On the Surface Permeability of Indiana Limestone

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ABSTRACT: This paper presents the results of a research investigation leading to the development of a technique for the measurement of the surface permeability of Indiana Limestone. The tests are conducted on a cuboidal block of the limestone with a flat surface that can be sealed to create a circular aperture through which water influx can take place. Computational results for Darcy flow are used to interpret the results of the experiments. The water entry area can be moved over the surface area of the block to investigate, quite conveniently, the distribution of permeability across the surface of the limestone block sample.

1 INTRODUCTION

Permeability is a key geomaterial property that is important to many problems in geoenvironmental and geotechnical engineering. Many problems in hydrogeology, including groundwater extraction, contaminant transport, aquifer contamination by sediments, etc., are controlled by the permeability characteristics of the porous materials involved. Permeability describes the ability of fluids to flow through the pore space of a geomaterial. In contrast to fluid conductivity, which can be influenced by the mass density and viscosity of the permeating fluid, the property of permeability or intrinsic permeability depends solely on the porous structure of the geomaterial. The intrinsic permeability has units of length squared (m^2). In hydrological geosciences in particular, the property of permeability is considered to be sessile, whereas in geomechanics factors such as the stress state acting on the geomaterial can have a significant influence on the permeability (Selvadurai and Głowacki, 2008). Relatively new areas in environmental geomechanics, including carbon dioxide sequestration, nuclear waste disposal and deep injection of hazardous wastes (Laughton et al. 1986; Chapman and McKinley 1987; Gnirk 1993; Selvadurai and Nguyen 1997; Apps and Tsang, 1996; Selvadurai, 2006), require accurate estimates of permeability of the geologic media encountered. The estimation of permeability of geomaterials becomes even more crucial when the information is used in computational models of flow and transport processes to predict the long-term performance of strategies for geoenvironmental remediation.

Permeability of rocks is scale-dependent. These can range from crustal scales of 0.5 km to 5.0 km to borehole scales ranging from 30 m to 300 m to laboratory scales of 5 cm to 15cm. The variability in the "permeability" is largely derived from natural inhomogeneities that can include fractures, fissures, damage zones, voids and vugs that have will have an influence on the interpretation of bulk permeability of the tested zone or element. The experimental work associated with this research focuses on the measurement of the permeability of a relatively intact block of Indiana Limestone. So why attention should be focused on the determination of the permeability of the intact units of a geologic material when variability can be encountered as the scale changes? The main reason is that in disposal endeavours, such as geologic sequestration of carbon dioxide, the intact units of rock offer a substantial pore volume for sequestration and access to this volume is controlled by the intact permeability of the rock. In cases where fractures and

other defects are present in the host geologic medium, these features will be sealed before any waste isolation activity can commence. Upon sealing, the main mode of fluid transport through the porous geologic medium is the intact material.

Laboratory procedures therefore offer the most convenient techniques for the experimental determination of natural geologic materials that are void of dominant defects such as fractures, fissures and other visually observable defects including solution channels. Several laboratory procedures have been developed, particularly for testing rock cores recovered from drilling operations associated with site investigations. The test methodologies invariably involve the application of steady state flow, usually in the longitudinal direction of the sample. The attainment of steady state conditions largely depends on the permeability characteristics of the rock itself. The main advantage of a steady state test in hydraulic property measurement is that the interpretation of the results is relatively straightforward, dependent only on the hydraulic boundary conditions of the test, the dimensions of the sample and a knowledge of the steady flow rate. In materials with low permeability an inordinate amount of time is required to attain steady state flow conditions, and recourse is usually made to transient methods that are referred to as hydraulic pulse tests. Pulse tests are rapid tests but require a significantly larger number of extraneous parameters including the compressibility of the fabric of the geologic medium to interpret the test data. Research dealing with the evaluation of permeability properties of geomaterials using both steady state and pulse tests is quite extensive and no attempt will be made to provide an exhaustive review of the area. References to historical and current literature in this area can be found in the articles by Brace et al. (1968), Hsieh et al. (1981), Selvadurai and Carnaffan (1997), Tokunaga and Kameya (2003), Suri et al. (1997), Selvadurai et al. (2005), Selvadurai and Selvadurai (2007), Selvadurai and Głowacki (2008) and Selvadurai (2009).

This paper is a departure from the use of rock cores for determining the permeability characteristics of geologic materials, in that the permeability tests are conducted on a substantially larger intact sample of the geomaterial. The accessibility to large samples is a prerequisite to the tests that are advocated in this research. In practical situations, large block samples can be obtained from either rock outcrops or from tunnels and adits and test pits that are used for other geological investigations. The test involves the application of a constant flow rate to an open region of the test specimen, which is provided with an adequate seal to enable the development of steady flow conditions in the flow domain. Tests along these lines were conducted by Tidwell and Wilson (1997) in their experimental work involving the measurement of air permeability of a large block of Berea Sandstone. These studies have been adapted and extended by a number of investigators who have examined the surface permeability measurement technique both from the view of theoretical relationships that are used to interpret the results to applications involving measurement of permeability using variations of the sealing technique. The paper discusses briefly the experimental procedure used in the research, the computational models used to interpret the data and the results of preliminary experiments conducted to estimate the surface permeability characteristics of the Indiana Limestone. A more detailed presentation that includes mathematical evaluations, computational developments, complete experimental data and their analysis will be presented in a forthcoming paper (Selvadurai and Selvadurai, 2009).

2 THE TEST FACILITY AND EXPERIMENTAL PROCEDURE

2.1 *The Test Specimen*

The Indiana Limestone used in the experiments was a 508 mm cube that was obtained from a local supplier. Indiana limestone has been used quite extensively in research investigations and the geomechanical and mineralogical characteristics of the rock are well documented in the open literature (Głowacki, 2007). The faces of the sample were saw cut to a surface texture consistent with an emery paper of a FEPA grade P120. Actual surface roughness characterizations we not required for the research investigation. The deformability, strength and other physical properties of the rock were determined in previous investigations. The colours of the limestone on the separate surfaces were not uniform and the demarcations were noted as supplementary information to assist in the interpretation of the test data.

2.2 *General Setup*

The test configuration involves the development of a perfect seal over an annular region in contact with the surface of the cuboidal specimen and the application of a constant potential within the internal circular region, to attain steady state flow conditions. The dimensions of the annular sealing region can vary depending on the experimental configuration, but the attainment of a perfect seal over the annular region is essential to the success of the experiments. Figure 1 shows a cross section through the experimental device used to provide the seal. The annular region was sealed against the surface of the rock by the application of a normal load to a confined rubber gasket. A schematic view of the experimental configuration is shown in Figure 2. It consists of a Bosch reaction frame for the sealing loads, a hydraulic cylinder (Miller Fluid HV3-50R2N) with a manual hand pump (Enerpac P391) to apply the sealing load, a load cell to measure the applied load, liquid chromatographic pump (Shimadzu LC-A3), a designed permeameter to allow sealing of the annular patch, a water supply to the Shimadzu pump and a water reservoir to maintain the cuboidal block under water. The test sample was placed in the reservoir which rests on the reaction frame. Special care was taken to ensure that the block is placed at the appropriated position with respect to the reaction frame. The top of the reaction frame can be moved to accommodate specific test locations on the surface of the sample, as shown in Figure 3. This is necessary in order to quantify the spatial distribution of the permeability and will be quite important for the computational analysis of the test. The hand pump allows for the accurate, constant load required to generate sealing to be maintained during a test. Once sealed, the inside annulus cavity was pressurized using a constant flow rate from the liquid chromatographic pump. Filtered water was chosen as the permeating fluid to ensure uniformity in the test procedure. The pressure induced during the attainment of a steady flow rate was monitored using a pressure transducer.

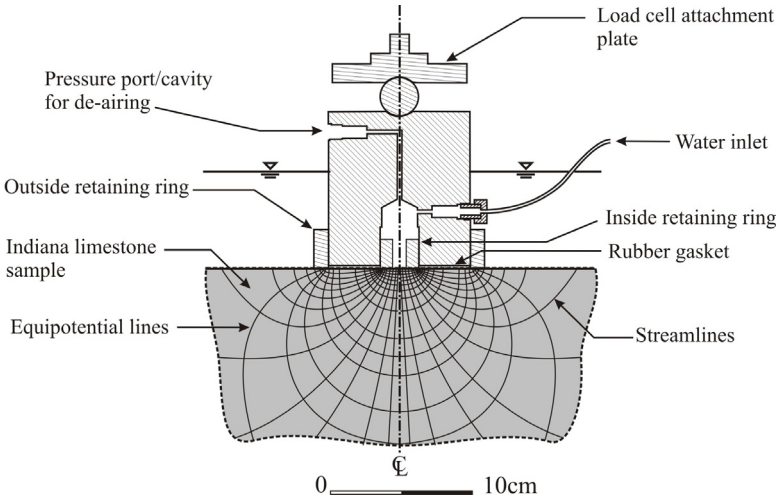

Figure1. A cross sectional view through the permeameter

2.3 *Permeameter*

Figure 1 shows the essential elements of the permeameter, which allows for the physical sealing of the annular region so that impermeable conditions are assured in experiments. Sealing is achieved by applying a load to a gasket made of natural gum rubber (40 Shore A durometer). The gasket (0.3175cm thick) was constructed using die-punches to obtain the proper inside and outside diameter (2.54cm and 10.16cm respectively). The interior to exterior diameter ratio (D_o/D_i) is also referred to as the "tip seal ratio". This value can be arbitrary, but was selected as 4, based on the work of Tartakovsky et al. (2000). The gasket, or sealing annulus, was confined internally and externally by retaining rings to prevent expansion of the gasket in the radial direction, in the plane of the gasket. The process for determining the correct load to obtain a proper seal will be discussed in the forthcoming paper. Once the seal was achieved, the inside cavity was de-aired by flushing the system with water via the pump. To begin the test an appropriate

flow rate was chosen to ensure that conditions conformed to Darcy flow and the internal cavity region was subjected to pressure to initiate flow. The permeameter was equipped with an online pressure transducer that recorded the steady state pressure.

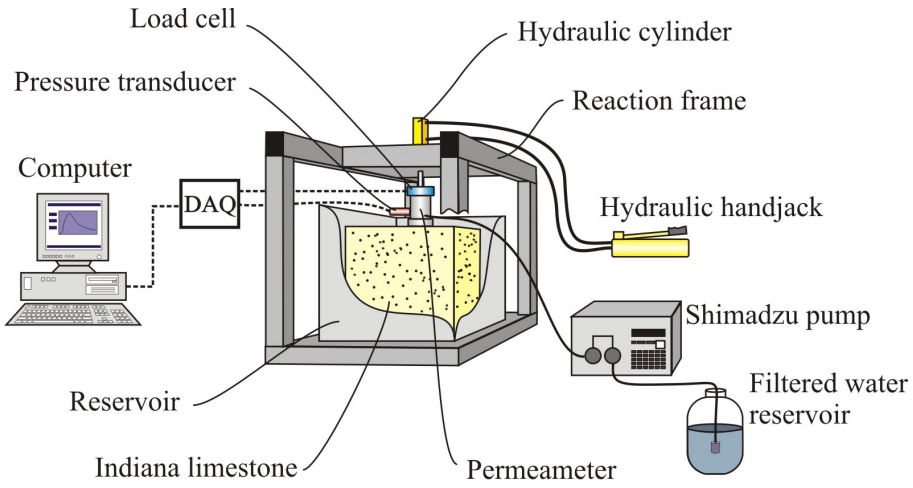

Figure 2. The general arrangement of the laboratory-scale surface permeability test

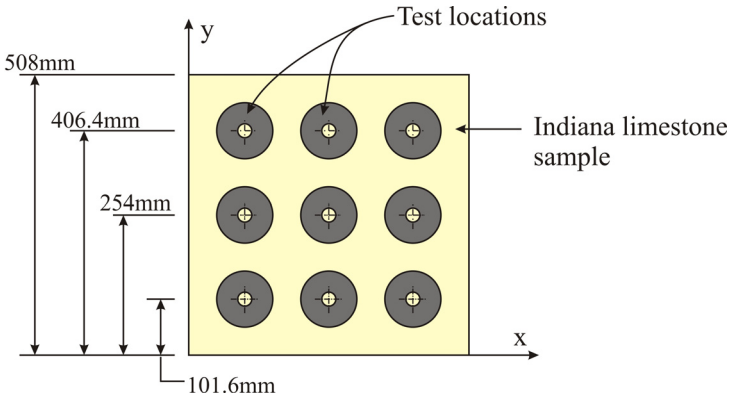

Figure 3. Plan view of test locations (nine total) on a cuboidal Indiana limestone sample. All dimensions given are typical and no two test locations are used at the same time.

2.4 *Sealing Procedure*

It is difficult to determine whether or not a sufficient load has been applied to the gasket to ensure sealing. In these experiments, we adopted the procedure outlined by Tidwell and Wilson (1997) to determine the adequacy of the sealing load. A separate experiment was conducted where the load was varied on the gasket and the permeability measured at the same location (Fig. 4). The observed reduction in permeability is directly related to the gasket deformations that conform to the surface topography of the limestone. This ensures that the fluid cannot migrate through the rock/gasket interface. At gasket compression stresses above 1.4MPa, we observed that the changes in permeability were independent of the compression stresses applied to the gasket (i.e. less than 2%). Since this test is done only once for the entire test program and knowing the sample is relatively homogeneous and with a uniform surface texture, we set the sealing pressure at 1.75MPa for any position on the sample. Triaxial tests reported by Selvadurai and Głowacki (2008)) showed the effects of a confining pressure on the permeability of Indiana limestone and concluded that confining pressures below 5MPa resulted in virtually no al-

teration in permeability; therefore it could be concluded that the loads applied to seal the annulus do not contribute to poroelastic deformations.

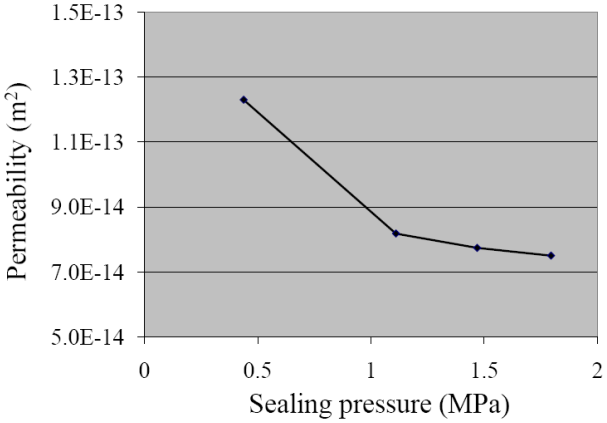

Figure 4. Data showing the relationship between "tip seal" and sealing pressure applied at a single fixed point on the Indiana limestone sample. This reaction is assumed to be caused by the penetration of the gum rubber into the surface contours of the rock.

2.5 *Darcy Flow in Support Volume*

For porous media Hornberger et al. (1998) defined the pore Reynolds number as:

$$\mathrm{Re} = \frac{\rho_f q d}{\mu_f} \tag{1}$$

where Re is the pore Reynolds number, ρ_f is the density of the fluid, q is the specific discharge, d is the mean grain size diameter and μ_f is the dynamic viscosity of the fluid. The Reynolds number defines the flow regime inside the fluid-filled pore space. There are four types of flow regimes found in porous media; we are concerned with Darcy flow (low Reynolds number). A Darcy or seepage flow regime can be characterized as dominated by viscous forces (i.e. laminar flow) and the exact nature of the velocity distribution is determined by local geometry. This type of flow occurs at $Re < 1$. In order to classify what flow was taking place in the limestone block it is necessary to know the mean grain size diameter. This was difficult since the purpose of this experiment was to be non-invasive and the size of the sample makes it very difficult to manipulate. An experiment was created to ensure a Darcy flow regime inside the support volume as outlined below.

While keeping the permeameter in one location and ensuring an adequate sealing pressure, the flow rates into the permeameter were varied and the corresponding steady state pressure observed. Darcy's law has a linear relation between the flow rate (q) and the hydraulic gradient (i.e. the doubling the flow rate should result in the doubling of the steady state pressure)

$$q = -\frac{k}{\mu}\nabla P \tag{2}$$

The experimental results performed with variable flow rates indicate that Darcy flow can be established within the tested block for flow rates lower than 15 ml/min. This limiting flow rate was adhered to in all the experiments.

2.6 *Test Procedure*

A complete record of the experiments will be presented in the companion paper. We report here the results of nearly 45 steady state permeability tests conducted on one face of the block of Indiana Limestone. Tests were conducted at nine locations and the test results interpreted using the computational approach described in the ensuing section. At each location the maximum

and minimum pressures reached during attainment of a steady state flow were recorded. The temperature of the percolating fluid was also recorded for each test since this information will be required for the computational analysis.

3 COMPUTATIONAL METHODS

Since the steady state Darcy flow problem associated with the surface permeability testing deals with a three-dimensional problem, the interpretation of the experimental data is more conveniently accomplished using a computational model of the steady state flow problem. The finite element code COMSOL Multiphysics® was used. Also, since the pressure heads applied to initiate steady flow are substantially greater than the datum head, the formulation of the problem can be in terms of the fluid pressure $p(x, y, z)$. For steady state flow, the partial differential equation governing the pressure $p(x, y, z)$ of the fluid migrating through the porous medium is given by (Bear, 1972; Selvadurai, 2000):

$$\nabla^2 p = 0 \tag{3}$$

where ∇^2 is Laplace's operator. The partial differential equation can be solved by prescribing suitable Dirichlet and Neumann boundary conditions on parts of the boundary of the flow domain. The computational code COMSOL solves the steady flow problem by employing a Galerkin finite element scheme. The computational procedures are well established and since the objective of the computational modelling is to develop the computational estimates for the interpretation of the test data, the relevant results can be obtained in a straightforward manner. The choice of finite element mesh refinement is important to the computational accuracy. To address this issue and to optimize computations, three models were generated that take advantage of symmetry based on the template shown in Figure 3. Boundary conditions were then applied to all surfaces; the central region of the annulus was given the constant velocity (flow rate / area [$m^3/s/m^2$]), the axes of symmetry and the annulus (or "tip seal region") were given Neumann ($\partial p / \partial n = 0$ i.e. impervious) boundary conditions and all other surfaces were assumed to be atmospheric (or $p = 0$). Mesh one, two and three consisted of 165 634, 102 237 and 101 463 elements respectively, densely located around the annulus. A robust solver SPOOLES, provided in COMSOL, was used to generate the steady state solution.

4 RESULTS

Two out of the six cuboidal faces of the Indiana limestone block have been tested to date using the technique described previously. Results vary over the face of the Limestone block (Table 1) but show very good repeatability. Repeatability, seen in Figure 5, was mainly due to the rigorous implementation of the procedure described in the previous sections.

Table 1. Results of surface permeability experiments conducted on face one of a cuboidal sample of Indiana limestone.

Location	Tests/Location	Flow rate	Temperature	Permeability ($*10^{-15}$ m^2)		
		(ml/min)	(^{o}C)	Min	Max	Mean
A	5	3	27.3	23.8	26.0	24.5
B	4	5	27.3	40.3	43.3	41.3
C	4	3.5	27.2	21.9	22.7	22.2
D	5	3	25.7	27.8	29.7	28.9
E	6	10	26.7	38.8	44.1	42.3
F	4	1.5	25.8	12.3	12.7	12.5
G	4	3	25.9	24.3	25.7	24.9
H	6	7.5	26	45.5	51.1	48.1
I	5	1	25.9	7.2	7.8	7.4

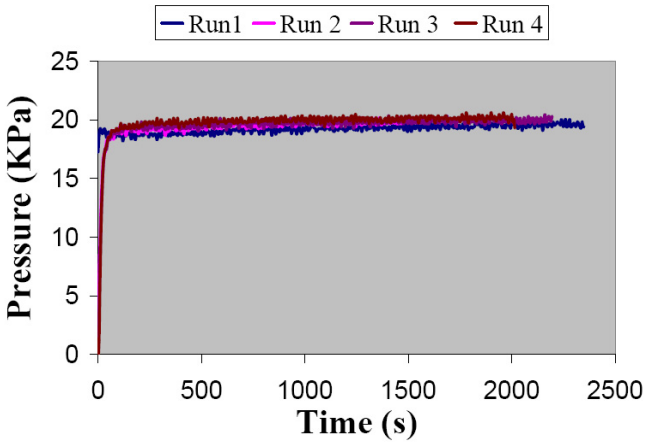

Figure 5. Test results for location F on face 1 of a cuboidal Indiana limestone sample. Tests were preformed over a span of 3 days.

Once the data was collected we were able to infer that the surface of the first face of the limestone block was relatively homogeneous; i.e. there is variability between the average values corresponding to the maximum and minimum permeability values (48.1×10^{-15} m^2 to 7.4×10^{-15} m^2). The average permeability for the face one was 29.4×10^{-15} m^2 with a standard deviation of 11.21×10^{-15} m^2. These are acceptable values and are in the range of the values obtained previously, 16×10^{-15} m^2 for Indiana limestone tested using axial flow tests (Selvadurai and Głowacki, 2008). Previous studies for Indiana limestone also found that values can range from 4×10^{-15} m^2 to 54×10^{-15} m^2 (Churcher et al., 1991). The second face showed the same spatial variability that was shown on the first face. The values of permeability for face two ranged from 97.8×10^{-15} m^2 to 17.2×10^{-15} m^2. This face averaged a permeability of 44.3×10^{-15} m^2 with a standard deviation of 19.1×10^{-15} m^2. Figure 6 shows graphically the spatial distribution of the permeability.

The test data from the series of tests on one face of the Indiana limestone block were compiled to visually show the spatial representation of the permeability over the surface area. This was done using the contour mapping algorithm in MATLAB. It was noted that the minor fluctuations in permeability corresponded to localized strata of the sample, observed through the visual discoloration of the sample in a somewhat "layered" fashion. Figure 7 shows the possible link between the slight changes in limestone composition to the changes in permeability. Although slight aberrations in permeability might occur locally, the representative volume element (RVE) of a sample this size still makes the block relatively homogeneous.

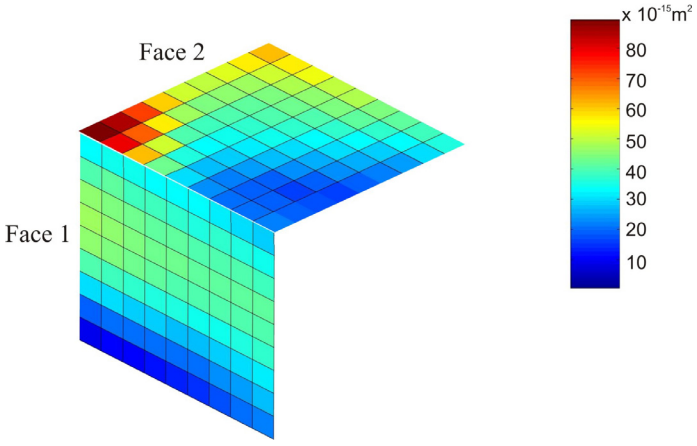

Figure 6. Spatial variation of permeability on the cuboidal limestone block for faces one and two.

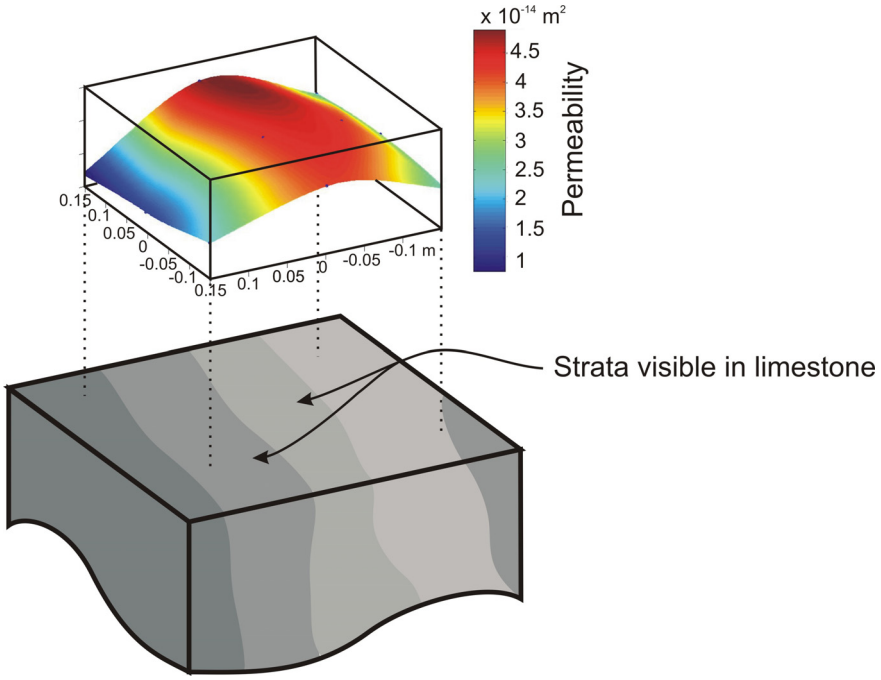

Figure 7. Spatial representation of permeability measured at the surface of a 508mm cuboidal Indiana limestone sample. The possibility of a link between localized strata change and the variation in permeability is suggested in this figure.

5 CONCLUSIONS

Results from experiments conducted on a cuboidal Indiana limestone sample using the surface permeability technique in a laboratory environment show excellent correlation to those obtained using traditional cored cylindrical samples. The average values for face one and two, 29.4×10^{-15} m^2 to 44.3×10^{-15} m^2 respectively, obtained from the surface permeability tests are comparable to those found previously using core samples of the same material (Selvadurai and Głowacki, 2008; Churcher et al., 1991). When performing the surface permeability test the confirmatory experiments (sealing test and Reynolds test) must be carried out to ensure the applicability of the test methodology and the underlying assumptions of the theoretical developments. The non invasive technique proposed in this paper may prove beneficial in situations where coring of the sample is not an option. These tests might take longer and be more costly, but for situations were the overall permeability is needed and the samples cannot be altered by drilling or coring, the surface permeability technique is considered to be a valuable approach. This research investigation will be extended to include the surface permeability measurement of the four remaining sides and an interpretation of the permeability of the cuboidal block in terms of global statistical estimates.

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