a freshly drilled hole was arrived at.

The relative humidity measurement should be taken over a period of not less than 30 minutes and no measurement may be deemed taken until the internal temperature in the hole has returned to within 0.5 °C of the ambient temperature.

In practice this has been found to vary between 30 and 60 minutes.

Test 2

Jaflus (1992) promoted the idea of using a shield device to isolate a small volume of air in direct contact with a controlled area of concrete at the base of a drilled hole.

Aim of test 2: To determine whether the use of a depth shield device (figure 3.07) was required to measure accurately relative humidity at depth in concrete.

Description of test 2: Two concrete specimens (0.45 and 0.65 w/c) were conditioned at a constant temperature of 20 ± 2 °C and 50% R.H. ± 5% for an extended period, as previous tests indicated that oven drying at an elevated temperature led to a significant quantity of water being absorbed to depths in excess of 30 mm (Nolan, 1994b). Two periods of wetting, 30 minutes for the 0.65 w/c specimen and 2 hours for the 0.45 w/c specimen, were achieved by placing the test face in a shallow bath of water. The excess moisture at the test face was then wiped off and the specimen surface was covered with plastic tape to prevent evaporation of moisture during testing. Relative humidity measurements were carried out in
drilled holes in the concrete specimens at depths of 10, 20 and 30 mm, both with and without the use of a simple depth shield device (figure 3.07(a)).

**Test 2 Conclusions:** The results of the experiment are presented in figure 3.08. As expected, both readings with and without the shield device identified a moisture condition in the concrete that had more moisture closer to the surface. The depth shield device was found to influence results at 10 and 20 mm depths, in that the shielded measurements tended to be higher than their unshielded counterparts. The influence of concrete's lack of homogeneity, slight differences in hole roughness, etc. on the results is unclear as the shielded and unshielded measurements were carried out in separate drilled holes, however the trend is consistent. The measurements carried out in the 30 mm hole show the unshielded measurements were higher than their shielded counterparts. The explanation for this may be that the moist concrete in the upper regions of the concrete influenced the relative humidity measurement at 30 mm depth. Therefore the use of a depth shield device was found to be necessary only at depths in excess of 20 mm.

**3.3.2.3 Measurement of relative humidity at the concrete surface**

Figure 3.07 shows the arrangement used to measure the relative humidity at the surface of the concrete. The surface shield device was used to segregate a small volume of air in contact with the surface of the concrete, which was then measured by the chilled mirror probe. The relative humidity probe has an expanding ring that allowed the probe head to provide an air tight seal with the sides of the shield device. The lower edge of the base of the surface shield had a small amount of 'bluetac' material in a recess, which under a small amount of pressure, moulded to the shape of the concrete surface providing an effective seal.
Thus a controlled quantity of air in contact with the surface of the concrete was isolated and could be measured by the relative humidity probe.

3.3.2.4 Measurement of internal relative humidity measurement at depth in concrete

Measurement of relative humidity at depth in concrete was undertaken in two separate ways in this investigation.

(i) R.H. measurement in a drilled hole.
(ii) R.H. measurement in a preformed hole.

Both methods are described in detail below.

(i) The drilled hole method was generally preferred because it mirrored the way in which relative humidity measurements would have to be used in any investigation of an existing structure. The procedure for the test was to drill a hole of 22 mm diameter to the required depth and to use a surface shield device as shown in figure 3.07 to seal the air in the hole. It was found necessary to use a tungsten tipped percussion masonry drill bit to form a well-defined hole of 22 mm diameter in concrete. After drilling the hole, the remaining dust particles were removed by use of a small dry brush. The probe was inserted into the hole, the surface shield device was secured to the surface of the concrete and the expanding seal on the relative humidity probe was expanded to isolate the air to be measured. In cases where the depth of the hole was in excess of 20 mm a shield device as shown in figure 3.07 was used to isolate the air to be measured.

(ii) The one test series in which pre-cast wells were used was a special case in that it precluded the drilling of holes in a small specimen that was also used for weight loss measurements. The drilling and re-filling of holes used for relative humidity would invalidate any weight loss measurements and so a pre-cast hole and stopper arrangement was used.
instead. This method has been used by other investigators (Parrott, (1991b) and Rolle, (1994)) and has been shown to produce reliable results.

3.3.3 Conductance / Resistance measurement

3.3.3.1 Conductance meter

The instrument used for measuring conductance was a 'Jenway' Model 4010 conductivity meter. Its features include a temperature compensator, calibration control, range switch and a digital display (Jenway, 1988).

The automatic temperature compensator probe was found to require a short period of time, prior to use, to achieve equilibrium with the ambient conditions. This was not mentioned in the operating manual and was generally only significant if the unit was moved from a temperature controlled laboratory area to a much cooler outdoor test site or vica versa. In all cases where it was thought that a significant temperature jump was encountered, the unit was given an extended period to reach thermal equilibrium with its surroundings.

The calibration of the unit was carried out by measuring the conductance across a resistor of known conductance and adjusting the calibration control switch to give the required reading. This was done before the start of any set of test measurements and at intervals thereafter. Re-calibration was generally carried out after a set of 16 measurements when measuring outdoors and at slightly less frequent intervals when measuring in a controlled temperature laboratory.

The range switch was used to produce the most accurate measurement of the capacitance and the final readings, when displayed, were recorded manually.
3.3.3.2 Conductance between embedded electrodes

The conductance measured between embedded stainless steel electrodes was one of the main methods used throughout this investigation to monitor the movement of water through concrete. Each separate set of tests had a unique arrangement of electrodes that were designed to give the optimum quantity and accuracy of data required for each separate experiment.

The electrodes were manufactured by cutting 1.5 mm diameter stainless steel rods to the required length. The rods were then covered with heat shrink tubing, leaving an exposed length in the centre, thus the effective length of the electrode in direct contact with concrete was accurately regulated. Figure 3.09 shows both the electrodes and the electrode arrangement used in the outdoor exposure test series, and is typical of the electrode arrangements used throughout. The electrodes were positioned in the concrete specimen mould prior to casting and were anchored either from the sides of the mould or using an additional perspex holder as shown in figure 3.09. When the mould was struck the ends of the electrodes projected from the concrete specimens. Prior to conductance testing the heat shrink tubing was stripped from a length of exposed rod and the conductance was measured between adjacent electrodes.

3.3.4 Temperature measurement

Temperature in air was measured by a digital thermometer. The thermometer was calibrated on a number of occasions during the duration of the experimental work and was found to be correct within its stated limits of accuracy (± 0.1 °C). Temperature was also measured at specific depths in concrete using embedded thermistors.
3.3.4.1 Embedded thermistors

The accurate and stable measurement of temperature at depth in concrete was achieved by using embedded thermistors. Before the thermistors were set into position in the concrete mould they were individually checked for accuracy. The thermistors are guaranteed not to deviate more than 2% from the supplied resistance response curve (figure 3.10). The copper alloy limbs leading from the head of the thermistor were soldered to a covered wire and the joint was made moisture tight by covering the section with heat shrink tubing and sealing the ends with adhesive (figure 3.11). The waterproofing of the leads from the measurement head of the thermistor ensured that moisture could not influence the resistance measurements when the unit was embedded in concrete. The thermistors were mounted on the perspex support blocks included in the concrete for the positioning of conductance electrodes (as shown in figure 3.09), thus insuring accuracy of depth. Resistance readings were taken between the two exposed wires by using a multimeter and were used to calculate the temperature in the concrete at the respective specific depths.
### Table 3.01 - Chemical composition of cement used in investigation

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<thead>
<tr>
<th>% $C_3S$</th>
<th>% $C_2S$</th>
<th>$C_3S/C_2S$</th>
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<tr>
<td>50</td>
<td>20</td>
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### Table 3.02 - Booking and calculation sheet for Autoclam results

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<th>Time (mins)</th>
<th>Pressure (mB)</th>
<th>Ln Pressure</th>
<th>Piston travel (mm)</th>
<th>Sorption Flow Cu.M X E-7</th>
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<td>310</td>
<td>5.737</td>
<td>0.831</td>
<td>1.469</td>
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</table>
Plate 3.01 - Autoclam permeability system

Plate 3.02 - Chilled mirror RH probe
Pressure applied to secure ring to concrete

(a) 'Clamped' ring - 'Autoclam' test arrangement

(b) 'Bonded' ring - 'Autoclam' test arrangement

Figure 3.01 - Autoclam ring test arrangements
**Test pressure**
Sorptivity test: 0.02 bar

**Test duration**
15 or 60 minutes

**Measurement**
Piston is pushed down to keep the pressure constant and the travel of the piston is monitored to determine the flow into the concrete.

**Display of pressure & piston travel**

---

**Test pressure**
Air Permeability test: 0.5 bar

**Test duration**
15 minutes

**Measurement**
Air pressure in the test area is increased to 0.5 bar and the pressure decay is monitored at every minute for a total duration of 15 minutes.

**Display of Pressure**

---

**Figure 3.02** - The Autoclam method of operation. After (Basheer, 1991)
Equation of the line \( y = 6.20 - 3.12e^{-2x} \) \( R^2 = 1.00 \)
Written as API = 3.12e-2 : \( R^2 = 1.000 \)

(A) Test No. 1 - Specimen air permeability calculation

Equation of the line \( y = -4.86e^{-2} + 0.39x \) \( R^2 = 0.999 \)
Written as SI = 0.39 : \( R^2 = 0.999 \)

(B) Test No 1 - Specimen sorptivity calculation

Figure 3.03 - Specimen 'Autoclam' results graphs for data in table 3.02
R.H. Probe Head sealed into the gas jar using the micro-climate sealing ring.

Display Unit

<table>
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<th>Int. Temp C</th>
<th>20.3</th>
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</thead>
<tbody>
<tr>
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</tr>
<tr>
<td>g/Kg</td>
<td>11.5</td>
</tr>
</tbody>
</table>

Known Relative Humidity entrapped in glass jar.

Super Saturated Salt Solution giving a known Relative Humidity at a known Temperature

Figure 3.04 - Chilled Mirror Probe - Calibration / verification
Figure 3.05 - Probe response to manufactured atmospheres
Figure 3.06 - Preliminary test 1 - Equilibrium and response time investigation
Rebate to accept 'blue-tac' material

Surface Shield Device

Soft rubber sealing ring

Shield Device #1

Screw action handle applies pressure to rubber ring

Shield Device #2

Figure 3.07 - Relative humidity measurement - shield devices
Figure 3.08 - Preliminary Test 2 - Shield Vs No shield studies
Electrodes exposed over a length of 40 mm

Electrodes held in place by mould and a perspex support.

Specimen mould 450 X 500 X 200 mm

Threaded holes allow fixing to formwork

5 mm depths

Concrete surface / Mould face

15 mm

15 mm

15 mm

Thermistor holders at set depths

heat shrink covered stainless steel electrodes

Electrodes held by 'pushfit' holes

Perspex Support

Figure 3.09 - Electrode Details
RS Thermistor (151-221)

Resistance = 17.103 - 0.83556T + 1.6185e-2T^2 - 1.0902e-4T^3

R^2 = 0.997; Where T is Temperature (C)

Data supplied by manufacturer

Figure 3.10 - Thermistor response curve

- 0.2 mm Dia. Tinned copper alloy leads
- Epoxy coated measurement head
- heatshrink tubing sealed with adhesive at either end
- Wires to concrete specimen surface
- 2.4 mm Curve matched Thermistor
- soldered joint

Figure 3.11 - Thermistor details
The influence of moisture on the permeation properties of concrete

4.1 Introduction

The results of laboratory based explorations into the influence of moisture on the Autoclam air permeability and sorptivity measurements are reported in this chapter.

4.2 Objectives of experimental programme

The main aim of the experimental programme was to investigate the influence of moisture in concrete on Autoclam air permeability and sorptivity measurements. To achieve this aim, the following specific objectives were identified:

(i) To examine the effect of different moisture conditions in concrete on Autoclam air permeability and sorptivity measurements.

(ii) To compare the effect of a uniform moisture condition and a moisture gradient on Autoclam permeation measurements.
4.3 Experiment programme

4.3.1 Experimental variables

The variables which were incorporated into the experiment are shown in full in table 4.01. A set of three specimens and two smaller measurement blocks was cast for each of the test variables shown in table 4.01.

4.3.1.1 W/C ratio

As discussed in chapter 2, the permeability of concrete is influenced by numerous factors from which w/c ratio was chosen as the most suitable variable in this experiment to provide a reasonable range of concrete qualities. The other mix variables, mix procedure and quality control procedures were as detailed in chapter 3.

4.3.1.2 Moisture conditions

The greatest emphasis was given to the number of moisture conditions. Initially all specimens were saturated by incremental immersion over a period of 3 days (described in figure 4.01) and were then subjected to drying (at 40°C) for periods of 1, 2, 7, and 14 days to give four different moisture contents in the concrete. Drying of the specimens was carried out in a fan assisted drying cabinet (plate 4.02).

4.3.1.3 Moisture distribution in concrete

The distribution of the moisture in the concrete at time of test was considered as a variable in this experiment. The test specimens that were tested immediately after the initial cooling period were considered to contain moisture gradients caused by uniaxial drying. The second half of the specimens were wrapped in polythene sheet and left for a period of
one year in the laboratory. During this time it was expected that the moisture in the capillary pores of the concrete would become distributed evenly throughout the specimens.

4.3.2 Tests methods

The following two test methods were used to monitor the moisture in concrete:

(i) Weight measurements. The quantity of evaporable water lost during drying was determined by weight measurements taken before (saturated surface dry) and after drying.

(ii) Relative humidity measurement at the test surface and in preformed holes of 10 and 20 mm depth. These measurements were carried out using the shield device #2 (see figure 3.07) for the 20 mm measurements and the surface shield device for surface and 10 mm depth measurements, as described in section 3.3.2.

In addition, the resistance / conductance between embedded electrodes in the concrete (3.3.3) was measured to obtain an indirect indication of moisture and moisture movement in concrete.

The permeation properties of the concrete were measured using following tests:

(i) Standard 'Autoclam' air permeability test (3.3.1.3).

(ii) Standard 'Autoclam' sorptivity test (3.3.1.2).

The quality control tests carried out on each mix were:

(i) 28 day compressive strength tests on 100 mm test cubes.

(ii) Standard slump tests.

Details of the test equipment and procedures are given in chapter 3.
4.3.3 Specimen details

In the selection of the test units two conflicting priorities emerged. The weight loss measurements required a relatively small sample so that small changes in weight could be measured accurately using a balance. However, the Autoclam tests required a surface area of roughly 200 X 200 mm with a depth in excess of 50 mm (i.e. a relatively large sample). In order to accommodate both priorities two sets of specimens were designed - a smaller test block to monitor the moisture loss from the concrete and a large specimen for Autoclam tests. The Autoclam test requires the average of three tests to be reported as the result, therefore three large specimens were manufactured for each variable. To monitor the moisture in the three Autoclam slabs, 2 moisture measurement blocks were manufactured from the same mix.

4.3.3.1 Moisture measurement blocks

The moisture measurement block (figure 4.02 and plate 4.01 ) was used to measure weight loss, resistance between embedded electrodes and relative humidity in preformed holes. Preformed holes were used for relative humidity measurement to ensure that the weight loss measurements would not be influenced by drilling. The moisture measurement block was designed to be representative of the larger test block. The moisture measurement block was the same depth as the Autoclam test block and to avoid any effect due to specimen size, the 5 non-test sides of each specimen were coated with epoxy paint. This meant that the specimen could be considered as a small section of a theoretical semi-infinite slab of thickness in excess of 160 mm. Since the five non-test faces were coated with epoxy paint, drying occurred exclusively through the test (cast) face.
The electrode arrangement (see figure 4.02) was designed to give primary measurements of resistance between 5 - 10, 20 - 25 and 35 - 40 mm depths in the concrete. Secondary measurements between 10 - 20 mm and 25 - 35 mm were also taken. The 10 mm coarse aggregate in the mix was expected to become fixed in the concrete matrix between electrodes 10 mm apart whereas between electrodes 5 mm apart no aggregate will influence resistance measurements (McCarter, 1995). Two arrays of electrodes were included in each block which allowed an average of four resistance measurements for each depth range and each experimental variable.

4.3.3.2 Autoclam test specimens

The Autoclam test specimens were of size 230 X 230 X 80 mm, with the five non-test faces painted with epoxy paint to ensure that moisture loss occurred only through the test face.

4.3.4 Curing and conditioning

After casting, the specimens were subjected to the following curing regime:

(i) Specimens struck from mould on day 1, 24 hours after casting. The specimens were then transferred to a water bath kept at 20 °C. Three days water curing is normally used in laboratory investigations by the SMRG in Queens University.

(ii) On day 4 the specimens were moved to a constant temperature laboratory environment of 20 °C ± 1 °C and 50% ± 5 % relative humidity.

(iii) Four of the non-test faces (those normal to the test face) of each specimen were painted with two coats of epoxy paint between day 18 and day 21. To allow for subsequent saturation by immersion the test face and the opposite face were left unpainted.
(iv) The specimens were re-saturated by incremental immersion in water for 3 days at the age of 25 days (See figure 4.01). The fifth non-test face (directly opposite the test face) was then painted with two coats of epoxy paint.

(v) Initial weight and resistance measurements were taken immediately and the specimens were placed on day 28 in a drying cabinet (plate 4.02) for a specified period of drying (table 4.01).

(vi) After conditioning the specimens were removed from the drier and were wrapped in polythene for a cooling period of 24 hours prior to testing or continued storage.

4.4 Presentation and analysis of results

The results from the experimental programme are presented in figures 4.03 - 4.37. In the following section the effect of each of the variables is presented and analysed. The experiment falls naturally into two distinct sections - tests on concrete with moisture gradients (table 4.01) and the tests on identical specimens after an extended period of storage.

4.4.1 Moisture gradients in concrete

The four periods of drying were 1, 2, 7 and 14 days. After the 24 hours cooling the concrete was tested immediately for weight loss, resistance between electrodes, RH on the surface and in pre-formed holes of 10 and 20 mm depth, air permeability and sorptivity of the cover concrete.
4.4.1.1 Weight loss measurements in concrete subjected to drying

The loss of water from the concrete due to the drying was determined from weight measurements taken before and after the conditioning. Figure 4.03 shows the weight loss with time for all 3 w/c ratio concretes investigated. Observations from figure 4.03 are:

(i) As expected the greatest weight loss occurred from the 0.65 w/c concrete.

(ii) The difference in weight loss between 0.65 w/c and 0.55 w/c concrete was shown to be considerably greater than the corresponding difference in weight loss between 0.55 w/c and 0.45 w/c.

The observation that the greatest quantity of moisture was lost by the 0.65 w/c concrete can be explained with reference to its the larger average pore size, greater pore connectivity and greater initial volume of capillary water. As a result, more moisture could be expected to be removed from a larger w/c concrete under a given drying regime.

The difference in weight loss between 0.65 w/c and 0.55 w/c and 0.55 w/c and 0.45 w/c concretes observed in figure 4.03 can be explained with reference to figure 4.04. This shows the frequency of occurrence of pore sizes in different w/c ratio concretes in a series of mercury intrusion porosimetry tests carried out by Nyame (1979). Generally, high w/c ratio concretes have a greater total pore volume (represented by the area under each curve) and have pores of larger diameter than concretes of lower w/c ratio. More specifically it can be seen that the difference in total pore volume between 0.71 and 0.47 w/c concrete is substantially greater than between 0.23 and 0.47 w/c concrete. This indicates that the increase in the quantity of evaporable moisture in concrete with increasing w/c ratio is non-linear. As discussed in 2.2.1.2, the continuity of capillary pores in cement paste increases with increasing w/c ratio, in a non-linear manner,
Therefore, the non linear influence of w/c ratio on the quantity of moisture lost during drying, as shown in figure 4.03, could be anticipated.

Figure 4.05 shows the weight of moisture loss versus the square root of the duration of drying under the stated conditions. This graph suggests that the loss of moisture from the concrete followed a square root time relationship. This relationship may indicate that the drying process was as described by the falling drying rate (stage 2 drying, described fully in 2.4.3.2) (Hall et al, 1984) where the rate of evaporation is thought to be determined or limited by the rate of unsaturated flow of the liquid towards the drying surface. This has been described by Hall et al (1984) as being analogous to a sorption process and is referred to as desorption. Therefore, the square root time relationship describes the unsaturated flow of moisture towards the surface of the concrete and as shown in figure 4.05 the rate of moisture loss is influenced by the physical characteristics of the pore structure of the concrete.

4.4.1.2 Relative humidity measurements in concrete with moisture gradients

Figure 4.06 shows the variation of relative humidity in concrete with the duration of drying for the three w/c ratio concretes. Each of the relative humidity (RH) values shown is the arithmetic average of two measurements (one measurement in each of the test blocks). For ease of discussion the RH reading in the 10 mm and 20 mm holes are referred to as the 10 mm and 20 mm RH's.

It is recognised that these measurements are not at a specific depth in the concrete but are influenced by the area of concrete surrounding the entrapped air. Jaflus, (1992) identified the concept of the area of concrete contributing to the relative humidity of the entrapped air and used a
shield arrangement to control it. In this investigation the RH measurement in the 10 mm hole is based on the moisture in the concretes pores between 0 and 13 mm while the RH measurement in the 20 mm hole is based on the concrete between 17 and 23 mm as illustrated in figure 4.07.

Salient observations from figure 4.06 are:

(i) The effect of one and two days drying on the relative humidity measurements at all depths was significant in the 0.45 w/c ratio concrete and became less significant with increasing w/c ratio.

(ii) In both the 0.55 and 0.65 w/c concrete the RH reading in the 20 mm hole remained relatively constant between one and two days drying.

(iii) The RH readings at the surface and in the 10 mm hole did not change significantly between 7 and 14 days drying in the 0.65 w/c concrete.

The significant reduction in the RH caused by one and two days drying in the 0.45 w/c ratio concrete may be explained by regard to the pore size distribution of the concrete. As stated in chapter 2 with reference to the Kelvin equation, relative humidity gives an indication of the largest sustainable meniscus in a porous body. Using the Kelvin equation [2.02] a maximum radius of curvature for the meniscus can be calculated for a given temperature and relative humidity (Penner, 1965 and Morrow, 1970). Table 4.02 shows a range of these pore sizes. If a parallel, circular cross sectional pore model of concrete is adopted then all pores of diameter greater than the values shown in table 4.02 are substantially empty of moisture. In reality, this is not strictly true as pores are generally described as being of variable cross sectional shape and area, however it serves as a good indication of what each relative humidity measurement means.

Defay et al (1966) identify the preferential condensation of moisture (based on the Kelvin equation) starting in the smallest pores and only filling larger pores after these pores had been filled. Conversely, in a
drying porous medium the largest pores could be expected to empty first. Therefore since the 0.45 w/c concrete has fewer or no large sized pores (as shown by figure 4.08, Nyame, 1979) these pores are quickly dried out and the removal of moisture starts from smaller sized pores. The 0.65 w/c ratio concrete has more large sized pores and this gives rise to the removal of moisture at a faster rate (shown by the weight loss measurements). However, despite the faster rate of moisture loss, the greater quantity of moisture that is contained in these large pores means that it takes longer to remove the moisture contained within them.

During the 1 to 2 day drying period the RH measurements in the 0.65 w/c ratio concrete was seen to remain relatively constant and were similar at all depths. This indicates that moisture which was removed from the concrete during this period was being lost from pores of roughly 0.02 μm size (90% RH) as predicted by the Kelvin equation and illustrated in table 4.02. While moisture was lost from the pore system on the second day of drying the size of pores losing the moisture was not significantly reduced which suggests that a large quantity of 0.02 μm diameter pores were present in the 0.65 w/c concrete. Referring to the work by Nyame (1979), figure 4.08 shows that in a cement paste specimen of 0.71 w/c ratio a large number of 0.2 μm diameter pores (shown as a radius of 1010 Å) existed thus providing evidence that this finding is plausible. The pore size distribution in a concrete could expect to contain the transition zone of increased porosity which is not present in a cement paste.

The RH measurements at the surface and 10 mm in the 0.65 w/c concrete showed no significant change over the seven to fourteen days drying period. This suggests that no overall loss of moisture occurred from the uppermost 10 mm of the 0.65 w/c concrete between 7 and 14 days drying. This is possible if the quantity of moisture entering this zone of concrete was equal to the quantity of moisture being evaporated, as shown
in figure 4.09. This indicates that a steady state of moisture flux occurred in this zone between 7 and 14 days, where moisture from the bulk of the concrete, travelled through the capillary pore system of the zone to the surface and was evaporated. This is consistent with the Hall et al (1984) description of the stage 2 drying processes.

The moisture profiles as measured by relative humidity due to the 4 periods of drying and the 3 w/c ratio concretes are shown in figure 4.10. Generally, the profiles are as expected with lower values of relative humidity closer to the drying surface.

Observations from figure 4.10:

(i) Drying for one day caused the development of shallow moisture profiles with depth in all specimens. The RH measurements in the 0.45 w/c concrete showed a significant difference between 0 and 20 mm depth while the difference in the 0.65 w/c specimen was less significant.

(ii) Drying for two days caused a significant moisture gradient in the 0.45 w/c ratio concrete. Drying for the same period result in a more shallow gradient in the 0.55 w/c concrete while the RH readings in the 0.65 w/c ratio concrete did not vary significantly with depth.

(iii) Seven days drying resulted in the establishment of constant gradients of moisture in the 0.45 w/c ratio concrete. The 0.55 and 0.65 w/c ratio concrete showed a slightly shallower gradient close to the surface and a larger gradient between 10 and 20 mm depth.

(iv) Fourteen days drying produced a constant gradient of moisture in the 0.65 w/c ratio concrete while the 0.45 and 0.55 w/c ratio concretes showed similar RH readings at the surface and in the 10 mm hole.

After both one and two days drying periods a significant moisture gradient in the 0.45 w/c ratio concrete was evident while more gentle gradients were identified in the 0.55 and 0.65 w/c concretes. The different moisture gradients in the concrete were most certainly caused by the
respective microstructures of the different concretes. The 0.45 w/c ratio concrete could be expected to have very few pores of 0.02 \( \mu \text{m} \) diameter (this corresponds to 90% RH as shown by table 4.02) and the continuity of the capillary pores in 0.45 w/c ratio concrete could be expected to be much less than that of the 0.65 w/c ratio concrete (Powers et al, 1959)(table 2.01). Therefore, as noted previously, drying could be expected to remove the moisture from the larger pores nearer to the surface while the reduced continuity of the pore system in the concrete would result in increased resistance to moisture flow from the deeper concrete towards the drying surface. This can be seen in the 0.45 w/c RH gradients shown in figure 4.10 where the moisture gradient after 2 days drying shows a significantly more saturated concrete at 20 mm depth.

Since the continuity of the capillary pore system increases with increases in the w/c ratio of the concrete (Table 4.02, Powers et al, 1959), the loss of moisture from depth in the 0.65 w/c ratio concrete over the 1 and 2 days drying period could be expected to be more significant. Also, in the 0.65 w/c concrete, moisture was still being removed from large pores which would have a reduced resistance to unsaturated flow compared to smaller pores. Therefore it is reasonable to suppose that the moisture gradient in the 0.65 w/c concrete would be much more gentle than in the 0.45 w/c concrete. This can be seen in figure 4.10 where there is no significant difference in the RH between the surface and the 20 mm RH readings in the 0.65 w/c concrete.

The moisture gradients caused by 7 days drying showed that the 0.45 w/c ratio concrete had a constant RH gradient of moisture. The 0.55 and 0.65 w/c ratio concrete showed a more gentle gradient close to the surface and a larger gradient between 10 and 20 mm depth. Why this should happen can be understood more fully by looking at what the RH values mean. The values of RH measured at the surface of all the concretes
indicated that all the free water in the concrete pores had been removed. Parrott (1994b) states, on the basis of experimental work, that the pore structure of concrete is substantially free from moisture at roughly 60% RH. Examining the pore size distribution described by Nyame (figure 4.08) shows that the smallest measured capillary pore in the concretes is roughly between 60 and 80 Å in diameter (which roughly corresponds to 70 - 74 % RH). Therefore in the region of 60 - 70 % RH the capillary pores in the concrete could be expected to be substantially free from moisture and relative humidity values below this could be expected to indicate the removal of some of the moisture adsorbed on the walls of the capillary pores.

Consequently, with reference to 4.07, the RH values in all concretes show substantial differences between the surface and 20 mm depth after 7 days drying. The 0.45 and 0.55 w/c ratio concretes show a loss of adsorbed moisture over the 10 mm of the concrete nearest to the drying surface. At 20 mm depth the RH values indicated that the free water in the concrete pores was substantially removed. The RH values in the 0.65 w/c ratio concrete after 7 days drying indicate some loss of adsorbed water over the upper 10 mm while the concrete at 20 mm depth still retained some moisture in its smallest pores.

The change in RH values over the 7 to 14 days drying period shows an unclear trend. In the 0.45 and 0.55 w/c ratio concrete the RH decreases at all depths although the surface RH measurement in the 0.45 w/c ratio specimens seems to be higher than could have been expected. The 0.65 w/c ratio concrete shows a small change in RH over the upper 10 mm while the RH at 20 mm depth reduces considerably to produce a very gentle moisture gradient. The apparent increase in the surface RH between 7 and 14 days drying is unlikely to be a natural phenomenon and may be caused by mix variation, or by the inhomogeniety of the concrete.
It may indicate that in 0.65 w/c ratio concrete subjected to extended drying caused little reduction in RH. This may be due to the large volume of pores in 0.65 w/c concrete which contain a large quantity of adsorbed water. The removal of adsorbed moisture from 0.65 w/c ratio concrete therefore could be expected to result in the slower decrease of RH.

4.4.1.3 Conductance / resistance measurements in concrete with moisture gradients

The conductance measurements taken in the experimental programme between the embedded electrodes were inverted to give resistance readings. The resistance ratio, $R_t/R_i$ (i.e. resistance after a period of drying $t$ / initial resistance) was plotted to show clearly the change in resistance from the initial state (Mc Carter et al, 1995).

In figure 4.11 each resistance value presented is the arithmetic average of four measurements (two from each of the moisture measurement blocks) and in figure 4.12 the gradients of resistance ratio after the periods of drying are shown.

Observations from figures 4.11 and 4.12:

(i) Common to all w/c ratio concretes is an increase in resistance ratio with increased drying time.

(ii) Figure 4.11 shows that resistance ratios for any depth range at any given time of drying were generally larger in the 0.65 w/c ratio concrete and smaller in 0.45 w/c ratio concrete.

(iii) Two days drying caused the formation of resistance ratio gradients in concrete which were more gentle in the 0.65 w/c ratio concrete and were more pronounced in the 0.45 w/c ratio concrete.

(iv) Prolonged drying for 7 and 14 days caused the formation of pronounced gradients of resistance ratio in all concretes.
The trends identified in the resistance ratio of the concrete suggest a link between resistance ratio values and relative humidity measurements.

The rise in resistance ratio with loss of moisture as shown in figure 4.11 is as a result in the loss of moisture from the concrete. Mc Carter et al (1995) states that the moisture in capillary pores acts as a conduit for the passage of electrical resistance through concrete. As the moisture is removed from the capillary pores in the concrete the electrical current flow path is lengthened and the resistance value increases.

The difference in resistance values identified between the different w/c ratio concretes, as shown in figure 4.11, are caused by the different microstructural properties of each concrete. The 0.65 w/c ratio concrete has pores of larger diameter, which lose moisture more readily than smaller pores, causing a greater increase in the electrical flow path length, than in concrete with smaller capillaries. The 0.65 w/c ratio concrete contains more large pores (figure 4.08) thus for a given period of drying the resistance of the concrete will be greater than that of a concrete with a lower w/c ratio. Figure 4.11, shows that with a few exceptions, the resistance of the 0.65 w/c concrete was greater than the corresponding values of resistance in the other concretes.

The gradients of resistance ratio in the concrete (figure 4.12), after one and two days drying, show that the 0.45 w/c ratio concrete had a steep gradient while the 0.65 w/c ratio had a shallow gradient. This observation confirms a similar finding from the RH measurements discussed above. The different resistance ratio gradients in different concretes can be explained by examining the differences in capillary continuity and the size of pores which are losing moisture, as discussed earlier.

After seven and fourteen days drying the steepest resistance gradient is evident in the 0.65 w/c ratio concrete. This finding can be explained by noting that during 7 and 14 days drying the removal of
adsorbed water was identified by RH results. The conductance of electrical current through a concrete is primarily through a continuous path provided by moisture. The removal of free water from the capillary pores causes an increase in the flow path (and removes the ionic component of current flow (section 2.4.2.2)) and so increases the resistance to the flow of electrical current. It seems likely that in this situation that the electrical current is conducted by the moisture adsorbed onto the walls of the pores. Further drying causes the loss of adsorbed water which in turn causes discontinuities in the adsorbed moisture paths which the electrical current follows. This forces the electrical current to pass through the CSH paste which has a much greater resistance to the flow of electrical current.

Therefore the large increases in resistance ratio evident after 7 and 14 days drying near to the drying surface seems to be caused by the removal of adsorbed moisture. After 14 days drying the resistance ratio values between 5 and 20 mm in all concretes show a large increase suggesting that the removal of some adsorbed water has taken place in this region. Figure 4.12 shows that the resistance ratio values increase with increasing w/c ratio of the concrete. The reason for this is possibly related to the rate of removal of adsorbed water from different sizes of pores, with the larger pores in the 0.65 w/c ratio concrete promoting a larger removal of adsorbed moisture and hence causing increased resistance ratios.

Many similarities can be identified between the resistance ratio values and the RH values obtained in this investigation. The values of RH in the 10 and 20 mm holes were compared to the resistance ratio values between the 5 - 10 and 15 - 20 mm and deep electrodes respectively. Figure 4.13 shows that the relationship was almost linear in all concretes in the range 100 - 60 % RH, after which the relationship breaks down. For a large change in relative humidity between 100 and 60 % there is a relatively small change in resistance ratio, and for RH values below
roughly 60% there is a significant increase in resistance ratio with a decrease in RH. This tends to suggest that the use of RH is more suitable for measuring the moisture condition of concrete with some free moisture in the capillary pores while the resistance ratio is extremely sensitive to the loss of adsorbed water from the capillary pores. Looking at figure 4.08 it should be noted that RH is more sensitive to the quantity of moisture in the smaller pores rather than to the quantity of moisture in the larger pores.

4.4.1.4 Autoclam air permeability of concrete with moisture gradients

The air permeability indices (based on the arithmetic average of three Autoclam tests) are presented in figure 4.14. As is normal practice when reporting Autoclam indices, in cases where a single permeability measurement was very different from the other two measurements, the outlying result was omitted from the average. These occurrences are highlighted in figure 4.14.

Observations from figure 4.14
(i) The air permeability indices of the concrete were higher for concretes of higher w/c ratio.
(ii) The 0.65 w/c ratio concrete did not exhibit a significant change for periods of drying in excess of 1 day.
(iii) The 0.45 and 0.55 w/c ratio concetes showed an increase in air permeability for the first 7 days drying after which there was no significant change.
(iv) The moisture condition of the concrete as shown by the RH measurements were shown to be related to the air permeability of concrete.
The air permeability of concrete was greater in the 0.65 w/c concrete and reduced with a decrease in w/c ratio. This is as expected and is caused by the different pore structures of the concretes, with the 0.65 w/c concrete having a greater volume of pores (figure 4.04) and containing pores of greater size (figure 4.08).

The 0.65 w/c ratio concrete did not show significant change with the different periods of drying. This can be explained with reference to the RH measurements in the 0.65 w/c concrete. The initial 1 day drying caused a uniform reduction of RH to roughly 90 % at a depth in excess of 20 mm. This meant that the largest pores were free from moisture and were able to provide a continuous path for the flow of air. The removal of moisture from the smaller capillary pores by subsequent drying did not result in a significant increase in air permeability as most of the air flow followed the path of least resistance through the more continuous large sized pores. This effect is also shown (but not discussed) by Parrott (1994b) in a revision to his original graph (figure 4.15) where the most permeable concretes attained an air permeability at 90 % internal RH roughly equal to that of the air permeability at 60 % and 20 % RH.

The air permeability of 0.45 and 0.55 w/c ratio concretes were shown to increase with time of drying until 7 days drying after which relatively little change in air permeability occurred. Again this can be examined with reference to the RH measurements shown in figure 4.10. The RH values after 1 day drying shows that only some of the pores had no meniscus in the upper 10 mm of the concrete, while the 10 - 20 mm zone of the concrete had a substantial quantity of moisture. The RH after 2 days drying is reduced significantly with the concrete in the 10 mm closest to the drying surface losing moisture and the 10 - 20 mm zone of the concrete staying largely unchanged in both the 0.45 and 0.55 w/c ratio concretes. This had the effect of increasing the air permeability of the concrete.
Further drying reduced the RH in the concrete to 70% or below at all depths in the concrete. This means that all the capillary pores in the uppermost 20 mm of the 0.45 and 0.65 w/c ratio concretes were free from moisture and were able to sustain the passage of air. The removal of moisture from the 10 - 20 mm depth zone of the concrete had a significant affect on the air permeability of the concrete. Further drying (until 14 days) caused no significant change in the air permeability of the 0.45 and 0.65 w/c ratio concretes as the removal of adsorbed water from the capillary pores could not be expected to affect the capacity of these pores to allow the passage of air.

Variations in the expected RH values were noted in figure 4.10 with the 0.65 w/c ratio concrete showing a slight decrease in air permeability for drying periods up to 7 days. It was considered that this slight reduction in permeability was due to effects such as the variability of concrete mixes and the inhomogeniety of concrete. In particular the air permeability result for 7 days drying seems to be low when compared to the other results nevertheless there was no reason for any of the air permeability test results from which this average value is composed. There also seems to be a slight decrease in air permeability in the 0.45 and 0.55 w/c concretes between 7 and 14 days drying. Again there seems to be little basis for expecting this result and the fact that the reduction was marginal tends to suggest that this is as a result of normal experimental error rather than a real phenomenon.

The relationship between RH and air permeability indices was investigated in figure 4.16. The overall trend of increasing air permeability with decreasing RH can be seen in each of the concretes although the trend becomes confused after the maximum permeability has been reached. The increase in air permeability due to the loss of moisture from the pore system can be seen to increase with increasing w/c.
ratio concrete and the ability of moisture to mask the true permeability of a concrete is quite evident. The rate of increase in permeability with decreasing RH, which is shown by the slope of the relationship, can be seen to be most steep in the 0.45 w/c ratio concrete and most shallow in the 0.65 w/c concrete. This reflects the different pore sizes in the different concretes and shows the importance of the large sized pores to the air permeability of concrete. Parrott (1994b) (figure 4.15) uses relative permeability term (relative to the permeability at 60% RH) to show the change in permeability with RH in concretes with different pore structures. The data from this investigation has been added to Parrott's graph and as can be seen from figure 4.15 the relationships between the air permeability and relative humidity agree closely. It should also be noted that this does not agree with the findings of Nagataki et al (1986) who found that the increase in air permeability in 0.65 w/c ratio concrete continued for decreases in relative humidity in the 40 - 60 % range. (See figure 4.17)

4.4.1.5. Autoclam sorptivity of concrete with moisture gradients

The sorptivity results shown in figure 4.18 and figure 4.19 show the change of sorptivity index with the duration of drying for each of the three w/c ratio concretes. Each value shown is the arithmetic average of three Autoclam tests. In accordance with Autoclam guidelines, outlying sorptivity results (in the order of 50% of the average) were not included in average sorptivity values and these cases are shown in figure 4.18.

Observations from figures 4.18 and 4.19

(i) The sorptivity of the concrete increased with w/c ratio.

(ii) The sorptivity of the concrete increased with the duration of drying.
(iii) The change in sorptivity with duration of drying was found to be related through a square root relationship, as shown in figure 4.19.

(iv) The moisture condition of the concrete was seen to be related to the sorptivity of the concrete.

(v) The 10 mm RH reading was shown to provide the best straight line fit with the Autoclam sorptivity index in all w/c ratio concretes.

The increase in the sorptivity of the concrete with w/c ratio was consistent with generally accepted views. The increased pore volume associated with higher w/c ratio concretes (shown in figure 4.04) is responsible for the absorption of a greater quantity of water. The greater continuity of the pore system (Table 4.02) (Powers et al, 1959) which is a feature of high w/c ratio concretes can be expected to promote the absorption of water to a greater depth in the concrete.

Figure 4.18 and 4.19 show that the sorptivity of the concrete increases with duration of drying. This is due to the loss of moisture from the capillary pores in the concrete which leaves capillary pore space free to absorb moisture into the concrete. The relative humidity and resistance ratio measurements indicated a loss of adsorbed water during the 7 to 14 days period in all concretes whilst the sorptivity of all the concretes increased during this period. The loss of adsorbed moisture can be expected to produce an increased water absorption since the absorbed water will replace any water (either adsorbed to the sides of the pore or contained within it) which has been lost by drying and will leave the capillary pore system substantially saturated. This is in contrast with the lack of increase in air permeability due to the loss of adsorbed moisture outlined above.

Figure 4.19 shows that the sorptivity index of the concrete displayed a linear relationship with the square root of the duration of drying. This finding indicates a more fundamental relationship which exists between weight loss and the sorptivity index which is shown in figure 4.21.
Theoretically a fully saturated concrete (0 days drying) could be expected to display no water absorption and for this reason the linear regressions were forced through the origin in figures 4.20 and 4.21. The straight line relationship between weight loss and sorptivity measurements illustrates the relationships between sorption and desorption in different concretes. As discussed in section 4.4.1.1, the desorption of concrete occurs in drying concretes where the rate of evaporation is thought to be determined or limited by the rate of unsaturated flow of the liquid towards the drying surface (Hall et al, 1984). Since the desorption process is known to have a square root time relationship (Hall et al, 1984) it seems probable that the square root relationship identified in figure 4.19 is caused by desorption. The fact that the 0.45 and 0.55 w/c ratio concretes display a similar rate of increase of sorptivity with increasing weight loss may be an indication that the differences in the continuity and tortuosity of the capillary pores may have been more significant between 0.55 and 0.65 w/c than between 0.45 and 0.55 w/c.

Figure 4.20 shows the relationship between relative humidity measurements and sorptivity for each of the w/c ratio concretes. Theoretically, each relationship could be expected to pass through 100 % RH, 0 sorptivity, as concrete which is saturated could not be expected to exhibit capillary rise. As can be seen, a straight line relationship is evident for all the relative humidity measurements at given depths. The difference in slope between each of the depths, at which relative humidity was measured, is a result of the moisture gradient due to drying in the concrete.

As can be seen in figure 4.20 sorptivity and relative humidity in a 10 mm hole consistently gave the best fit relationship and this would suggest that the depth of penetration of the waterfront from the Autoclam sorptivity test was in the order of 10 mm in each of the tests carried out in
this investigation. It also suggests that for site investigations in drying concrete that a RH measurement at 10 mm may provide a better indication of the moisture in the volume of concrete influenced by Autoclam sorptivity tests.

4.4.2 Moisture in concrete after a storage period

The specimens reported in section 4.4.1 were tested immediately after drying whereas their counterparts were wrapped in polythene sheets and were left in a laboratory for one year. Over this period the resistance between the electrodes embedded in the test blocks was monitored at periods of approximately 3 months. It was expected that this procedure would identify the gradual redistribution of moisture in the concrete and the eventual decay in the moisture gradients.

4.4.2.1 Monitoring of moisture movement in concrete during storage

The moisture movement in concrete was monitored by measuring resistance between the embedded electrodes in the test blocks at intervals of 3 months over a 12 month period. It should be noted that the resistance between embedded electrodes is dependent on the distance between the electrodes (Monfore, 1968). Therefore resistance readings over a 10 mm depth (i.e. 10 - 20 and 25 - 35 mm) were generally greater than those over a 5 mm depth (i.e. 5 -10 and 20 - 25 mm). To aid clarity of trends and to avoid confusion, the readings taken over the 10 mm depth were omitted from figures 4.22 - 4.24. Four sets of graphs numbered a to d are presented in figures 4.22, 4.23 and 4.24 and each refer to one of the four drying durations, 1, 2, 7 and 14 days drying in each of the w/c ratio concretes. Each resistance ratio value is obtained from an arithmetic average of four resistance readings.
Observations from figure 4.22, 4.23 and 4.24

(i) Generally the resistance ratio increased with time spent in storage.
(ii) Comparable resistance ratio values generally increased with increasing w/c ratio.
(iii) The resistance ratio of the concrete was lower with increasing depth from the surface in many specimens, especially those initially dried for 7 and 14 days.

The increase in resistance ratio with time spent in storage indicates a loss of moisture from the capillary pores over the storage period. As explained above, the loss of moisture from capillary pores increases the electrical flow path between the embedded electrodes. The rise in resistance could also be attributed to a change in the structure of the concrete. The resistance of the concrete to the flow of electrical current rises with increased cement paste hydration (Monfore, 1968), therefore a continuation of the hydration process may also be responsible for the rise in resistance ratio over the storage period. The notable exception to the trend of increasing resistance ratio with time of storage is the fall in resistance ratio in the 0.45 w/c ratio specimen (figure 4.22 (d)) close to the drying surface. This indicates a rise in the moisture content of the concrete between the period of 9 and 12 months.

The almost universal rise in resistance ratio of the concrete was unexpected. In concrete, redistribution of moisture could be expected to cause an increase in moisture at a specific level in the concrete which would be offset by a moisture decrease in another volume of the concrete. This would have the effect of producing both increases and decreases in resistance ratio in each concrete specimen. Figures 4.22, 4.23 and 4.24 show, with one exception, that this did not occur. This suggests that excess moisture was used in a continuing hydration reaction in the cement paste or that it escaped from the concrete.
Comparable resistance ratio values generally increased with increasing w/c ratio. The larger increase in resistance ratio evident in the 0.65 w/c concrete can be explained with reference to the loss of moisture from the capillary pores. As stated above, the 0.65 w/c ratio concrete has larger pores and therefore when substantially empty of moisture the larger pores cause a larger increase in the electrical flow path which in turn causes a larger increase in resistance ratio.

The increase in resistance ratio of the concrete with increasing depth from the surface in many specimens indicates that a gradient of either moisture or properties of the cement paste may be present in the concrete. In concrete subjected to drying, a gradient of moisture is inevitable with the concrete closest to the drying surface being drier than concrete at depth, as was shown in 4.4.1. This can be seen in the gradients of resistance ratio with depth evident immediately after the end of the 7 and 14 days drying periods in figures 4.22, 4.23 and 4.24. The resistance gradient due to moisture could be expected to reduce with time as moisture redistribution occurs however as can be seen this is not evident in any of the concrete specimens with the notable exception of 4.16(d). 0.45 and 0.55 w/c ratio specimens dried for 1 and 2 days show an increase in the difference in resistance ratio at different depths.

Gradients of physical properties in the concrete could also be expected to produce a gradient of resistance ratio. Patel et al (1985) showed that there were significant microstructural gradients in drying (over an extended period at room temperature) concrete with the surface region having a lower degree of hydration and a higher volume of large pores. This indicates that the removal of moisture from capillary pores can influence the physical microstructure of concrete.
4.4.2.2 Relative humidity measurements in concrete after storage

The change in relative humidity in the concrete over the storage period is shown in figure 4.25. Figure 4.26 shows the relative humidity values in the concrete before and after the storage period while figures 4.27 to 4.29 show sketches of the moisture gradients in the concrete.

Observations from figures 4.25, 4.26, 4.27, 4.28 and 4.29.
(i) A substantial decrease in relative humidity occurred in the specimens dried for 1 and 2 days.
(ii) The drop in relative humidity over the storage period decreased with the increasing duration of initial drying. Some of the specimens dried for 7 and 14 days showed increases in relative humidity.
(iii) The reduction in relative humidity was greater in the 0.65 w/c ratio concrete.
(iv) Gradients of relative humidity were still evident in most concretes after the storage period.
(v) The 0.65 w/c ratio concrete initially dried for 14 days showed no substantial change over the storage period.

The largest drop in relative humidity over the storage period was evident in the specimens that had most moisture to lose, i.e. the specimens dried for 1 and 2 days. Sketches of the initial and final moisture gradients are presented in figures 4.27, 4.28 and 4.29 and these show that the final relative humidity values fell to 51 - 69 % irrespective of the initial moisture condition. Relative humidity values of 60 - 70% indicate a capillary pore system that is substantially free from water and relative humidity values lower than 60% relative humidity indicates that some adsorbed water has been removed from the walls of the pores (Parrott, 1994b), (section 4.4.1.2). The large loss of moisture from the uppermost 20 mm of the concrete indicates that it is unlikely that the moisture lost from
the pore system is exclusively through hydration especially in the 0.45 w/c ratio concrete (which could be expected to be substantially mature after 3 days water immersion and 14 days laboratory exposure (Powers et al, 1959)).

The other explanation for where this moisture has gone is that it was removed from the concrete. While examining this possibility it was noted that, although the specimens were wrapped in polythene sheet to counter moisture loss during the storage period, the way in which the moisture blocks were wrapped may have allowed the loss of moisture from the concrete. As shown in figure 4.30 the lifting hooks included in the concrete plugs caused a pocket of air to be entrapped. In the equilibrium process between this pocket of air and the concrete moisture may have been removed from the concrete. It should be noted that this wrapping was removed periodically to allow the resistance measurements during the storage period. This would have effectively replaced the moisture laden entrapped air with room air (approximately 60% RH) and more moisture would have been lost from the concrete as the equilibrium process between the concrete and the entrapped air restarted. This was not foreseen in the planning of the experimental programme and may have been responsible for the loss of much of the free moisture from capillary pores in concretes that had free moisture to lose. A similar investigation by Parrott (1994b) used a plastic container to avoid moisture loss (at an elevated temperature), however the container was opened each time relative humidity measurements were taken and no unexplained moisture loss was reported.

The drop in relative humidity over the storage period decreased with the increasing duration of initial drying. This effect can be explained by the observation that the quantity of free moisture that was available to be lost in each of the concretes was different. The specimens which were
dried initially for 1 day had the most moisture while specimens dried for 14 days had least.

The increase in relative humidity in some of the 7 and 14 days drying specimens are all in concretes that had initially lost water adsorbed on the sides of the pore walls. Therefore redistribution of moisture from the lower regions of the concrete appears to have replaced the adsorbed water. The entrapped air noted above could also have provided the moisture responsible for the increase in relative humidity however the increase in relative humidity at the surface was accompanied by a decrease in relative humidity at lower levels in all but one of the cases (0.45 w/c ratio concrete dried for 14 days). The initial relative humidity readings indicate that although most of the free water was removed from the 0.65 w/c ratio concrete dried for 14 days, little adsorbed moisture was removed and, as can be seen from figure 4.29, minimal moisture change was seen to have taken place over the storage period.

Observing the relative humidity gradients (illustrated in sketches in figures 4.27, 4.28 and 4.29) it becomes clear that while most relative humidity values changed over the storage period, only the 0.65 w/c ratio concrete dried for 14 days achieved a similar relative humidity over the 3 depths measured. This suggests that the redistribution of moisture was incomplete after 1 year or that the moisture was lost from the capillary pores (either through hydration or from the concrete). It can also be seen that the shape of final humidity gradients commonly consists of lower relative humidity at the surface and a greater RH at depth which again may indicate a loss of moisture from the concrete surface or a lack of redistribution of moisture from depth.

The general trends in figures 4.27, 4.28 and 4.29 show that the 10 and 20 mm RH values became more uniform during the storage period showing that some redistribution of moisture did occur, however the
difference in RH between the surface and 10 mm often remained significant. This may indicate a concrete that is losing moisture close to the surface while the bulk of the concrete (i.e. 10 and 20 mm measurements) has a substantially uniform distribution of moisture. The relative humidity gradients also show quite clearly that the loss of moisture from the specimens subjected to 1 and 2 days duration of drying was quite substantial. Further, there is no indication of this moisture relocating in the concrete and the obvious conclusion was that this moisture was lost from the capillary pore system (either used by a hydration process or lost from the concrete). The concrete dried for 7 and 14 days showed some moisture distribution with moisture gains and losses indicated in most specimens.

4.4.2.3 Conductance / resistance measurements in concrete after storage

Each resistance value is the arithmetic average of four resistance readings (from two moisture measurement blocks). From these results resistance ratios were calculated with the initial value (Ri) taken as the resistance value after drying and the final value (Rt) taken as the resistance value obtained after the 12 months storage period. Thus figures 4.31 and 4.32 illustrate the changes in resistance measurements during the storage period.

Figure 4.31 shows the resistance ratio variation for the different durations of drying and figure 4.32 shows the gradients of resistance ratio in the concretes after the storage period.

Observations from figures 4.31 and 4.32

(i) All resistance ratio values are in excess of 1.
(ii) The 0.65 w/c ratio concretes show an increase in resistance ratio values between 1 and 2 days duration drying and a subsequent decrease in resistance ratio values for longer periods of drying.

(iii) The 0.45 and 0.55 w/c ratio concretes show a large decrease in resistance ratio between 7 and 14 days duration of initial drying.

(iv) Pronounced gradients of resistance ratio are evident in the 0.45 and 0.55 specimens which were dried for 14 days.

The observation that all the resistance ratio values recorded were in excess of 1 indicates that a loss of moisture occurred from the capillary pores in the concrete during the storage period. This is not what could have been expected as the redistribution of moisture in the concrete pores would involve the movement of moisture from areas with more moisture to areas of less moisture thus giving a number of resistance ratio readings of less than 1. As discussed in 4.4.2.2 in relation to the relative humidity results, the loss of moisture from the specimens dried for 1 and 2 days was large. However the concrete dried for 7 and 14 days did show an increase in relative humidity close to the surface. This increase in relative humidity could have been expected to have produced resistance ratio results lower than 1, which was not the case. This indicates that the decrease in resistance ratio caused by the increased moisture was offset by another factor. As noted in 4.4.2.1, the resistance of the concrete to the flow of electrical current rises with increased cement paste hydration (Monfore, 1968) therefore the resistance ratio of concrete that had continued hydration during storage could be expected to rise. This may well have offset the reduction in the resistance ratio of the concrete caused by the loss of moisture from the capillary pores.

Considering that the resistance of a concrete is influenced by the nature of the concrete as well as the moisture condition of its pores and relative humidity is influenced by the moisture in the concretes pores
alone, it follows that in regions of the concrete where the RH and resistance ratio give conflicting results that an alteration to the structure of the concrete occurred. This does not quantify the amount of hydration that occurred nor does it preclude the continued hydration of concrete in other areas of the concrete. However, it does show that modification of the structure of the concrete may well have occurred in some of the concrete. One could expect that hydration would occur preferentially in the 0.65 w/c ratio concrete in the concretes which had the greatest quantity of free water (Powers, 1958) therefore it would be reasonable to presume that further hydration occurred to some extent in most concrete specimens.

The 0.65 w/c ratio concretes show an increase in resistance ratio values between 1 and 2 days duration drying. The reason for this is not clear however it may be partially caused by the marginally larger loss of moisture shown by the specimen that was dried for 2 days (shown in figure 4.29). The reduction in resistance ratio values seen in the 0.65 w/c concrete (figure 4.31) decreased with increasing duration of initial drying in excess of 2 days. This may be due to increased hydration of the concrete in the capillary pore systems which had more free moisture, i.e. the 0.65 w/c ratio concrete dried for 1 and 2 days. It may also be a reflection of the fact that over the storage period there was no significant increase in moisture in the 0.65 w/c ratio concrete dried for 7 and 14 days, at 10 mm depth (as shown in figure 4.29). This is in contrast to the increases in RH identified in the corresponding 0.45 and 0.55 w/c ratio concretes.

Figure 4.31 shows that the 0.45 and 0.55 w/c ratio concretes exhibit a large decrease in resistance ratio between concretes dried for 7 and 14 days initial drying between the 5 and 10 mm deep electrodes. This can be linked to the significant increases (15.5 and 13.7% increases for 0.45 and 0.55 w/c ratio concretes respectively) in RH in the 10 mm hole shown in
the specimens dried for 14 days. This increase in moisture could be expected to reduce the resistance ratio as explained above.

The increase in moisture over the storage period in the 0.45 and 0.55 w/c specimens at 10 mm (dried for 14 days) is also responsible for the large gradients of resistance ratio evident between the 5 - 10 mm electrode pairs in the corresponding concretes (See figure 4.32). The orientation of the other resistance ratio gradients are roughly vertical with no discernible trends distinguishable. The resistance ratio values were generally lower in concretes that were dried for longer periods (this is not an absolute rule) and is probably a reflection of the amount of moisture in the concrete at the start of the storage period. The concretes with more moisture to lose generally lost it with a resulting increase in resistance ratio. Any hydration of the concrete could be expected to disturb these trends since, as discussed previously, the hydration of the concrete also causes an increase in the resistance ratio.

4.4.2.4. Autoclam air permeability of concrete after storage

The air permeability values (figures 4.33 and 4.34) are the average of three Autoclam measurements except where indicated. Again, outlying results were omitted from the average value. Figures 4.33 and 4.34 show the air permeability indices after storage and the change in air permeability over the storage period, respectively.

Observations from figures 4.33 and 4.34

(i) The 0.65 w/c concrete displayed the largest air permeability with the air permeability decreasing with decreasing w/c ratio of the concrete.
(ii) The air permeability of the concrete generally increased over the storage period.
The effect of the initial duration of drying was evident in the air permeability of 0.45 and 0.55 w/c ratio concrete, while this was not clear in the 0.65 w/c ratio concrete.

The results (figure 4.33) show that the air permeability of the concrete was greatest for the 0.65 w/c specimens and lowest for the 0.45 w/c concrete, as could be expected. This finding was also evident in 4.4.1.4 (figure 4.14) and it is discussed in that section.

The 0.55 w/c ratio concrete shows an increase in air permeability with the drying duration. Since the relative humidity measurements show no large differences (varied between 51 - 65%) in the final moisture condition of all the concretes, the increase in air permeability with the duration of drying must be caused by the concretes microstructure. The concrete with most moisture in the capillary pores may well have hydrated to a greater extent causing a reduction in the permeability which partially offset the increase in permeability due to the loss of the moisture from the capillary pores. The greatest hydration could be expected to take place in the concrete with the most moisture initially, therefore the lower permeability could be expected to be evident in the concrete dried for 1 day as was noted in figure 4.33.

The 0.45 w/c ratio concrete dried for 14 days showed a lower than expected air permeability in figure 4.33 and figure 4.34 shows that the air permeability of the concrete decreased slightly over the storage period. This again may be, at least, partially due to the moisture change and hydration effects over the storage period. The 0.45 w/c concrete dried for 14 days gained moisture (Figure 4.27) at the surface (8%) and at 10 mm (15.5%) which may have reduced the air permeability slightly. The 0.45 w/c ratio concrete could be expected to hydrate very little since full hydration could be expected to occur in the preliminary conditioning period prior to drying (Powers, 1958). Also the effect of moisture on the air
permeability could be expected to be small in the 60 - 65 % range in a 0.45 w/c ratio concrete rather than negligible as in a higher w/c ratio concrete (Parrott, 1994b).

The 0.65 w/c ratio concrete showed no increase in air permeability as a result of the duration of initial drying in excess of 2 days. This finding mirrors the findings of the air permeability tests carried out immediately after the initial drying (section 4.4.1.4). The change in air permeability over the storage period (figure 4.34) in the 0.65 w/c ratio concrete shows a small decrease in permeability in the concrete dried for 1 day and substantial increases in permeability for all other durations of drying. As the concrete dried for 1 day lost a substantial quantity of moisture during the storage period (figures 4.25 and 4.29), the increase in air permeability associated with this loss of moisture may well have been offset by the decrease in permeability associated with the hydration. In the concrete dried for 2 and 7 days the loss of moisture may have produced the increase in air permeability over the storage period evident in figure 4.34. The 0.65 w/c ratio concrete dried for 2 days could be expected to experience a similar amount of hydration as the concrete dried for 1 day since the moisture profiles both before and after storage are similar, however this seems not to have taken place. Also the increase in air permeability (figure 4.34) of the 0.65 w/c ratio concrete dried for 14 days is not due to moisture loss as no significant change in moisture occurred in the concrete over the storage period (figure 4.29). No explanation for this increase is manifest.

4.4.2.5. Autoclam sorptivity of concrete after storage

The sorptivity indices presented in figure 4.35 are the arithmetic average of three Autoclam tests except where indicated. Outlying results were not included in the average. The change in sorptivity index over the storage period is shown in figure 4.36.
Observations from figures 4.35 and 4.36

(i) The sorptivity index increased with increasing duration of initial drying.

(ii) The expected trend of increasing sorptivity index with increasing w/c ratio of the concrete was not evident in figure 4.35.

(iii) Changes in sorptivity of the concrete over the storage period showed no discernible absolute trend.

The sorptivity index increased with increased drying time (figure 4.35). This trend was identified in section 4.4.1.5 and it was considered a result of the free moisture in the capillary pores. After drying, however, there was little difference in humidity between specimens of a similar w/c ratio and hence the differences in sorptivity after the storage period may be due to hydration during the storage period. The greatest hydration could be expected to take place in the 0.65 w/c ratio concrete dried for 1 day with the least hydration in the 0.45 w/c ratio concrete dried for 14 days. The effect of hydration alone is unclear as the sorptivity of the concrete is also influenced by the loss of moisture from the capillary pores in the concrete.

The interrelationship between the sorptivity indices in the different w/c concretes shown in figure 4.18 is not evident after the period of storage (figure 4.35), i.e. a 0.45 w/c ratio concrete dried for a specified period did not always exhibit a lower sorptivity than the 0.55 w/c ratio concrete dried for a similar period. The differences in moisture contents (ranging from 51.2 - 68.5% RH) between the concrete specimens could be expected to influence the sorptivity indices as shown in figure 4.20 (unlike air permeability). This, along with the twin effects of moisture loss and continued hydration may be responsible for the lack of this trend in the sorptivity index results after the storage period.

The sorptivity indices and relative humidity measurements after storage are shown to exhibit a range of sorptivity indices for similar
relative humidities (figure 4.37). The graph suggests that concrete with a similar w/c ratio and similar moisture content can give different sorptivity indices. This may be true only if the concrete pore structures have been influenced by different moisture and drying histories.

No clear trend in the change of sorptivity over the storage period was evident as several of the concretes showed a decrease in sorptivity over the storage period whilst some showed a modest increase. As the relative humidity measurements indicated that the pore system was substantially free from moisture after the storage period the decrease in sorptivity is another indicator that hydration during the storage period changed the physical characteristics of some concretes.
4.5 Summary of findings

The major findings of the investigation are summarised below:

(i) It was shown that the measurement of the electrical resistance between embedded electrodes was most sensitive to changes in moisture adsorbed onto the walls of the pores whereas relative humidity was more sensitive to the moisture in smaller pores in the concrete (pores of approximately 0.2 μm diameter and smaller).

(ii) The different moisture gradients investigated in this experiment were identified by relative humidity measurements at three depths and by resistance measurements between embedded electrodes. The effect of each of the gradients on the Autoclam permeation properties was identified and the drying regime categorised in terms of change in sorptivity and air permeability in three w/c concretes.

(iii) Air permeability indices were shown to fit the general trend of Parrott's air permeability versus relative humidity graph. A straight line relationship between sorptivity and internal and surface relative humidity was identified from the experimental results.

(iv) The relative humidity measurements and the relationship between relative humidity and permeation properties was described with reference to Kelvin's equation.

(v) Water adsorbed on the walls of the capillary pores was found to influence the sorptivity of concrete whereas the adsorbed moisture had no significant influence on the air permeability indices.

(vi) Relative humidity measurement in a 10 mm hole was found to be the most suitable for comparison with Autoclam sorptivity indices.
(vii) The redistribution of moisture in the concrete over a period of unheated storage was shown to be only partially successful with the loss of most of the free moisture from the capillary pores.

(viii) The effect of the initial drying period was not evident in the moisture measurements after one year's storage.

(ix) The moisture history of the concrete specimens was shown to influence the Autoclam measurements after a one year storage period.
Table 4.01 - Experimental variables

<table>
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<th>Variable</th>
<th>Levels</th>
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<tr>
<td>W/C ratio</td>
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<tr>
<td>Moisture conditions</td>
<td>Saturated + 1, 2, 7, 14 days drying</td>
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<tr>
<td>Moisture distribution</td>
<td>Uniform, Gradient</td>
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Table 4.02 - Relative Humidity for different pore size. (at 20°C)

<table>
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<th>Relative Humidity (%)</th>
<th>Radius of Curvature (µm)</th>
<th>Pore diameter capable of sustaining meniscus</th>
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</thead>
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<tr>
<td>100</td>
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<td>∞</td>
</tr>
<tr>
<td>99.999</td>
<td>109.3896</td>
<td>218.8 µm</td>
</tr>
<tr>
<td>99.9</td>
<td>1.09</td>
<td>2.18 µm</td>
</tr>
<tr>
<td>95</td>
<td>0.0213</td>
<td>426 Å</td>
</tr>
<tr>
<td>90</td>
<td>0.0104</td>
<td>208 Å</td>
</tr>
<tr>
<td>85</td>
<td>6.731 E-3</td>
<td>135 Å</td>
</tr>
<tr>
<td>80</td>
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<td>51 Å</td>
</tr>
<tr>
<td>60</td>
<td>2.141 E-3</td>
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</tr>
</tbody>
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Plate 4.01 - Moisture measurement block moulds

Plate 4.02 - Specimens in drying cabinet
Concrete specimen

Test (cast) face

Specimen raised from base of the water bath

Saturation of the concrete through cast test face of specimen

Water level increased daily over a period of 3 days

Concrete saturated after 3 days

Figure 4.01 - Incremental saturation of concrete specimens.
Electrodes covered in heatshrink
20 mm bare electrode in centre

5 mm
10 mm
5 mm
10 mm
5 mm

Aluminium alloy void formers
for 10 and 20 mm deep holes

Film faced plywood mould

Concrete 'plugs'

Test face / Drying surface

Embedded electrodes

Five non test surfaces painted with epoxy coating

Test Block - dimensions 100 X 80 X 80 mm

Figure 4.02 - Moisture measurement block details
Figure 4.03 - Percentage weight loss with duration of drying
Figure 4.04 - Effect of w/c ratio on the cumulative pore size distributions of hardened cement pastes. Nyame, (1979).
Figure 4.05 - Weight Loss versus square root of duration of drying.
Figure 4.06 - Relative humidity variation in concrete for different durations of drying.
Figure 4.07 - Relative humidity measurement in concrete.
Figure 4.08 - Effect of w/c ratio on the differential pore size distributions of hardened cement paste. Nyame, (1979).
Evaporation of moisture from concrete surface

For volume A
Moisture evaporated = Moisture crossing A-B boundary

For volume B
Moisture crossing A-B boundary > Moisture crossing B-C boundary.

Figure 4.09 - Moisture equilibrium in concrete near surface
Figure 4.10 - Relative Humidity gradients in concrete for different durations of drying.
Figure 4.11 - Resistance Ratio variation with time drying.
Figure 4.12 - Resistance ratio gradients due to drying.
Figure 4.13 - Comparison of resistance ratio and RH readings in drying concrete.
Figure 4.14 - Air permeability indices with duration of drying
Figure 4.15 - Effect of relative humidity upon permeation data. Permeability values normalised by dividing by the corresponding to 60% RH. With added data. Revised from Parrott (1994b).
Figure 4.16 - Air Permeability indices versus RH.
Figure 4.17 - Air Permeability and relative humidity in drying concrete. Nagataki et al (1986).
Figure 4.18 - Sorptivity indices with the duration of drying.

Figure 4.19 - Sorptivity Index with root duration of drying
Figure 4.20 - Sorptivity Index results with relative humidity
Figure 4.21 - Sorptivity Index with Weight loss
Figure 4.22 - 0.45 w/c ratio concrete - Resistance ratio variation during storage period.
Figure 4.23 - 0.55 w/c ratio concrete - Resistance ratio variation during storage period.
Figure 4.24 - 0.65 w/c ratio concrete - Resistance ratio variation during storage period.
Note: Negative relative humidity = a drop in relative humidity over storage period

(A) 0.45 w/c concrete

(B) 0.55 w/c concrete

(C) 0.65 w/c concrete

**Figure 4.25 - Change in relative humidity over storage period**
Figure 4.26 - Relative humidity with duration of initial drying. Before and after storage period.
Figure 4.27 - Changes in moisture gradients in 0.45 w/c ratio concrete over storage period. Moisture gradient sketches.
Figure 4.28 - Changes in moisture gradients in 0.55 w/c ratio concrete over storage period. Moisture gradient sketches.
Figure 4.29 - Changes in moisture gradients in 0.65 w/c ratio concrete over storage period. Moisture gradient sketches.
Section through a wrapped concrete moisture measurement block

Figure 4.30 - Moisture measurement block during storage.
Figure 4.31 - Resistance ratio variation at the end of the storage period
0.45 w/c concrete

0.55 w/c concrete

0.65 w/c concrete

Figure 4.32 - Resistance ratio moisture gradients due to drying after storage period.
Change in Air Permeability Index

![Graph showing Air Permeability indices after storage period.](image)

**Figure 4.33 - Air Permeability indices after storage period.**

![Graph showing change in air permeability index over storage period.](image)

**Figure 4.34 - Change in air permeability index over storage period**

- 0.45 w/c concrete
- 0.55 w/c concrete
- 0.65 w/c concrete

Note: Negative value = reduction in air permeability over storage period

* Denotes average of two tests
Figure 4.35 - Sorptivity indices after storage period.

Figure 4.36 - Change in Sorptivity over drying period
Relative Humidity (%)

RH @ 0 mm $y = 59.876 - 2.7561x$ $R^2 = 0.355$
RH @ 10 mm $y = 64.802 - 2.4434x$ $R^2 = 0.989$
RH @ 20 mm $y = 65.397 - 0.14341x$ $R^2 = 0.015$

\(JL\) ~

(A) 0.45 w/c Concrete    Sorptivity Index (Cu.M/Sqr(mins))

RH @ 0 mm $y = 51.579 + 1.4627x$ $R^2 = 0.570$
RH @ 10 mm $y = 59.702 - 0.51389x$ $R^2 = 0.027$
RH @ 20 mm $y = 66.452 - 2.5322x$ $R^2 = 0.890$

(B) 0.55 w/c concrete    Sorptivity Index (Cu.M/Sqr(mins))

RH @ 0 mm $y = 61.042 - 1.8541x$ $R^2 = 0.552$
RH @ 10 mm $y = 67.695 - 2.8955x$ $R^2 = 0.704$
RH @ 20 mm $y = 69.020 - 2.5831x$ $R^2 = 0.814$

(C) 0.65 w/c concrete    Sorptivity Index (Cu.M/Sqr(mins))

Figure 4.37 - Sorptivity indices after storage versus relative humidity.
5. Outdoor exposure trials

5.1 Introduction

The temperature and the moisture condition of the concrete at the time of an in-situ permeation test are highly variable and dependent on ambient weather and exposure conditions. This chapter considers the feasibility of accounting for the influence of environmental factors from in-situ permeation measurements, by investigating:

(i) The effect of ambient temperature on permeation properties.
(ii) The effect of moisture on permeation properties.

The results of outdoor exposure trials are used to satisfy the above objectives. To assist in the interpretation of results, relevant laboratory studies were carried out and these results are presented in the following section.

5.2 The effect of temperature on Autoclam measurements

5.2.1 The effect of temperature on air permeability

The effect of temperature on the flow of air though an ideal concrete pore is examined in this section and the theoretical trend observed is compared with the results of a short series of tests.
5.2.1.1 Theoretical study of the effect of temperature on air permeability

The permeability of air through concrete is generally held to be described by Darcy's equation

\[
Q = A \frac{-k \partial p}{\mu} \frac{\partial l}{l}
\]

Where \(Q\) is the flow rate (m\(^3\)/s),
\(A\) is the cross sectional area (m\(^2\)),
\(\mu\) is the viscosity of air (N.s/m\(^2\)),
\(k\) is the intrinsic permeability (m\(^2\)) and
\(\partial p\) is pressure loss over the flow path of length \(\partial l\).

Adopting a single cylindrical capillary pore model \([5.01]\) may be rewritten as

\[
Q = -k \pi r^2 \frac{\Delta P}{\mu L}
\]

where \(\Delta P\) is the change in pressure over the length of the pore,
\(L\) is the length of the pore (m) and
\(r\) is the radius of the pore (m).

The effect of temperature on the rate of air-flow through concrete can be estimated by determining the change in variables with temperature. In an Autoclam site test the overpressure pressure is measured on the basis of a pressure transducer reading which is at thermal equilibrium with its surroundings. If it is accepted that the temperature is constant during the 15 minute Autoclam test then the pressure term \(\Delta P\) will not be influenced by temperature.

A change in temperature will alter the viscosity of the air (values are given table 5.01). Typical values for the variables in equation \([5.02]\) were selected (Table 5.02) and the resulting relationship between air flow and temperature is shown in figure 5.01. Figure 5.01 shows the trend that
the air flow through a single ideal capillary pore, under a pressure head, could be expected to decrease with increasing temperature.

The relationship can be described by a linear equation given in [5.03]

\[
\text{Rate of air flow} = 1.466 \times 10^{-16} - 3.756 \times 10^{-19} (T)
\]

Eqn. 5.03

Where \( T \) is the temperature of the system and the rate of air flow is in an ideal capillary pore of specific dimensions.

This indicates that an increase in temperature will result in a modest decrease in the rate of air flow through the concrete. The difference in air flow between the temperatures of 0 and 30 °C is in the order of 8%. This indicates that the variation of the air permeability of concrete with temperature may not be significant when compared to the variability of the concrete (and the resultant variability in permeability).

5.2.1.2 Experimental study of air permeability variation with temperature

(i) Aim of test

A short series of tests was undertaken to verify the effect of temperature on Autoclam air permeability tests.

(ii) Description of test

18 Autoclam test slabs of 230 X 230 X 75 mm deep were cast - 9 No. X 0.45 w/c ratio slabs and 9 No. X 0.65 w/c ratio slabs. The procedure for casting is given in chapter 3. The slabs were cured for 3 days in water after which they were stored in the laboratory until 28 days old. The specimens were then dried at 105 °C for 3 days to remove free water from all capillary pores. The slabs were cooled to 20 °C and then conditioned to different test temperatures over a 3 day period. An Autoclam air permeability test was then carried out at this temperature.
(iii) Conclusions from test

Figure 5.02 shows the results from these tests. The air permeability indices quoted are the arithmetic average of three Autoclam air permeability tests. As can be seen, there was little difference in the air permeability indices when tested at different temperatures. The slight decrease shown in the air permeability indices of both concretes, with the increase in temperature, is in accordance with the theoretical prediction in section 5.2.1.1. It should be noted, however, that these results are not significantly different and the decrease in air permeability noted in figure 5.02 could also be attributed to specimen to specimen variability. This variability may be the cause of the 20 °C air permeability index which was lower than expected. Therefore, the effect of temperature on Autoclam air permeability indices was found not to be significant. The results of a similar investigation by Bungey et al (1989) (shown in figure 2.31) into the effect of temperature on Figg air permeability tests indicated ' that the Figg test is not significantly affected by the temperature of testing' over a temperature range of 20 to 50 °C.

5.2.2 The effect of temperature on absorption

5.2.2.1 Theoretical study of water absorption variation with temperature

Using the Poiseuille equation (also known as the Hagen-Poiseuille equation) (Scheidegger, 1963), the rate of capillary flow through a straight capillary pore can be expressed as:

$$Q = \frac{\pi r^4}{8 \eta} \frac{dp}{dx}$$  \hspace{1cm} \text{Eqn. 5.04}

Where $Q$ is the flow rate (m$^3$/s),
\( \frac{dp}{dx} \) is the pressure gradient over the length of the filled pore, 

\( r \) is the radius of the capillary (m) and 

\( \eta \) is the viscosity (Ns/m²).

If the pressure head is \( H \) (in m) and the length of the capillary pore filled with water is \( L \) (in m), then \( \frac{H}{L} \) can be used to replace \( \frac{dp}{dx} \):

\[
\therefore Q = \frac{\pi r^4 H}{8 \eta L}
\]

Eqn. 5.05

Where \( Q \) is the flow rate of absorbed water (m³/s).

If it is taken that the total head (\( H \)) resulting from the Autoclam sorptivity test consists of

(i) \( 0.2 \) m applied pressure head (\( H_a \)).

(ii) Head resulting from capillary forces (\( H_c \)).

Therefore,

\[
H = H_a + H_c
\]

Eqn. 5.06

Where, \( H_a = 0.2 \) m,

and,

\[
H_c = \frac{2 \gamma \cos \theta}{g \rho r}
\]

Eqn. 5.07

(Jouren Equation (C.f. Chapter 2)).

Where \( g \) is acceleration due to gravity (taken as 9.81 m/s²),

\( \rho \) is the density of the pore fluid (Kg/m³),

\( \gamma \) is the surface tension of the pore fluid, (N/m) and

\( \theta \) is the angle of contact between the pore wall and the pore fluid (taken as 0).

Thus, it is possible to express the total head resulting from the Autoclam sorptivity test as
\[ H = 0.2 + \left( \frac{2 \cdot \gamma \cdot \cos \theta}{g \cdot \rho \cdot r} \right) \]  
\text{Eqn. 5.08}

Substituting for \( H \) in equation [5.02] gives the quantity of moisture flow in an ideal capillary pore resulting from an Autoclam sorptivity test,

\[ Q = \frac{\pi \cdot r^4}{8 \cdot \eta} \left( 0.2 + \frac{2 \cdot \gamma \cdot \cos \theta}{g \cdot \rho \cdot r} \right) \]  
\text{Eqn. 5.09}

Sorptivity is proportional to the flow rate of absorbed water Hall, (1981)

\[ i = S \sqrt{t} \]  
\text{Eqn. 5.10}

Where \( i \) is the cumulative absorbed volume per unit area of the inflow surface, \( S \) is the sorptivity of the material and \( t \) is the elapsed time. Therefore [5.09] can be rewritten as:

\[ S \propto \frac{\pi \cdot r^4}{8 \cdot \eta} \left( 0.2 + \frac{2 \cdot \gamma \cdot \cos \theta}{g \cdot \rho \cdot r} \right) \]  
\text{Eqn. 5.11}

The effect of temperature on sorptivity can be determined by a consideration of the effect of temperature on each of the factors in equation [5.11]. Assumptions relevant to this case which are additional to those stated in chapter 2 for the Jouren and Poiseuille equations are

(i) The effect of ions in the absorbed water is neglected.

(ii) The angle of contact between the pore wall and the absorbed water does not change with temperature (taken as 0).

(iii) The effect of lateral spread which occurs in the Autoclam sorptivity test is not considered.

Given the above assumptions, the following three physical properties could be expected to change with temperature

(i) The density of the absorbed water.

(ii) The viscosity of the absorbed water.
(iii) The surface tension of the water in contact with the air.

Table 5.03 shows published values of the change in the physical properties of water. Using values for variables from table 5.03 the change in absorption flow for an ideal capillary pore may be estimated using equation [5.11]. For a typical capillary radius of 2.0 E-6 m and length of water-filled pore of 2.0 E-3 m figure 5.03 shows the change in the rate of flow (Q) with change in temperature. Equation [5.12] describes this variation and the relationship is plotted in figure 5.03.

\[
\text{Flow rate} = 1.419 \times 10^{-14} + 4.236 \times 10^{-16} (T) + 2.625 \times 10^{-18} (T^2) \quad \text{Eqn 5.12}
\]

Where the flow rate is the rate of absorption flow in an ideal capillary, and T is the temperature (°C) of the system.

Figure 5.03 indicates that an increase in temperature will result in an increase in the rate of absorption flow into the concrete. The difference in absorption flow between the temperatures of 0 and 30 °C is in the order of 100% (from figure 5.03). This indicates that the effect of temperature on the sorptivity of concrete may be significant.

5.2.2.2 Experimental study of water absorption variation with temperature

(i) Aim of test

A short series of tests was undertaken to verify the effect of temperature on Autoclam sorptivity tests.

(ii) Description of test

The 18 Autoclam test slabs used for the air permeability investigation (section 5.2.1.2) of 230 X 230 X 75 mm deep were used in this investigation - 9 No. X 0.45 w/c ratio slabs and 9 No. X 0.65 w/c ratio slabs. The procedure and details of the manufacture are given in chapter 3. The slabs were conditioned for 3 days in water, after which they were stored in
the laboratory until day 28. The specimens were then dried at 105 °C for 3 days which is considered to result in a capillary pore system unobstructed by free moisture. Three slabs of each w/c ratio concrete were then conditioned to 10 °C, 20 °C and 30 °C over a 3 day period. Autoclam sorptivity tests were carried out at the respective temperature. Care was taken to keep the water used in the test at the same temperature as the test slab.

(iii) Conclusions from test

Figure 5.04 shows the results of the Autoclam sorptivity tests. The increase in sorptivity with an increase in temperature is similar to the theoretically determined relationship above. Overall the results confirm the findings of the theoretical investigation shown in section 5.2.2.1. as the effect of temperature can be seen to be significant. The results of a set of tests by Bungey et al (1989) show a similar increase in ISAT results with increasing temperature (Figure 2.32).

The increase in sorptivity with increasing temperature displayed by the 0.65 w/c ratio concrete is larger than the increase displayed by the 0.45 w/c ratio concrete (figure 5.04). This may indicate that the quantity of water flowing into the concrete may be related to the effect of temperature. Bungey et al (1989) showed that the moisture condition of the concrete influenced the effect of temperature on ISAT sorptivity measurements (figure 2.32). Since the moisture condition, like the w/c ratio, of a concrete influences the quantity of water flowing into the concrete, this may indicate that the effect of temperature on the sorptivity measurements may be linked to the amount of water being absorbed. The theoretical study into the effect of temperature (section 5.2.2.1) on sorptivity indicates that the variables which were considered to be temperature dependent were all associated with the water which was being absorbed. This again lends credence to the theory that the effect of temperature on sorptivity
measurements becomes more significant as the quantity of water being absorbed increases.

5.2.2.3 Development of a temperature correction process for Autoclam sorptivity tests

The theoretical relationship [5.11] between absorption moisture flow and temperature was used to develop a temperature correction procedure for Autoclam sorptivity measurements.

\[
\text{Flow rate} = 1.419 \times 10^{-14} + 4.236 \times 10^{-16} (T) + 2.625 \times 10^{-18} (T^2) \quad \text{Eqn 5.12}
\]

Factors such as the lateral spread of the wetted region and the geometry of the finite water source, which are associated with in-situ sorptivity tests such as the Autoclam and ISAT tests, were examined by Hall, (1981). He found that the sorptivity index obtained in the ISAT test was related to the absorption flow rate despite these factors. Using the fact that sorptivity is proportional to the flow rate (Hall, 1981) a correction factor calculated on the basis of absorption flow rates is applicable to sorptivity measurements. Using 20 °C as a standard temperature, the rate of absorption flow at other temperatures was related to the flow at 20 °C to give a temperature correction factor. The resultant correction graph is shown in figure 5.05 and typical values are given in table 5.04. The correction factor can be expressed as:

\[
\text{CF}_{20} = 1.6385 - 3.8834 \times 10^{-2} (T) + 3.5132 \times 10^{-4} (T^2) \quad \text{Eqn. 5.13}
\]

Where \( \text{CF}_{20} \) is a correction factor which converts both the rate of absorption flow and the sorptivity index at a given temperature to the equivalent temperature at 20 °C and \( T \) is the temperature of the system (°C).
5.2.2.4 Application of the correction factor for temperature effect on sorptivity measurements

Figure 5.06 shows the results from a study on the effect of temperature on Autoclam sorptivity measurements (Basheer, 1992). The measurements were carried out in an environmental chamber and illustrate the influence of temperature on Autoclam sorptivity measurements, similar to the results presented in figure 5.04. The specimens were subjected to a standard 3 day 105 °C conditioning prior to the test and the temperature conditioning was carried out at a RH of 55 ± 5%. The 20 °C correction factor [5.13] was used to transform the data to equivalent sorptivity indices at 20 °C (figure 5.07). It may be noted that the variation of sorptivity index for each temperature from the average value for all three temperatures is within the usual variation which is expected from any test on concrete. Therefore the correction factor may be considered to have eliminated the effect of temperature on the Autoclam sorptivity index.

5.2.2.5 The effect of ambient temperature and internal RH on Autoclam sorptivity measurements

Figure 5.08 shows the results from an investigation (Basheer, 1992) which studied the influence of ambient temperature and RH on the Autoclam sorptivity index of a 0.55 w/c ratio concrete. The specimens were initially dried at 105 °C for three days, wrapped in plastic and allowed to cool prior to three days conditioning at the appropriate temperatures and RH values. The concrete which was conditioned to 80 % RH shows little variation in sorptivity index with temperature while the concrete which was conditioned to 40% RH shows a much larger increase in sorptivity with increasing temperature. These findings are consistent with Bungey et al (1989) who found a similar trend in ISAT sorptivity
measurements between 20 and 50 °C (figure 2.32). This adds further supporting evidence to the argument that the effect of temperature on sorptivity is related to the quantity of water being absorbed (discussed in section 5.2.2.2).

5.3 Autoclam measurements on exposed concrete

5.3.1 Objectives
The main objectives of the investigation were:
(i) Measure and monitor the fluctuations of moisture in concrete exposed to an outdoor condition over an extended period.
(ii) Establish the influence of changing moisture conditions in concrete on the measured permeation properties.
(iii) Develop a procedure to eliminate the influence of temperature and moisture on Autoclam air permeability and sorptivity measurements for tests carried out on site.

5.3.2 Experimental programme

5.3.2.1 Experimental variables
The variables in the experiment are shown in table 5.04.

(i) W/C ratio
The permeability of concrete is influenced by many different factors, and in this experiment w/c ratio was chosen to provide concretes of different quality for the experiment. Three concretes of varying quality were produced using w/c ratios of 0.45, 0.55 and 0.65 w/c. The concrete constituents, mix procedure and quality control procedures are detailed in chapter 3.
(ii) Exposure conditions

The exposure conditions were initially chosen to be exposed and sheltered however experience of the testing regime showed that in reality the actual exposure conditions could be described more correctly as exposed and partially exposed. The specimens were left outside (plate 5.01) with the exposed side facing West-North-West, and the sheltered side of the specimens facing the wall (3 storeys in height) of the David Keir Building, Stranmillis Rd. Belfast (approx. Lat. 54° 39 N, Long. 6° 12' W). The specimens were kept approximately 1 m from the building (plates 5.01 and 5.02). After a period of 4 months it was noticed that the winter sun did not reach the slabs therefore they were moved to the centre of the 'quad', keeping the orientation and distance from the wall similar. Both of these exposure sites are shown in figure 5.09 as sites 1 and 2 respectively.

5.3.2.2 Specimen details

The use of large scale test specimens was considered appropriate in an experimental series which sought to provide data which was relevant to in-situ structures. In total, three 450 X 200 X 500 mm slabs were cast (figure 5.10), one slab made from each w/c ratio concrete. The concrete was compacted using a poker vibrator, as detailed in chapter 3.

Figure 5.10 illustrates that the stainless steel electrodes were cast into the slab at different depths from the surface to allow the measurement of resistance variation in the concrete with depth (plate 5.03). The electrodes were sleeved except for a length of 20 mm at the tip (figure 3.09). Themistors were also embedded at different depths from each test face to measure the temperature variation with depth (C.f. chapter 3). The edge areas of the slab were not used for testing (figure 5.10) as the concrete in these areas is recognised not to be representative of the slab as a whole (Dewar, 1985).
5.3.3 Curing and conditioning

The specimens were struck from the formwork 18 hours after casting and were subjected to the following curing and conditioning regime:

(i) After the formwork was stripped, the specimens were cured in damp hessian for 3 days. The hessian was wetted twice each day during this period.

(ii) On day 4 the specimens were removed to a constant temperature laboratory (20° C ± 2 °C) and stored for approximately 28 days.

(iii) The specimens were moved to the outdoor exposure site at the age of 28 days.

5.3.4 Tests methods

The following tests were carried out on each of the test specimens, each month, for a period of 18 months. The concrete specimens were tested on 3 consecutive days each month. The 0.55 w/c ratio concrete specimen was tested on the first day, the 0.45 w/c ratio specimen on the second day and the 0.65 specimen tested on the third day.

(i) Temperature measurements using embedded thermistors at 5, 15 and 25 mm depth (section 3.3.4.1).

(ii) Conductance measurements between embedded electrodes (Section 3.3.3.2).

(iii) RH measurements at the surface and at 10 mm depth (detailed in section 3.3.2.3).

(iv) 'Autoclam' air permeability and sorptivity tests (Sections 3.3.1.3 and 3.3.1.2 respectively).
5.3.4.1 Thermistor temperature measurements

Thermistor resistance measurements were taken with a voltmeter unit, at approximately 9.30am on the day of test. Details of the thermistor units at 5, 15 and 25 mm depth shown in figure 5.10 are given in section 3.3.4.1. Readings were taken on both the exposed and sheltered faces of each specimen.

5.3.4.2 Conductance between embedded stainless steel electrodes

Measurements of conductance between the electrodes were carried out as described in chapter 3. The measurement unit was kept outside for at least one hour to allow the temperature compensator probe to come to thermal equilibrium with its surroundings. Conductance measurements were carried out at around 5 pm on the day of test.

5.3.4.3 Relative humidity measurements

Relative humidity was measured on the surface of the concrete and in a 10 mm deep drilled hole. The relative humidity measurements were both carried out using a surface shield device as preliminary studies (section 3.3.2.2) indicated that the use of a depth shield was not required for depths less than 20 mm. The procedure for measuring the relative humidity described in chapter 3 was adhered to for all tests. RH measurements were carried out, on both sheltered and exposed faces of the specimens, from approximately 9.30am to 4.00pm on the day of test.

5.3.4.4 Autoclam air permeability and sorptivity tests

In order to carry out the Autoclam air permeability and sorptivity tests on the specimens each month (plate 5.04), six Autoclam test sites were marked on each face of the test specimens (shown in figure 5.10). Three of the test locations were used on alternative months, i.e. locations
1, 3 and 5 one month and 2, 4 and 6 on the following month. The reason for adopting this methodology was allow the longest possible time for the moisture state in the concrete (altered by the Autoclam sorptivity test) to return to equilibrium with its surroundings. The air permeability test was carried out first and after a period of one hour a sorptivity test was carried out at the same location. The one hour period between the air and sorptivity tests was adopted to avoid the possibility of increased air pressure in the concrete pores influencing the sorptivity measurement (Basheer, 1991). The water used for the sorptivity test was brought to thermal equilibrium with the surface of the concrete by keeping it beside the test specimens. Exposed face Autoclam measurements were carried out between 9.30am and 12.30pm and the sheltered face was tested between 12.30 and 4.00pm on the day of test.

5.4. Presentation and analysis of results

The results are presented and analysed in terms of the test methods used in the investigation.

5.4.1 Temperature measurements during the exposure period

Figure 5.11 shows the variation in temperature, at approximately 9.30am on the day of test, throughout the exposure period. The results from the different concrete specimens are not comparable as the specimens were tested on consecutive days.

Observations from figure 5.11:

(i) The temperature at 9.30am varied between 0 and 20 °C over the 18 months exposure period.

(ii) A seasonal variation in temperature is evident in the results.

(iii) There is no significant gradient of temperature at the time of test.
Significant differences in temperatures were noted between the different test slabs tested on consecutive days.

The temperature variation in the concrete specimens over the exposure period varied between 0 and 20 °C. This is in general agreement with the observation 'For most of the year the temperature at sea-level lies between 5°C and 20 °C' (for Northern Ireland), (Chandler et al, 1976). The December 1994 and January 1995 measurements were carried out during a period of especially cold weather, while the summer months of 1995 were generally warmer than normal. This is reflected in the temperature data (taken at approximately 9.30 am on the morning of test) for these months. Figure 5.11 shows the seasonal nature of the variation in temperature. The two main drops in temperature correspond the two winter periods during the investigation, while the highest temperatures correspond to the summer of 1995.

The gradients of temperature in the concrete specimens can be seen to be generally insignificant. The summer sun typically did not directly shine on the exposed faces of the specimens until approximately noon each day, therefore gradients of temperature in the concrete may well have formed after this period.

A difference in temperature is evident in some instances between specimens that were tested on consecutive days. This shows the variable nature of the ambient temperature which changes on an hourly and daily basis. Thus, the data presented in figure 5.11 can not be thought of as average monthly values of temperature but rather a series of discrete temperature measurements.

A typical daily variation in air temperature at the exposure site (measured using a standard mercury thermometer) in August 1996 is shown in figure 5.12.
Observations from figure 5.12:

(i) The daily temperature range was approximately 12 to 19.5 °C.
(ii) A maximum temperature can be seen to occur at 15.30 (3.30pm).
(iii) A minimum temperature occurs at 04.30.

These observations show a typical daily cycle of temperature fluctuation, the loss of heat during the night and rise in temperature during the day. The constant change in ambient temperature shows the importance of considering the effect of temperature on Autoclaml measurements.

5.4.2 Conductance measurements during the exposure period

Conductance measurements were taken at monthly periods across a series of embedded electrodes (details in section 3.3.3.2) on both the exposed and sheltered faces of the test specimens. As the values are significantly different for different w/c ratio concretes (see figure 5.13) the results are presented with appropriate scales in figures 5.14, 5.15 and 5.16. These figures show the resistance ratio to illustrate the change in conductance / resistance with regard to the initial measured values. The resistance ratio values presented in the graphs are an average of three or four measurements (depending on the depth of the electrodes).

Observations from figures 5.14, 5.15 and 5.16:

(i) Higher w/c ratio concrete specimens show greater increases and decreases of resistance ratio values close to the surface of the concrete.
(ii) The value of maximum resistance ratio recorded over the exposure period is generally inversely related to the depth of the electrode pair from the surface of the concrete.
(iii) The resistance ratio was influenced to a significant extent up to a depth of 20 mm in all concrete specimens.
The maximum resistance ratio values in the 0.65 w/c ratio specimen can be seen to be at the 10 - 15 mm depth range.

Higher w/c ratio concrete specimens show greater increases and decreases of resistance ratio values close to the surface of the concrete. This is due to the greater pore volume, larger pore size and greater pore connectivity associated with higher w/c ratio concretes (Nyame, 1979), (Powers, 1968). As stated in section 4.4.1.3, moisture can be removed more readily from larger pores in a concrete (stage II drying described by Hall et al, (1984)) (more common in higher w/c ratio concrete) than from smaller pores. Similarly, the quantity of water penetrating into larger pores is greater than that penetrating into smaller pores. Consequently, the fluctuations in moisture in higher w/c ratio concretes becomes more significant than that in lower w/c ratio concrete when exposed to outdoor conditions. This, in turn, results in larger increases and decreases in resistance ratio in higher w/c ratio concrete than in lower w/c ratio concrete.

The value of maximum resistance ratio recorded over the exposure period is inversely related to the depth of the electrode pair from the surface of the concrete. This indicates that gradients of moisture, of the form shown in figure 5.17(a), were in the concrete on the day of test each month over the exposure period. This is typical of a concrete which is losing moisture through the concrete surface. The trend is not found in all cases; for example in figure 5.14 (0.55 w/c ratio concrete), the resistance ratio values are greater at a depth of 10 - 15 mm than 5 - 10 mm in the months April, May, August 1995 and February 1996. These results indicate that the concrete at 10 - 15 mm has less moisture than the concrete at 5 - 10 mm relative to their initial values and hence considered to have formed the type of moisture gradient shown in figure 5.17(b). This may be caused
by light rain immediately prior to testing which would wet the surface region while it might not influence the deeper zones.

It should be noted that the 25 - 40 mm depth zone was not significantly influenced by the environment compared to the 5 - 10 and 10 - 15 mm zones of concrete. Therefore, in exposed concrete, the typical depth to which moisture movement occurs could be considered as 15 - 20 mm due to variations in ambient conditions. Bakker, (1994) reported results from exposed concrete tests in the Netherlands. He stated that moisture fluctuations caused by wetting and drying extended to a depth of 15 - 20 mm from the surface of the concrete. Significant resistance ratio fluctuation (and hence moisture fluctuation) to a depth of 20 - 25 mm was recorded in the 0.65 w/c ratio concrete (August '95).

Small variations in resistance ratio are evident at all depths investigated in all concrete. This indicates that moisture was at least partially mobile at depths of 40 mm. As stated chapter 4, the loss of moisture could be expected to occur preferentially from the largest pores in concrete, therefore, it seems logical to assume that this small movement of moisture was confined to the largest pores in each concrete. Bakker (1994) states that the concrete below a depth of 15 - 20 mm dries out with time irrespective of the exposure conditions of the concrete. The resistance ratio results from figures 5.14, 5.15 and 5.16 indicate that no large loss of moisture occurred from the 20 - 40 mm depth of the exposed concrete. This is in direct conflict with the findings of Bakker.

The resistance ratio results from the 0.65 w/c ratio specimen (figure 5.16) show that after February 1995 that the 10 - 15 mm electrode pair often gave larger values than the 5 - 10 mm depth resistance ratio values. There was no evidence of cracking (one possible explanation for this anomaly) and no explanation for this anomaly is apparent.
5.4.3 Relative humidity measurements during the exposure period

The variation in relative humidity in a 10 mm drilled hole and at the surface of the concrete specimens over the exposure period is presented in figures 5.18, 5.19 and 5.20.

Observations from figures 5.18, 5.19 and 5.20:

(i) Generally, the relative humidity measurements were lower during the summer months and higher during the rest of the year. Recorded values ranged from 45% to 100% RH.

(ii) The differences between the relative humidity measurements taken on the exposed and sheltered faces of the concrete specimens were often significant.

(iii) The relative humidity measurements carried out at 10 mm depth were generally greater than those recorded at the concrete surface.

The general seasonal variations in the moisture condition of the concrete identified in section 5.4.2 (conductance measurements) are evident in the relative humidity measurements shown in figures 5.18, 5.19 and 5.20. The concrete was shown to generally dry out in the summer months and to become more saturated during the winter months. The relative humidity measurements indicate the moisture condition of the concrete at the time of the test alone and, as shown below, the variation of relative humidity in concrete throughout a daily cycle can be significant.

The relative humidity was significantly different between the exposed and sheltered faces of the concrete specimens, with the exposed face generally having a higher RH. This is probably a result of the orientation and positioning of the concrete faces and is similar to the findings of Parrott (1991b). The sheltered face received little direct wind driven rain as it faced onto the wall of a building whereas the exposed face was open to wind driven rain. Casual observation of the specimens during the exposure period showed that rain commonly fell vertically,
wetting first the top and eventually both the exposed and sheltered faces of the concrete at a similar rate. The exposed face of the concrete was open to the direct sunlight whereas the sheltered face of the specimens received only reflected light. Osborne, (1994) indicates that this should lead to a significantly different microclimate in the concrete.

The formation of moisture gradients in the concrete over the exposure period was identified from the conductance measurements presented in section 5.4.2. The gradients of moisture can also be seen in the relative humidity results with the measurements in the 10 mm hole commonly being greater than those at the surface of the concrete. This is typical of a gradient of the form shown in figure 5.17(a) (of the type identified by Terril et al (1986)) which is caused by drying though the concrete surface. September 1995 RH measurements in the 0.45 w/c ratio concrete (figure 5.18) is an example of a reversal of the normal moisture gradient where the exposed face of the concrete showed an appreciably higher RH than that measured at 10 mm depth. As shown below, the relative humidity in the concrete varies on an hourly basis and the data presented in figures 5.18, 5.19 and 5.20 should be considered as 'snapshots' of the condition of the concrete at the time of RH and Autoclam tests each month.

Figures 5.21 and 5.22 show the variation in RH and temperature at the surface of the concrete and at 10 mm depth respectively over a 24 hour period during August 1996 (Measurements carried out on the 0.55 w/c ratio concrete specimen). Prior to testing the concrete was lightly wet by a shower of rain which meant that the surface of the concrete contained more moisture than that at 10 mm depth.

Observations from figures 5.20 and 5.21:
(i) The concrete showed a range of RH values over the 24 hour period ranging from 75% RH to 100% RH.
(ii) The drop in ambient temperature which occurred over night was coincident with an increase in RH to saturation (100 % RH) at both the surface and 10 mm depth.

(iii) The RH values taken at 18.30 on the first day are higher than the RH values taken 24 hours later.

The concrete showed a wide range of RH values over the 24 hour test period. The Kelvin equation (C.f. chapter 2), predicts that a reduction of temperature will cause an increase in RH. Figures 5.20 and 5.21 illustrate this, as the ambient temperature drop overnight caused an increase in the RH to 100 % at both the surface of the concrete and at 10 mm depth. The increase in temperature in the morning caused the concrete to return to the initial partially saturated state.

The difference between final and initial RH values over the 24 hour period was not caused by the temperature difference at the time of measurement, since the initial temperature was higher. This indicates that a loss of moisture occurred from the concrete over the 24 hour test period at both at the surface of the concrete and in the uppermost 10 mm. The weather during the test was dry (although overcast) and windy which would promote the loss of moisture by type I drying (C.f. 2.3.3.2.) Hall et al (1984). As stated earlier, there was a light shower of rain before the test started and, hence, his moisture was lost during the 24 hour test period. In addition a significant reduction in RH occurred at a depth of 10 mm (figure 5.21). This is more surprising as moisture at such depths should not be readily available for direct evaporation. It is unclear as to the explanation for the apparent loss of moisture at 10 mm depth. Hall et al (1984) indicates that the ambient RH will also influence the amount of moisture lost from the concrete however this was not measured as only two chilled mirror test probes were available to carry out the work.
Ambient RH values were recorded at 1.30pm on the day of test during part of the exposure period (August 1995 to February 1996). These values are shown in figure 5.22. The monthly readings were taken on specimen test days, therefore the ambient RH values were taken on consecutive days each month. The values show the, sometimes extreme, daily variability of the ambient RH at the exposure site. The seasonal variation in ambient RH values is less clear although many of the lowest recorded ambient RH values were recorded in the summer months and the highest in the winter months. This trend is in general agreement with Meteorological Office data published by Chandler et al, (1976) (figure 5.23). Ambient RH could be expected to influence the amount of moisture lost by a concrete (Hall et al, 1984). This shows that the model of the environment that was proposed by Bakker (1994) to assess the fluctuations of moisture gradients in concrete (C.f. 2.3.3.6), is at best simplistic.

The results presented in figures 5.21 and 5.22 have important implications for the development of a methodology to eliminate the effect of the moisture condition of the concrete on the measured Autoclam indices. The results indicate that:

(i) There could be daily variation of RH as high as 25 % at 10 mm depth from the surface of the exposed concrete.

(ii) The ambient RH could vary from 50 to 100 % within a day.

(iii) The gradient of moisture in concrete could be reversed due to a shower of rain on the concrete surface.

Therefore, a measurement of ambient conditions at the time of test is not sufficient to propose a correction factor for in-situ tests carried out with the Autoclam permeability system. Instead, the RH values measured in the concrete at the time of the Autoclam test should be used. This means that the RH values in figures 5.18, 5.19 and 5.20 could form the basis of a correction procedure for in-situ Autoclam permeation tests.
5.4.4 Air permeability measurements during the exposure period

The variation of air permeability over the exposure period is shown in figure 5.25

Observations from figure 5.25:

(i) The 0.65 w/c ratio concrete displays a significantly larger variability of air permeability with time than the 0.45 and 0.55 w/c ratio concretes.

(ii) The air permeability of all concretes was shown to decrease initially after the start of the exposure period.

(iii) Sheltered faces generally displayed higher air permeability values than their exposed counterparts.

The variation of air permeability with time in the 0.65 w/c ratio concrete was substantially greater than variation of air permeability in the 0.45 and 0.55 w/c ratio specimens. The larger pores that are present in the 0.65 w/c ratio concrete allow the passage of air freely and therefore when moisture in a pore acts to block this the flow of air it causes a large reduction in the air permeability. Hence, a larger variation in air permeability is caused by different moisture conditions in 0.65 w/c ratio concrete.

The air permeability values of the 0.65 w/c ratio concrete decreased markedly between September 1994 and January 1995. Moisture in the concrete increased slightly over this period (figure 5.20) however when the concrete dried out subsequently (summer months of 1995) the concrete did not display a corresponding increase in air permeability indices. The most likely explanation for this is that the 0.65 w/c ratio concrete did not finish hydration until December 1994 or January 1995. Powers (1958) predicts that the hydration of 0.6 w/c ratio cement paste will continue for 6 months after casting and the hydration in a 0.7 w/c ratio cement paste will continue for 1 year. Even though Powers observations are based on the hydration of cement paste in a laboratory it seems reasonable that his...
findings could be applicable, to some extent, for concrete exposed to the environment. For both 0.55 and 0.45 w/c ratio concrete there was a decrease in air permeability soon after the exposure outdoors. Again, this can be related to their continued hydration.

Sheltered faces of the concrete specimens generally gave higher air permeability values than their exposed counterparts. This observation can be explained with reference to the RH values presented in figures 5.18, 5.19 and 5.20. These graphs showed that the RH values on the sheltered faces were generally lower than their exposed counterparts. It is known that moisture in the capillary pore system impedes the passage of air. Consequently, concrete with less moisture (the sheltered faces of the specimens) displays a higher air permeability. This finding suggests a dependence of air permeability on internal RH which was also indicated by Parrott et al (1991d).

Figures 5.26 and 5.27 show plots of air permeability versus the corresponding RH measurements at the surface and 10 mm respectively. Bands representing the different concrete specimens are superimposed on the data to aid clarity. Following the finding that the 0.55 and 0.65 w/c ratio concretes may have experienced hydration during the early months of exposure the relevant values were omitted from figures 5.26 and 5.27.

Observations from figures 5.26 and 5.27

(i) Distinct bands of RH versus air permeability for all three w/c ratio concretes for values of RH less than 80%.

(ii) Between 80 and 100% RH the decrease in air permeability due to an increase in RH is proportional to the w/c ratio of the concrete.

(iii) There is high proportion of overlap between the three specimen bands in air permeability values above 90% RH.

(iv) Similar trends were observed between the air permeability of the concrete and the RH measured at the surface and in a 10 mm hole.
Using a classification of concrete based on Autoclam air permeability indices (based on both freeze-thaw and carbonation results) proposed by Basheer, (1991) (see figures 5.26, 5.27 and table 5.06) it may be seen that the moisture condition of the concrete could alter the classification of a concrete. Basheer used specimens oven dried at 105 °C (RH = 20 %) to measure air permeability, which means that the classification may not be completely reliable due to the possibility of microstructural damage (see figure 2.36). However, the spread of the classification of the same concrete (from 'very poor' to 'good' in 0.65 w/c ratio concrete) at different RH values makes it necessary to eliminate the effect of moisture on air permeability measurements before the air permeability results can be used to classify concrete for its durability.

From figures 5.26 and 5.27 it can be seen that three distinct bands of results are visible for RH values less than approximately 80%. This indicates that when the capillary pores are substantially free from moisture, in-situ air permeability tests can be used to differentiate between concretes of different qualities.

Between 80 and 100 % RH the decrease in air permeability due to an increase in RH is proportional to the w/c ratio of the concrete. The relationship proposed by Parrott (1994b) (shown in figure 2.36) and confirmed in chapter 4 indicates that larger w/c ratio concretes show a greater variation in air permeability caused by moisture loss and gain. The reason for this phenomenon is that when fully saturated (pores fully blocked by moisture), all concretes have an air permeability approaching zero. When the capillary pores of a concrete are free from moisture the flow of air through the concrete is possible. Nyame (1979) shows that pores of approximate diameter greater than 1 E-2 μm (corresponds to > 80% RH) are more numerous in higher w/c ratio concrete. Therefore, at a RH between 80 and 100% larger w/c ratio concretes could be expected to
have a greater number of pores free from moisture and available to allow the flow of air. Thus larger w/c ratio concretes display a greater the change in air permeability due to moisture fluctuations in concrete.

In concrete with a RH in excess of 80% a significant overlap between the different bands is evident. From figures 5.26 and 5.27 it can be seen that 0.65 w/c ratio concrete with a RH greater than 90% could be confused with both 0.45 and 0.55 w/c ratio concretes. Similarly the 0.45 and 0.55 w/c ratio concretes can not be differentiated with certainty in concrete with a RH greater than 80%. This means that insitu air permeability measurements can not be used to give an indication of the quality of the concrete if the RH is greater than 80%.

The range of air permeability indices associated with each specimen band may be caused by some variation in the concrete at test sites and by any variation in clamping the clam ring to the surface. The air permeability values in each band or results may also be influenced by the way in which RH measures the moisture in a non cylindrical capillary pores. Figure 5.28 shows two pores which would produce a similar RH but a different permeability to pressurised air. Air under pressure may dislodge the small neck of moisture in figure 5.28. This could be expected to occur in a substantially dry concrete that experienced a modest amount of capillary condensation. Conversely, a similar pore that was initially saturated and was subjected to some drying would not lose moisture until the neck region of the pore lost moisture. This large pore would not allow the flow of air even though a RH measurement would suggest that it should be free from moisture.

Thus, the concrete will display a different air permeability for a similar RH depending on whether the concrete is drying out, has just been rained upon or is experiencing capillary condensation. This may be partly overcome by stipulating that an in-situ test should only be carried out after
a given period of dry weather, however the effect of capillary condensation
due to a change in temperature cannot be eliminated. Figure 5.27 shows
that below a RH of 80 % moisture did not obscure the differences in air
permeability between the different quality concretes and hence the
relationship between air permeability and RH may be used assess the
quality of concrete.

The relationships between air permeability and the RH at the
surface of the concrete and in a 10 mm hole are similar and it is unclear as
if one is more suitable for use than the other. Both graphs are equally
valid however the measurement of RH in the 10 mm hole is influenced
by a greater depth of concrete. As the Autoclam air permeability test is
influenced by concrete to a depth of 40 mm and added to the fact that the
laboratory sorptivity test correlates better with the RH at 10 mm depth (C.f.
Chapter 4) it was decided to adopt the 10 mm RH measurement as
standard for moisture estimation in the air permeability test.

5.4.4.1 Correction of in-situ Autoclam air permeability
measurements

Using the results presented in figure 5.27 a method of removing
most of the influence of moisture from Autoclam air permeability
measurements can be proposed:
(i) The concrete surface should be sheltered from rain for an extended
period until the RH measurement in a 10 mm hole is less than 80%.
(ii) 3 No. X Autoclam Air permeability tests to be carried out on the
concrete under investigation.
(iii) At least 1 No. X RH measurement taken in a 10 mm drilled hole in
concrete which is representative of the Autoclam test area at the time of
the tests.
Using the RH measurement and the average Autoclam air permeability index an assessment of the quality of the concrete can be made from figure 5.27.

5.4.5 Sorptivity measurements during the exposure period

The variation of sorptivity (uncorrected for temperature effects) over the exposure period is shown in figure 5.29.

Observations from figure 5.29:

(i) All concretes displayed a significant seasonal variation in sorptivity.
(ii) The sorptivity of all concretes was shown to decrease initially after the start of the exposure period.
(iii) Sheltered faces generally gave higher sorptivity indices than their exposed counterparts.

The variation of concrete sorptivity can be seen to be linked to the seasonal changes in ambient conditions. The summer months of 1995 show a large sorptivity compared to the sorptivity measured during the winter months. This variation in sorptivity is significant as the lowest sorptivity indices in the 0.65 w/c ratio (February 1995) are similar to the largest recorded sorptivity indices in the 0.45 w/c ratio concrete (June 1995). This means that the results of standard sorptivity measurements to evaluate the quality of in-situ concrete do not give an indication of concrete quality.

Figure 5.29 shows that the sorptivity of all concretes decreases initially after the start of the exposure period. The continued hydration of the concrete is considered to be the cause of decrease in sorptivity over as a similar reduction in air permeability (see section 5.4.4) during the months up to December 1995 was observed. The high sorptivity measurements recorded in the first months of exposure were not repeated even after a sustained period of dry and hot weather over the summer of 1995.
therefore it again seems likely that the drop in sorptivity was caused by continued hydration of the concrete.

From figure 5.29 it can be seen that higher sorptivity indices were generally measured on the sheltered faces of the specimens which was also found by Parrott (1992). This can be attributed to the lower RH values that were generally recorded on the sheltered faces of the specimens. The moisture condition of the concrete, as measured by RH, was related to the Autoclam sorptivity indices in laboratory investigations presented in chapter 4. This relationship shall be verified for exposed concrete.

Figure 5.30 and 5.31 show the relationship between the RH measurements and Autoclam sorptivity indices. Bands representing the three different w/c ratio concretes are superimposed on the data to aid clarity. Figure 5.32 and 5.33 show the relationship between RH and the 20 °C equivalent Autoclam sorptivity indices (using equation [5.17]). Measurements which were influenced by continued hydration of the concrete were omitted from the graphs.

Observations from figures 5.32 and 5.33:
(i) Distinct bands of RH versus sorptivity for all three w/c ratio concretes for values of RH less than 80 %.
(ii) There is high proportion of overlap between the three specimen bands in sorptivity values above 90 % RH.
(iii) Between 80 and 100 % RH the decrease in sorptivity due to an increase in RH is proportional to the w/c ratio of the concrete.
(iv) The temperature correction factor made little difference to the overall trends identified between RH at 10 mm depth and sorptivity.
(v) Relationship between RH and sorptivity in laboratory concrete was shown not to describe the relationship between RH and sorptivity in exposed concrete.
Below a RH of 80% distinct bands of RH versus sorptivity are clearly visible (figures 5.32 and 5.33). This indicates that in-situ sorptivity measurements may be used to classify the quality of the concrete when the RH is less than 80% RH. The classification of concrete proposed by Basheer (1991) is based on sorptivity measurements at approximately 20% RH which indicates that the concrete may have been microstructurally damaged by dying. As the sorptivity of a concrete (unlike air permeability) could be expected to vary with RH between 20 and 70% (C.f. chapter 4) the classification is of very limited use for exposed concrete in Northern European climates.

Conversely, for measurements carried out in concrete with a RH in excess of 90% no judgement as to the quality of the concrete may be made as there is a significant overlap between the three bands. The 0.65 w/c ratio concrete band can be confused with both the 0.45 and 0.55 w/c ratio concretes. This means that sorptivity measurements in exposed concrete with a RH in excess of 80 - 90% cannot be used to give an indication of the likely durability of a structure.

Each concrete showed a measurable sorptivity in concrete at a RH of 100% in figure 5.33. This finding is not consistent with the generally accepted theory of capillary absorption which states that a concrete will have no capillary flow when fully saturated. The reason for this anomaly is because of the fact that a RH of 100% does not always a mean a fully saturated concrete. No surface RH value of 100% was recorded in the investigation (see figure 5.32) although several measurements of 100% were recorded in the 10 mm hole. This means that although figure 5.33 shows a RH value of 100% the surface concrete had some of its largest pores free from moisture.

The applied pressure may be sufficient to force moisture from the neck of an 'ink bottle' pore (figure 5.28) and thus allow water absorption
through the open length of pore whereas in a saturated pore the applied pressure would not permit any water absorption. This hysteresis effect could be expected only in a concrete which was subjected to wetting and drying, i.e. in-situ concrete. The effect could also be expected to be more significant in a concrete with a greater number of large pores as the difference in pore size diameter along the length of the pore creates 'ink bottle' pores. This is supported by figure 5.33 where the 0.65 w/c ratio concrete shows a greater deviance from the trend of expected sorptivity (based on the laboratory investigation) than 0.55 and 0.45 w/c ratio concretes.

A comparison between figures 5.30, 5.31, 5.32 and 5.33 shows that range of air permeability values in the 0.65 w/c ratio concrete band was increased after the application of the temperature correction factor to the result. Generally, there was little difference between the trends identified between RH and sorptivity, with (figures 5.32 and 5.33) and without (figures 5.30 and 5.31) the temperature correction factor, in the 0.45 and 0.55 w/c ratio concretes. This implies that the influence of temperature on Autoclam sorptivity indices may be less significant than the influence of moisture.

Chapter 4 identified a linear relationship between the RH at 10 mm depth and the Autoclam sorptivity index of each test concrete. The relationships for 0.45, 0.55 and 0.65 w/c ratio concrete are reproduced in equations [5.14], [5.15] and [5.16].

\[
\begin{align*}
\text{RH @ 10 mm (0.45 w/c ratio concrete)} &= 100 - 28.35 \text{ (SI)} \\
\text{RH @ 10 mm (0.55 w/c ratio concrete)} &= 100 - 23.67 \text{ (SI)} \\
\text{RH @ 10 mm (0.65 w/c ratio concrete)} &= 100 - 16.29 \text{ (SI)}
\end{align*}
\]

Where RH is the relative humidity (%) and
SI is the Autoclam sorptivity index.

These relationships are plotted in figure 5.33, which shows that the relationship between sorptivity and RH measurements in exposed concrete cannot be represented by these equations. The reason that the results of the laboratory investigation were not directly transferable to in-situ measurements may be partially due to incomplete hydration of concrete specimens in the laboratory investigation being compared to fully hydrated in-situ concrete (C.f. chapter 4 and above). However, a principal reason may be the moisture hysteresis effect in exposed concrete.

The relationships between sorptivity and the RH at the surface of the concrete and in a 10 mm hole are similar and it is unclear as if one is more suitable for use than the other. Both graphs are equally valid however the measurement of RH in the 10 mm hole is influenced by a greater depth of concrete. Given that the laboratory sorptivity test correlates better with the RH at 10 mm depth (C.f. Chapter 4) and the 10 mm RH measurement could be expected to measure the RH of the concrete which influences a 15 minute sorptivity test, it was decided to adopt the 10 mm RH measurement as standard for moisture estimation in both air permeability and sorptivity tests.

5.4.5.1 Correction of in-situ Autoclam sorptivity measurements

Using the results presented in figure 5.33 and the temperature correction procedure developed in section 5.2.2.3 a method of removing the influence of both temperature and moisture from Autoclam sorptivity measurements can be proposed:

(i) The concrete to be tested should have a RH of less than 80%.
(ii) 3 No. X Autoclam sorptivity tests to be carried out on the concrete under investigation.
(iii) At least 1 No X Relative humidity measurement taken in a 10 mm drilled hole in the concrete at the time of the Autoclam tests.

(iv) The ambient temperature and the temperature of the test water should be kept similar. This temperature should be recorded.

(v) Using the recorded temperature and the measured sorptivity index an equivalent 20 °C sorptivity index (SI_{20}) should be calculated from equation [5.13], which is:

\[ SI_{20} = SI \times [1.6385 - 3.8834e-2 \times (T) + 3.5132e-4 \times (T^2)] \]

where SI is the measured Autoclam sorptivity index, SI_{20} is the equivalent sorptivity index at 20 °C and T is the temperature at the time of test (°C)

(vi) Using the equivalent sorptivity index (SI_{20}) and the RH measured in a 10 mm hole, a determination of the quality of the concrete may be made from figure 5.33.

5.4.6 Significance of test results

Both the air permeability and the sorptivity of in-situ concrete with a RH at 10 mm in excess of 80% cannot be used to estimate the likely durability of a reinforced structure. Reviewing the RH results in figures 5.18, 5.19 and 5.20 it can be seen that the exposed concrete had a RH (at 10 mm depth) of less than 80% for three months (on average) during the summer of 1995. Therefore, for most of any year, the concrete could be expected to contain too much moisture to allow meaningful Autoclam tests to be carried out. The most obvious way to overcome this problem is to precondition the in-situ concrete, to remove moisture, prior to Autoclam testing.
5.5 Summary of findings

The major findings of the investigation presented in this chapter are summarised below.

(i) Theoretical and experimental studies indicated that the effect of temperature on Autoclam air permeability measurements was negligible.

(ii) Theoretical and experimental studies indicated that the effect of temperature on Autoclam sorptivity measurements was significant.

(iii) A temperature correction factor to transform an Autoclam sorptivity index to an equivalent sorptivity at 20 °C was developed.

(iv) In exposed concrete, the temperature was seen to vary seasonally between 0 and 20 °C, resulting in no significant gradients of temperature in the concrete at 9.30am.

(v) Recorded values of relative humidity in exposed concrete ranged from 45% to 100%.

(vi) The variation of RH measurements due to a change in temperature during a 24 hour period was assessed as being significant.

(vii) A relationship between measured RH and in-situ Autoclam air permeability indices was identified in concrete with a RH less than 80%. A methodology for removing the influence of different moisture conditions in concrete with a RH (measured at 10 mm) of less than 80% on Autoclam air permeability measurements was proposed.

(viii) A relationship between RH measured in a 10 mm hole and in-situ Autoclam sorptivity indices was identified in concrete with a RH of less than 80%. A methodology for removing the influence of different ambient temperatures and different moisture conditions in concrete with a RH of less than 80% on Autoclam sorptivity measurements was proposed.
Laboratory studies of the relationship between sorptivity and relative humidity were shown not to be applicable to concrete exposed to the environment.

To evaluate the quality of concrete with an internal RH of greater than 80%, a method of reducing the internal RH to less than 80% is required.
### Table 5.01 - Dynamic viscosity of air at different pressures and temperatures.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Viscosity at 1 bar absolute pressure (N.s/m²)</th>
<th>Viscosity at 10 bar absolute pressure (N.s/m²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>-3.15</td>
<td>16.96 E-7</td>
<td>17.11 E-7</td>
</tr>
<tr>
<td>6.85</td>
<td>17.46 E-7</td>
<td>17.60 E-7</td>
</tr>
<tr>
<td>16.85</td>
<td>17.96 E-7</td>
<td>18.10 E-7</td>
</tr>
<tr>
<td>26.85</td>
<td>18.46 E-7</td>
<td>18.59 E-7</td>
</tr>
<tr>
<td>46.85</td>
<td>19.45 E-7</td>
<td>19.58 E-7</td>
</tr>
</tbody>
</table>

Adapted from Marshall (1976)

### Table 5.02 - Assumed values of variables used in equation [5.01]

<table>
<thead>
<tr>
<th>Variable</th>
<th>Assumed value</th>
</tr>
</thead>
<tbody>
<tr>
<td>(k) Permeability</td>
<td>1.6 E-13</td>
</tr>
<tr>
<td>(r) Capillary pore radius</td>
<td>2.0 E-6 m</td>
</tr>
<tr>
<td>(µ) Dynamic viscosity</td>
<td>Various (see table 5.01)</td>
</tr>
<tr>
<td>(ΔP) Pressure difference</td>
<td>0.5 Bar (≈ 5 m head)</td>
</tr>
<tr>
<td>(L) Length of ideal pore</td>
<td>0.04 m</td>
</tr>
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### Table 5.03 - Physical properties of water. Adapted from Evett (1989).

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Density (kg/m³)</th>
<th>Viscosity (N.s/m²)</th>
<th>Surface tension (N/m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>1000</td>
<td>1.75 X E-6</td>
<td>0.0756</td>
</tr>
<tr>
<td>10</td>
<td>1000</td>
<td>1.30 X E-6</td>
<td>0.0742</td>
</tr>
<tr>
<td>20</td>
<td>998</td>
<td>1.02 X E-6</td>
<td>0.0728</td>
</tr>
<tr>
<td>30</td>
<td>996</td>
<td>8.03 X E-7</td>
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<td>40</td>
<td>992</td>
<td>6.51 X E-7</td>
<td>0.0696</td>
</tr>
<tr>
<td>50</td>
<td>988</td>
<td>5.41 X E-7</td>
<td>0.0679</td>
</tr>
</tbody>
</table>

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### Table 5.04 - Temperature correction factor for sorptivity indices

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>CF&lt;sub&gt;20&lt;/sub&gt; - Correction Factor (to 20 °C)</th>
</tr>
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<tbody>
<tr>
<td>5</td>
<td>1.44</td>
</tr>
<tr>
<td>10</td>
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<td>15</td>
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<td>20</td>
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</tr>
<tr>
<td>25</td>
<td>0.89</td>
</tr>
<tr>
<td>30</td>
<td>0.80</td>
</tr>
<tr>
<td>35</td>
<td>0.73</td>
</tr>
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</table>

### Table 5.05 - Experimental variables.

<table>
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<tr>
<th>Variable</th>
<th>Level</th>
</tr>
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<tbody>
<tr>
<td>W/C ratio</td>
<td>0.45, 0.55, 0.65</td>
</tr>
<tr>
<td>Exposure Conditions</td>
<td>Full exposure, Sheltered exposure</td>
</tr>
</tbody>
</table>

### Table 5.06 - Classification of concrete (Basheer, 1991)

<table>
<thead>
<tr>
<th>Classification (Predicted Risk)</th>
<th>Autoclam Air Permeability Indices</th>
<th>Autoclam Sorptivity Indices</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low</td>
<td>&lt; 0.1</td>
<td>&lt; 1.3</td>
</tr>
<tr>
<td>Medium</td>
<td>0.1 - 0.5</td>
<td>1.3 - 2.6</td>
</tr>
<tr>
<td>High</td>
<td>0.5 - 0.9</td>
<td>2.6 - 3.4</td>
</tr>
<tr>
<td>Extremely High</td>
<td>&gt; 0.9</td>
<td>&gt; 3.4</td>
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Plate 5.01 - Specimens at exposure site 2

Plate 5.02 - View W-N-W from exposure site 2
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Plate 5.04 - Specimen testing.
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Figure 5.02 - Variation of 'Autoclam' Air permeability indices with temperature.
Figure 5.03 - Theoretical relationship between absorption flow in an ideal capillary pore and temperature.

Figure 5.04 - Variation of 'Autoclam' Sorptivity indices with temperature.
Correction factor to equivalent sorptivity at 20 °C

\[
y = 1.639 - 3.883e^{-2x} + 3.513e^{x^2} \quad R^2 = 0.998
\]

Figure 5.05 - Correction factor to obtain the equivalent sorptivity at 20 °C.
Sorptivity index with temperature variation

- 0.45 w/c ratio concrete
- 0.65 w/c ratio concrete

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Rear face tested in an identical manner

Area of concrete not used for testing (lighter shade)

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Concrete Relative Humidity (%) surface

RH gradient caused by surface drying

increasing depth

(A) Moisture gradient typical of a concrete subjected to drying.

Concrete surface Relative Humidity (%)

RH gradient caused by surface drying Moisture from light surface wetting

increasing depth

(B) Moisture gradient typical of a concrete subjected light rain.

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Air/water under pressure displaces moisture in the neck of the pore to allow flow.

Dry capillary pore subjected to some capillary condensation

RH measurement gives similar value

(A) Pore subjected to 'Wetting'

Air/water under pressure does not displace moisture in the pore. No flow.

Saturated capillary pore subjected to some drying.

(B) Pore subjected to 'Drying'

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Relative humidity (%) at 10 mm depth

Sorptivity Index V RH at 0 mm depth

- 0.45-Exposed Face
- 0.45-Sheltered Face
- 0.55-Exposed Face
- 0.55-Sheltered Face
- 0.65-Exposed Face
- 0.65-Sheltered Face

<table>
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<th>Very Good*</th>
<th>Good*</th>
<th>Poor*</th>
<th>Very Poor</th>
</tr>
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</table>

Sorptivity Index (Cu.M/Sqrt(mins)) X E-7


Figure 5.30 - Uncorrected Sorptivity Indices V Relative Humidity at 0 mm

Sorptivity Index V RH at 10 mm depth

- 0.45-Exposed Face
- 0.45-Sheltered Face
- 0.55-Exposed Face
- 0.55-Sheltered Face
- 0.65-Exposed Face
- 0.65-Sheltered Face

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<th>Very Good*</th>
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<th>Poor*</th>
<th>Very Poor</th>
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20°C equivalent Sorptivity Index (Cu.M/Sqr(mins)) X E-7


Figure 5.31 - Uncorrected Sorptivity Indices V Relative Humidity at 10 mm.
Figure 5.32 - 20 °C Equivalent Sorptivity Indices V Relative Humidity at 0 mm

Figure 5.33 - 20 °C Equivalent Sorptivity Indices V Relative Humidity at 10 mm
6. Preconditioning concrete for permeation tests

6.1 Introduction

Concrete exposed to the natural environment inevitably contains gradients of moisture in the near surface region. As found in Chapter 5, the removal of moisture from in-situ concrete with a RH in excess of 80% is required to allow the meaningful use of in-situ Autoclam measurements. Two disparate strategies for removing the influence of moisture on the air permeability and sorptivity of in-situ concrete have been suggested by Hudd (1989) and Dhir (1993):

(i) The moisture state of the cover concrete can be measured and used with the in-situ permeation properties to give an estimate of concrete quality, as examined in chapter 5 (Hudd, 1989).

(ii) The moisture state of the near-surface concrete can be altered by preconditioning so that the moisture state is similar for all permeation measurements (Dhir, 1993).

The findings of chapter 5 indicate that a combination of these techniques may be required to remove the influence of moisture from in-situ Autoclam tests, since in exposed concrete with a RH above 80% no estimation of the concrete quality can be made using Autoclam tests. This chapter details an investigation into the feasibility of altering the moisture state of a concrete using a number of preconditioning techniques.
6.2 Objectives of experimental programme

The main objective of this programme of tests was to investigate the feasibility of using a preconditioning method to remove the influence of moisture on the Autoclam permeation properties of concrete. Based on the literary review, four separate preconditioning methods were identified and their effectiveness was investigated and compared.

6.3 Experiment programme

6.3.1 Experimental variables

Table 6.01 shows the variables used in this experimental programme.

6.3.1.1 W/C ratio

In this investigation, the quality of the concrete was varied by choosing two w/c ratio viz. 0.45 and 0.65 w/c ratio concrete. Other particulars of the mixes are as detailed in chapter 3.

6.3.1.2 Preconditioning methods

The four preconditioning methods investigated were:

(i) Hot air application.
(ii) Dried compressed air application.
(iii) Vacuum application.
(iv) Solvent replacement.

Schonlin et al (1987) and Basheer (1991) used the application of heated air to the surface of the concrete to remove the influence of moisture on permeation measurements. The principle used was applied
to form the basis of the hot air preconditioning method investigated here. The application of heat at the surface of the concrete for a period of 12 hours was adopted as a method of preconditioning concrete. As shown in figure 6.01, heat was applied to the centre of a standard Autoclam ring by means of a heat gun. The distance of the gun from the concrete was adjusted to achieve 100°C at the concrete surface directly below the heat source. This procedure was adopted to ensure that any damage to the structure of concrete caused by the application of heat was minimised. It was presumed that a thermal gradient would be set up in the near surface concrete and that this would result in the evaporation of water from the test region. The concrete was allowed to cool for 12 hours before carrying out Autoclam tests.

(ii) Dried Compressed Air application

The Autoclam air permeability index of saturated concrete is not always zero. It was assumed that the reason for this flow of air may be caused by the pressurised air displacing water from the largest pores in the concrete. The application of dried compressed air to the surface of the concrete was achieved using an Autoclam ring and a pressurised bottle of compressed air, as shown in figure 6.02. This preconditioning method was expected to force moisture, contained within in the capillary pores, out of the concrete test area using an overpressure. Preliminary tests indicated that the apparatus could sustain an air pressure of 6 Bar, which was governed by a regulator attached to the cylinder of dried compressed air. The application of compressed air was continued for 24 hours. The Autoclam air permeability test was carried out one hour after the end of the compressed air application to allow the dissipation of air pressure in the capillary pores (Basheer, 1991).
(iii) Vacuum application

Dhir et al (1993) used a vacuum applied through an ISAT apparatus (Described in 2.3.4.1) to remove the influence of moisture from concrete prior to ISAT tests. Using the same principle a vacuum was applied through an Autoclam base ring to remove moisture from the capillary pores in the concrete. Shown in figure 6.03, a vacuum generator was attached to a plate covering the Autoclam ring and a vacuum of 745 mm of mercury (980 mBar) applied to the surface of the concrete. A dessicator was linked to the vacuum generator to prevent moisture from entering the vacuum pump. A vacuum gauge was fixed to the covering plate to monitor the level of vacuum achieved at the concrete surface. As illustrated by figure 6.03, the principle of this preconditioning method was to draw moisture from the volume of concrete, directly below the Autoclam test location, thereby removing the influence of moisture on Autoclam permeation measurements.

(iv) Solvent replacement methods

Marsh, (1996) applied solvent at pressure, to replace moisture in mercury intrusion porosimetry test samples, and using this principle a method for applying solvent through an Autoclam ring was developed. This method is based on the introduction under pressure of a solvent that is immiscible with water, thus it could be expected to displace moisture in the capillary pores. Two solvents were used in this investigation - diethyl ether and isoproponol. The solvents were chosen to have a low boiling point as this indicates that it could be easily removed from the concrete prior to permeation testing. The initial application of solvent was achieved by using a piston apparatus which was fixed onto an Autoclam ring, as shown in figure 6.04. Preliminary tests indicated that an application pressure of 1 bar would be sufficient to force the diethyl ether into the concrete whereas isoproponol required 2 Bar pressure. To allow
comparison with other preconditioning techniques the application of solvent was continued for 24 hours and the removal of the solvent was achieved by evaporation in a laboratory (Temperature 20 °C ± 2°C, RH 55% ± 5%) over 48 hours.

6.3.2 Specimen details

Four 215 X 215 X 75 mm deep slabs were cast for each preconditioning method (two slabs for each w/c ratio). The concrete was compacted using a vibrating table as detailed in chapter 3.

The test specimens used in this investigation were of the form shown in figure 6.05. The arrangement of the stainless steel electrodes (figure 6.06 and plate 6.01) enabled the monitoring of the change in electrical resistance at specific depths in the concrete during preconditioning. This gave an indication of the movement of moisture in the volume of concrete that influences Autoclam tests. The electrodes were covered over with a heat shrink material except for a central region of 20 mm (figure 6.06), which resulted in the electrical resistance measurement over a length of 20 mm at the test location. Table 6.02 shows the specimen numbers, the corresponding the w/c ratio and preconditioning technique which was carried out on the specimen.

6.3.3 Curing and conditioning

The specimens were removed from the mould after 24 hours and exposed to the following conditioning regime

(i) Water storage at 20 °C for 3 days.

(ii) On the fourth day the slabs were placed in a controlled environment of 20 °C ± 2°C and 50 ± 5% relative humidity.

(iii) Commencing on day 28, the specimens were oven dried at 70 ± 2 °C for 7 days, to remove all free moisture from the samples.
(iv) The specimens were then wrapped in polythene to prevent moisture ingress and were transferred to a constant temperature laboratory at 20 °C for a cooling period of 24 hours.

(v) Initial 'oven dried' permeability and resistance measurements were carried out on the specimens.

(vi) After initial testing, the specimens were re-saturated. Saturation was achieved by incremental immersion of the test slab over a four day period, as described in chapter 4. The fully saturated condition was adopted as the initial moisture state prior to preconditioning as it was deemed to be the most extreme moisture condition that could be encountered on-site.

(vii) The preconditioning methods were applied to the saturated concrete specimens and the resistance was monitored over the period of application.

(viii) Final permeability and relative humidity tests were carried out after preconditioning, as described below.

6.3.4 Tests methods

The following methods were used to evaluate and quantify the effectiveness of the preconditioning techniques:

(i) Resistance between embedded stainless steel electrodes (described in Section 3.3.3).

(ii) Autoclam air permeability tests (as described in section 3.3.1.1).

(iii) Relative humidity measurements (section 3.3.2).

6.3.4.1 Resistance between embedded stainless steel electrodes

After oven drying, resistance measurements were taken vertically between the embedded electrodes in each of the slabs. Measurements of resistance between consecutive electrodes (vertically) were also taken
during the application of the preconditioning procedures to give an indication of the effectiveness of each method (plate 6.02). Final resistance measurements were carried out after the preconditioning method had finished.

6.3.4.2 Autoclam air permeability tests

Autoclam air permeability tests were carried out on the oven dried specimens and on the concrete after each of the preconditioning procedures were carried out. The air permeability test was chosen as it is influenced by a larger volume of concrete than the 15 minute sorptivity test. The use of the Autoclam sorptivity test was discounted as it changes the moisture content of the concrete and would therefore preclude the measurement of relative humidity.

6.3.4.3 Relative humidity measurements

Relative humidity was measured in a drilled hole at 15 mm or 30 mm depth from the surface using the chilled mirror concrete probe. The relative humidity test was carried out inside the clam ring on each specimen, after the completion of the final Autoclam air permeability test.

6.3.4.4 Order of testing

The tests carried out on each specimen were carried out in the following order.

(i) Following the oven drying and subsequent 24 hour cooling period, electrical conductance measurements were carried out in each specimen.

(ii) Air permeability measurements were then carried out on the oven dried concrete specimens.
(iii) For the duration of each preconditioning technique (with the exception of hot air application, see 6.4.1.1) conductance measurements were taken at intervals.

(iv) Final air permeability and RH measurements were taken although the differences between the preconditioning necessitated a different procedure. In general, the air permeability test was carried out as soon as was expedient. The specimens subjected to hot air preconditioning were cooled for 12 hours prior to final conductance measurements, and the air permeability test was carried out immediately afterwards. Air permeability tests were carried out on the compressed air and vacuum specimens after one hour (to allow the dissipation of air pressure in the pores). The specimens which were preconditioned using a solvent replacement technique were tested for air permeability 48 hours after the end the application of solvent. This was to allow the solvent to evaporate from the pores in the concrete (Marsh, 1996).

(v) The relative humidity measurements were carried out immediately after the air permeability test.

6.4 Presentation and analysis of results

The results of the resistance measurements, Autoclam air permeability tests and the internal relative humidity measurements are presented and discussed with regard to each preconditioning technique.

6.4.1 Resistance measurements

6.4.1.1 Hot air preconditioning

The resistance of concrete is reported to be temperature dependent (Bungey et al, 1989). Therefore, final resistance measurements were
carried out after preconditioning with hot air for 12 hours and a 12 hour cooling period. Measurements of resistance were not taken during the period of hot air preconditioning. The resistance ratio plots for the array of electrodes, directly below the centre of the test ring, are shown in figures 6.07 and 6.08.

The following observations from figures 6.07 and 6.08:

(i) The resistance ratio increased, after the hot air preconditioning regime, indicating a loss of moisture from the concrete.

(ii) At a given depth the resistance ratio in the 0.65 w/c ratio concretes is greater than in the 0.45 w/c ratio concretes.

(iii) The resistance ratio of the concrete increased at depths in excess of 35 mm due to the hot air preconditioning.

(iv) A significant variation in resistance ratio measurements between specimens made from the same concrete mix was evident in both figures 6.07 and 6.08.

The increase in resistance ratio during the hot air preconditioning is due to the loss of moisture from the pores in the concrete. As discussed in chapters 2 and 4, the resistance of concrete to the flow of electrical current is altered significantly by the quantity of moisture in the capillary pores of the concrete. The conductance of a cement paste comprises of two components, Monfore, (1968).

(i) Ionic conductance through the free capillary pore water.

(ii) Conductance through the CSH matrix (including bound water).

In substantially moist concrete, the conductance is primarily through ions in the free water. When this moisture is removed the ionic conductance through the capillary free water is removed and the resistance of the concrete increases. Monfore (1968) states that the increase in resistivity between saturated concrete and oven dried (at 105 °C) concrete is in the order of 10 million times.
Figures 6.07 and 6.08 show that the resistance ratio values in the 0.65 w/c ratio concrete is greater than in the 0.45 w/c ratio concrete for any given electrode group. This is caused by the different pore sizes in each of the concretes, as shown by figure 6.09. The 0.65 w/c ratio concrete has capillary pores of larger diameter than the 0.45 w/c ratio concrete (figure 4.08). As the removal of moisture from concrete occurs from the largest pores first, the hot air application removes moisture faster from 0.65 w/c ratio concretes (which has relatively large pores) than from 0.45 w/c ratio concretes (which have relatively small pores). This causes an increased electrical flow path which in turn causes an increase in the resistance between embedded electrodes and hence an increased resistance ratio value.

The resistance ratio of the concrete increased for all electrode groups monitored during the hot air preconditioning. This indicates a change in the moisture condition of all the specimens to depths in excess of 35 mm. As stated previously, the largest pores in the concrete could be expected to lose moisture first (stage II drying as described by Hall et al, (1984) is dependent on material properties, i.e. pore size and continuity) and it seems likely that the large sized pores in all the concretes lost some moisture to a depth in excess of 35 mm. Figure 6.07 shows that the depth range 15 - 40 mm in the 0.45 w/c ratio concrete showed a modest increase in resistance ratio. This suggests that below 15 mm in the 0.45 w/c ratio concrete only moisture from the largest pores was removed and the increased resistance ratios shown by the 5 - 10 and 10 - 15 mm depth ranges were as a result of drying occurring from smaller pores. The 0.65 w/c concrete (figure 6.08) shows a significant influence of hot air preconditioning on smaller diameter pores to a depth of roughly 30 mm in specimen 2. The resistance ratios below this depth are similar which suggests that only the largest more continuous pores had moisture
removed. The depth range between 5 and 30 mm in specimen 2 shows an increase in resistance ratio inversely proportional to the distance from the drying surface. This indicates a gradient of moisture over a greater depth than exhibited by the 0.45 w/c concrete (figure 6.07).

Figures 6.10 and 6.11 show contour plots of the final resistance ratio values in relation to the Autoclam test ring. Observations from figures 6.10 and 6.11 are as follows:

(i) There is evidence of a shallow bowl of increased resistance ratio underneath the Autoclam ring, in the 0.45 w/c ratio concrete.

(ii) The hot air preconditioning was seen to produce an increase in resistance ratio under the outer edge of the Autoclam ring.

From work by Basheer, (1991) and Arbaoui, (1988) it was expected that the removal of moisture from the capillary pore system would need to extend to the outside of the Autoclam ring (in plan) and to a depth of 40 mm directly below the centre of the clam ring, to eliminate the influence of moisture on the air permeability measurements. As can be seen from figures 6.11 and 6.12, both w/c ratio concretes had a gradient of resistance ratio and hence presumably a gradient of moisture up to a depth of 40 mm. This indicates that the air permeability measurements could be expected to be influenced by the moisture remaining in the concrete (Cf. chapter 4).

The volume of concrete influenced by the hot air preconditioning can be seen to extend beyond the outside edge of the Autoclam ring (in plan) for both w/c ratio concretes. This is unusual as it could be expected that a bowl of influence would be centred on the middle of the clam ring where the hot air was applied. The explanation for the spread of the zone of influence under the clam ring may be that the steel clam ring transmitted heat into the concrete underneath. An escape surface for the moisture in the concrete directly underneath the clam ring may have been
provided by the lateral component of the capillary pore system. This lack of a defined bowl shape is important as it indicates that a hot air preconditioning may be able to fulfil the requirement (for Autoclaim air permeability measurements) that the reduction of moisture should extend beyond the plan area of the clam ring.

6.4.1.2 Compressed air preconditioning

Resistance ratio graphs for the centre array of electrodes during the compressed air preconditioning are presented in figures 6.12 and 6.13. Observations from figures 6.12 and 6.13 are as follows:

(i) Figure 6.12 shows that the resistance ratio in the 0.45 w/c ratio concrete was only slightly altered over the period of preconditioning.

(ii) Initially, the resistance ratio of the concrete decreased due to compressed air preconditioning.

(iii) The 0.65 w/c ratio concrete shows larger increases in resistance ratio than the 0.45 w/c ratio concrete.

It was observed that over the preconditioning period a relatively small increase in the resistance ratio occurred in the 0.45 w/c specimen. Specimen 10 (figure 6.12) shows a slightly greater loss of moisture from the 5 - 10 mm region compared with the other depths, however this was not significant. This indicates that the moisture in the 0.45 w/c ratio concrete was altered only slightly by the compressed air preconditioning with no significant moisture gradients resulting. It is probable that only the largest pores in the concrete experienced a loss of moisture. The small pore diameter and low pore continuity associated with 0.45 w/c ratio concrete is likely to have resulted in a capillary pore system which did not allow significant moisture movement under the applied air pressure of 6 Bar.

The 0.45 w/c ratio specimen (figure 6.12) had resistance ratios less than unity immediately after the start of the preconditioning (generally in
the depth ranges further from the drying surface). The decrease in resistance ratio in an initially saturated concrete seems implausible as it indicates an increase in the quantity of moisture in the concrete pores (Mc Carter et al, 1995). The most likely reason for this increased resistance ratio may be that the capillary pores were not fully saturated initially and that the applied pressure forced water from the 0 - 5 mm zone of concrete into small capillary spaces at depth in the concrete which had not been fully saturated by capillary action. The initial decrease in resistance ratio values is shown to approach unity with time which may be due to the removal of moisture from the largest pores which offsets the initial increase in moisture.

The 0.65 w/c specimen generally showed a more significant increase in resistance ratio over the 40 mm depth when compared to the 0.45 w/c specimen. Again, as explained above, this can be related to the larger size and greater continuity of pores in the 0.65 w/c concrete. A large diameter pore that has lost most of its free water presents a larger obstacle to the flow of electrical current than a smaller pore.

Contours of final resistance ratio from specimens 16 and 3 are presented in figures 6.14 and 6.15. The 'shallow bowl' feature is evident in both specimens 10 and 3. Of the form shown in figure 6.16, this moisture profile is a direct result of the way in which the compressed air preconditioning was applied to the centre of the clam ring. The largest reduction in moisture being directly below the surface of the concrete where the preconditioning is applied. The amount of moisture removed from the capillary pores is reduced with increased depth from the surface and increased lateral distance from the centre of the clam ring. The electrode arrangement adopted for this experiment did not allow the measurement of resistance for depths less than 5 mm, nevertheless the
results presented in figures 6.14 and 6.15 suggest that the bowl shape is centred on the test area of the clam ring.

6.4.1.3 Vacuum preconditioning

The resistance ratio results for the centre array of electrodes in the 0.45 and 0.65 w/c ratio concretes are presented in figures 6.17 and 6.18 respectively. The corresponding contour diagrams of final resistance ratio are presented in figures 6.21 and 6.22 respectively. The results are taken during the application of a vacuum through an Autoclam ring and the final resistance measurement corresponds to the end of the vacuum preconditioning.

(i) The 0.45 and 0.65 w/c ratio concretes displayed similar increases in resistance ratio over the preconditioning period in the 5 - 10 mm region of the concrete.

(ii) Resistance ratio values did not increase significantly for depths in excess of 25 mm.

(iii) After an initial decrease the resistance ratio values generally increased with time.

The rise in resistance ratio in each of the w/c ratio concretes in the 5 - 10 mm depth zone varied between 1.5 and 2.0. Although the final resistance ratios for both w/c concretes are similar (in the 5 - 10 mm region) it does not necessarily imply that the same quantity of moisture has been removed from each specimen (as their porosities are different)(c.f. chapter 4, figure 4.13).

Very few resistance ratio values significantly exceeded unity which indicates that the vacuum preconditioning was generally ineffective in removing moisture from the capillary pores of the concrete. The 0.45 w/c ratio specimen had a significant reduction in resistance ratio up to 15 mm. The effective depth of significant moisture loss in the 0.65 w/c ratio
specimens was up to 20 mm. This implies that, the effective depth of the vacuum preconditioning was greater in the 0.65 w/c ratio concrete. This is a reflection of the greater pore size and continuity associated with the concrete with the greater w/c ratio, as discussed previously. This depth is in excess of 40 mm therefore it could be expected that the air permeability of the concrete would be influenced by the moisture remaining in the pore system (Basheer, 1991 and Arbaoui, 1988).

All concretes preconditioned using the vacuum technique showed an initial drop in resistance ratio values (especially at deeper electrodes in the concrete). This is probably due to the nature of the preconditioning technique because the vacuum could be expected to pull moisture towards the test surface which led to an increase in the quantity of moisture in the capillary pores of the concrete. This initial decrease in resistance ratio (increase in saturation) was degraded during the preconditioning with the resistance ratio values increasing towards unity before the end of the 24 hours test. This indicates that the moisture was being removed from the capillary pore system at all depths, although in many cases the removal of moisture was only sufficient to return the concrete to its initial 'saturated' state. Larger increases in resistance were evident in the concrete nearest to the surface with final resistance values indicating a net loss of moisture (resistance ratio greater than unity).

The exception to this general trend was displayed in one of the 0.45 w/c ratio specimens (Specimen 14 - presented in figure 6.19) which showed a very significant initial decrease in resistance ratio in the 10 - 25 mm depth region. The continued application of a vacuum did not influence these readings significantly. The resistance ratio of the concrete between 5 and 10 mm depth increased over this period and that the resistance ratio of the concrete below a depth of 25 mm showed no large deviation from unity. This identifies three specific depth regions which
were affected by the vacuum preconditioning very differently. As shown in figure 6.20, the regions can be categorised in terms of the moisture movement over the preconditioning period.

Figure 6.21 shows the contours of final resistance ratio in specimen 14 and it highlights an area of concrete that is more saturated than the initial condition around the 15 and 20 mm electrodes under the centre of the clam ring. Although saturated over 4 days by capillarity, the concrete could be expected to contain small voids unfilled with water, therefore the decrease in resistance ratio is possible upon the application of a pressure gradient. Above 10 mm depth there is a reduction in the quantity of moisture in the concrete. One explanation for this unusual moisture profile is that initially moisture was drawn towards the vacuum surface and after this period air from outside the clam ring began to displace moisture in the pores of the uppermost 10 mm of the concrete as shown in figure 6.19. Concrete below 25 mm at the centre seems to have been reasonably unaffected by the vacuum preconditioning.

The following observations can be made from figures 6.21 and 6.22 which present contour plots of the final resistance ratio values in specimens 11 and 1:

(i) A shallow bowl of reduced resistance ratio is visible in specimen 11.

(ii) An area of high resistance underneath the body of the clam ring in specimen 1.

Figure 6.21 shows that a bowl of increased resistance ratio (and hence reduced moisture) is clearly visible in the uppermost 15 mm of the concrete underneath the clam ring. It is most probable that the vacuum removed moisture from directly underneath the test area and also laterally from underneath the clam ring from the direction of the nearest free air surface (outside the clam ring).
The resistance ratio contour graph for the 0.65 w/c ratio specimen (figure 6.22) shows an elongated area of higher resistance ratio (lower moisture) underneath the clam ring. This indicates that a lateral movement of moisture was prevalent. The depth of influence of the vacuum preconditioning is substantially greater than in the 0.45 w/c ratio concrete and this may be due to the more open pore system which allowed more vertical movement of moisture towards the vacuum surface.

### 6.4.1.4 Solvent replacement preconditioning - Diethyl Ether

It was found during testing that the concrete exhibited signs of hairline cracking directly above the electrodes near to the surface of the concrete. At the sides of the specimen where the electrodes protruded there was evidence of solvent weeping out. Both concrete and mild steel bars were subsequently exposed to solvent separately and it was found that both of them were damaged by the solvent, thus the observed cracking was attributed to the diethyl ether solvent. Cracking of the concrete on the surface (plate 6.03) led to a large ingress of solvent into the concrete over the full 24 hour period of the preconditioning test.

The resistance ratio variation with time for the centre array of electrodes for the solvent replacement technique is shown in figures 6.23 (0.45 w/c concrete) and 6.24 (0.65 w/c concrete). The final resistance value is taken at the end of the solvent application period.

Observations from figures 6.23 and 6.24

(i) An instantaneous increase in the resistance ratio of the concrete occurred to a depth of at least 15 mm in all concretes.

(ii) Increases in resistance ratio were recorded in all concretes to depths of 40 mm over the 24 hour preconditioning period.
Resistance ratio values recorded were generally found to be in excess of the corresponding values recorded after hot air, vacuum and compressed air preconditioning.

Significant increases in resistance ratio values were recorded at a range of depths after a period of 30 minutes solvent which indicates a very rapid advance of the solvent into the concrete. Increases in resistance ratio were recorded in all concretes to a depth of 40 mm during the preconditioning period. This indicates that the solvent reached depths in excess of 40 mm over the 24 hours preconditioning period. The lower resistance values, common at greater depth (seen in figures 6.23 and 6.24) in the concretes, suggest that the solvent displaced less capillary water in this region.

Resistance ratio values recorded were generally found to be in excess of the corresponding values recorded after hot air, vacuum and compressed air preconditioning. This may indicate that the moisture in the smaller capillary pores of the concrete was displaced by the solvent.

The following observations can be made from the final resistance ratio contour plots of specimens 8 and 9 are presented in figures 6.25 and 6.26.

(i) Specimen 9 shows a bowl of increased resistance ratio.
(ii) Specimen 8 (0.65 w/c ratio concrete) shows a confused pattern of high resistance ratio values.

The bowl of increased resistance ratio evident in specimen 9 is centred on the test area inside the clam ring. The rise in resistivity ratio to depths in excess of 40 mm along the centre array of electrodes could imply that there was preferential ingress of solvent at the centre of the clam ring. Significant increases in resistance ratio can also be seen under the edge of the clam ring to a depth of 15 mm which may be inconsistent with the expected 'shallow bowl' of solvent ingress. However, the evidence is not
conclusive as much of the necessary detail is contained in the uppermost 5 mm of the concrete (an area not covered by electrodes).

Specimen 8 (figure 6.26) shows a much more confused pattern of final resistance ratio where the 'shallow bowl' feature is not evident. Four areas of high resistance ratio can be identified in the concrete at various depths and at various positions relative to the clam ring. This suggests that the solvent moved preferentially into these areas, perhaps caused by inhomogeniety of the concrete pore system. It seems unusual that the shallow bowl which could be expected to be related to the ingress of a solvent into concrete is not present. The reason why the concrete at these specific areas should have exhibited a larger increase in resistance than surrounding concrete is unclear.

6.4.1.5 Solvent replacement preconditioning - Isoproponol

The fact that the diethyl ether caused damage to the concrete meant that no conclusions could be drawn as to the suitability of a solvent replacement technique for preconditioning concrete. Thus, the investigation into the solvent replacement preconditioning method was repeated with similar specimens using isoproponol as the solvent. It should be noted that the specimens used were cast using the same procedure and constituents as the other specimens investigated in this chapter. The replacement specimens were, however, considerably more mature (approximately one year) and so direct comparisons with the results of other preconditioning techniques should be considered tentative.

It was found in preliminary tests (using spare specimens with embedded electrodes) that the ingress of isoproponol under a driving pressure of 1 Bar was very modest, and as a result, the application pressure was increased to 2 Bar. Figures 6.27 and 6.28 show the final resistance ratio
variation for the centre column of electrodes in the 0.45 and 0.65 w/c ratio concretes, respectively.

Observations from figures 6.27 and 6.28:

(i) Increases in resistance ratio at any given depth were generally larger in the 0.65 w/c ratio concrete than in the 0.45 w/c ratio.

(ii) The time to the first increase in resistance ratio was related to the depth of the electrode pair from the concrete surface.

(iii) Large increases in resistance ratio ($R_t/R_i > 5$) were recorded in specimens at 5 to 10 mm depths in the 0.65 w/c ratio concrete.

The greater increase in resistance ratio in the 0.65 w/c ratio concrete is due to the more continuous pores (Powers, 1958) of larger diameter (Nyame, 1979) which are present in the concrete. The larger pore continuity facilitates the ingress of solvent (and the consequent removal of moisture) to a greater depth in the concrete. This can be seen by comparing the depth of concrete which exhibits a rise in resistance ratio - 10 mm in specimen 18 (figure 6.27) compared to 20 mm in specimen 19 (figure 6.28). The solvent has a large resistance to the flow of electrical current and therefore a solvent filled pore acts in much the same way as an air filled pore. The larger pore presents a larger obstruction to the passage of current through the concrete and therefore is responsible for a larger increase in resistance ratio.

The time to the first increase in resistance ratio was dependent on the depth of the electrode pair from the concrete surface in concretes subjected to isoproponol replacement preconditioning. This is a feature which can be explained as it represents a reasonably well defined front of solvent advancing into the concrete over the preconditioning period. The resistance ratio of the concrete increases after the passing of the solvent front which suggests that the solvent ingress may occur preferentially in the largest capillary pores and that the smaller capillary pores become...
filled later after the front has past. Therefore the rate at which the solvent moves into the concrete may be proportional to the diameter of the capillary pore. The Poiseuille equation [6.01] for fluid flow in a fine tube is,

$$\frac{dv}{dt} = \frac{5 \times 10^4 \pi H r^4}{4L \eta}$$ \hspace{1cm} \text{Eqn. 6.01}

Where \( \frac{dv}{dt} \) is the flow rate,
H is the head of pressure (with the applied head significant in this case),
r is the radius of the capillary,
L is length of the capillary pore filled with liquid and
\( \eta \) is the viscosity of the liquid.
Dividing by the cross sectional area of the pore to obtain the velocity of flow gives,

$$V = \frac{5 \times 10^4 \pi H r^4}{4L \eta (\pi r^2)} = \frac{5 \times 10^4 H r^2}{4L \eta}$$ \hspace{1cm} \text{Eqn. 6.02}

For a given pore system and applied head, the length of the pore filled with liquid, the viscosity of the liquid and the head are constant, then:

$$V \propto r^2$$ \hspace{1cm} \text{Eqn. 6.03}

This indicates that the larger the pore the greater the speed of fluid flow (both solvent and capillary water) which could be expected. This may explain the form of the resistance ratio graphs shown in figures 6.27 and 6.28.

Large increases in resistance ratio (\(R_t/R_i > 5\)) were recorded in specimens in the 5 - 10 mm depth zone. If the findings of chapter 4 are considered then this indicates that the solvent replaced much of the moisture in the capillary pores in this zone of the concrete. The smaller increases in resistance ratio in areas below 15 mm indicate that only the isoproponol may have only penetrated the larger pores in this region.
damage to the concrete specimens was observed in the isoproponol preconditioning tests.

Contour plots for the final resistance ratio values in specimens 18 and 19 are shown in figures 6.29 and 6.30. The final conductance measurements were taken at the end of the solvent application period and therefore the resistance values are influenced by the solvent which was in the pores of the concrete.

Observation from figures 6.29 and 6.30:

(i) A well defined 'bowl shape' of resistance ratio is apparent under the clam ring in each concrete.

(ii) A gradient of resistance ratio is evident in both the concretes investigated.

The well-defined 'shallow bowl' of increased resistance ratio gives a clear indication of the volume of concrete which the solvent occupied after the 24 hours of application. The maximum depth of the bowl of solvent ingress is in the order of 15 - 20 mm in both specimens 18 and 19. This may be related to the long period of water curing which the isoproponol specimens were subjected as capillary discontinuity may have developed in both the 0.45 and 0.65 w/c ratio concretes. This is in contrast to the specimens subjected to other preconditioning techniques which indicated very dissimilar capillary pore systems in the 0.45 and 0.65 w/c ratio concretes.

For the removal of the influence of moisture on Autoclam air permeability measurements the moisture would have to be removed from pores of size 0.021 μm in 0.65 w/c ratio concrete (RH of 90%, c.f. chapter 4). The corresponding diameter is 0.006 μm diameter (70% RH) for the 0.45 w/c ratio concrete (chapter 4). The 0.45 w/c ratio concrete (max. Rt/Ri >1.8) shows a smaller rise in resistance ratio compared to the 0.65 w/c ratio (max. Rt/Ri >8) which may indicate that it was only in the largest
pores that the water in the capillary pores of the 0.45 w/c ratio concrete was replaced by solvent. This indicates that the 0.45 w/c ratio concrete may require a longer period of solvent application or a higher application pressure than the 0.65 w/c ratio concrete.

6.4.2 Air permeability measurements

The first test carried out on the preconditioned specimens was an Autoclam air permeability test. The full results of these tests are presented in figures 6.31 and 6.32. Each value in these diagrams represents an average of the two test results from the corresponding two specimens. A similar concrete specimen which was dried for 7 days drying at 70°C was shown to have RH of 35.5% at 30 mm depth, and therefore was considered to have a capillary pore system free from moisture. Air permeability measurements were not carried out on the specimens preconditioned using diethyl ether solvent as the specimens were surface cracked.

Observations from figures 6.31 and 6.32 are as follows:

(i) No preconditioning method resulted in air permeability values comparable to ovendried concrete.
(ii) The air permeability of the 0.45 w/c ratio concrete subjected to vacuum preconditioning was similar to that of its oven dried counterpart.
(iii) The application of hot air could distinguish the effect of w/c.
(iv) Variability in oven dried air permeability indices between each of the concretes is evident.

Figures 6.31 and 6.32 indicate that no preconditioning method was completely successful in removing the influence of moisture on the air permeability of concrete. As discussed in previous chapters, the air permeability of the concrete can be expected to be reasonably constant in a concrete with no free capillary water and a RH between 20 and 65%, irrespective of the w/c ratio of the concrete (figure 2.36, Parrott, 1994b).
Therefore the air permeability of oven dried concrete which had a RH of 35% at 30 mm could be expected to give a good indication of what the air permeability of a concrete without moisture in the capillary pore system. Thus, by comparing the air permeability results between preconditioned and oven dried concrete, the presence of moisture in the capillary pore system, which influences the air permeability of concrete can be identified. The finding that no preconditioning method was successful in removing the influence of moisture on the air permeability was predicted from the results of the final resistance ratio plots underneath the clam ring (section 6.4.1). These plots showed that the volume of concrete from which the significant removal of moisture caused by preconditioning did not extend to the limits of influence of the Autoclam apparatus (40 - 50 mm) as determined by Basheer (1991) and Arbaoui (1988) (Section 2.3.5.1).

That the vacuum preconditioning method was seen to be ineffective at removing the influence of moisture is in direct conflict with the findings of Dhir (1993). He found that a vacuum applied through an ISAT apparatus (figure 6.33(a)) could reduce the influence of moisture on sorptivity indices. Dhir's work does not state categorically that the preconditioning procedure removed the effect of moisture on absorption tests, although there is an implied inference that it did (See section 6.4.4). The geometry of the ISAT apparatus (figure 6.33(b)) compared to that of the Autoclam test ring means that the depth of influence of a preconditioning technique could be expected to be considerably deeper in the Autoclam vacuum preconditioning. Since Dhir (1993) does not report any measurement of moisture in the preconditioned concrete, it is uncertain how much moisture was removed or to what depth the preconditioning extended.

That a concrete contains pores of different size (Nyame, 1979) also tends to suggest that vacuum preconditioning may be unsuitable for the
removal of all the moisture from concretes capillary pores since the largest pores could be expected to empty first. These large pores once empty would provide a path for air from the surface of the concrete to reduce the vacuum and thus the efficiency of the preconditioning method could be expected to fall off with the increased removal of moisture. This may result in the moisture from smaller pores never being removed (depending on the distribution of pore sizes in the concrete). The 0.45 w/c ratio concrete has smaller range of pore sizes, (Nyame, 1979) and hence could be expected to result in a greater removal of moisture due to vacuum preconditioning. Figures 6.31 and 6.32 show that the air permeability index of 0.45 w/c ratio concrete is considerably closer to the oven dried result than the corresponding 0.65 w/c ratio concrete (larger range of pore sizes). This is contrary to what could have been normally been assumed since 0.45 w/c ratio concretes must have a RH of 65 % (0.65 w/c ratio concrete requires a RH of 90%) to avoid the influence of free moisture in the capillary pores (Parrott, 1994b and figure 2.36). The concept of using an overpressure of air to force moisture from the concrete could also be expected to be influenced by the distribution of pore sizes in a concrete (Compressed air preconditioning).

The hot air preconditioning method could distinguish between the two w/c ratio concrete concretes (figures 6.31 and 6.32). This is important in so much as it allows a concrete with a comparatively continuous (Powers, 1958) system of large (Nyame, 1979) capillary pores (0.65 w/c ratio concrete) to be differentiated from a concrete with a less continuous system of smaller sized pores (0.45 w/c ratio concrete) irrespective of the initial moisture condition. The pore systems of the 0.45 and 0.65 w/c ratio concretes could be expected to be relatively extreme examples of the range of pore continuity and size encountered on site therefore it can only be claimed that the hot air preconditioning can be used to distinguish
between very disparate concrete pore structures irrespective of the initial moisture condition. In direct contrast to the vacuum preconditioning, the hot air preconditioning was most effective on the 0.65 w/c ratio concrete (figure 6.32). This is presumably due to the larger pore sizes that are common in the 0.65 w/c ratio concrete which could be expected to lose the ability to sustain a meniscus more readily than smaller pores. In addition, figure 2.36 indicates that the loss of moisture from larger (> 90% RH = > 0.021μm diameter for 0.65 w/c ratio concrete) pores is sufficient to achieve an air permeability relatively free from the effects of moisture.

Basheer, (1991) and Schonlin, (1987) used a hot air preconditioning method to remove or reduce the influence of moisture on permeation tests. Schonlin makes no claim as to the effectiveness of his treatment nor does he present any data to support its use. Basheer (1991) did a short series of tests which investigated the length of surface drying using a gas heater on permeation test results and concluded that for periods of drying in excess of 20 minutes no significant change occurred. This was taken to mean that the influence of moisture was removed from Autoclam measurements however no measurement of the moisture condition of the concrete before and after was taken. It is conceivable that much moisture remained in the concrete and that the rate of moisture loss became very small after 20 minutes. The conclusion that the influence of moisture on the Autoclam test results was removed by 20 minutes application of surface heat is not supported by the results of this investigation.

A significant variability in the oven dried air permeability indices can be seen in the 0.65 w/c ratio concrete (figure 6.32). In theory, these values could be expected to be similar (with the exception of the isoproponol solvent replacement specimens which had an extended period of curing). In practice this may not have occurred due to a
combination of the inhomogeneity of concrete or specimen-to-specimen variation. In addition, the air permeability indices were calculated as the average of two air permeability tests, while Basheer (1991) states that the average of three results is required to avoid errors. Since all the 0.65 w/c ratio concrete was manufactured from the one mix of concrete, the constituents were similar. The compaction was to a standard procedure (outlined in chapter 3) and therefore the specimen-to-specimen variation was minimised. Despite this, it can be seen from figure 6.32 that the oven-dried air permeability in the 0.65 w/c ratio concrete showed significant variability.

The difference between the air permeability after oven drying between the specimens is most marked in the 0.65 w/c ratio concrete used for isopropanol solvent preconditioning. As noted earlier, the replacement specimens were made separately for the isopropanol replacement technique and although they were manufactured using the same constituent materials, mix and casting procedures the specimens were water cured for an extended period. This can be assumed to have promoted extensive hydration and from Powers (1958) the pore system in the 0.65 w/c ratio concrete could be expected to have become discontinuous over this period. This is reflected in the low air permeability measured after oven drying in the 0.65 w/c ratio concrete when compared to the other oven-dried air permeability indices (figure 6.32).

6.4.3 Internal relative humidity measurements

Relative humidity measurements were carried out in a hole drilled off centre of the Autoclam test area, as shown in figure 6.06, in order to avoid the embedded electrodes. As a result of the confined test area and the requirement to avoid the embedded electrodes only one relative
humidity measurement per test slab was carried out. Therefore the RH measurements in the 15 mm deep hole were carried out on one slab while the RH measurement in a 30 mm hole was carried out on its counterpart. One consequence of the site of the RH measurement is that it cannot be said to be representative of the concrete at the centre of the test area.

Table 6.03 shows the relative humidity values at 15 mm and 30 mm depths from the test surface for the four preconditioning methods.

Observations from table 6.03

(i) All values of RH are equal to or in excess of 90 % RH.
(ii) The lowest values of RH were generally caused by hot air preconditioning.

If 65% RH is taken as the condition at which all of the capillary pores in a concrete are substantially free from moisture (Parrott, 1994 and Hansen, 1987) none of the preconditioning techniques were successful in achieving this state at either 15 or 30 mm depth. Chapter 5 indicated that below a RH of 80% at 10 mm depth, the influence of moisture could be removed by using the established relationships between in-situ Autoclam test results and RH. As can be seen from table 6.03 the values of RH indicate that the RH at 15 mm depth measured in concrete subjected to any preconditioning were not close to 80%. This indicates that the preconditioning methods would not always be suitable for use on-site in cases where the concrete had an initial RH greater than 80%.

That the electrodes prevented the measurement of RH at the centre of the clam ring meant that the RH measurements, shown in table 6.03, were influenced by concrete close to the edge of the clam ring (see figure 6.06). This probably caused RH measurements to be higher than if they were taken at the centre of the ring since concrete away from the centre of the ring was generally found to exhibit less moisture loss ('shallow bowl' identified in section 6.4.1). It should be noted that the RH measurements
were inside the volume of concrete which influences Autoclam tests, as found by Basheer (1991) and Arbaoui (1988).

The hot air preconditioning technique was responsible for the lowest RH measurements obtained in the investigation (Table 6.03). This supports the finding that the hot air preconditioning was the most successful at removing the influence of moisture on the 0.65 w/c ratio concrete. Parrott (1994b) and the findings of chapter 4, indicate that high w/c ratio concretes of 90% RH have an air permeability index which is similar to that of the same concrete at lower RH (but greater than 20% RH). Therefore the finding that 90% RH at 30 mm depth indicates that the air permeability value may have been free from a substantial amount of the influence of moisture. The RH measurement at 15 mm in the similar specimen was 98% which does not indicate a similar moisture profile in the other 0.65 w/c ratio specimen.

6.4.4 Significance of test results

The effectiveness of the hot air preconditioning is unexpected in that the period of heat application was 12 hours while the application of the other preconditioning techniques continued for 24 hours. This indicates that the application of hot air to the surface of the concrete may be the most effective method of preconditioning concrete prior to permeation tests. The time of hot air preconditioning required to remove the influence of moisture from the air permeability indices (with a maximum concrete temperature of 100° C) is in excess of 12 hours and this seems impractical for use on-site. The time required for the compressed air, vacuum application and solvent replacement techniques to remove the free moisture from the volume of concrete influencing an Autoclam air permeability test could be expected to be more than 24 hours. These
techniques could be expected to be less practical than the hot air preconditioning technique.

It has been shown by the investigation that the removal of moisture from the capillary pores requires a significant input of energy to the concrete. This, coupled with the absolute requirement to avoid damage to the microstructure of the concrete, suggests that a preconditioning procedure would need to be applied over a considerable period of time, perhaps days. As the prospect of preconditioning concrete for periods of days prior to one Autoclaim measurement appears unattractive the obvious conclusion is that the concept of using short term preconditioning prior to permeation tests to remove the influence of moisture seems impractical.

Another method of preconditioning which may reduce the internal RH in exposed concrete to below 80% at 10 mm, would be the use of long-term shielding of the test concrete. A shield device could be affixed to the test area and left for an extended period, until the RH measurements at 10 mm decreased to below 80% RH. Insitu Autoclaim tests could than be carried out and assessed using the procedures detailed in chapter 5.

6.5 Summary of findings

The major findings of the investigation are listed below:

(i) Hot air preconditioning was found to be the most efficient method of removing moisture from concrete.
(ii) The vacuum and compressed air preconditioning techniques were shown to be less effective than hot air preconditioning and there is doubt as to whether these methods are suitable for use on a porous body with a distribution of pore sizes.
(iii) The use of diethyl ether as a solvent for replacing the free moisture in the capillary pores of concrete was found to cause significant cracking in the concrete.

(iv) A 'shallow bowl' of reduced moisture in the concrete caused by many of the preconditioning tests was identified by resistance measurements between embedded electrodes.

(v) On the basis of the results obtained in this investigation, the concept of short-term preconditioning concrete prior to permeation tests appears impracticable.
### Table 6.01 - Experimental variables.

<table>
<thead>
<tr>
<th>Variable</th>
<th>Levels</th>
</tr>
</thead>
<tbody>
<tr>
<td>W/C ratio</td>
<td>0.45 and 0.65</td>
</tr>
<tr>
<td>Preconditioning Method</td>
<td>Hot Air application, Compressed Air application, Vacuum application and Solvent Replacement</td>
</tr>
</tbody>
</table>

### Table 6.02 - Specimen details

<table>
<thead>
<tr>
<th>Specimen No.</th>
<th>w/c ratio</th>
<th>Preconditioning method</th>
</tr>
</thead>
<tbody>
<tr>
<td>2, 6</td>
<td>0.65</td>
<td>Hot air application</td>
</tr>
<tr>
<td>3, 7</td>
<td>0.65</td>
<td>Compressed air application</td>
</tr>
<tr>
<td>1, 5</td>
<td>0.65</td>
<td>Vacuum application</td>
</tr>
<tr>
<td>4, 8</td>
<td>0.65</td>
<td>Diethyl ether solvent replacement</td>
</tr>
<tr>
<td>12, 15</td>
<td>0.45</td>
<td>Hot air application</td>
</tr>
<tr>
<td>10, 16</td>
<td>0.45</td>
<td>Compressed air application</td>
</tr>
<tr>
<td>11, 14</td>
<td>0.45</td>
<td>Vacuum application</td>
</tr>
<tr>
<td>9, 13</td>
<td>0.45</td>
<td>Diethyl ether solvent replacement</td>
</tr>
<tr>
<td>17, 18</td>
<td>0.45</td>
<td>Isoproponol solvent replacement</td>
</tr>
<tr>
<td>19, 20</td>
<td>0.65</td>
<td>Isoproponol solvent replacement</td>
</tr>
<tr>
<td>Depth from surface (mm)</td>
<td>W/C ratio</td>
<td>Precondition Method</td>
</tr>
<tr>
<td>------------------------</td>
<td>-----------</td>
<td>---------------------</td>
</tr>
<tr>
<td>15</td>
<td>0.45</td>
<td>Vacuum</td>
</tr>
<tr>
<td>15</td>
<td>0.45</td>
<td>Hot Air</td>
</tr>
<tr>
<td>15</td>
<td>0.45</td>
<td>Compressed. Air</td>
</tr>
<tr>
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<td>15</td>
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<td>15</td>
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</tr>
<tr>
<td>30</td>
<td>0.65</td>
<td>Isoproponol</td>
</tr>
</tbody>
</table>
Plate 6.01 - Electrodes in specimen mould prior to casting

Plate 6.02 - Conductance measurement during preconditioning
Plate 6.03 - Surface cracking of the concrete caused by diethyl ether
Figure 6.01 - Applied hot air preconditioning apparatus

Figure 6.02 - Compressed air preconditioning technique
Figure 6.03 - Vacuum preconditioning technique

1 Bar for Diethyl ether, 2 Bar for Isoproponol.

Piston arrangement bolted onto ring

Standard Autoclam base ring bonded to concrete surface

The advance of solvent forces moisture out of concrete test zone

Figure 6.04 - Solvent replacement preconditioning technique
Figure 6.05 - Specimen details

Figure 6.06 - Electrode Details
Resistance Ratio (Rt/Ri)

4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0 250 500 750 1000 1250 1500
Time (mins)

0.45 w/c concrete centre-line electrodes - Specimen 12
Note: Figure 6.11 shows final resistance ratio contours for specimen 12

Figure 6.07 - 0.45 w/c concrete resistance ratio variation before and after hot air preconditioning. Specimen 12.

0.65 w/c concrete centre-line electrodes - Specimen 2
Note: Figure 6.12 shows final resistance ratio contours for specimen 2.

Figure 6.08 - 0.65 w/c concrete resistance ratio variation before and after hot air preconditioning. Specimen 2.
Moisture gradient over a smaller depth

Slower moisture removal from smaller pores

Preferential moisture loss from largest pores

Lower w/c concrete = smaller pore diameters & reduced total pore space

Cement paste + aggregate
Moisture in capillary pores

Moisture gradient over a larger depth

Quicker moisture removal from larger pores

Preferential moisture loss from largest pores

Higher w/c concrete = larger pore diameters & increased total pore space

Figure 6.09 - Moisture removal from concrete - diagrammatic representation
Figure 6.10 - Resistance Ratio contour plot.
Hot air preconditioning. Specimen 12.

Figure 6.11 - Resistance Ratio contour plot.
Hot air preconditioning. Specimen 2.
0.45 w/c concrete centre-line electrodes - Specimen 16
Note: Figure 6.15 shows final resistance ratio contours for specimen 16

Figure 6.12 - 0.45 w/c concrete resistance ratio variation during compressed air preconditioning Specimen 16.

0.65 w/c concrete centre-line electrodes - Specimen 3
Note: Figure 6.16 shows final resistance ratio contours for specimen 3

Figure 6.13 - 0.65 w/c concrete resistance ratio variation during compressed air preconditioning Specimen 3.
Figure 6.14 - Final resistance ratio contour plot. Compressed air preconditioning. Specimen 10.

Figure 6.15 - Final resistance ratio contour plot. Compressed air preconditioning. Specimen 3.
Figure 6.16 - Moisture distribution in concrete after preconditioning
Resistance Ratio (Rt/Ri)

- 5 - 10 mm
- 10 - 15 mm
- 15 - 20 mm
- 20 - 25 mm
- 25 - 30 mm
- 30 - 35 mm
- 35 - 40 mm

Time (mins)

0.45 w/c concrete centre-line electrodes - Specimen 11
Note: Figure 6.22 shows final resistance ratio contours for specimen 11

Figure 6.17 - 0.45 w/c concrete resistance ratio variation during vacuum preconditioning. Specimen 11.

0.65 w/c concrete centre-line electrodes - Specimen 1
Note: Figure 6.23 shows final resistance ratio contours for specimen 1

Figure 6.18 - 0.65 w/c concrete resistance ratio variation during vacuum preconditioning. Specimen 1.
Figure 6.19 - Schematic section of specimen 14 after 24 hours vacuum preconditioning

Figure 6.20 - Contour diagram of specimen 14 after 24 hours vacuum preconditioning
Figure 6.21 - Resistance Ratio contour plot.
Vacuum preconditioning. Specimen 11.

Figure 6.22 - Resistance Ratio contour plot.
Vacuum preconditioning. Specimen 1.
Resistance Ratio (Rt/Ri)  

Diethyl Ether solvent used.

0.45 w/c concrete centre-line electrodes - Specimen 9

Note: Figure 6.26 shows final resistance ratio contours for specimen 9

Figure 6.23 - 0.45 w/c concrete resistance ratio variation during solvent replacement preconditioning. Specimen 9.

0.65 w/c concrete centre-line electrodes - Specimen 8

Figure 6.27 shows final resistance ratio contours for specimen 8

Figure 6.24 - 0.65 w/c concrete final resistance ratio variation during solvent replacement preconditioning. Specimen 8.
**Figure 6.25** - Final resistance ratio contour plot - Diethyl ether solvent replacement technique. Specimen 9.

**Figure 6.26** - Resistance Ratio contour plot - Diethyl ether solvent replacement technique. Specimen 8.
**0.45 w/c concrete centre-line electrodes - Specimen 18**

Note: Figure 6.30 shows final resistance ratio contours for specimen 18

**Figure 6.27** - 0.45 w/c concrete resistance ratio variation during isoproponol replacement preconditioning Specimen 18.

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**0.65 w/c concrete centre-line electrodes - Specimen 19**

Note: Figure 6.31 shows final resistance ratio contours for specimen 19

**Figure 6.28** - 0.65 w/c concrete final resistance ratio variation during isoproponol replacement preconditioning Specimen 19.
Specimen 18 - 0.45 w/c concrete

**Figure 6.29** - Final resistance ratio contour plot. Isopropyl alcohol replacement preconditioning. Specimen 18.

Specimen 19 - 0.65 w/c concrete

**Figure 6.30** - Final resistance ratio contour plot. Isopropyl alcohol replacement preconditioning. Specimen 19.
Preconditioning

Note: Diethyl ether results unavailable due to specimen damage

0.45 w/c ratio concrete - Air Permeability results

Figure 6.31 - Autoclam air permeability indices - Oven dried and after preconditioning

Preconditioning

Note: Diethyl ether results unavailable due to specimen damage

0.65 w/c ratio concrete - Air Permeability results

Figure 6.32 - Autoclam air permeability indices - Oven dried and after preconditioning
(A) Vacuum applied through an ISAT apparatus (Dhir, 1993)

Wider seal causes removal of moisture to a greater depth

(B) Vacuum applied through a clam ring

Figure 6.33 - Differences between ISAT and Autoclam vacuum preconditioning methods.
Conclusions and recommendations for further research

7.0 Conclusions

Conclusions based on the investigations presented in this thesis:

7.0.1 Investigation into the relationship between air permeability, sorptivity and the moisture condition of concrete

(i) Air permeability indices of laboratory specimens were shown to fit the relationship between air permeability and relative humidity proposed by Parrott, (1994b).

(ii) A straight line relationship between sorptivity and the relative humidity in a 10 mm drilled hole was identified in the laboratory investigation.

7.0.2 The relationship between permeation measurements and environmental conditions in exposed concrete

(i) Theoretical and experimental studies indicated that the effect of temperature on Autoclam air permeability measurements was negligible. The effect of temperature on Autoclam sorptivity measurements was found to be significant and a temperature correction factor was developed to transform Autoclam sorptivity indices to an equivalent sorptivity index at 20 °C. Thus the effect of ambient temperature on Autoclam measurements can be removed.
(ii) The existence of relationships between RH measured in a 10 mm hole and in-situ Autoclam air permeability indices and in-situ sorptivity indices were identified in concrete with a RH of less than 80% at a depth of 10 mm. In concrete with a RH greater than 80% (at 10 mm depth) the differences in permeation properties between concretes of different quality are obscured. Methodologies for removing the influence of different moisture conditions on Autoclam air permeability and sorptivity measurements in exposed concrete with a RH less than 80% at 10 mm depth, were developed.

(iii) The results of the outdoor trials indicate that exposed concrete should not be tested before the age of, at least, 6 months in order to prevent the possibility of incomplete hydration influencing the Autoclam measurements.

7.0.3 Preconditioning to remove moisture from the concrete prior to permeation testing

(i) A method of preconditioning concrete prior to permeation tests to remove the influence of moisture was not identified in this investigation.

(ii) Hot air preconditioning was found to be the most efficient method of removing moisture from concrete. The vacuum and compressed air preconditioning techniques were assessed as being unsuitable for use on a porous body with a range of pore sizes.

7.1 Recommendations for insitu permeation testing

On the basis of the findings of this thesis the following guidelines for permeation testing of exposed structures are proposed:

(i) The tests should be carried out after an extended period of dry weather, normally during the summer months of the year.
(ii) In general, the ambient temperature at the time of RH measurement should be as high as possible. If sunlight shines directly onto the test area then it may prove beneficial to test the concrete during this time.

(iii) The RH in a 10 mm drilled hole in the concrete should be less than 80% to allow permeation testing. If the RH measurement is in excess of 80% then the test area should be shielded from rain for a period, after which the RH should be tested again. The period of time between RH measurements is arbitrary and, although the period of roughly one month is suggested, longer or shorter periods may be used as appropriate.

(iv) Permeation tests should be carried out recording the value of RH at 10 mm depth (less than 80%) and the ambient temperature.

(v) Autoclam sorptivity measurements at specific temperatures should be converted to equivalent values at 20 °C using:

$$SI_{20} = SI \times [1.6385 - 3.8834e-2 (T) + 3.5132e-4 (T^2)]$$

where SI is the measured sorptivity index at temperature T (°C).

(vi) Using the air permeability and/or temperature corrected sorptivity indices with the RH measured at 10 mm depth, an estimation of the quality of the concrete can be made from figures 5.27 and 5.33.

7.2 Recommendations for further research

The use of RH and Autoclam permeation measurements has been shown to provide an indication of the quality of reasonably dry in-situ concrete. However, the following require further research before these two together can be used to qualify in-situ concrete for durability:

(i) Preconditioning of in-situ concrete.

(ii) Classification of concrete for durability.

(iii) The effect of temperature and moisture on sorptivity indices.
7.2.1 **Preconditioning of in-situ concrete**

Autoclaim measurements were shown not to be useful for assessing the quality of exposed concrete which has a RH in excess of 80%. A methodology for reducing the internal RH of the concrete to a value less than 80% should be investigated. A possible solution to the problem may be to cover or shield the Autoclaim test area for an extended period of time until the RH has decreased to below 80% before carrying out in-situ Autoclaim tests. An assessment of the effectiveness and practicality of such a procedure should be carried out.

7.2.2 **Classification of concrete for durability**

The classification of concrete for durability proposed by Basheer (1991) was shown to be limited because it was based on oven dried concrete. A more realistic classification system should be developed using concrete subjected to moisture conditions which are more representative of those experienced on-site.

7.2.3 **The effect of temperature and moisture on sorptivity indices**

The investigation into the effect of temperature and moisture on Autoclaim sorptivity indices carried out in this work was limited by the amount of time available. A comprehensive experimental study of the effect of moisture and temperature on the Autoclaim sorptivity indices should be undertaken. This may lead to a revision of the temperature correction process proposed in this thesis.
7.2.4. Monitoring the microstructure of the near surface concrete in real structures

Using the temperature and moisture correction procedures developed in this thesis an investigation into changes in the near surface microstructure of a reinforced concrete structure could be undertaken. This could be expected to identify the time taken for the concrete in a real structure to achieve maturity and the microstructural damage caused by the corrosion process at the end of its life span.
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