ANISOTROPIC PERMEABILITY CHARACTERISTICS OF INDIANA LIMESTONE: EXPERIMENTAL AND COMPUTATIONAL STUDIES

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August 31\textsuperscript{th}, 2009

A thesis submitted to McGill University
in partial fulfillment of the requirements of the degree of
Master of Engineering
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Abstract

This thesis presents the development of an experimental methodology to determine the fluid transport characteristics of geomaterials whether they are homogenous or inhomogeneous and directionally dependent. The experimental concepts, set up and procedures for different configurations of permeability testing are discussed. In this research, a steady state technique is deployed to evaluate the permeability characteristics of Indiana Limestone, which can exhibit hydraulic properties that are non-isotropic and an associated solution to analyze experimental data in steady state permeability test conditions is established. The developments presume that one of the principal directions of hydraulic conductivity coincides with the axis of the cylindrical specimen that is being tested.

The experimental procedure involved testing cylindrical samples of the Indiana Limestone of 100 mm diameter and about 60 mm height subjected to steady state flow configurations in the axial and radial directions. The results of certain preliminary tests show that the intrinsic permeability of Indiana Limestone is in the order of $10^{-15}$ m$^2$ and exhibits some extent of anisotropy: the ratios of the permeability in the vertical direction and bedding direction $K_z / K_r$ range from 1.8 to 2.6.
Résumé

Cette thèse présente le développement d’une méthode expérimentale de détermination des caractéristiques de transport fluide des géomateriaux (homogènes ou non homogènes et dépendants de la direction). Elle décrit les concepts, installations et procédures expérimentaux pour différentes configurations d'essai de perméabilité.


Le procédé expérimental consiste à examiner les échantillons cylindriques du calcaire de l'Indiana (diamètre de 100 mm et longueur d'environ 60 mm) qui sont soumis à des configurations d'écoulement en régime permanent dans les directions axiales et radiales. Les résultats de certains tests préliminaires montrent que la perméabilité intrinsèque du calcaire de l'Indiana est de l'ordre $10^{-15}$ m² et la mesure de l'anisotropie du rapport de la perméabilité dans la direction verticale et la direction de stratification $K_z/K_r$ varie de 1.8 à 2.6.
Acknowledgement

The author would like to express his great attitude to his thesis supervisor, Professor A.P.S. Selvadurai, *William Scott Professor and James McGill Professor*, Department of Civil Engineering and Applied Mechanics at McGill University, for the topic of research, support, guidance, reviewing with patience the author’s basic background of the field throughout the research program. It has been a great honour for the author to work with an internationally and nationally well-known researcher of his calibre.

The author also would like to acknowledge his deep attitude to Professor Van Thanh Van Nguyen, *Endowed Brace Professor Chair of Civil Engineering*, McGill University for his emotional and spiritual support from the beginning and throughout his study at McGill University.

The author would like to thank the technical support staff: Dr. William Cook, Mr. Marek Przykoski, Mr. Ronald Sheppard, Mr. Damon Kiperchuck in Civil Engineering laboratories, Mr. Georges Tewfik of Mechanical Engineering laboratories, Mr. Steve Godbout of Physics laboratory, Ms. Helen Campbell and Dr. Florance Paray of Materials Testing Services, especially Mr. John Bartczak at Civil Engineering laboratories, for help in designing, assembling and running experiments. The assistance from technical support staff of Mandel Scientific and Cole-Parmer Instruments is also acknowledged.

The author would like to offer many thanks to all his fellow colleagues and friends: Dr. Quifeng Yu, Dr. Wenjun Dong, Dr. Ali Shirazi, Dr. Hani Ghiabi and Mr. Jason Kaden, Mr. Paul Selvadurai, and Mr. Adrian Glowacki for help, guidance, suggestions and
cooperation and sharing the hard-working times at McGill University during the program. Their friendship made the stay in Montreal a wonderful and unforgettable experience.

The author is also grateful for the help of Mrs. Sally Selvadurai for her corrections of this thesis. The author also would like to acknowledge the help of fellow students Dr. Vanessa Lysakoune in French revision of the abstract and Mr. Kien Dang for proof-reading the draft.

The research work is supported by a Discovery Grant (NSERC) and the Max Planck Fellowship in the Engineering Science awarded to Professor A.P.S. Selvadurai.

The author wishes to take this opportunity to express his deepest appreciation to his parents and younger sister, for their endless love, support, patience, encouragement, and motivation not only in this study but also throughout his life. Without their loving support and continuous encouragement, it would not be possible for the author to complete this work. The author would like to dedicate this thesis to them.
Dedicated to:

My parents and sister, with affection
Table of Content

Abstract............................................................................................................................................. I

Resumé.................................................................................................................................................. II

Acknowledgement.......................................................................................................................... III

Table of Content.......................................................................................................................... VI

List of Figures ...................................................................................................................................... IX

List of Tables ........................................................................................................................................ XI

1. Introduction ....................................................................................................................................... 1
   1.1. Rationale for characterizing the hydraulic properties of geomaterials ....................... 1
   1.2. Literature Review .................................................................................................................. 3
   1.3. Objective and Scope of the thesis ...................................................................................... 10

2. Theoretical Background .............................................................................................................. 12
   2.1. Permeability and Hydraulic Conductivity ........................................................................ 12
   2.2. Darcy’s Law and General Assumptions ........................................................................... 13
       2.2.1 General Assumptions .................................................................................................. 13
       2.2.2 Darcy’s Law .............................................................................................................. 13
   2.3. Governing Equations of fluid transport in a transversely isotropic material ............... 15
   2.4. Solutions of governing equations for permeability tests .............................................. 16
   2.5. Effect of temperature on the estimation of permeability .............................................. 18

3. Indiana Limestone .................................................................................................................... 20
   3.1. General introduction and physical properties of Indiana Limestone .......................... 20
   3.2. Measurement of physical properties and porosity ......................................................... 21
   3.3. Penetration of epoxy into Indiana Limestone using Scanning Electron Microscope (SEM) .......................................................... 25

4. Experimental Facilities .............................................................................................................. 28
   4.1. Triaxial Cell for Axial Permeability Testing ................................................................. 28
4.1.1 Overall setup ................................................................................................. 28
4.1.2 Technical Specifications of Experimental Facilities ................................. 32
4.1.3 Geotextile ................................................................................................. 36
4.2 Radial Flow Test ............................................................................................. 38
4.2.1 Overall setup ............................................................................................. 38
4.2.2 Technical Specifications of radial permeability experiment ........................ 44
4.3 Other Experimental Facilities ........................................................................ 47
4.3.1 Distilled/De-aired water ............................................................................ 47
4.3.2 Technical Specifications of other facilities in the experiment ...................... 48
5. Experimental Procedures .................................................................................. 49
5.1 General Testing Conditions .......................................................................... 49
5.2 Sample Preparation ...................................................................................... 50
5.2.1 Test specimens ......................................................................................... 50
5.2.2 Epoxy sealing ........................................................................................... 51
5.2.3 Drilling central cavity ............................................................................... 53
5.2.4 Saturation process .................................................................................... 54
5.3 Experimental Procedures for Axial Permeability Tests ................................ 54
5.4 Experimental Procedures for Radial Permeability Test ................................ 58
6. Interpretation of Acquired Data and Results .................................................. 63
6.1 Results of Axial Permeability Tests ............................................................... 63
6.2 Results of Fully-Drilled Permeability Tests ................................................... 65
6.3 Steady State Flow Tests on Surface-Drilled and Partially-Drilled Cylinders... 67
  6.3.1 Computational modeling of steady flow in partially drilled samples with
     COMSOL ® Multiphysics ................................................................................. 68
  6.3.2 Results of surface cavity tests ................................................................. 72
  6.3.3 Results of partially-drilled cavity tests .................................................... 73
6.4 Discussion of Results .................................................................................... 75
6.5 Tests of transient decay from steady state .................................................... 76
7. Conclusions and Future work ......................................................................... 79
7.1. Conclusions ............................................................................................................... 79
7.2. Future Work ........................................................................................................... 80

References ........................................................................................................................ 81
List of Figures

Figure 1-1 Permeability cell using water and gas (after Bamforth, 1987) ....................... 5
Figure 1-2 Radial flow permeater (after Heystee and Roegiers, 1981) ......................... 6
Figure 1-3 Configuration of transient experiment after Brace et al. (1968) .................... 8
Figure 2-1 Uniaxial flow model .................................................................................... 16
Figure 2-2 Hollow specimen and radial flow test ....................................................... 18
Figure 3-1 Block of Indiana Limestone with 8 cored samples: cores were marked with
reference to their original location .................................................................................. 23
Figure 3-2 Coring Indiana Limestone sample from the block ..................................... 23
Figure 3-3 Scanning specimens from sealed surface .................................................. 25
Figure 3-4 Au/Pd coated scanning specimen ............................................................... 26
Figure 3-5 Images of epoxy penetration into Indiana Limestone ................................. 26
Figure 4-1 Overall setup of axial permeability in the Environmental Geomechanics
Laboratory, McGill University (after Glowacki, 2006) .............................................. 29
Figure 4-2 Cross-sectional view of the Triaxial Cell showing important components and
their location (after Glowacki, 2006) .......................................................................... 31
Figure 4-3 Diagram of the experimental setup (after Glowacki, 2006) ......................... 32
Figure 4-4 Geotextile used in experiment ................................................................... 37
Figure 4-5 Overall test setup of radial flow test at the Geomechanics Laboratories,
McGill University ......................................................................................................... 38
Figure 4-6 Schematic diagram of radial flow test and the detail of test sample ............ 39
Figure 4-7 Assembly of the loading frame and water chamber with top beam .......... 41
Figure 4-8 Plan and cross-sectional view of top cylinder ............................................ 41
Figure 4-9 Detail on test configuration for radial flow .................................................. 43
Figure 5-1 A sample of Indiana Limestone for uniaxial permeability experiment ...... 51
Figure 5-2 Set up and procedure of epoxy sealing ....................................................... 52
Figure 5-3 Sample before and after surface-drilling .................................................... 54
Figure 5-4 Saturation of sample in vacuumed water chamber with vibrating table ....... 55
Figure 5-5 A specimen of Indiana Limestone was in place on pedestal with hose clamps (after Glowacki, 2006) ............................. 57

Figure 5-6 Photograph of the ports prepared for pressurization of the chamber and the valve setup for the permeating fluid ................................................................. 57

Figure 5-7 Priming procedure of the radial permeability test ........................................ 60

Figure 6-1 Recorded pressure development in Indiana Limestone sample 2-12-1 in uniaxial permeability test .............................................................. 64

Figure 6-2 Recorded pressure development in Indiana Limestone sample 2-12-3 in a radial permeability test ................................................................. 66

Figure 6-3 COMSOL mesh for sample 2-12-1 with a 3 mm cavity (3216 elements) ...... 69

Figure 6-4 COMSOL mesh for sample 2-12-1 with a 36.5 mm cavity (4408 elements) . 70

Figure 6-5 COMSOL mesh for sample 2-12-2, 2-12-3, 2-12-4 with a 30 mm cavity (4276 elements) ........................................................................................................... 70

Figure 6-6 Variation of cavity pressure and flow rate to axial and transverse permeability ........................................................................................................... 70

Figure 6-7 Water pressure in the 3 mm cavity of sample 2-12-3 at 1 ml/min flow rate... 72

Figure 6-8 Water pressure in the 30 mm cavity of sample 2-12-2 at 1 ml/min flow rate. 74

Figure 6-9 Time history of pressure decay from a steady state test ............................. 77

Figure 6-10 Normalized pressure decay curve from a steady state test ...................... 78
List of Tables

Table 3-1 Physical and mechanical properties of Indiana Limestone (ILIA, 1998)........ 21
Table 3-2 Chemical analysis of Indiana Limestone (ILIA, 1998) ................................... 22
Table 3-3 Mechanical, physical and chemical properties of Indiana Limestone .......... 24
Table 4-1 Summary of technical specifications of Polyfelt TS 750 (Polyfelt, 2006): ...... 37
Table 6-1 Coefficients of axial permeability estimated from permeability tests......... 65
Table 6-2 Coefficients of transverse permeability estimated from radial permeability tests ........................................................................................................................................... 67
Table 6-3 Surface cavity permeability tests of Indiana Limestone samples............... 72
Table 6-4 Estimated transverse permeability from surface cavity tests ..................... 73
Table 6-5 Permeability tests on partially-drilled Indiana Limestone samples.............. 74
Table 6-6 Estimated transverse permeability from partially-drilled cavity tests and
COMSOL ® results .......................................................................................................... 75
Table 6-7 Summary of estimated axial and transverse permeabilities from all tests....... 75
1. Introduction

1.1. Rationale for characterizing the hydraulic properties of geomaterials

The movement of fluids through porous geological media is governed by the intrinsic permeability, of the porous medium. The term permeability is distinct from the term hydraulic conductivity of a porous medium. The former is an intrinsic physical property of a porous medium, whereas the latter depends on both the properties of the pore space and the permeating fluid, which is usually ground water. The measurement of permeability or hydraulic conductivity of geomaterials has been important not only in petroleum engineering for a long period of time, but also in civil engineering endeavours. In geotechnical applications the fluid transport characteristics of porous media are important to areas such as ground water hydrology, environmental geotechnique, geothermal energy extraction, engineered geotechnical facilities including earth dams, excavations and underground construction (Harr, 1962; Bear, 1972; Barenblatt et al., 1990). The design of earth dams, tunnels or excavations requires information on the hydraulic conductivities to estimate the quantities of flow, hydraulic gradient, instabilities, erosion etc.

Other civil engineering applications that require knowledge of permeability or hydraulic conductivity include flow through cementitious materials such as concrete and grout used both in new construction and infrastructure rehabilitation (Selvadurai and Carnaffan, 1997).
Recently, in the context of geoenvironmental engineering, the assessment of permeability characteristics of natural rock has become increasingly important in investigating the transport of chemical or hazardous materials in ground water and its affect on the environment. The Canadian context of storage of hazardous nuclear fuel waste, both natural geological materials such as rocks and cementitious material (i.e. concrete or grout) used as the barrier for nuclear waste disposal (Selvadurai and Carnaffan, 1997) require a thorough assessment of their permeability characteristics.

Despite its potential importance to many civil and geological engineering applications, the measurement and interpretation of permeability characteristics of geotechnical materials, notably rocks, is far from routine. One of the key factors that influences the interpretation of the fluid transport characteristics for geologic media is the choice of scale. This can range from geological crustal scales of 0.5 km to 5 km, to borehole scales ranging from 30 m to 300 m and laboratory scales that can range from 5 cm to 15 cm (Selvadurai et al., 2005). The bulk fluid transport characteristics of naturally occurring geologic media will be influenced by factors such as fractures, fissures, inclusions, dissolution channels and other anomalies. This will invariably depend on the type of geomaterial being investigated, but materials such as sandstone, limestone, shale or even granite can contain local scale stratifications and inhomogeneities that can influence the measurement and interpretation of the fluid transport characteristics at the laboratory scale (Harr, 1962; Tarbuck, 2002). Recognizing these difficulties, efforts are continually being made to develop methodologies for the accurate characterization of porous media through accurate measurement and interpretation. Various techniques have been used to measure the permeability of low permeable materials but currently there are no
standardized laboratory tests for measuring the characteristics of rocks or cement grout. The measurement of the fluid transport characteristics of a porous medium in a laboratory setting also has to address the possible influences of defects and other imperfection that become insignificant at larger scales. Only limited number of techniques have been developed in the laboratory to address the anisotropy or inhomogeneity of hydraulic characteristics of geomaterials.

1.2. Literature Review

The need for the accurate determination of permeability or hydraulic conductivity has been discussed earlier in this thesis. The basic theory governing movement of a fluid through a porous medium by virtue of a hydraulic potential was first proposed by Darcy (1856). Darcy’s Law forms the basis for evaluating many experimental approaches for determining either the permeability or hydraulic conductivity of porous media. Since this research relates to a laboratory experimental technique to evaluate of the fluid transport characteristics of rocks and other low permeability materials, a brief overview of the related experiment methodology will be discussed.

The most common approaches for the measurement of the fluid transport characteristics of porous media focus on the one-dimensional steady state and transient tests. Each method has both advantages and disadvantages and is suitable for certain types of geomaterials.

The steady state test is more suitable for the measurement of the fluid transport properties of relatively porous geological materials such as sandstones, sedimentary shale and carbonate rocks. The main advantage of the steady state test is its simplicity of analysis,
as it only requires application of Darcy’s law and the geometrical arrangements of the test and the boundary conditions associated with the test configuration. The method relies on the testing of a sample in a fully saturated condition and the possible attainment of a steady state fluid flow condition in the sample with no alteration in the fabric of the rock due to an applied pressure gradient. Steady state permeability tests involve either the application of a constant pressure head (constant head technique) across the sample and the measurement of the corresponding constant flow rate in steady state, or the application of a constant flow rate of permeating fluid through the sample and measurement of the corresponding steady pressure that builds up during the test (flow pump technique).

Early permeability tests were usually performed using the constant head technique. Examples of the steady state fluid flow laboratory test conducted on rocks by constant head technique are given by Daw (1971), Poon et al. (1986), Bamforth (1987), Banthia and Mindess (1989) and Ahmed et al. (1991). Bamforth (1987) performed the test using water and nitrogen as the permeating fluid with the pressure up to 1 MPa for a concrete specimen of 100 mm diameter and 50 mm thick (Figure 1-1). Heystee and Roegiers (1981) used a steady state, constant pressure gradient permeability test with radial flow condition. In their experiment, Indiana Limestone, granite and red sandstone samples of dimension about 64 mm in diameter by 100 mm in length have been tested in radial flow permeameter as in Figure 1-2.
Figure 1-1 Permeability cell using water and gas (after Bamforth, 1987)
A different approach to the steady state test is flow pump technique. The technique was first introduced by Olsen (1966), used by Olsen et al. (1985) for silty sand and silty clays and was applied to low permeability materials by Aiban and Znidarcic (1989) and Zhang et al. (1998). In this test, the sample is subjected to a constant flow rate and the variation in the pressure head is monitored using a pressure transducer. The advantage of this test method over the conventional constant head test is that the error associated with the direct measurement of the flow rate can be avoided, since such measurement results can be affected by other factors such as evaporation, capillary and surface tension effects. Another advantage of the flow pump technique is that the permeability test can be performed much faster with a small pressure gradient, and errors in the permeability measurement can be recognized (Zhang et al., 1998). When conducting permeability tests
on low permeability materials such as rock, concrete or cement grout, the constant flow rate is extremely small and difficult to measure, while the pressure can be measured accurately with higher resolution and conveniently with a pressure transducer and automatic data acquisition system (DAQ). The only disadvantage of the method is the high cost of the devices (pump, DAQ, pressure transducer, etc.). However, faster and more accurate performance, more convenient and cheaper data acquisition systems are being developed so that this method is the preferred testing method nowadays for low permeability materials (Aiban and Znidarcic, 1989).

For very low permeability materials, it is generally time consuming to reach steady state and the flow generated is too small for accurate measurement (*constant head technique*) or excessive pressure is developed (*flow pump technique*). The alternative *transient tests* are generally referred to as “*pulse tests*”, which are conducted on geological materials such as granite, limestone and other relatively low permeability materials. The sample configuration usually tends to be one-dimensional and the earliest application of the transient pulse test for the measurement of fluid transport characteristics of Westerly granite is due to Brace *et al.* (1968). In their work, a jacketed cylindrical specimen was subjected to a uniaxial fluid pressure pulse on one of its plane surfaces and the time-dependent decay of the pressure pulse was used to evaluate the fluid transport parameters as shown in Figure 1-3.
The one-dimensional transient pressure pulse technique has been extensively investigated by Zoback and Byerlee (1975), Krantz et al. (1979), Hsieh et al. (1981), Neuzil et al. (1981), Bourbie and Walls (1982) and Martin (1986) to determine the fluid transport characteristics of geologic materials under both un-stressed and stressed conditions. The transient pulse testing of hollow cores was first performed by Selvadurai and Carnaffan (1997) who used the technique to determine the fluid transport characteristics of a cement grout. The hollow cylinder concept is a laboratory extension of the packer testing of
boreholes or the “Slug Test Method” used for determining the in-situ fluid transport characteristics of both soils and rocks. The primary advantage of the transient pulse method is that the test itself can be performed relatively quickly, provided that the sample can be kept in a saturated condition with little or no residual fluid pressure from the saturation process. The constraint on the method primarily relates to the additional information required, including knowledge of the porosity and the medium, the compressibility of both porous skeleton and permeating fluid, the fluid viscosity and a complete mathematical analysis of a pressure diffusion equation, in order to interpret the experimental data (Selvadurai and Carnaffan, 1997; Selvadurai et al., 2005).

Most of the permeability tests at the laboratory scale make assumptions that the isotropic and homogenous characteristics of the porous media. But natural sedimentary geological rocks, such as sandstone, limestone, shale or even granite, may contain local scale stratifications and inhomogeneities that influence the measurement and interpretation of the fluid transport characteristics at the laboratory scale (Harr, 1962; Tarbuck, 2002). Therefore measurement of the fluid transport characteristics of a porous medium in a laboratory setting also has to address the possible influences of directionally dependent and other imperfections on the permeability. Wright et al. (2002) performed permeability tests on 6 Limestone samples in both vertical and horizontal directions in a modified-Wykeham Farrance triaxial and Hassler-type permeater (API, 1998); they compared the horizontal hydraulic conductivities in the test samples to the hydraulic conductivities in the vertical direction of the other samples at different stress states. The ratios varied from 0.5 to 24, from a shallow depth to a greater depth (155 m) with stress increasing from 200 kPa to about 2 MPa.
1.3. Objective and Scope of the thesis

The objective of the research in this study is to develop an experimental methodology to investigate the fluid transport characteristics of geomaterials at the laboratory scale: experimental concepts, set up and procedures for different configurations of permeability testing using the steady state technique. The permeability of a geomaterial depends on various factors (Heiland, 2003): stress states and conditions (Brace et al., 1968; Heystee and Roegiers, 1981; Suri et al., 1997; Wright et al., 2002; Selvadurai and Glowacki, 2008), degree of saturation (Bamforth, 1987; Banthia and Mindess, 1989; Hearn and Mills, 1991), permeating fluid which can be either gas that can cause the Klinkenberg effect, or liquid (Bamforth, 1987; Ahmed et al., 1991) or even the temperature of the permeating fluid as in ASTM D5084-97, 2000 or ASTM D5084-03, 2006.

In this research, experiments were carried out to examine cylindrical samples recovered from a large Indiana Limestone block. The experiments were performed on samples of Indiana Limestone measuring 100 mm in diameter and approximately 60 mm in height, free of radial stress with a small axial stress of less than 300 kPa. Using water as the permeating fluid; the permeabilities were corrected to the standard laboratory temperature of 20°C. The assumption of transverse isotropy is a plausible approximation for stratified geological rock (Selvadurai, 2004). Axial permeabilities in the direction normal to the bedding direction of the rock were measured from tests conducted on saturated jacketed samples under constant flow conditions and the end pressure of samples at steady state conditions was recorded. A central cavity then drilled in each of the testing samples of approximately 10 mm in diameter. Radial flow was initiated by subjecting the cavity to a constant flow rate, and the cavity pressure at the steady state condition of the saturated
sample was recorded. The transverse permeabilities were estimated independently from radial flow experiments on samples with a drilled central core. The radial flow tests were also conducted on partially-drilled samples at different stages to compare the results.

This thesis is organized into seven chapters as follows:

Chapter 1 presents the introduction of the research: the importance and necessity of permeability measurement of geomaterials, review of previous research and the objectives and the scope of the thesis.

Chapter 2 provides the background to the research: definition and properties of the variables and constants encountered in the testing and computation of permeability, general assumptions and governing equations of fluid in transport in porous media, solutions of the equations related to the experimental data from the permeability tests.

Chapter 3 presents an introduction to Indiana Limestone, the material used as the test samples and its physical properties that relate to the hydraulic characteristics of the geological rock.

Chapter 4 describes the experimental setup and specification of the testing facilities for uniaxial and radial flow test of Indiana Limestone samples.

Chapter 5 presents the experimental procedure: sample preparation and testing procedures for axial and transverse permeability tests.

The interpretation of the experimental data through both analytical and finite element solutions using the COMSOL® MULTIPHYSICS software, will be presented in chapter 6. Discussion of the results and recommendations for future work are presented in chapter 7.
2. Theoretical Background

2.1. Permeability and Hydraulic Conductivity

*Absolute permeability* \((K)\), or *intrinsic permeability*, referred to as permeability in this thesis is the rate at which fluid passes through the pore space of a material, which depends only on the material properties. The unit of permeability coefficient is length squared \((m^2)\).

The term *hydraulic conductivity* \((k)\), is a parameter that reflects the ability of the porous medium, such as rock or soil, to transmit specific fluid (normally water). The hydraulic conductivity depends both on the permeating fluid and its mechanical properties and has units of velocity \((m/s)\).

The hydraulic conductivity \((k)\) of a porous geomaterial is related to its permeability \((K)\) through the relation:

\[
K = \frac{k \gamma}{\mu}
\]  

(2.1)

where \(\mu\) is the dynamic viscosity of the permeating fluid, and \(\gamma\) is the unit weight of water.

The dimensionless *hydraulic gradient* \((i)\), is defined as the ratio of the difference in the hydraulic potential causing the flow between two points and the length over which the difference occurs (Terzaghi and Peck, 1967):

\[
i = \frac{\Delta h}{L} = \frac{\Delta p}{\gamma L}
\]  

(2.2)
where $h$ (m) is the *hydraulic head*, the height from the datum to the piezometer height and $p$ (N/m$^2$) is fluid pressure, $L$ is the distance between two points.

### 2.2. Darcy’s Law and General Assumptions

#### 2.2.1 General Assumptions

This research, as with other permeability investigations, considers the problem of fluid flow using the following assumptions:

- Porous medium is *saturated*, the pore space in the medium is fully occupied by permeating fluid.

- Fluid flow through the pore structure is assumed to be *compressible* and governed by *Darcy’s Law* (discussed in the next section).

- The material composing the porous medium is *non-deformable* in comparison with the pore fluid and the skeleton of the porous solid.

#### 2.2.2 Darcy’s Law

The most fundamental and basic law governing fluid flow through a porous medium proposed by the French researcher H.P.G. Darcy (1856); Darcy’s Law, can be expressed in the form:

$$\mathbf{v} = -k \nabla \phi$$  \hspace{1cm} (2.3)

where $\mathbf{v}$ is the *spatially averaged fluid velocity vector*, $\nabla \phi$ is the gradient of the hydraulic potential and $k$ is the hydraulic conductivity tensor. In general $k$ is a function of the position and orientation, and from thermodynamic considerations, it can be shown
that $k$ is symmetric (Yeung, 1990). When the axes of the reference coordinate system align with the axes of the principal hydraulic conductivities $k_i (i = 1, 2, 3)$ we have

$$
k = \begin{bmatrix}
k_1 & 0 & 0 \\
n0 & k_2 & 0 \\
n0 & 0 & k_3
\end{bmatrix}
$$

(2.4)

As mentioned previously, here we consider that the saturated porous medium to be \textit{hydraulically transversely isotropic}. The assumption of transverse isotropy for Indiana Limestone is an approximation but is realistic for stratified geological materials (Selvadurai, 2003, 2004). It is convenient to develop a governing equation in cylindrical polar coordinates ($r$, $\theta$ and $z$), where $v$ is the velocity tensor that consists of spatially averaged fluid velocity components $v_r$, $v_\theta$ and $v_z$. The generalized Darcy’s Law for fluid flow through a porous medium (2.3) for transverse isotropy material, where $k$, the hydraulic conductivity matrix:

$$
k = \begin{bmatrix}
k_r & 0 & 0 \\
n0 & k_r & 0 \\
n0 & 0 & k_z
\end{bmatrix}
$$

(2.5)

can be written of the form:

$$
v_r = -k_r \frac{\partial \phi}{\partial r}; \quad v_\theta = 0; \quad v_z = -k_z \frac{\partial \phi}{\partial z}
$$

(2.6)

It is noted that Darcy’s Law (2.3) can only be applied when a representative volume element is much larger than the pore and grain size, and the fluid is in a \textit{laminar flow condition}. The fluid dynamics shows that Darcy’s Law is valid when the Reynolds number ($Re$) is small ($Re=1\div10$) (Bear, 1972).
2.3. Governing Equations of fluid transport in a transversely isotropic material

Considering the equation of mass conservation and Darcy’s Law (Selvadurai, 2000), the partial differential equation governing the steady state condition of a hydraulic potential can be written in the form:

\[ \nabla^2 \varphi = 0 \]  
(2.7)

where the operator \( \nabla \) taken the form:

\[ \nabla^2 = k_r \left( \frac{\partial^2 \varphi}{\partial r^2} + \frac{1}{r} \frac{\partial \varphi}{\partial r} \right) + k_z \frac{\partial^2 \varphi}{\partial z^2} \]  
(2.8)

When the porous medium is hydraulically isotropic, \( \nabla \) reduces to the classical Laplace’s operator (Selvadurai, 2000).

The partial differential equation (2.8) can be solved by considering the relevant Dirichlet boundary condition \( S_D \) of prescribed potential and Neumann boundary condition \( S_N \) (impervious boundary) applicable to the domain i.e.:

\[ \varphi(r, z) = \varphi_0 \quad (r, z) \in S_D \]  
(2.9)

\[ \n \nabla \varphi = 0 \quad (r, z) \in S_N \]  
(2.10)

where \( n \) is the unit normal to boundary \( S_N \).

In general, the permeability of geomaterials can be obtained from permeability tests by combining the solution of the governing equation (2.7) or (2.8) with the corresponding
boundary conditions and the equation of the total flux $Q$ measured on an entire external surface $S$:

$$Q = \iiint_S v \, dS$$

(2.11)

The solution of the governing partial differential equation can be approached in a variety of ways, which include analytical methods and computational methods based on finite element and boundary element schemes.

2.4. Solutions of governing equations for permeability tests

In the case of the one-dimensional test configuration as shown in Figure 2-1, when the axis of sample being tested coincides with one of the principal axis of permeability or hydraulic conductivity, the solution of the steady state test can be expressed in the form (Terzaghi and Peck, 1967; Freeze and Cherry, 1979; Selvadurai, 2000):

$$K_z = \frac{Q \mu H}{(\Delta p) A}$$

(2.12)

where $Q$ (m$^3$/s) is the flow rate, $\mu$ is dynamic viscosity of water, $H$ (m) is the length (i.e. height) of the sample, $A$ (m$^2$) is the cross-sectional area through which flow passes, and $\Delta p$ (N/m$^2$) is the difference of hydraulic pressures of the two ends of the sample.

![Figure 2-1 Uniaxial flow model](image-url)
In some testing configurations the system resistance due to friction, head losses at inlets and outlets cannot be neglected; the system resistance must be determined and taken into consideration in the calculation of permeability.

Head losses may occur in the one dimensional permeability test due to compression of the geotextile used, Glowacki (2006) developed an equation to account for the system resistance ($k_{sys}/L_{sys}$):

$$k = \frac{L}{\left(\frac{L_{eq}}{k_{eq}} - \frac{L_{sys}}{k_{sys}}\right)}$$

(2.13)

where $k_{sys}/L_{sys}$ must be determined through experiments and the ratio of equivalent hydraulic conductivity and equivalent length can be found experimentally (see Glowacki, 2006; Selvadurai and Glowacki, 2008):

$$\frac{k_{eq}}{L_{eq}} = \frac{Q\gamma}{A\Delta p}$$

(2.14)

For the determination of permeability in the transverse direction, when the permeability test is in a radial flow condition, by pumping fluid though the central cavity of a cylindrical specimen as shown in Figure 2-2, the resistance of the system is negligible, the permeability is determined from the analytical solution (Carslaw and Jaeger, 1959; Selvadurai, 2000):

$$K_r = \frac{Q\mu\ln\left(\frac{b}{a}\right)}{2\pi H\rho_0}$$

(2.15)
where $Q$ (m$^3$/s) is the steady flow rate, $\mu$ is the dynamic viscosity of water (Ns/m$^2$); $H$ (m) is the height of the sample; $2a$ (m) and $2b$ (m) are the interior and exterior diameters of the sample; $p_0$ (N/m$^2$) is the internal fluid pressure and $K_r$ (m$^2$) is the transverse permeability.

For the permeability test configuration where the pressurized central cavity of the cylindrical specimen is only partially drilled, the coupled flow condition of axial and radial flow occurs; the solution of equation (2.8) requires the numerical technique, and is analyzed using the finite element scheme discussed later in the thesis.

![Hollow specimen and radial flow test](image)

**Figure 2-2 Hollow specimen and radial flow test**

### 2.5. Effect of temperature on the estimation of permeability

Although all the experiments were performed in laboratory, the temperature of the water used as permeating fluid and the temperature of the ambient environment in which the test was carried out varied for different experiments. For easy comparison, all the permeability results at different temperatures have been converted to the permeability at reference laboratory room temperature of 20°C. At the standard room temperature of 20°C, the dynamic viscosity of water $\mu_{20}=1.002\times10^{-3}$ (Pa.sec/m$^2$), and the viscosity at
other temperatures $T$ ($^\circ$C) can be estimated through the formula (ASTM D5084-97, 2000):

$$
\mu_{20} = (1.495 - 0.02452T) \times \mu
$$

(2.16)

Therefore the conversion to permeability at 20$^\circ$C is as follows:

$$
K_{20} = (1.495 - 0.02452T) \times K_T
$$

(2.17)

where $K_{20}$ is the coefficient of permeability adjusted to 20$^\circ$C, $T$ is the water temperature of the permeability test, $K_T$ is the permeability obtained from the test.
3. **Indiana Limestone**

In this research, Indiana Limestone, or Bedford Limestone, a popular natural light-colored, fine-grained stone used extensively in construction in North America was selected as the testing material. The following paragraph will briefly introduce the general properties of Indiana Limestone base on the information from Indiana Limestone Institute of America (ILIA, 1998), and the results of some laboratory tests to measure the physical parameters that may relate to the hydraulic characteristics of the rock. Other investigations that deal with the measurement of the permeability of Indiana Limestone are discussed by Heystee and Roegiers (1981), Wright et al. (2002) and Selvadurai and Glowacki (2008).

3.1. **General introduction and physical properties of Indiana Limestone**

Indiana Limestone, the common term of Salem Limestone, is a natural product that dated back about 330 million years to the Paleozoic period (ILIA, 1998). Since the 19th century, Indiana Limestone has been increasingly used as a material for construction in the United States and other places in North America. Indiana Limestone has been found in massive deposits located almost entirely in the Owen, Monroe, and Lawrence counties of Indiana. Like other limestones, Indiana Limestone is a monomineralic rock, primarily composed of calcium carbonate (CaCO₃) or **calcite** that resulted from deposition of shell and shell fragmented from shallow sea covered Midwest of America. Its beautiful and neutral colour, versatility, durability, uniform texture has allowed it to gain world-wide acceptance as a premier dimension stone (Indiana Limestone Institute of America,
The physical and mechanical properties make it very adaptive to changes in the ambient temperature; it is easy to machine to any desirable shape so that it can be used in construction of piers, tiles and facades. The hydraulic characteristics of Indiana Limestone are relatively homogeneous due to the high calcite content, uniform and consistent deposition; it is virtually non-crystalline and contains no cleavage plane. With all the listed advantages and the availability of material in the market, Indiana Limestone was chosen as the specimen for permeability testing. Tables 3-1 and 3-2 from ILIA Handbook, 1998 listed the properties for two typical types of Indiana Limestone: Buff and Gray.

Table 3-1 Physical and mechanical properties of Indiana Limestone (ILIA, 1998).

<table>
<thead>
<tr>
<th>Properties</th>
<th>Value</th>
<th>Standard</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ultimate compressive strength of dry specimens</td>
<td>27.5 MPa minimum</td>
<td>ASTM C170</td>
</tr>
<tr>
<td>Modulus of rupture of dry specimens</td>
<td>4.8 MPa minimum</td>
<td>ASTM C99</td>
</tr>
<tr>
<td>Absorption</td>
<td>7.5 percent minimum</td>
<td>ASTM C97</td>
</tr>
<tr>
<td>Bulk Specific Gravity</td>
<td>2.1 minimum to 2.75 maximum</td>
<td></td>
</tr>
<tr>
<td>Modulus of Elasticity</td>
<td>22.7 GPa min. to 37.2 GPa max.</td>
<td></td>
</tr>
<tr>
<td>Ultimate Shear Strength</td>
<td>6.2 MPa min. to 12.4 MPa max.</td>
<td></td>
</tr>
<tr>
<td>Ultimate Tensile Strength</td>
<td>2 MPa min. to 4.9 MPa max.</td>
<td></td>
</tr>
<tr>
<td>Weight</td>
<td>2306 kg/m³</td>
<td></td>
</tr>
</tbody>
</table>

3.2. Measurement of physical properties and porosity

Although the data on Indiana Limestone was readily available from ILIA, the laboratory tests on the Indiana Limestone used in the current research investigation were performed.
to obtain the properties to complement this research and to confirm the properties of the material from previous studies.

Table 3-2 Chemical analysis of Indiana Limestone (ILIA, 1998).

<table>
<thead>
<tr>
<th>Content</th>
<th>Indiana Limestone (Buff)</th>
<th>Indiana Limestone (Gray)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CaCO₃</td>
<td>97.39</td>
<td>97.07</td>
</tr>
<tr>
<td>MgCO₃</td>
<td>1.20</td>
<td>1.20</td>
</tr>
<tr>
<td>Silica (SiO₂)</td>
<td>0.69</td>
<td>0.80</td>
</tr>
<tr>
<td>Alumina (Al₂O₃)</td>
<td>0.44</td>
<td>0.68</td>
</tr>
<tr>
<td>Iron Oxide (FeO or Fe₂O₃)</td>
<td>0.18</td>
<td>0.12</td>
</tr>
<tr>
<td>Water and loss</td>
<td>0.1</td>
<td>0.13</td>
</tr>
</tbody>
</table>

All specimens used for this thesis were Indiana Limestone cylinders cored out from a single large block. The stone was purchased from a construction supplier in Montreal, Primcar Inc. Les Pierres. Cylindrical samples were cored using a diamond bit corer with an internal diameter of 108 mm and tap water was used as the cooling fluid (see Figures 3-1 and 3-2) along the direction normal to the bedding direction of the stone. The bedding direction was easy to recognize visually. After coring, each core had a diameter of 107 mm and a height of 305 mm.

The next step was to grind the two end surfaces to prepare a perfectly cylindrical sample; this was accomplished using a grinding machine (Marui & Co. Ltd, Japan), located in the McGill University Materials Laboratory, used to prepare concrete samples. The Indiana Limestone cores then were machined down to 100 mm in diameter on a lathe. This
resulted in a smooth-surfaced cylindrical sample with the following dimensions: diameter of 100 mm and height from 200 mm to 280 mm.

Figure 3-1 Block of Indiana Limestone with 8 cored samples: cores were marked with reference to their original location.

Figure 3-2 Coring Indiana Limestone sample from the block
The cores were tested in a rock testing machine to determine the physical and mechanical properties, as well as chemical analysis according to ASTM procedures. Table 3-3 shows the results of the tests at McGill University Structural Engineering Laboratories by the thesis author and Mr. A. Glowacki, which is also presented more detail in the M.Eng. thesis by Glowacki (2006).

The porosity of Indiana Limestone sample was determined through the Standard Test for Laboratory measurement of moisture content in soil and rock, according to procedure D2216-05 by ASTM 2006 (Vol.4.8), measurement of water content of rock by mass method. A fully saturated test specimen was dried in the oven at 120°C to a constant mass. The loss of mass due to drying is considered to be from the water. The volume of water lost is equal to the volume of voids in the specimen.

Table 3-3 Mechanical, physical and chemical properties of Indiana Limestone

<table>
<thead>
<tr>
<th>Properties</th>
<th>Unit</th>
<th>Average Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Modulus of Elasticity</td>
<td>GPa</td>
<td>24</td>
</tr>
<tr>
<td>Ultimate Compressive Strength</td>
<td>MPa</td>
<td>37.5</td>
</tr>
<tr>
<td>Ultimate Tensile Strength</td>
<td>MPa</td>
<td>3.6</td>
</tr>
<tr>
<td>Weight</td>
<td>kg/m³</td>
<td>2243</td>
</tr>
<tr>
<td>Poisson’s Ratio</td>
<td></td>
<td>0.14</td>
</tr>
<tr>
<td>Porosity</td>
<td>%</td>
<td>16.60</td>
</tr>
<tr>
<td>pH</td>
<td>pH</td>
<td>7.4</td>
</tr>
<tr>
<td>Harden Test (CaCO₃ concentration)</td>
<td>ppm</td>
<td>28</td>
</tr>
</tbody>
</table>
3.3. Penetration of epoxy into Indiana Limestone using Scanning Electron Microscope (SEM)

When preparing specimens for radial flow testing of transverse permeability, the end surface of the cylindrical sample was sealed with Bondo Marine Resin to create an impervious boundary; more details will be given later. The Bondo Resin Filler-Fiberglass 402C from Bondo Corp. was found to provide a strong and smooth surface sealant for the test specimen, as used by Carnaffan (1994) for cement grout specimens. In order to determine whether the epoxy has any effect on the accuracy of the permeability test results, tests were undertaken to see how deep the sealing epoxy penetrated into the intact rock sample. These tests were carried using Scanning Electron Telescopes (SEM) at McGill University’s Materials Testing Services. The sealed part of specimen was cut into different samples as shown in Figure 3-3. Before scanning in the SEM, each sample was coated with a gold and palladium alloy (Au/Pd coating), as shown in Figure 3-4.

Figure 3-3 Scanning specimens from sealed surface
Figure 3-4 Au/Pd coated scanning specimen

Figure 3-5 shows some examples of the images from SEM and traces the penetration of the epoxy resin into the rock.

Figure 3-5 Images of epoxy penetration into Indiana Limestone
In all the tested specimens, the resin was traced to a deepest depth of less than 1 mm into the Indiana Limestone surface. For the test specimens which will be presented later in the thesis, the cavity depths were extended to 3 mm or deeper to ensure that the test results were not affected by the penetration of epoxy into the stone.
4. Experimental Facilities

In this research, the axial permeability and transverse permeability were measured independently in two separate testing systems; the former in a GDS Rock-Testing Triaxial Cell following the procedure for a one-dimensional test specimen with an isotropic confining pressure of 1 MPa and the latter performed in a stainless steel chamber with a radial flow, as described latter in this chapter, with an axial stress of about 300 kPa. From the results of Selvadurai and Glowacki (2008), the variation of permeability for the stress range from 0 to 1 MPa is small and it is assumed that the comparison of axial and transverse permeability is a reasonable judgment. This chapter provides the descriptions of the function and technical specifications of the facilities making up each system.

4.1. Triaxial Cell for Axial Permeability Testing

4.1.1 Overall setup

In the axial permeability test, the procedure used in this investigation is similar to that mentioned in Selvadurai and Glowacki (2008). The information on the experimental setup and components in this section is primarily from Glowacki (2006) with some changes to adapt to the test conditions of this research.

The following components are shown in Figure 4.1:
Figure 4-1 Overall setup of axial permeability in the Environmental Geomechanics Laboratory, McGill University (after Glowacki, 2006).

The modular and adjustable data acquisition system (Techmatron) is designed to acquire, amplify, filter and store the voltages received from two pressure transducers, PT 10000 psi that measured the confining pressure and PT 300 psi that measured the fluid pressure initiating flow through the sample. Readings of pressures from the GDS controller and the Shimadzu pump LC-8A were also used to support and confirm the pressures obtained from the PT 10 000 and PT 300 pressure transducers. The Techmatron DAQ can only record the data at a maximum of 1 Hz. Since experiments are conducted in a steady state condition, the scanning rate of channels, as well as the computing power, can be acquired at a basic level.

The GDS controller has the ability to build up and maintain the confining pressure in the apparatus up to 64 MPa. The controller is connected to the Triaxial Cell via specially
rated stainless steel tubing and Swagelok valves. An elaborate setup of valves and tubing was necessary for the following reasons (see Figure 4-3):

- The GDS controller is a one way pump, with the inlet for water the same as the outlet. Therefore, when the GDS controller is empty it needs to be refilled while the pressure has to be maintained inside the Triaxial Cell.
- Since the actuator in the GDS controller is slow and its reservoir has a volume of only 207 ml, it would take many hours to fill up the Triaxial Cell. It was therefore necessary to use an auxiliary reservoir as the flooding apparatus.

A digital scale was used to check if the outflow was the same at the flow rate of the test (1 ml/min) and to confirm that there were no leaks. By taking several measurements during testing, the proper functioning of the Shimadzu pump was confirmed, as well as indicating whether the pore pressure had stabilized.

The reservoir situated on top of the yellow frame (see Figure 4-1) is made of Plexiglas with a removable cover; it was used to fill the Triaxial Cell (see Figure 4-3) with water. The water used throughout the experiments was distilled/de-aired water described more detail later in the thesis.

The stainless steel Triaxial Cell was adapted and modified to easily accommodate the filling, emptying and pressurizing of the chamber without stopping an experiment in progress, as shown in Figure 4-2
Figure 4-2 Cross-sectional view of the Triaxial Cell showing important components and their location (after Glowacki, 2006).

The balanced ram assembly prevents vertical movement of the ram while the chamber is being pressurized (see Figure 4-3). This is achieved by balancing the pressure between both chambers, and it could also be used with a hydraulic piston to apply deviatoric stress in the vertical direction.
The chromatograph Shimadzu pump LC-8A was used to provide the pore water at a specified flow rate for the permeability test, and to re-saturate the Limestone sample after it was mounted on the Triaxial Cell pedestal.

![Diagram of the experimental setup](image)

**Figure 4-3** Diagram of the experimental setup (after Glowacki, 2006).

### 4.1.2 Technical Specifications of Experimental Facilities

The specifications of the facilities used in the experimental program are as follow:

Data acquisition system: from Techmatron, is composed of the following parts:

Personal Measurement Device (PMD)-1608FS

- 16 bit analog input resolution
- 8 analog to digital independent channels at 50 KHz scanning rate
- Accuracy to 2.98 mV on a 5 V range; or 2 kPa on 2.1 MPa scale
- USB compatible
Two Isolated Process Control Signal Conditioning Modules or SCM-5B38
by DATAFORTH CORP.

- Input range: ± 10 to ± 100 mV
- Accuracy of ± 0.08% over the full span
- Output range ± 5V
- 300 Ω to 10 KΩ Full-bridge input
- Nonlinearity of ± 0.02% over the full span

8-Position Analog I/O Backpanel, Non-Multiplexed

- Working temperature range: -40 to 85°C
- Can accept up to 8 SCM-5B signal conditioners

Shielded cable C37FFS-one open end

- 37-pin female to connect to backpanel
- Open end with 37 cables to connect to PMD

Swagelok supplies included brass valves, ferrules, port connectors, tube adaptors.

- Maximum working pressure for brass valves: 21 MPa;
- Working temperature range: 10°C to 65°C;
- Working pressure for brass connectors and fittings: 28 MPa
- Working pressure for stainless steel connectors and fittings: 70 MPa

Swagelok High-Pressure 83 Series Trunnion Ball Valve

- 2 × Three-Way Valves and 1 × Two-Way Valve

- Maximum working pressure: 70 MPa
316 Stainless Steel

Working temperature range: -17 °C to 232 °C

Pump tubing: SCIVEX Upchurch Scientific 1/16" OD PEEK™ Polymer Tubing

- Internal diameter: 0.5 mm
- Maximum internal pressure: 49 MPa
- Maximum temperature: 100 °C

Stainless steel tubing: from PINACLE Acier Inoxydable based in Montreal, Canada

- Seamless tube
- Outside diameter: 3.1 mm
- Internal diameter: 0.9 mm
- Maximum internal pressure: 72 MPa

GDS Advanced Digital Controller for confining pressure regulation (hereafter referred to as GDS controller) from GDS Instruments Ltd., UK. This is a digital controller with a microprocessor-controlled hydraulic actuator for precise regulation and measurement of liquid pressure and liquid volume change. The manufacturer’s specifications are as follow:

- Pressure readings are based on a moving average
- Resolution is ± one in 10000 of the full scale
- Its resolution being that one step of the stepper motor displaces 0.02 mm³ of fluid.
- The volume capacity of the piston is 207 ml.

GDS High Pressure Balanced Triaxial Cell, all stainless steel (Figure 4-2)
- Maximum working pressure that can be applied to the cell: 64 MPa
- Axial maximum working load: 250 kN
- Specimen sizes allowed: 100 mm diameter x 200 mm long
  
  70 mm diameter x 140 mm long
  
  50 mm diameter x 100 mm long

- The fluids used to apply the cell pressure should be either: silicone oil, hydraulic oil or water.

Pressure transducer: SENSOTECH (referred to as PT10000)

- Excitation: 10 V DC
- Range: 0 to 10000 psi (0 to about 70 MPa)
- Serial Number: 636357
- Output: 2.8 mV/V

Pressure transducer: DCT INSTUMENTS (referred to as PT300)

- Series: PZA 300AC
- Excitation: 10 V DC
- Range: 0 to 300 psi (0 to 2.1 MPa)
- Serial Number: 006512
- Output: 7.65 mV/V

Shimadzu LC-8A Preparative LC Pump

- Double plunger, reciprocating pump delivering 250 μl by stoke per head.
- Operating temperature is between 10 °C and 35 °C.
- Accuracy in flow rate setting: ± 2%
- Flow rate stability: ± 0.5%
- The lowest flow rate that can be set is 0.1 ml/min
- Flow rate ranges available:
  - 0.1 to 50 ml/min (1 to 30 MPa)
  - 0.1 to 100 ml/min (1 to 18 MPa)
  - 0.1 to 150 ml/min (1 to 8 MPa)
- Pressure range: 1 MPa to 30 MPa

Membrane: Nitrile rubber sleeve

Supplier: Design Rubber Ltd., Montreal

- 99.6 mm inside diameter x 3 mm thick x 250 mm in height
- Rubber type: 70A Nitrile

Grey geotextile TS 750 (see section 4.1.3: Geotextile)

There are other facilities and parts related to sample preparation and the testing procedure; these will be presented later in the thesis: Scale, De-airing apparatus, vacuum pump, vibrating table for sample saturation.

4.1.3 Geotextile

The geotextile used during testing was as Polyfelt TS 750 a product from Polyfelt Geosynthetics, Austria and distributed in Quebec by SOLMAX Texel (see Figure 4-4). It is described as polyfelt mechanically bonded, continuous-filament, nonwoven textile, made from 100% UV-stabilized polypropylene (Polyfelt, 2006). The main application of
this geotextile in the experiment is to uniformly distribute and collect water on the upper and lower surfaces of the sample.

Table 4-1 Summary of technical specifications of Polyfelt TS 750 (Polyfelt, 2006):

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight</td>
<td>g/m²</td>
<td>350</td>
</tr>
<tr>
<td>Resistance to perforation</td>
<td>N</td>
<td>3000</td>
</tr>
<tr>
<td>Thickness ((t_g))</td>
<td></td>
<td></td>
</tr>
<tr>
<td>under 2 kPa</td>
<td>mm</td>
<td>3.00</td>
</tr>
<tr>
<td>under 0.1 MPa</td>
<td>mm</td>
<td>1.55</td>
</tr>
<tr>
<td>under 0.2 MPa</td>
<td>mm</td>
<td>1.25</td>
</tr>
<tr>
<td>Permeability radial to layer ((K_h))</td>
<td>cm/sec</td>
<td></td>
</tr>
<tr>
<td>under 2 kPa</td>
<td>cm/sec</td>
<td>0.80</td>
</tr>
<tr>
<td>under 0.1 MPa</td>
<td>cm/sec</td>
<td>0.10</td>
</tr>
<tr>
<td>under 0.2 MPa</td>
<td>cm/sec</td>
<td>0.08</td>
</tr>
<tr>
<td>Permeability normal to layer ((K_v))</td>
<td>cm/sec</td>
<td></td>
</tr>
<tr>
<td>under 2 kPa</td>
<td>cm/sec</td>
<td>0.40</td>
</tr>
<tr>
<td>under 0.1 MPa</td>
<td>cm/sec</td>
<td>0.10</td>
</tr>
<tr>
<td>under 0.2 MPa</td>
<td>cm/sec</td>
<td>0.06</td>
</tr>
</tbody>
</table>

Figure 4-4 Geotextile used in experiment
4.2. Radial Flow Test

4.2.1 Overall setup

The general facility used in this study was developed by Carnaffan (1994) and summarized by Selvadurai and Carnaffan (1997).

Figure 4-5 Overall test setup of radial flow test at the Geomechanics Laboratories, McGill University

Figure 4-6 shows a schematic diagram of the radial permeability test. The computerized data acquisition system is a package from National Instruments Corp. (NI) and consists of an AMUX-64T analog multiplexer with temperature sensor, an AT-MIO-16F-5 data acquisition board, which plugged directly into a slot in a Dell Pentium III computer installed with the NI Labview 6i software package. This software allows the data acquisition program to be custom designed. The program displayed the time history of both the axial load and the fluid pressure in the central cavity of the specimen.
The recording interval can be prescribed by user, and can be set at scanning rate of 100 Hz. The pressure transducer and load cell both needed excitation voltages of 24 and 10
VDC, respectively, which was provided by connecting the power leads to DC power supply boxes.

The stainless steel water chamber, the base of the water chamber and the loading frame (Figure 4-7) were previously used by Carnaffan (1994) to conduct “transient pulse test”. The water chamber has an inside diameter of 292 mm and a depth of 305 mm, the wall thickness is 6.4 mm with the volumetric capacity of the chamber 20.4 litres. Stainless steel flanges were welded on the top and bottom of the stainless steel tube which forms the wall of the water chamber.

The base of the water chamber was a stainless steel plate 400 mm by 460 mm by 12.7 mm which was machined smooth over the entire top surface. There are two O-ring grooves in the top surface of the plate; one provides the seal against the bottom flange of the water chamber and a 50.7 mm outside diameter groove around the centre provides a seal between the plate and the bottom of test specimen for longer specimens that can reach the base. A hole was drilled and tapped through the centre of the base plate that was used to attach the pressure transducer that would measure the pressure in the central cavity of the test specimen in the “transient pulse test”. In this experiment, the measurement of the central cavity pressure was adapted to the top of specimen; in this case, the hole was bolted and sealed.

The top loading cylinder served both as a rigid loading head against which the top end of the test specimen was sealed and the inlet manifold for the fluid pressure to the specimen cavity and the bleeder port. A plan view and a cross-sectional view through the middle of the cylinder are shown in Figure 4-8.
Figure 4-7 Assembly of the loading frame and water chamber with top beam

Figure 4-8 Plan and cross-sectional view of top cylinder
Two flattened areas were made at the edge of the top cylinder. The flattened areas were opposite to each other and provided areas for the connection of fittings. Two holes of diameter 1/8” (3.2 mm) were drilled in the cylinder: one at mid-height of the plate from the centre of one flattened area through to the centre of the other flattened area and the second hole upwards from the centre of the base to intersect with the cross-drilled hole. The ends of the cross-drilled hole were tapped and fabricated to accommodate an 1/8” NTP thread for connection of the elbows. One elbow was connected to a 3-way valve and in line with the Cole Parmer 100 psi (690 kPa) pressure transducer to measure fluid cavity pressure, while the other elbow was connected to another 3-way valve and tube for bleeding the air in the system (Figure 4-9). At the center of the top face of the loading cylinder, a depression was machined to seat the stainless steel ball bearing of the load cell. At the base of the cylinder, a groove of dimensions as shown in Figure 4-8 was made to seat the Neoprene O-ring size 213.

The load was applied by the reaction frame through a 1 ½ inch (38.1 mm) diameter stainless steel ball bearing which rested in the depression in the top cylinder (Figure 4-8). The reaction frame consisted of a rigid base, two threaded rods and a rigid top beam. More detail of the loading frame can be found in Carnaffan (1994).

The base was fabricated by welding two steel plates with a central hole 225 mm in diameter to the top and bottom of 100 mm long piece of steel pipe. The steel pipe had an inside diameter of 225 mm and an outside diameter of 250 mm. Four holes were drilled in the corners on the bottom plate of the rigid base and aluminum cylinders were attached to provide a clearance of 100 mm as shown in Figure 4-7.
The top load beam was fabricated by welding 76.2 mm by 610 mm by 12.7 mm strips of steel onto the top and bottom sides of a 76.2 mm by 101.6 mm by 6.35 mm hollow structural section. A small hole was drilled through the bottom of the rigid beam at the midpoint between the two ends. A bolt was passed through the small hole from the inside of the hollow beam and threaded into a 50 mm diameter rod, 95 mm long. The rod served as a spacer to apply the axial load to test specimens that sat below the top flange of the water chamber. A load cell was attached to the bottom of the 50 mm diameter rod (Figure 4-7). The base of the load cell had a dimpled load head which rested on the steel ball bearing.

In this experiment, samples of 60 mm in height cannot reach the base of water chamber for sealing the cavity, so the bottom of the specimen was seated on a stainless steel pedestal 100 mm diameter and about 150 mm in height. There is a groove for O-ring size 213 in the center of the top face of the pedestal, as shown in Figure 4-8.

Figure 4-9 Detail on test configuration for radial flow
The LabAlliance Serie I pump was connected to the top cylinder through the PEEK tubing to supply the water into the cavity of Indiana Limestone sample. In order to prevent the an unfavorable working condition that may occur during testing at high back pressures, a Upchurch Scientific back pressure regulator of fixed pressure 1000 psi (≈7 MPa) was used to stabilize the back pressure between the pump and the Limestone cylinder as shown in Figure 4-6.

4.2.2 Technical Specifications of radial permeability experiment

The specifications of the facilities used in the experimental program are as follow:

Data acquisition system: from National Instruments Corp., is composed of the following parts:

Dell Pentium III computer, LABVIEW 6i

AT-MIO-16F-5 analog data acquisition board

- 12 bit analog input resolution
- 16 single ended or 8 differential input channels
- Gain of 0.5, 1, 2, 5, 10, 20, 50, 100;
- Output relative accuracy (nonlinearity) ±1.5 LSB maximum, ±0.25 LSB typical
- Differential analog input ranges ±5 V or 0 to +10 V, software-selectable
- Output voltage ranges 0 to 10 V, unipolar mode; ±10 V, bipolar mode
- Counter input frequency 6.9 MHz maximum
AMUX-64T analog multiplexer with 64 channels and temperature input.

- Gain of 0.5, 1, 2, 5, 10, 20, 50, 100, 500;
- Input range: ± 10 V, ± 5 V, 0 to 10 V (selectable on MIO board)
- 64 single ended or 32 differential input channels
- Power requirement ± 5 V (± 5%), 150 mA typical
- Maximum working voltage ± 10 V
- Dimensions: 324.3 by 96.5 mm
- I/O connector: Two 50-pin male ribbon-cable connectors, one 68-pin male, shielded or ribbon-cable connector 78-screw terminals
- Operating temperature: 0° to 70°C
- Storage temperature: −55° to 150 °C
- Relative humidity: 5% to 90% noncondensing

Water chamber and the base

- Water chamber: Outside diameter 305 mm, height 305 mm, volume 20.4 litres
- Base: stainless steel plate 400×460×12.7 mm and 4 bolts

Stainless steel pedestal 100 mm in diameter and 150 mm in height

Loading frame:

- Two base plates 600×600×25 mm
- 100 mm length steel pipe with 225/250 mm inside/outside diameter
- 4× 100 mm length aluminum cylinder
- Rigid beam length 610 mm of hollow section: outside dimension 127×76.2 mm
- 2 1"-8 UNC threaded rods, 900 mm in length

Interface 1210 Load cell 0-10,000 lbs
- Output: 0-2.7 mV/V
- Excitation voltage (supply voltage): 0-20 V (use 10 V)
- Accuracy: ±0.05% full-scale

Top loading cylinder:
- Diameter 35 mm, height 70 mm,
- 2 stainless steel elbows 1/8”
- 2 Swagelok brass three-way valves,
- Stainless steel tubing ¼”

Stainless steel ball 1 ½”

Cole Parmer Pressure Transducer 100 psi (690 kPa)
- Serial number: 21042299175
- Output: 0.5-5.5 VDC
- Excitation voltage (supply voltage): 24 V
- Global error: ±0.25% full-scale
- Working temperature: -40° to 125°C
- Connection: ¼’ NPT (M)

Upchurch Scientific Back pressure regulator P-455 1000 psi (fixed)

Economical PEEK HPLC pump Serie I
- Manufacturer: LabAlliance/Cole Parmer
- Flow rate range: 0.01-10 ml/min
- Minimum/maximum back pressure: 100 psi/2500 psi (700 kPa/17.2 MPa)
- Typical back pressure: 1000 psi (7 MPa)
- Accuracy: ±2%
- Repeatability: 0.5%
- Working temperature: 0° to 80°C, fluid maximum 100°C

Pump tubing: SCIVEX Upchurch Scientific 1/16” OD PEEK™ Polymer Tubing

- Internal diameter: 0.5 mm
- Maximum internal pressure: 49 MPa
- Maximum temperature: 100°C

Neoprene O-rings standard size 213

4.3. Other Experimental Facilities

4.3.1 Distilled/De-aired water

In order to obtain accurate results as well as carefully maintaining the sensitive equipment (chromatograph pumps, valves, transducers etc.), the experiment used distilled/de-aired water as the operating (i.e. permeating and flooding) fluid. The 95% ion free distilled water was obtained from the McGill University Environmental Engineering Laboratories, the Department of Civil Engineering and Applied Mechanics. The water was transferred by special plastic containers to a SOILTEST de-airing apparatus at the Geomechanics Laboratories. Distilled water was vacuumed for a period of 24 hrs before it was used in the experiment. This distilled/de-aired water was also used to saturate the
sample while it was on the vibrating table, and as the flooding fluid was pumped into the Triaxial Cell by the GDS controller or the sample cavity by LabAlliance pump.

4.3.2 Technical Specifications of other facilities in the experiment

Other facilities used in experiment or in the preparation of the test samples are as follow:

Scale: Denver TR-2102

- Maximum weight: 2100 g
- Resolution: 0.01 g

De-aired Apparatus: SOILTEST LT-150 for the de-aired distilled water used as saturating and permeating fluid

Vacuum pump: Precision Scientific Inc. Model D25

- Vacuum rate: 0.024 m$^3$/min

Vibrating table: FMC Technologies SYNTRON

- Maximum vibration per minute: 3600 vpm
- Maximum amplitude: 1.6 mm

Distilled water supply: Barnstead FI-STREEM GLASS STILL: Model A56228-857

- Cleans tap water by removing contaminants from vapor-borne water droplets with patented vapor trap.

Bondo Resin Filler-Fibre Glass 402C from Bondo Corp. for sealing

Other supplies included ferrules, port connectors, tube adaptors, graduated cylinders, thermometer, timer watch, spirit level.
5. Experimental Procedures

5.1. General Testing Conditions

As discussed earlier in the thesis, the permeability tests on Indiana Limestone samples for both axial and transverse directions were performed under steady state conditions, using the constant flow rate technique (i.e. the experiment involved the establishment of conditions with constant inflow and outflow of water and stabilized fluid pressure). The stabilized fluid pressure and fluid temperature in the steady state conditions were required for the evaluation of permeability. In this research, all the experiments were performed at a constant flow rate of 1 ml/min. This flow rate was been selected according to the working ranges of the pumps, and the Reynolds number satisfied the condition where Darcy’ Law is valid.

In the uniaxial permeability tests, to ensure that there was no leakage at the interface of specimen and membrane, a trial test was run on the 100 mm diameter aluminum cylinder with Nitrile membrane (see Glowacki, 2006). An isotropic compressive stress of 1 MPa was determined and applied to all samples in the experiments since it was adequate for sealing with some factor of safety, and minimized the possible reduction in the coefficient of permeability due to the effect of confining stress. A trial test was also performed for axial stress to seal the hollow cylinder of the radial flow tests by Neoprene O-ring; an axial stress of approximately 300 kPa was selected for sealing purpose.

In this research, 4 specimens of diameter of 100 mm and heights of 60 mm were tested. Each specimen of Indiana Limestone in this study was first tested in uniaxial permeability test using the Triaxial apparatus and then the radial permeability tests were
performed on the same set of samples prepared according to the procedure described previously.

In radial permeability tests, both the end surfaces of the cylindrical samples were sealed with epoxy, and tested in the three stages of drilled central cavity: the surface cavity, the cylindrical cavity extended to about half the height of the sample, and the cavity extended right through the height of specimen (i.e. the drilled-through cavity). The experimental procedures consisted of preparation of the testing sample (including sealing the samples with epoxy and drilling the cavities), saturating the samples, assembling the test facilities and obtaining experimental results for data.

5.2. Sample Preparation

5.2.1 Test specimens

A cylindrical core of Indiana Limestone, recovered from the Indiana Limestone block 2 at Geomechanics Laboratories, McGill University, was machined down to a diameter of 100 mm with a relatively smooth surface; 4 samples were cut from the long core marked 2-12, indicating that specimens were produced from block # 2 and the indexes 1,2 show the position of the core as shown in Figure 3-1. These samples were labeled as 2-12-1, 2-12-2, 2-12-3, 2-12-4. The next step was to grind these samples to the height of 60 mm, giving two flat parallel end surfaces to the perfectly cylindrical specimens, as shown in Figure 5-1. This was accomplished using a concrete grinding machine from the McGill University Materials Laboratory.
5.2.2 Epoxy sealing

After uniaxial permeability testing in the GDS Triaxial system, the specimens were left to dry to prepare for the radial permeability testing. Both ends of the samples were sealed with Bondo epoxy using the following procedure:

Figure 5-1 A sample of Indiana Limestone for uniaxial permeability experiment

At one end of the clean and dry Indiana Limestone specimen, a circular piece of duct tape of 10 mm diameter was attached right at the center of cross-section of the surface. This tape layer was used to prevent the epoxy being absorbed into the testing area of the central cavity, and served as a guide to locate the central cavity later in the drilling process. The sample was placed inside two hollow plastic tubes, standing on two circular smooth plastic plates (non-stick Teflon) on a flat and level surface as shown in Figure 5-2. These tubes were cut to create the gaps of about 3 mm along the height of the tube. The sealing surface of sample was put in contact with the two flat plastic plates. The clamp outside circumference of outer tube was tightened so that the gaps on the hollow
plastic tubes close, that held the sample fixed to the cylindrical tubes. The top plastic plate (in contact with the sample bottom) was then taken out. The thickness of this top plastic plate ($\approx 1.5$ mm) controlled the thickness of the epoxy layer, so that it was thick enough to avoid delaminating of epoxy at the specimen surface at high pressure.

Figure 5-2 Set up and procedure of epoxy sealing
Bondo Marine Resin was mixed with a hardener, according to manufacturer’s instructions, and stirred well for about 5-10 minutes. The mixed resin was applied evenly on the surface of the Teflon plate. The sample and tubes were placed at the center on top of the bottom plastic plate and left for the resin to harden in at least 5 hours before removing the plastic plate from sample. The clamp was then un-tightened to remove the specimen from the plastic tubes. The sealed surface was machined in the lathe to remove the epoxy outside the cross-sectional area of the sample and then polished using 600 grit sandpaper.

5.2.3 Drilling central cavity

In radial permeability tests, the sample was drilled at the centre through the epoxy layer using a drill bit of 3/8” (≈9.525 mm) diameter; the size of the actual cavity was 10 mm. The bit drilled through the resin layer into the Limestone cavities to achieve depths of 3 mm, 30 mm and through the height of sample for different stages of radial flow testing: surface cavity, partially drilled cavity and drilled-through cavity. After drilling, the cavity of the specimen was cleaned using metal brusher to remove any debris that may have penetrated into the material during drilling process. Figure 5-3 shows a prepared specimen before and after drilling a surface cavity.
5.2.4 Saturation process

For the steady state permeability test, the specimen had to be fully saturated before performing the testing. Figure 5-4 shows the set up for the saturation procedure: the prepared test specimen was transferred to a stainless steel container, covered with transparent Plexiglas, that was filled with distilled water for the saturation process. During saturation, the vacuum pump operated intermittently while the water chamber was put on the Sytron Vibrating Table (CT-164 from ELE International). When the vacuum pump was operating, vibration of the water chamber accelerated the removal of the air from the sample. The saturation was considered to be complete when no air bubbles were visible through the Plexiglas top lid of the water chamber. Normally, it took about 24 hours for an Indiana Limestone specimen to reach close to the fully saturated state.

5.3. Experimental Procedures for Axial Permeability Tests

The testing procedure for determining axial permeability followed the same procedures used by Glowacki (2006) and is only briefly reviewed here.
Figure 5-4 Saturation of sample in vacuumed water chamber with vibrating table

The experimental technique for axial permeability measurement is outlined in the following step-by-step procedures:

1) Saturated the samples using the vibrating table and vacuum pump (see section 5.2.4).

2) Primed the Shimadzu pump with 10% Methanol solution before and after testing.

3) Filled GDS controller with distilled/de-aired water from the reservoir until it was full.

4) 2 disks of TS 750 Polyfelt geotextile, 100 mm in diameter were cut from geotextile cloth. A Nitrile rubber membrane 30 mm longer than the sample height needed to be prepared and soaked in water.
5) The Indiana Limestone cylinder was removed from the saturation bath and inserted midway inside the rubber membrane as quickly as possible. Geotextile disks, soaked in water until saturated, were inserted at the lower and upper surfaces of the cylindrical sample.

6) The parts assembled in step 5 were placed onto the base of the Triaxial Cell, and four hose clamps were installed, see Figure 5-5.

7) The top cap was mounted into place with the outflow line fastened to its port. A hose clamp was positioned below and above a geotextile disk, and then the hose clamps were tightened to ensure a good seal.

8) Started the pump to run at 1 ml/min. Check on all sides to ensure that there were no leaks. The sample was left on the pedestal for a period of at least 72 hrs with the pump operating to ensure complete saturation of sample (see section 5.4 for further detail).

9) Upon completion of step 8, stopped the pump and lowered the outer stainless steel chamber into place with the clamp rings and the retaining ring.

10) Filled the empty chamber with distilled/de-aired water when the valve and the port of chamber were open. Once the chamber was filled, closed the GDS ports and the pressure was raised to 200 kPa using the GDS controller. To release the entrapped air, the upper port nut was loosened until the pressure inside dropped to zero; the nut was then tightened to achieve a seal (see Figure 5-6). At this point careful inspection was made to locate and remove all air bubbles from connections, tubing or valves using the pressure head from the reservoir and the GDS controller. Before the confining pressure was raised any further, the outflow
valve was opened and the inlet valve closed; this minimized any component failures (see Figure 5-6).

Figure 5-5 A specimen of Indiana Limestone was in place on pedestal with hose clamps (after Glowacki, 2006)

Figure 5-6 Photograph of the ports prepared for pressurization of the chamber and the valve setup for the permeating fluid
11) The desired confining pressure of 1 MPa was obtained with the GDS controller using the “Pressure Target” mode. Once the specified confining pressure was attained the sample was left for 10-15 minutes to stabilize before any permeability testing begun. The inlet valve was opened and the Shimadzu pump started at the desired flow rate of 1 ml/min. During the first run the outlet valve was also opened to remove air from the tubing and connections.

12) Started the data acquisition system and recorded the atmospheric pressure for about 2-3 minutes as the offset for the pressure when the outlet valve was opened. Closed the outlet valve and started recording the cavity pressure. The confining pressure and the pumped fluid pressure were recorded at 1 Hz until a constant outflow was measured and a stable outflow pressure was established.

13) Stopped the pump and closed the inlet valve once constant flow was confirmed.

14) Test was repeated 3 times for each sample from step 11 onward after the sample was recovered and free from residual fluid pressure, normally after leaving it for 2-3 hours.

5.4. Experimental Procedures for Radial Permeability Test

1) Saturated the sealed/drilled test specimen in water container using vibrating table.

2) Transferred the sample to the test chamber, which was already filled with water, and placed it on top of pedestal and O-ring, in the centre of water chamber. The sample must always be submerged in water.

3) Placed the top cylinder, with O-ring attached, to bleeder valves, connector and pressure transducer on the top of the specimen, at the center of specimen. The
outflow tubing was always kept submerged in the water chamber. The ball bearing was placed in the depression in the centre of the top plate.

4) Placed the load head (top beam, cylindrical spacer and load cell) into the frame guided by two threaded rods. The load head was lowered and adjusted as evenly as possible by adjusting the nuts at the bottom of the beam and using the spirit level until it reached the steel ball. While the weight of the load head kept the steel ball and the top cylinder in place, the cylinder could still be adjusted so that it was vertically straight and at the right center of the specimen.

5) Connected the pump, after it was primed with Methanol 5% solution, to the stainless steel tubing through a connector as shown in Figure 4-9. The data acquisition system was connected to the load cell and pressure transducer. The data acquisition was activated to record the applied load for sealing. The load head was tightened down using the nuts on top of the beam. The nuts were tightened as evenly as possible by tightening one nut a fraction of a twist, then the other nut by an equal amount. The nuts were tightened until an axial load of 2500 N was recorded by the load cell.

6) Removing the air bubbles in the line of tubing, connections, pressure transducer and cavity is a very important procedure to obtain accurate and consistent results. The valve of outflow tubing was opened, and the valve of priming tube was closed while the line from the pump was opened. The pump delivered distilled/de-aired water into the system, first filling up the cavity and the tubing, and pushing air bubbles out through the outflow tubing. It took about 10-15 min to push the air bubbles out.
7) Stopped the pump, connected a syringe filled up with distilled/de-aired water to the primer tubing (Figure 5-7), opened valve to connect this tubing to the line of outflow tubing, the top cylinder and central cavity. Valve for supplying water from the pump was closed. The system was primed by sucking and pushing the syringe, pressing out the air bubbles in case there were any left in the line until no air bubbles were observed through the tubing.

8) Closed the valve for the primer tubing, and opened the valve for inflow PEEK tubing; started the pump at 1 ml/min to supply water into the cavity. The data acquisition system started to record the data from the load cell and pressure transducer while the outflow valve was opened for 2-3 minutes and then the valve was closed to start pressurizing the cavity of the sample.

![Swagelok 3-way valve](image1)

![Plastic tubing for priming connected to syringe](image2)

![3-way valve and outflow tubing submerged in water](image3)

Figure 5-7 Priming procedure of the radial permeability test.

9) As also observed in the uniaxial permeability tests conducted by Glowacki (2006), although the sample had been pre-saturated in a vibrating water chamber, there was a trend for the built-up cavity pressure to increase slightly at the start of
the test, this was not as noticeable over the short term but was visible after a long period of time; the sample did not seem to be completely saturated. This may lead to the inconsistent results of some permeability tests. After a certain period of time which varied from 2 to 3 days depending on the sample) of continuous pressurization by de-aired water, the built-up pressure started to drop and then stabilized. This observation meant that the first pressurization run had to last at least 72 hours before the pump was stopped, i.e. until the cavity pressure “dropped” and stabilized. Then the outflow valve was opened and the sample was left to dissipate the pressure back to original state for at least 2 hours. Outflow water can be removed from water chamber by vacuum to prevent overflow during this long initial run.

10) After the sample was fully saturated and free of residual pressure, the permeability testing commenced. The pump started supplying a constant flow rate of 1 ml/min into the cavity. The data acquisition was activated, start record the load and pressure for a few minutes while the outflow valve was opened; atmospheric pressure recorded at this initial stage set the baseline of data for the recorded pressure. The outflow valve was then closed and recording of the evolution of cavity pressure started. Measurement of the inflow pumping rate using data from the scale was compared to the flow rate of the pump. Although the outflow rate could not be measured in this test, the steady state condition could easily be recognized by the stabilized pressure in the cavity in the recorded data.
11) The “load” and “pressure” history records were acquired using the DAQ at a recording rate of 1 sec, until the pressure was stabilized; these results were saved in the computer. A normal data set was recorded for 1 to 1.5 hours for each test. The temperature of the outflow water was also recorded by averaging the thermometer readings from the start, middle and end of the test.

12) The test was repeated twice more from step 10 onwards. This gave three sets of results for each specimen under the same conditions. Between each test, the sample has been left to recover for 2-3 hours. During the test, reloading might be needed by tightening the nuts; when the loading frame was relaxed, which can lead to the reduction in the applied sealing stress.
6. Interpretation of Acquired Data and Results

6.1. Results of Axial Permeability Tests

The pressure data from axial permeability test were collected at 1Hz by the pressure transducers and the Techmatron DAQ and have been saved as an EXCEL spreadsheet. In each experiment, three data sets were collected to confirm the repeatability of the results.

The recorded data acquired were raw data in voltage units (V). The stabilized pressure at steady state can be converted to pressure (kPa), using a conversion factor for the pressure transducer used in the permeability tests that is dependent on the gain and calibration factor of the devices (Glowacki, 2006).

The conversion of the raw data to pressure in kPa can be calculated as follow:

\[
P(kPa) = \left( \frac{V_1 - V_0}{G} \times 1000 \right) \frac{mV}{V} \times \frac{P_{max}}{CF}
\]

(6.1)

where

- \(V_1\) is the recorded reading of pressure in volts,
- \(V_0\) is the offset recorded reading (baseline) of atmospheric pressure,
- \(G\) gain depends on DAQ system, in this case \(G=50\)
- \(P_{max}\): capacity of the pressure transducer (\(P=300\) psi)
- \(CF\): calibration factor of the pressure transducer (\(CF=76.511\) mV)

Figure 6-1 shows an example of the data set recorded for an axial permeability test.
The hydraulic conductivity has been estimated using the equation (2.13):

\[
k_z = \frac{L}{\left(\frac{L_{eq}}{k_{eq}} - \frac{L_{sys}}{k_{sys}}\right)}
\]

where

\[
\frac{k_{eq}}{L_{eq}} = \frac{Q\gamma}{Ap}
\]

The system resistance parameter \(k_{sys}/L_{sys}\) at the isotropic confining pressure of 1MPa determined from the experiments is \(6.6 \times 10^{-6}\), and

\[A=7.853982 \times 10^{-2} \text{ m}^2\]
\[ \gamma = 9.8 \times 10^3 \text{ N/m}^3 \]

\[ Q = 1 \text{ ml/min} = 1.667 \times 10^{-8} \text{ m}^3/\text{s} \]

The permeability coefficient was obtained from the hydraulic conductivity using equation (2.1)

\[ K_z = \frac{k_z \gamma}{\mu} \]

and was adjusted to the reference temperature (equation 2.17):

\[ K_{20} = (1.495 - 0.02452T) \times K_T \]

Table 6-1 shows the result of estimated permeability for axial flow tests conducted at \( T = 20^\circ \text{C} \).

<table>
<thead>
<tr>
<th>Sample</th>
<th>Number of tests</th>
<th>Average stabilized pressure (kPa)</th>
<th>( T_w(\circ \text{C}) )</th>
<th>( K_{20} (10^{-15} \text{ m}^2) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-12-1</td>
<td>3</td>
<td>6.72\pm0.07</td>
<td>26</td>
<td>(23\pm0.3)</td>
</tr>
<tr>
<td>2-12-2</td>
<td>3</td>
<td>8.9\pm0.4</td>
<td>26</td>
<td>(13.7\pm0.7)</td>
</tr>
<tr>
<td>2-12-3</td>
<td>3</td>
<td>8.36\pm0.15</td>
<td>26</td>
<td>(13.8\pm0.3)</td>
</tr>
<tr>
<td>2-12-4</td>
<td>3</td>
<td>7.92\pm0.22</td>
<td>27</td>
<td>(14.3\pm0.4)</td>
</tr>
</tbody>
</table>

### 6.2. Results of Fully-Drilled Permeability Tests

The pressure data from the radial permeability test of a fully-drilled sample were also collected at 1 Hz and have been saved as an ASCII file. Three data sets were collected to
confirm the repeatability of the results. Figure 6-2 shows one data set for the sample 2-12-3 with drilled-through cavity and the pump flow rate at 1 ml/min.

![Graph showing recorded pressure development in Indiana Limestone sample 2-12-3 in a radial permeability test](image)

Figure 6-2 Recorded pressure development in Indiana Limestone sample 2-12-3 in a radial permeability test

For the fully drilled cavity, the test involves flow only in the radial direction; the permeability from a steady state test of the Indiana Limestone in the radial (transverse) direction can be estimated from (2.15):

$$K_r = \frac{Q \mu \ln \left( \frac{b}{a} \right)}{2\pi L \rho}$$

Table 6-2 shows the complete results for 4 samples:

$L=0.06$ m, $b=0.005$ m, $a=0.05$ m

$Q=1.667 \times 10^{-8}$ m$^3$/s

$\mu_{20}=1.002 \times 10^{-3}$ (Pa.sec/m$^2$)
Table 6-2 Coefficients of transverse permeability estimated from radial permeability tests

<table>
<thead>
<tr>
<th>Sample</th>
<th>Number of tests</th>
<th>Average Pressure (kPa)</th>
<th>$T_w$(°C)</th>
<th>$K_{r20}$(10^{-15} m²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-12-1</td>
<td>3</td>
<td>10.2±0.43</td>
<td>25</td>
<td>(8.82±0.43)</td>
</tr>
<tr>
<td>2-12-2</td>
<td>3</td>
<td>11.±0.6</td>
<td>27</td>
<td>(7.72±0.4)</td>
</tr>
<tr>
<td>2-12-3</td>
<td>3</td>
<td>14.5±0.5</td>
<td>27</td>
<td>(5.89±0.3)</td>
</tr>
<tr>
<td>2-12-4</td>
<td>3</td>
<td>10.7±0.4</td>
<td>27</td>
<td>(7.9±0.3)</td>
</tr>
</tbody>
</table>

6.3. Steady State Flow Tests on Surface-Drilled and Partially-Drilled Cylinders

In order to interpret the experimental data from the experimental configurations of surface cavity and partially-drilled cavity, it is necessary to solve Laplace’s equation (2.7) with mixed boundary conditions at the outer surface since an analytical solution results in a complicated final result, which also requires numerical evaluation (Goggin et al., 1988). The mathematical analysis of the problem is not as straightforward as a one-dimensional or purely radial problem since it has to recognize the singularities in the solution when the boundary condition changes from a Dirichlet to a Neumann type. Therefore a computational technique in a finite element program has been used to interpret the data. This section presents the use of COMSOL® Multiphysics (COMSOL, 2006) to solve the problem of groundwater transport to evaluate the permeability from the experimental data.
6.3.1 Computational modeling of steady flow in partially drilled samples with COMSOL ® Multiphysics

COMSOL ® Multiphysics (formerly FEMLAB) is a finite element code based on MATLAB platform developed by Germund Dahlquist at the Royal Institute of Technology (KTH) in Sweden. The code is a useful tool for modeling the physical processes and phenomenal in multiphysics, which can be described with partial differential equations (PDEs) (Earth Science and Hydrology, Process and Chemical Engineering, Acoustics, Heat Transfer etc.). The Earth Science Module in COMSOL ® Multiphysics can be used in this study to solve the pseudo-Laplace’s governing equation for experimental permeability tests.

The Earth Science module in COMSOL ® Multiphysics (http://www.comsol.com/) can be used to model 3D, 2D, 1D and 1D or 2D axi-symmetric problems of groundwater flow. This study deals with the problems concerning flow through porous media of assumed transversely isotropic. The COMSOL code is capable of using two different values of transverse $K_r$ and vertical $K_z$ permeability of transversely isotropic media, which is specified in the code as the vertical permeability $K_z$ and the ratio $K_z/K_r$.

The problem of a cylindrical specimen with a central cavity in this study can be modeled in COMSOL ® as a two-dimensional axi-symmetric problem of finite extent, as shown in Figure 6-3, Figure 6-4 and Figure 6-5. The geometrical region of 2D axi-symmetric was discredited into triangular finite elements in COMSOL ®, using the free mesh (or unstructured mesh) generation of triangular elements. The mesh generation in the code can be controlled through parameters such as element size, the element growth rate for
graded mesh generation, mesh curvature etc., and can be adjusted to mesh different regions to the desired refinement. Figures 6-3, 6-4 and 6-5 show the mesh generation for samples with a surface cavity and a partially-drilled cavity to the mid-height of the samples.

The two types of boundary conditions of the tested sample can be prescribed in the COMSOL ® Earth Science module: the prescribed pressure head (Dirichlet boundary) and the impervious boundary (Neumann type). The solution of the problem in the code is capable of estimating the total flow for any specific surface regions. This total outflow on the free surface of specimen is compared to the inflow supplied to the cavity (pump flow rate), which must be balanced in a steady state condition.

Figure 6-3 COMSOL mesh for sample 2-12-1 with a 3 mm cavity (3216 elements)
Figure 6-4 COMSOL mesh for sample 2-12-1 with a 36.5 mm cavity (4408 elements)

Figure 6-5 COMSOL mesh for sample 2-12-2, 2-12-3, 2-12-4 with a 30 mm cavity (4276 elements)
The results for the axial permeabilities from the uniaxial tests were combined with the test results for the surface cavity or partially-drilled cavity tests and a computational model in COMSOL ® was generated to “back estimate” the radial permeability of the Indiana Limestone sample as follows:

For the sample 2-12-1 with 36.5 mm cavity depth, using an axial permeability $K_a=2.3 \times 10^{-14}$ m$^2$ and the built-up water pressure $p=13.68$ kPa applied to the entire cavity surface, the radial permeability $K_r$ must be estimated so that the total flux on the free surface (i.e., outflow for steady state) was equal to the inflow pumping rate $1.666 \times 10^{-8}$ m$^3$/s ($Q=1$ ml/min).

Figure 6-6 shows the variation of the ratio $p/Q$ to the ratio of $K_z/K_r$ generated by COMSOL ®, while $K_z=2.3 \times 10^{-14}$ m$^2$ and the pressure head in cavity $p=13.68$ kPa.

With $p=13.68$ kPa and $Q=1$ ml/min, $K_z/K_r=2.71$, i.e. $K_r=8.83 \times 10^{-15}$ m$^2$

![Figure 6-6 Variation of cavity pressure and flow rate to axial and transverse permeability](image-url)
6.3.2 Results of surface cavity tests

Figure 6-7 shows a set of recorded data for sample 2-12-3 tested at a cavity depth of 3 mm and a flow rate of 1 ml/min.

![Figure 6-7 Water pressure in the 3 mm cavity of sample 2-12-3 at 1 ml/min flow rate](image)

Table 6-3 summaries the results of the permeability tests for 4 samples.

**Table 6-3 Surface cavity permeability tests of Indiana Limestone samples**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Number of tests</th>
<th>Cavity depth (mm)</th>
<th>Cavity diameter (mm)</th>
<th>Average Pressure (kPa)</th>
<th>$T_w$ (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-12-1</td>
<td>3</td>
<td>3</td>
<td>10</td>
<td>112±5.2</td>
<td>26</td>
</tr>
<tr>
<td>2-12-2</td>
<td>3</td>
<td>3</td>
<td>10</td>
<td>75±3</td>
<td>27</td>
</tr>
<tr>
<td>2-12-3</td>
<td>3</td>
<td>3</td>
<td>10</td>
<td>63.6±5</td>
<td>27</td>
</tr>
<tr>
<td>2-12-4</td>
<td>3</td>
<td>3</td>
<td>10</td>
<td>83±2</td>
<td>26</td>
</tr>
</tbody>
</table>
Using the same modelling procedure that was discussed in section 6.3.1, permeabilities of transverse direction of Indiana Limestone samples with surface cavity were “back estimated”. Table 6-4 shows the results of transverse permeabilities estimated using COMSOL ® modeling and surface cavity test results for all the samples.

Table 6-4 Estimated transverse permeability from surface cavity tests and COMSOL ® results

<table>
<thead>
<tr>
<th>Sample</th>
<th>Number of tests</th>
<th>Cavity depth (mm)</th>
<th>$K_z$ (axial tests) $10^{-14}$ m$^2$</th>
<th>$K_r$ (COMSOL) $10^{-15}$ m$^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-12-1</td>
<td>3</td>
<td>3</td>
<td>2.3±0.03</td>
<td>1.5±0.07</td>
</tr>
<tr>
<td>2-12-2</td>
<td>3</td>
<td>3</td>
<td>1.37±0.07</td>
<td>3.42±0.1</td>
</tr>
<tr>
<td>2-12-3</td>
<td>3</td>
<td>3</td>
<td>1.38±0.03</td>
<td>4.47±0.3</td>
</tr>
<tr>
<td>2-12-4</td>
<td>3</td>
<td>3</td>
<td>1.43±0.04</td>
<td>3.0±0.1</td>
</tr>
</tbody>
</table>

6.3.3 Results of partially-drilled cavity tests

Figure 6-8 shows a set of recorded data for the sample 2-12-2 tested at cavity depth 30 mm and the flow rate of 1ml/min.
Figure 6-8 Water pressure in the 30 mm cavity of sample 2-12-2 at 1 ml/min flow rate

Table 6-5 summaries the permeability test result for 4 cylindrical specimens.

Table 6-5 Permeability tests on partially-drilled Indiana Limestone samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>Number of tests</th>
<th>Cavity depth (mm)</th>
<th>Cavity diameter (mm)</th>
<th>Average Pressure (kPa)</th>
<th>$T_w$(°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-12-1</td>
<td>3</td>
<td>36.5</td>
<td>10</td>
<td>13.68±0.2</td>
<td>26</td>
</tr>
<tr>
<td>2-12-2</td>
<td>3</td>
<td>30</td>
<td>10</td>
<td>31.3±2.3</td>
<td>27</td>
</tr>
<tr>
<td>2-12-3</td>
<td>3</td>
<td>30</td>
<td>10</td>
<td>43.9±0.5</td>
<td>26</td>
</tr>
<tr>
<td>2-12-4</td>
<td>3</td>
<td>30</td>
<td>10</td>
<td>19.5±1.2</td>
<td>26</td>
</tr>
</tbody>
</table>

Table 6-6 shows the estimated transverse permeability derived from the partially-drilled cavity test data.
Table 6-6 Estimated transverse permeability from partially-drilled cavity tests and COMSOL® results

<table>
<thead>
<tr>
<th>Sample</th>
<th>Number of tests</th>
<th>Cavity depth (mm)</th>
<th>$K_z$ (axial tests) $10^{-15}$ m²</th>
<th>$K_r$ (COMSOL) $10^{-15}$ m²</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-12-1</td>
<td>3</td>
<td>36.5</td>
<td>23.0±0.3</td>
<td>8.83±0.04</td>
</tr>
<tr>
<td>2-12-2</td>
<td>3</td>
<td>30</td>
<td>13.7±0.7</td>
<td>4.2±0.03</td>
</tr>
<tr>
<td>2-12-3</td>
<td>3</td>
<td>30</td>
<td>13.8±0.3</td>
<td>2.5±0.02</td>
</tr>
<tr>
<td>2-12-4</td>
<td>3</td>
<td>30</td>
<td>14.3±0.4</td>
<td>6.21±0.02</td>
</tr>
</tbody>
</table>

6.4. Discussion of Results

Table 6-7 shows the results of axial and transverse permeabilities estimated from all tests.

Table 6-7 Summary of estimated axial and transverse permeabilities from all tests

<table>
<thead>
<tr>
<th>Sample</th>
<th>$K_z$ (axial tests) $10^{-15}$ m²</th>
<th>$K_r$ (10$^{-15}$ m²) (drilled-through)</th>
<th>$K_r$ (10$^{-15}$ m²) (partially drilled)</th>
<th>$K_r$ (10$^{-15}$ m²) (surface cavity)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-12-1</td>
<td>23±0.3</td>
<td>8.82±0.43</td>
<td>8.83±0.04</td>
<td>1.5±0.07</td>
</tr>
<tr>
<td>2-12-2</td>
<td>13.7±0.7</td>
<td>7.72±0.4</td>
<td>4.2±0.03</td>
<td>3.42±0.1</td>
</tr>
<tr>
<td>2-12-3</td>
<td>13.8±0.3</td>
<td>5.87±0.3</td>
<td>2.5±0.02</td>
<td>4.47±0.3</td>
</tr>
<tr>
<td>2-12-4</td>
<td>14.3±0.4</td>
<td>7.9±0.3</td>
<td>6.21±0.02</td>
<td>3.0±0.1</td>
</tr>
</tbody>
</table>

The results from all the experiments are within the range of permeability of Indiana Limestone given in textbook by Freeze and Cherry, 1979 and comparable to the results.
from previous study by Selvadurai and Glowacki, 2008 of similar conditions. The test results show that the estimated permeabilities obtained from the uniaxial tests and complete radial tests on Indiana Limestone are fairly consistent although then exhibits variability, while the estimated transverse permeabilities from the surface cavity and partially drilled cavity tests are quite scattered; the transverse permeability estimate is much less than estimates from the radial flow tests. The reason for this fluctuation is not known, but is most probably due to the drilling process that produced micro particles that were able to penetrate into the surface of the cavity, blocking the flow of water and generating high cavity pressures.

6.5. Tests of transient decay from steady state

The recorded data from the test at steady state was normally recorded at 1 Hz, as these data were adequate for steady state analysis. As a supplement to the results from the steady state condition, another set of data also was acquired from the tests: the time history of cavity pressure decay from the steady state condition, while the pump was stopped. Those transient data were different from those of conventional “pulse test”. The data can be used in future investigation to provide a confirmation with the results obtained from steady state tests.

The acquisition of transient data is described as follow:

1) When the cavity pressure stabilized after recording the time history, and a steady state pressure was achieved, the data acquisition at 1 Hz was stopped while the test was still running.
2) The DAQ was reactivated at a faster rate, 100 Hz, and began to record the steady pressure in a short period of time.

3) The pump was stopped and the decay of cavity pressure was recorded until it reached atmospheric pressure.

Figure 6-9 shows an example of pressure decay data of the sample 2-12-3 with 30 mm cavity and Figure 6-10 presents the normalized decay curve which may be used for further study.

Figure 6-9 Time history of pressure decay from a steady state test
Sample 2-12-3 (partially-drilled)
$Q=1\ \text{ml/min}$
$T_w=26^\circ\text{C}$

Figure 6-10 Normalized pressure decay curve from a steady state test
7. Conclusions and Future Work

7.1. Conclusions

The research developed and applied a methodology and experimental technique for permeability measurement of both axial and transverse permeability of Indiana Limestone in laboratory in the same sample using the constant flow rate technique in a steady state condition. The experimental facilities and procedures set the basis for steady state technique for measurement of hydraulic characteristics of not only Indiana Limestone but other stratified geological rocks of transversely isotropic hydraulic characteristics.

The estimated permeabilities of Indiana Limestone derived from the tests, using small axial stress of about 300 kPa in radial direction or 1 MPa isotropic confining pressure in axial direction, are in the order of $10^{-15}$ m$^2$. The results from preliminary tests indicate some slight level of anisotropy in Indiana Limestone specimens at the laboratory scale, with ratios of axial and transverse permeabilities $K_z/K_r$ vary from 1.8 to 2.6. And the obtained results show that the axial permeability $K_z > K_r$ of bedding direction, while most hydrogeologic situation find the opposite, with $K_r$ are greater than $K_z$. More tests are needed to conclusively estimate the level of anisotropy of the hydraulic characteristics of Indiana Limestone.

The results from partially drilled cavity tests when back calculated using COMSOL ® modelling to estimate $K_r$, do not provide a reliable set of data. However, investigation can be carried out to check if the preparation and testing method can be improved. The tests performed at different stages of drilling, with numerical modeling, are a possible alternate
means of evaluating the permeability in the axial and transverse directions, without the use of one-dimensional tests.

7.2. Future Work

i. As mentioned in the previous section, any further studies using the experimental facilities can be improved by modifying the technique for successive drilling of the cylindrical sample; this would be another way to confirm the results of the permeability estimation. The tests can be continued on other specimens to get more consistent results with the partially drilled cavity, especially in radial flow to confirm the hydraulic characteristics of geomaterials.

ii. The hydraulic transport characteristics of rocks can be validated through the development of transient tests and analytical (or computational) modeling using the transient data in section 6.5.

iii. A transient test can be developed using the same facilities and concepts for geomaterials of the very low permeability where the steady state is not applicable.

iv. Develop and extend the experimental method to address the directionally-dependent permeability of non-isotropic materials.
References


ASTM D 5084-97, 2000, **4.08**.

ASTM D 2216-05, 2006, **4.08**, 205-211.

ASTM D 5084-03, 2006, **4(8)**, 1000-1022.


