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Challenges from North to South
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The Effect of Flow Reversal on Permeability Measurement in Rocks

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ABSTRACT
This paper presents the results of an experimental evaluation of flow reversal on the alteration of "permeability" in both saturated and unsaturated rock samples subjected to constant flow tests. Cylindrical samples of the rock (Indiana limestone, Stanstead granite and Rudna Sandstone measuring 50 mm in diameter and 20 mm in length) were tested. The samples were epoxy coated on the cylindrical surfaces and capped with acrylic disks, allowing only one-dimensional flow. A constant flow rate was applied at the upstream and the downstream was kept at atmospheric pressure. The inlet fluid pressure response was recorded with time. The samples were tested under different conditions: initially dry, initially saturated and flow reversal all using distilled de-aired water. The pressure response due to flow reversal was noted to be significant and dependent on the rock type. Sandstone was the least influenced by flow reversal, whereas granite and limestone showed an increase in pressure due to flow reversal.

RÉSUMÉ
Cet article présente les résultats d'une étude expérimentale sur la "perméabilité" dans la roche soumise à des tests à débit constant. Des échantillons de roche cylindriques mesurant 50 mm de diamètre et 20 mm de longueur de calcaire d'Indiana, de granite de Stanstead et du grès de Rudna ont été analysés. Les échantillons ont été scellés sur les surfaces latérales avec de l'époxy et les surfaces de chaque extrémité avec des disques en acryliques, permettant ainsi un écoulement unidirectionnel. Un écoulement constant a été appliqué à l'amont et la pression atmosphérique est maintenue à l'aval. La pression d'écoulement résultante à l'amont a été enregistrée. Les échantillons ont été testés dans des conditions différentes: initialement sec, initialement saturé et en inversant la direction d'écoulement, le tout en utilisant de l'eau distillée et désaérée. La variation de la pression causée par l'inversion de l'écoulement était significative et dépendante du type de roche. Le grès a été moins influencé par l'inversion de la direction d'écoulement, tandis que le granite et le calcaire ont montré une augmentation de la pression due à cette inversion.

1 INTRODUCTION

The study of rock permeability is an important topic in many fields such as: gas sequestration (Bachu et al. 2000), groundwater flow (Bouwer, 2000), resource extraction (Boyer et al., 2006), nuclear waste disposal (Selvadurai and Nguyen, 1996), waste water disposal (Tsang et al., 2008) and earthquake triggering (Keranen et al. 2014).

The estimation of permeability of rocks is a difficult task that requires an understanding of many factors that can influence the permeability. This study investigates the role of flow reversal during a steady state experiment on the inflow pressure in three different rocks (Indiana Limestone, Rudna Sandstone and Stanstead Granite). The investigation is aimed at determining if there is a significant change in permeability when the steady state flow direction is reversed (i.e. once the inflow pressure has stabilized) and whether any differences can be attributed to the rock type. The topic is especially of interest in the field of deep injection wells and oil production wells, as their operation generates waste water as a by-product. Hence, arises the need for injection wells also called brine disposal wells, and are officially known as class II underground injection wells to be subjected to flow reversal. They can take any fluid related to oil and gas drilling, including frack waste water (Enviromental Protection Agency, 2015).

In general, there is no direct research that the authors were able to find that examined the influence of flow reversal on the estimation of permeability in rocks; however, there are studies that have dealt with this as a secondary topic of the main research (some notable examples are mentioned in the text the follows).

The effect of flow reversal during permeability testing was noted by Gloyan and Reynolds (1961) when testing rock salt. They concluded that clogging was causing a constant drop in permeability and when they reversed the flow, the permeability reverted to its minimum initial value. In the experimental work by Wright et al. (2002) on limestones from Australia near Bolivar on the Northern Adelaide Plains, there is a section dealing with the effects of reversing flow direction on the hydraulic conductivities (k_x and k_y). The authors state that there is no significant change in hydraulic conductivities in comparison with variations between cores. However, they mention that for one test there was a slight increase in hydraulic conductivity upon flow reversal.

2 METHODOLOGY

2.1 Theoretical background

The experiments were organized to conduct steady state constant flow tests. In such a configuration (see Figure 1) Darcy's Law can be used to estimate the permeability of a porous medium; it is essential that the

entire pore space is fully saturated and that the flow velocities at the pore scale are within the limits that ensure laminar flow (i.e. should satisfy a criterion based on the Reynolds Number) (see Selvadurai and Selvadurai, 2010).
Since the experimental investigations presented in this paper were restricted to one-dimensional flow in the axial direction, permeability K (L^2) can be estimated from the relationship:

$$K = \frac{Q\mu L}{A(p_i - p_o)} \quad [1]$$

where Q is the flow rate (L^3/T), μ is the dynamic viscosity of water (M/TL), L is the length of the sample (L), A is the cross-sectional area of the sample perpendicular to the nominal flow direction (L^2) and the fluid pressures are prescribed at the inlet p_i in (M/T^2L) and the outlet p_o in (M/T^2L).

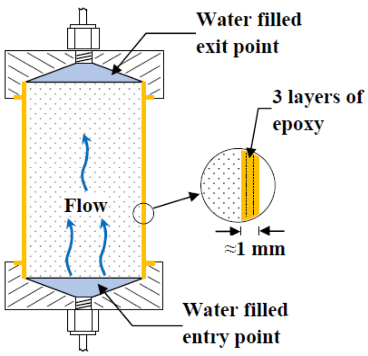

Figure 1: Typical cross-section of the rock sample and the acrylic disks which were tapered in order to funnel the water as well as an NPT-threaded opening for connections.

2.2 Samples

Indiana Limestone (Indiana Limestone Institute of America handbook from 2007, 22th edition), is also known as Salem Limestone and its formation dates back 330 million years to the Paleozoic era. It is a monomineralic rock, mostly composed (97%) of calcium carbonate ($CaCO_3$), a mineral also referred to as calcite.
Stanstead Granite is a rock from Devonian period, fine to coarse-grained rock, found in the Beebe region of the Eastern Townships in Quebec, Canada. The main minerals are clear, sharply defined quartz (25%); feldspar (65%), which are semi-transparent to milky white and biotite in flakes (9%) (Nasseri et al., 2007).
The Rudna Sandstone was obtained from a copper mine in Poland. The sandstone is part of the Permian strata (Suchan, 2001).
In Table 1 the porosity and density of the three rocks are presented. The porosity was determined by initially oven drying the samples followed by vacuum saturation using a venturi pump (Vaccon JS-200-AA6) and calculating the mass difference.

Table 1: Characteristics of tested rocks

	Stanstead Granite	Rudna Sandstone	Indiana Limestone	Units
Porosity	0.002	0.023	0.161	-
Density	2607	2322	2210	kg/m^3

2.3 Sample preparation

Samples of Indiana Limestone were obtained from slabs (91.5x46.0x33.0cm) supplied by Les Carrières Ducharme inc., a local Montreal, Quebec supplier. Using a large drill press core samples of 50 mm in diameter by 330 mm in height were obtained. The final samples were then cut to size using a circular diamond saw and, if required, turned using a lathe. A similar procedure and process was used to obtain the sandstone and granite samples. The samples had a finished diameter 50.1±0.1mm and an average height of 20.5 mm. Unless otherwise specified, the surfaces of all samples were cleaned with tap water using a stainless steel brush to remove any debris produced during the coring and machining process. The samples were then air dried for 24hrs before the application of the epoxy layers (Bondo Fibreglass Resin). All epoxy was applied manually using a brush and each layer was allowed to dry for 24 hrs before applying a subsequent layer. Typically 3 layers of epoxy were applied to the surface, followed by the gluing of the top and bottom acrylic caps (see Figures 1 and 2).

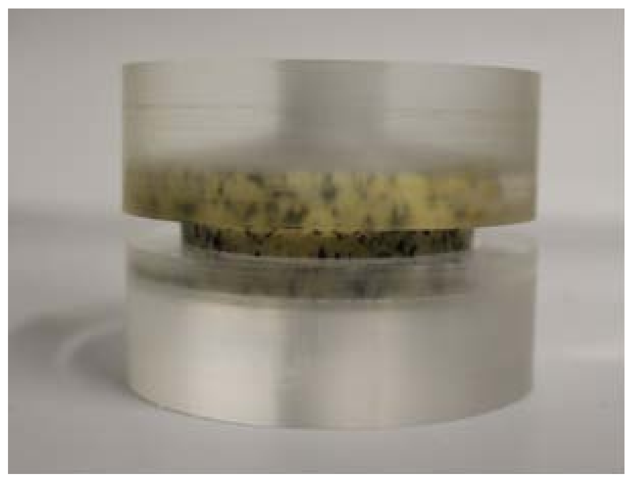

Figure 2: Stanstead granite sample epoxied and capped with two acrylic disks.

2.4 Experimental setup

An epoxied sample, in either an initally saturated or initially dry condition, was connected to the in-line pump (Jasco PU-2085 Semi-micro HPLC Pump) using Swagelok fittings. The inflow pressure was monitored via a pressure transducer (Honneywell TJE 300PSI) and the temperature of inflowing water was measured with a type K thermocouple. The data was collected using a data acquisition system (InstruNet or Dataforth) and stored on a computer. The flow rates were adjusted according to the rock type (i.e. 0.1ml/min for limestone; 0.01ml/min for granite and sandstone) in order to avoid detachment of the epoxy coating. Typically, the inflow pressure was maintained at 50% of the epoxy delamination pressure, which was established by testing several samples, of different sizes and types, and running a step by step increase in inflow pressure until a pressure drop was recorded and leakage observed.

2.5 Experimental procedure

A total of 5 samples (two of granite and sandstone, one initially dry of Indiana Limestone) were tested. Saturation of the samples was accomplished by submerging them in distilled water and applying a negative pressure using a venturi vacuum pump (see Figure 3). Samples were subjected to a constant flow and the pressure monitored until it stabilized. Once a stable inflow pressure was observed the flow ports were switched (i.e. the inflow became the outflow) and the process was repeated in order to determine the reversed inflow pressure. In order to dissipate the created pressure gradient, within the samples, a period of 5 hrs was allocated for each sample. This 5 hours period was confirmed to be sufficient by monitoring the inflow pressure during the dissipation period. All samples were tested using distilled de-aired water. The de-aired water was obtained by using a degasser (Jasco DG-2080-54); this apparatus effectively reduces the dissolved oxygen content in the water from 8ppm to 2ppm. The use of oxygen-deprived water during testing permits the water to absorb air bubbles that are occluding the interconnected flow paths, helping in attaining a saturated pore space; as well as obtaining a more accurate pump performance (Shimadzu Corporation, 1991).

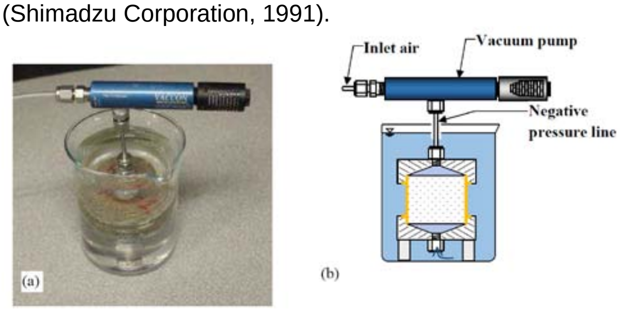

Figure 3: (a) The saturation setup; the sample is subjected to negative pressure (-81 kPa) at the top using a venturi vacuum pump, (b) a typical cross-section of the sample submerged in water.

The outflow rate was not constantly measured during the tests due the relatively low flow rates, such measurement would be difficult to conduct and inaccurate, considering the equipment at hand, for thorough analysis. However, sporadically during the experiment, when the inlet pressure was stable, a short measurement (validation) of the outflow rate was made (by weighting the mass of outflow water) in order to confirm that no leakage was occurring and that the pump was still functioning properly.

3 RESULTS

The results of inflow pressure vs time for all three rocks types are presented in the Figures 4 to 6.

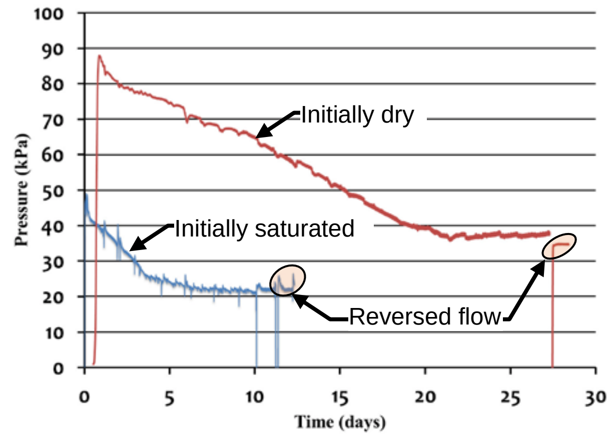

Figure 4: Inflow pressure vs time for saturated (blue) and dry (red) samples of Rudna Sandstone.

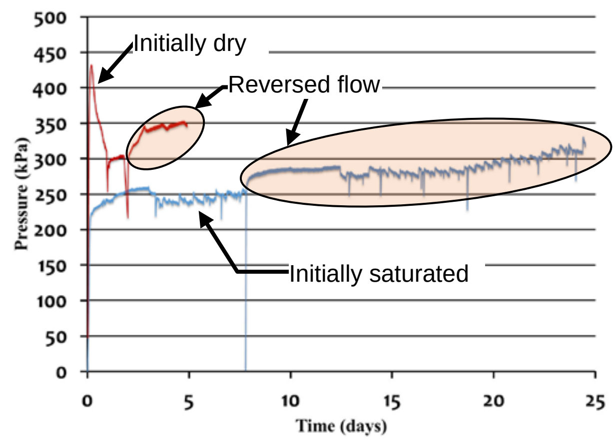

Figure 5: Inflow pressure vs time for saturated (blue) and dry (red) samples of Stanstead Granite.

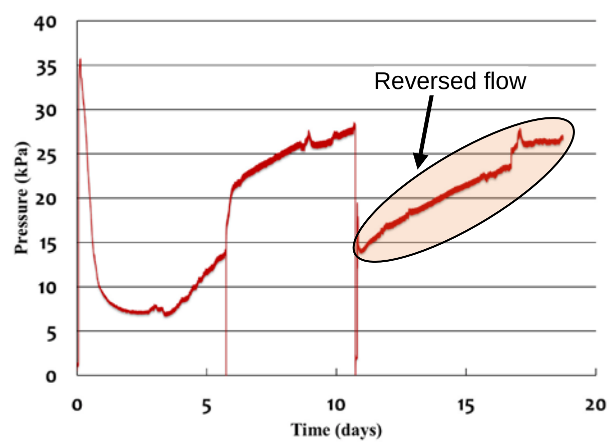

Figure 6: Inflow pressure vs time for initially dry sample of Indiana Limestone.

4 DISCUSSION

The initially dry sample of Rudna Sandstone took about 3 weeks of continuous influx of water to reach a steady state pressure, before any reversal of flow could be done. Comparing this 3 weeks period to a period of 4 days needed to saturate the same rock sample using the saturation setup in Figure 3, points to the fact that vaccum saturation and saturation by continuous flow will have different inflow pressure responses. Hence, if it assumed that the sample should be saturated within the first week of testing (based on the amount of influx), then the question is why the inflow pressure has not stabilized? The reason could be that vacuum saturation method pulls the occluding air bubbles out from the flow paths whereas the continuous flow fill up the conductive flow paths and pushes air bubbles, creating large pockets of air that effectively partially block a particular flow path. The work by Makhnenko and Labuz (2013) investigates the influence of air entrapment in a Berea Sandstone when trying to estimate the Skempton's B coefficient. The authors experiment with different methods (i.e. by applying a gradually increasing back pressure to the fluid) in order for the air-water mixture to behave as a fluid. In a similar case during the triaxial testing of soils, where air entrapment was an issue for Mendes et al. (2012), the authors concluded that a certain air percentage will remain in the sample; hence to overcome this issue they forced the air to dissolve into water by applying a back pressure to the fluid, however this process was time consuming and with reservations. The Rudna Sandstone results show the least significant change in inflow pressure response when the flow was reversed. The inflow pressure results for Stanstead Granite, see Figure 5, show a quick stabilizing inlet pressure; however this pressure tends to slowly rise with time. This rise was correlated to a general increase in water temperature (the room temperature increased). The erratic behaviour shown on Figure 6 for the Indiana Limestone can in part be due to the calcium carbonate reaction with the inflowing water and the creation of precipitate that could partially obstruct the flow paths. Research by Zhang and Spiers (2005) on intergranular pressure solution involving calcite and the effects of pore fluid chemistry on the mechanical compaction behaviour of granular calcite, revealed porosity reduction and precipitation of calcite on pore walls. Furthermore, the particulates can influence the pressure history during permeability testing of Indiana Limestone as seen in Figure 7, where a dry sample, covered with particulates from the machining, was tested and a large drop in pressure was observed while the pressure was rising. Chitty et al. (1994) hypothesized that the Salem Limestone specimens tested, in order to estimate the permeability, were being somehow altered by the testing procedure, where the pores of the intact material were being clogged by particles either originating internally or externally.

Initial higher permeabilities are achieved in Stanstead Granites and Rudna Sandstones that were initially saturated, however over the testing period time the permeabilities tend to converge towards the same value (see Figures 8 and 9). The test results show that there is a significant reduction in inlet pressures when samples of granite and sandstone are initially saturated. The initial peak threshold pressure, in initially dry samples, has an important consideration for the design of testing apparatus and data acquisitions, which could potentially be damaged by these high threshold pressures developed during the initial stages of testing dry samples.

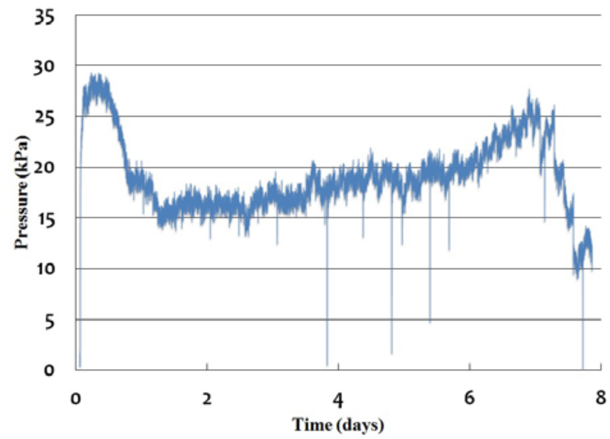

Figure 7: Inflow pressure vs. time; initially dry sample of Indiana limestone (ILH1SP1, 49 mm in diam. x 20.0 mm in height) was not cleaned after machining (on the circular planes).

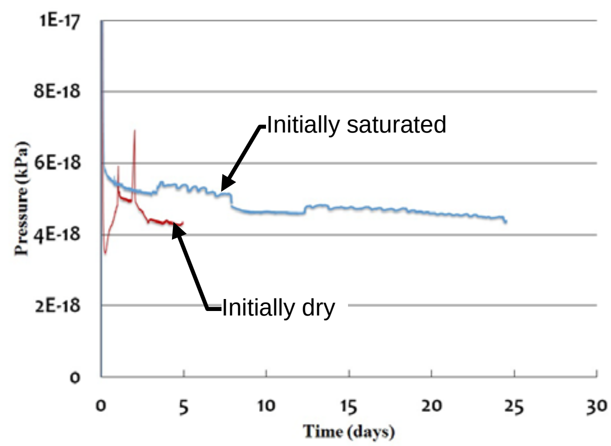

Figure 8: Permeability vs time, Stanstead Granite samples.

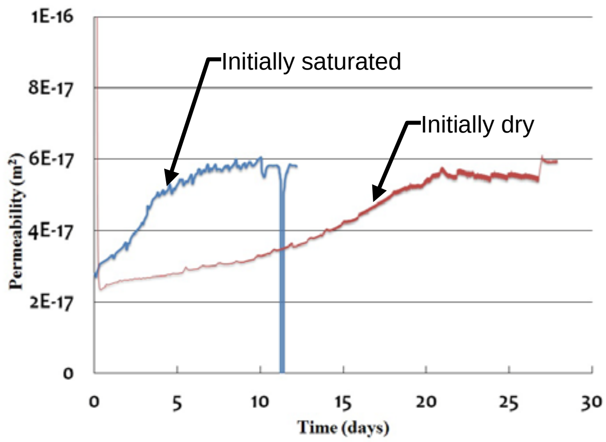

Figure 9: Permeability vs time, Rudna Sandstone samples.

5 CONCLUSIONS

Based on the results presented it can be concluded that reversing the flow has an influence on the inflow pressure response during constant flow steady state experiments. The inflow pressure changes (-4% for sandstones, +12% for granites) indicate that there is some particulate movement inside the flow channels even for a sample of 20 mm in height by 50 mm in diameter. The results show that for Rudna Sandstone the flow reversal has a very limited effect on the inflow pressure as opposed to a more chemically dissolvable rock such as Indiana Limestone in which inflow pressure was unstable. The Indiana Limestone showed the most unstable pressure response during long term testing. As an organic rock, limestones are known to exhibit very unpredictable results during permeability testing, as shown by Bulnes and Fitting (1945). In all rock types, the initially dry samples give a more significant inflow pressure drop than the initially saturated samples. An extension of this experimental investigation can be the measurement of the outflow and inflow rates with highly accurate devices; such results could, for example, provide a better understanding as to the more exact time when the stead state conditions occur.

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