PERMEABILITY MEASUREMENTS FROM A POTENTIAL DEEP GEOLOGICAL REPOSITORY SITE

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The Bruce nuclear site in Canada has been proposed to host a Deep Geologic Repository (DGR) for Low and Intermediate Level Radioactive Waste (L&ILW). The repository would be constructed within a low permeability, argillaceous limestone, the Upper Ordovician age Cobourg Formation. Here, we present the results of two steady-state laboratory hydraulic conductivity tests performed to measure the intrinsic permeability of rock core samples from the Cobourg and overlying Queenston shale formations; both samples were measured under an isotropic confining pressure using a constant head approach. Pump pressures and volumes were recorded for upstream and downstream pumps, throughout testing. The resulting hydraulic inflow and outflow rates were measured for each sample under two different pressure gradients, yielding exceptionally low values of permeability (on the order of 10^{-22} m^2 or 0.1 nD). These data provide further evidence of the applicability of existing steady-state experimental methods to obtain reliable estimates of extremely low permeabilities from rock core samples under re-established in-situ stress conditions. The exceptionally low permeability of these formations, consistent with in-situ testing and formation scale estimates obtained during the site characterisation program, along with their low porosities, renders them an effective barrier to hydraulic flow for the purpose of geological isolation.

I. INTRODUCTION

The Bruce nuclear site, situated on the eastern flank of the Palaeozoic age intracratonic Michigan Basin, near Tiverton, Ontario, Canada has been proposed by Ontario Power Generation (OPG) to host a Deep Geologic Repository (DGR) for its Low and Intermediate Level Radioactive Waste (L&ILW). The DGR would be built adjacent to the Western Waste Management Facility, which currently manages the interim storage of L&ILW waste from the Bruce, Pickering and Darlington nuclear power stations. The DGR concept includes multiple barriers to contain and isolate the L&ILW, key amongst them are Upper Ordovician age sediments, at a depth of 680 m, within the 840 m thick sedimentary sequence underlying the site. At this depth, the repository would be constructed within the low permeability Cobourg Formation that is overlain by upper Ordovician shales. These shale cap rocks comprise, in descending order, the Queenston, the Georgian Bay and the Blue Mountain formations.

In this study, we present the results of two steady-state laboratory hydraulic conductivity tests performed to estimate the intrinsic permeability of samples from the Cobourg and Queenston Formations. The Cobourg was selected because it has been identified as the potential repository host formation; the Queenston Formation was chosen for testing because it forms part of the overlying geology that will act as a cap rock to the repository and an additional barrier to fluid migration.

II. MATERIALS AND METHODS

II.A. Site Geology

The Bruce nuclear site is located on the eastern side of Lake Huron in Southern Ontario. Ontario Power Generation is proposing to build a DGR at this site, situated within the Cobourg Formation at a depth of 680 m below ground surface. The regional geology comprises mostly marine sediments of Cambrian to Devonian age[1,2] that strike approximately NNW-SSE and dip gently to the SW.

The Cambrian rocks are found in the eastern part of Ontario and rarely crop out; in the subsurface Cambrian sandstones and dolostones overlie the altered Precambrian surface[2,3]. The subsequent Ordovician sediments are a sequence of calcareous shales and limestones, laid down during a large marine transgression that occurred in the Mid-Ordovician[4,5] (Figure 1). The repository is planned to be constructed within the Cobourg Formation, a massive argillaceous limestone with a thickness of 27 m that underlies a number of formations of indurated illitic shales totaling about 200 m thickness. At the Bruce nuclear site, these include the Queenston Formation (70 m thick), the Georgian Bay Formation (90 m thick) and the Blue Mountain Formation (45 m thick), which are Upper Ordovician age (Figure 1). Whole rock analyses conducted by scanning electron microscope (SEM) and X-Ray Diffraction (XRD) along with information regarding clay mineralogy and fractionalogy are presented by Intera[2] using cores from 4 deep vertical boreholes

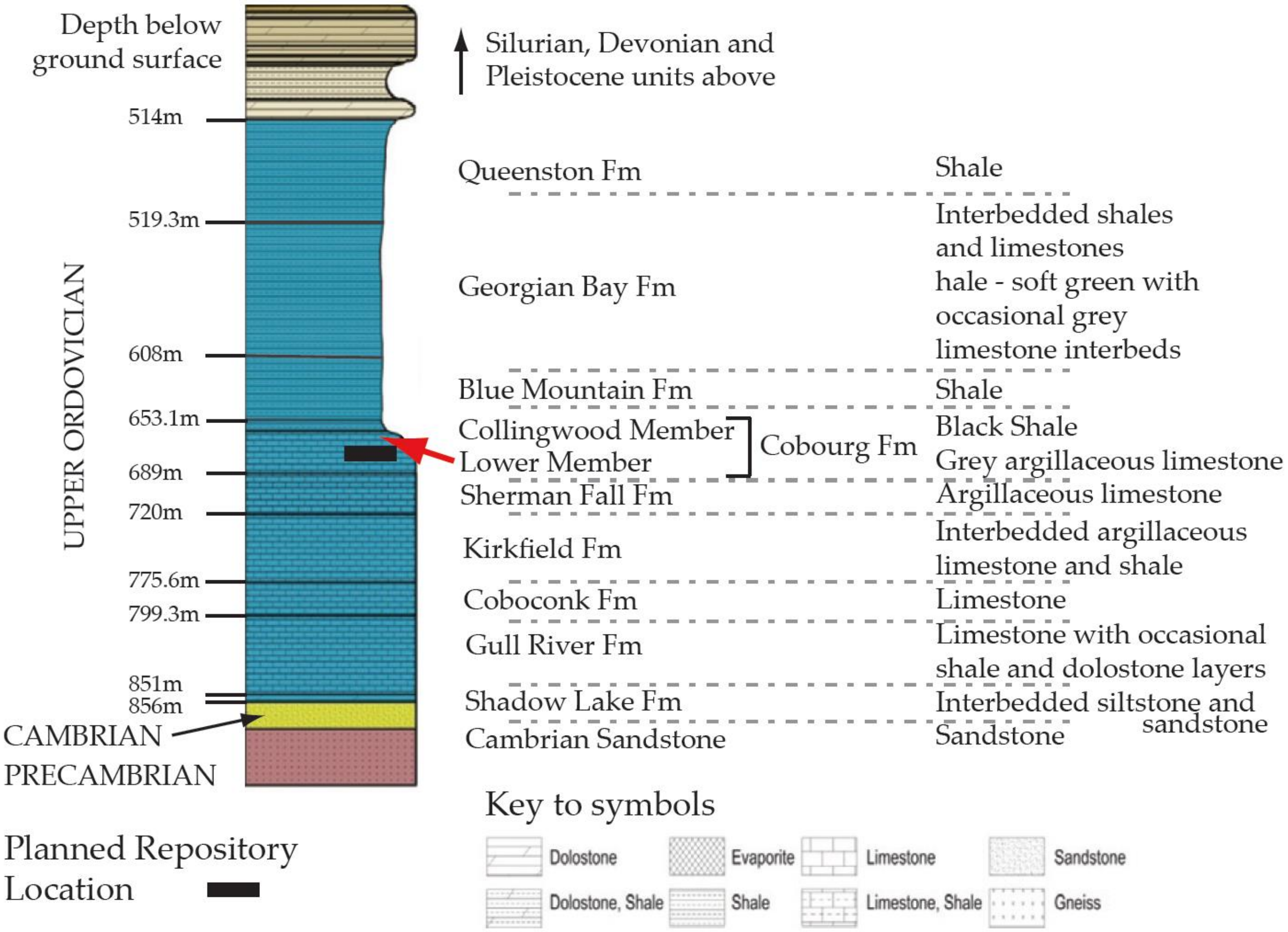

Fig. 1. Stratigraphic sequence of the rocks from Southern Ontario. The depths are metres below ground surface (mBGS). Originally, only the Queenston, Georgian Bay and Blue Mountain formations were classified as the Upper Ordovician, whilst the limestones comprising the lower half of the Upper Ordovician sediments were classified as the Middle Ordovician. The chronostratigraphic divisions have since been revised and are now as presented above. Not to scale. (Adapted from Brookfield[6] and Intera[2]).

(DGR-1 to DGR-4) and 2 deep inclined boreholes (DGR-5 and 6). The analyses give average values of the samples constituents, and each unit is described in detail.

Measurements of hydraulic conductivity and permeability for two samples from the Bruce nuclear site have been made during this study. The samples are from the Upper Ordovician Queenston Formation and the Cobourg Formation (from borehole DGR-3). The Queenston Formation is calcareous and dolomitised[2]; whilst the Cobourg Formation (part of the Trenton Group Carbonates) is mostly calcite rather than dolomite and contain comparatively little quartz. The Queenston Formation can be subdivided into two groups, an abundant red-maroon shale and a grey-green shale that form layers and lenses within the red shale. At the location of the DGR, the green shale contains interbedded fossiliferous limestone[2,6]. The Cobourg Formation comprises an upper (Collingwood) and lower (Cobourg) member; the Collingwood is an organic rich calcareous shale that is dark grey to black in colour and contains thin interbedded limestone layers. The Cobourg Member is a fossiliferous argillaceous limestone that is grey-blue to grey-brown in colour and has a low average porosity (1.9%)[7]. It has a high uniaxial compressive strength value (average 113 MPa) that renders it mechanically stable[7] and it is intended as the host rock for the DGR[2].

II.B. Sample Preparation

The laboratory samples were prepared from preserved rock core (73 mm dia.) from the Queenston and Cobourg Formations, drilled and stored in June 2008. The Queenston and Cobourg preserved cores came from borehole DGR-3 at depths of 519.65 m and 674.34 m, respectively. All core samples that were shipped offsite for analyses or placed in archive were preserved by placing the core sub-sample in a polyethylene (PE) bag, flushing with nitrogen, vacuum sealing the PE bags, and vacuum sealing in aluminum-PE-nylon bags. All efforts were made to begin breaking, photographing and preserving of core within 15 minutes of core retrieval and to complete these steps within 30 minutes of core retrieval from the borehole.

The samples were sub-cored at the British Geological Survey to fit the experimental apparatus. On opening, the Queenston Formation core fractured into 5 pieces, some of which were not large enough to prepare a sample for testing. Piece 4 was used to prepare the sample for testing. Piece 3, just above Piece 4 in the succession, was used to obtain the grain density and moisture content. The Cobourg Formation core was intact and the top 6 cm was removed with a circular rock saw to make the test sample. Offcuts from this process were used to obtain the Cobourg's grain density and moisture content. The end faces and circumference of the samples were finished using a machine lathe to give dimensions of 50 mm length and 50 mm diameter. The preparation was conducted under dry conditions to prevent contamination of the samples, and when not being used, they were kept in vacuum-sealed bags to minimise moisture loss. The samples were produced parallel to the core axis and perpendicular to bedding structure. The samples were X-rayed prior to testing to confirm their interior structure, and their weight and dimensions were recorded (Table I). The Queenston Formation exhibited distinct bedding features and a number of natural and induced fractures. The Cobourg Formation was bioturbated (Figure 2). Upon test termination, the post-test Queenston and Cobourg samples were dried for 25 and 17 days respectively at 105°C, until no more moisture was lost from the sample. This allowed the calculation of the moisture content from the wet and dry weights and the dry density from the dry weight and initial volume.

TABLE I. Sample dimensions, geotechnical properties and stress conditions experienced by the samples during the testing. The parameters given should be considered as estimates because of the sensitivity of the calculated values to the very low porosity and moisture content of these materials.

		Queenston Formation	Cobourg Formation
Rock Type		Upper Ordovician Shale	Upper Ordovician Limestone
Sample Length (mm)	Pre-test	42.38	48.54
Sample Diameter (mm)	Pre-test	50.25	49.08
Sample Weight (g)	Pre-test	226.74	259.41
Bulk Density[a] (mg/m^3)		2.70	2.69
Dry Density[b] (mg/m^3)		2.68	2.67
Grain Density[c] (mg/m^3)		2.76	2.71
Moisture Content[b]		0.73 %	0.78 %
Effective Porosity		5.05 %	1.08 %
Confining Stress		13 MPa	13 MPa
Differential pressure	Hydration	0 MPa	0 MPa
	Stage 1	4 MPa	4 MPa
	Stage 2	6 MPa	6 MPa
Effective Stress	Hydration	8 MPa	8 MPa
	Stage 1	6 MPa	6 MPa
	Stage 2	5 MPa	5 MPa

[a] bulk density was calculated from the sample's pre-test wet mass and initial volume. [b] dry density and moisture content were derived from the sample's dried weight and initial volume. [c] grain density was measured from an offcut.

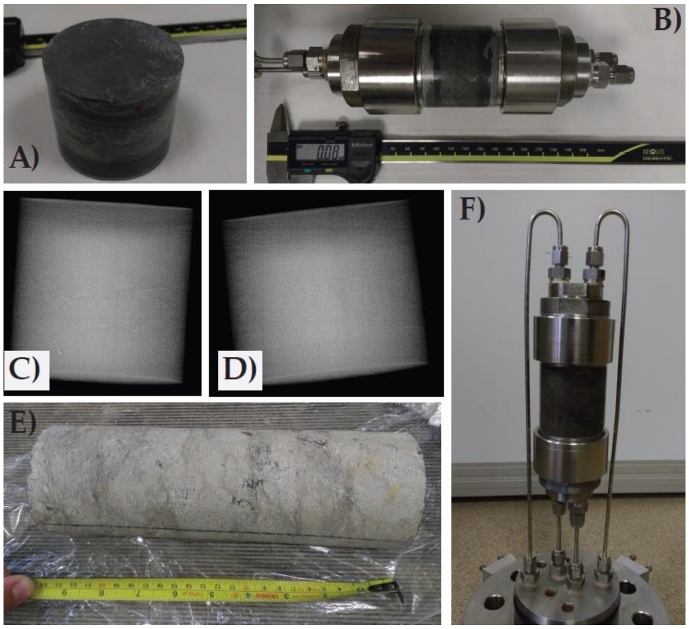

Fig. 2. Experimental apparatus for the tests. A) The Queenston Formation sample. B) The sample assembly including the Queenston sample. C) and D) X-Ray radiographs of the Queenston Formation and Cobourg Formation samples, respectively. E) The Cobourg Formation sample. F) The sample assembly with the Cobourg Formation sample attached to the base of the pressure vessel lid with stainless steel tubing.

II.C. Test Fluid Composition

A synthetic saline groundwater solution was used as the experimental fluid in the injection and backpressure pumps; the same fluid was used for both experiments. This solution had a salinity of 3.8 M and was produced to match the groundwater chemistry within the sedimentary sequence[2]. The groundwater in the DGR-3 borehole (from which the samples used in this study were derived) has been measured at 3 depths in the succession[2]: the Salina

TABLE II. Major element cation concentrations in the synthetic saline groundwater solution used in the experiments.

Element	Na	Cl[a]	Mg	Ca	K	S
Concentration (ppm)	88238	136074	7591	6224	4830	559

[a]Cl concentration is calculated based on the assumption that all of the Na is present as NaCl.

Upper (338-342 m), Guelph (387-393 m) and Cambrian (852-869 m) formations. The Guelph Formation was closest to the target depth and the major element chemistry from this analysis was used to generate the synthetic solution used in this study. Due to the differences between laboratory and *in-situ* conditions, the solution reached calcium saturation before the correct concentration of calcium or any of the strontium had been added. The solution was left on a magnetic stirrer for 2 weeks and its chemistry was then analysed prior to use in the experiments. The major element cation chemistry of the synthetic saline solution is given in Table II.

II.D. Method – Isotropic Confining Pressure Testing

The permeability of both samples was measured under an isotropic confining pressure using the constant head approach outlined in Harrington and Horseman[8]. The samples were isolated between two custom-built steel end filters and platens and sealed within a Teflon sheath that was heat-shrunk against the sample. This prevented the ingress of confining fluid into the injection and backpressure system. The assembly was then suspended from the lid of a single-closure pressure vessel, within the confining fluid which was de-ionised water (Figure 2). All air was removed from the apparatus before the tubing was connected between the pumps and the vessel lid. Each of the pumps was calibrated before the experiment. Volumetric flow rates were monitored using a pair of high precision syringe pumps operated from a single digital control unit. Movement of the pump piston is controlled by a micro-processor which continuously monitors and adjusts the rate of rotation of an optically encoded disc (graduated in segments equivalent to a change in volume of <16.6 nL) using a DC-motor connected to the piston assembly via a geared worm drive. This allows each pump to operate in either constant pressure or constant flow modes and provides an accurate measure of flow. The injection and backpressure pumps were calibrated between 0 and 12 MPa at 20°C, whilst the confining was calibrated to 14 MPa. The pressure vessel itself was not calibrated for its compliance under pressure because it is run at a constant pressure and therefore has no bearing on the results of the study.

The samples were hydrated until the injection and backpressure pump volumes had reached an equilibrium; the Queenston Formation sample was hydrated for 28 days and the Cobourg Formation sample for 20 days. Hydration was conducted with the synthetic saline solution at a confining pressure of 13 MPa and an effective stress of 8 MPa (Table I). Pump pressures and volumes were recorded for upstream and downstream pumps, throughout testing at a logging rate of 60 s for the Queenston sample and 120 s for the Cobourg sample. The rate was reduced because 60 s was deemed to be unnecessarily fast. The resulting hydraulic inflow and outflow rates were measured for each sample under two different pressure gradients; the backpressure was maintained at 5 MPa throughout the test whilst the injection pressure was first set to 9 MPa and then increased to 11 MPa.

III. RESULTS

Using Darcy's Law, the hydraulic conductivity (K) and intrinsic permeability (κ) for both samples were calculated from

$$\mathrm{K} = \frac{Q\,L\,\rho\,g}{\Delta P\,A} \qquad (1)$$

and

$$\kappa = \frac{Q\,\mu\,L}{\Delta P\,A}. \qquad (2)$$

Therefore:

$$\kappa = \frac{\mathrm{K}\,\mu}{\rho\,g} \qquad (3)$$

where Q is the flow rate, μ and ρ are the fluid viscosity and density respectively, L is the sample length, A is the sample cross sectional area, g is gravitational acceleration and ΔP is the differential pressure across the sample. Measured inflows and outflows for both pressure gradients yielded similar values of permeability, which were exceptionally low for both samples; values were on the order of 10^{-22} m^{2} or 0.1 nD (Table III).

TABLE III. Measured permeabilities and hydraulic conductivities for the Queenston and Cobourg Formation samples at both 6 MPa and 5 MPa effective stress.

	Effective Stress	Queenston Formation	Cobourg Formation
Inflow Permeability	6 MPa	1.7E^{-22} m^{2}	1.7E^{-22} m^{2}
	5 MPa	5.6E^{-22} m^{2}	1.9E^{-22} m^{2}
Outflow Permeability	6 MPa	1.2E^{-22} m^{2}	1.5E^{-22} m^{2}
	5 MPa	2.0E^{-22} m^{2}	2.0E^{-22} m^{2}
Average Permeability	6 MPa	1.5E^{-22} m^{2}	1.6E^{-22} m^{2}
	5 MPa	3.8E^{-22} m^{2}	1.9E^{-22} m^{2}
Inflow Hydraulic Conductivity	6 MPa	1.2E^{-15} m s^{-1}	1.2E^{-15} m s^{-1}
	5 MPa	4.0E^{-15} m s^{-1}	1.3E^{-15} m s^{-1}
Outflow Hydraulic Conductivity	6 MPa	8.5E^{-16} m s^{-1}	1.1E^{-15} m s^{-1}
	5 MPa	1.4E^{-15} m s^{-1}	1.4E^{-15} m s^{-1}
Average Hydraulic Conductivity	6 MPa	1.0E^{-15} m s^{-1}	1.2E^{-15} m s^{-1}
	5 MPa	2.7E^{-15} m s^{-1}	1.4E^{-15} m s^{-1}

III.A. Queenston Formation

In the Queenston Formation test, the fluid in the monitored injection and backpressure systems showed a constant volume change with time for both hydraulic gradients, after a short period where steady-state was achieved (Figure 3). The measured flow rates for both effective stresses were used to calculate the hydraulic conductivity and intrinsic permeability of the sample. The permeabilities calculated at the lower effective stress (and higher hydraulic gradient) were higher than for the higher effective stress (and lower hydraulic gradient) (Table III), with the inflow measurements showing a greater difference between the two effective stresses. After 80 hours at the lower effective stress, the injection pump volume change dropped off very rapidly suggesting that at this point the injection side of the sample assembly developed a significant leak. Closer inspection of the mass balance from both test stages indicated a very small background leak from the injection system existed from the start of testing, but was more obvious at the higher injection pressure. The experiment was then terminated.

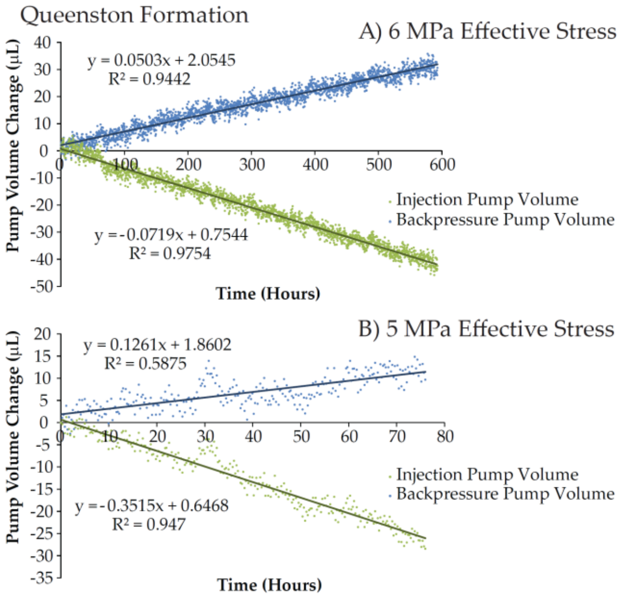

Fig. 3. Volume change (μL) against time (hours) in the Queenston Formation sample for an effective stress of A) 6 MPa (4 MPa differential pressure) and B) 5 MPa (6 MPa differential pressure).

III.B. Cobourg Formation

Like the Queenston Formation test, the fluid in the injection and backpressure systems of the Cobourg Formation test also showed a constant volume change with time for both hydraulic gradients. Similarly, a short period of time was required for the volume change to reach a steady-state, especially for the 5 MPa effective stress stage (Figure 4). As with the Queenston Formation sample, the hydraulic conductivity and intrinsic permeability were calculated from the measured flow rates for both effective stresses. The lower effective stress always gave higher values of hydraulic conductivity and intrinsic permeability than those obtained during the higher effective stress stage (Table III). As with the Queenston Formation data, the measurements derived from the inflow showed a greater variation with effective stress than the outflow measurements. Small step changes in the fluid volume observed in the data (Figure 4A) from the injection pump can be attributed to small fluctuations in the ambient temperature. The experiments were performed in a temperature controlled room which was maintained at 20°C ±0.2°C.

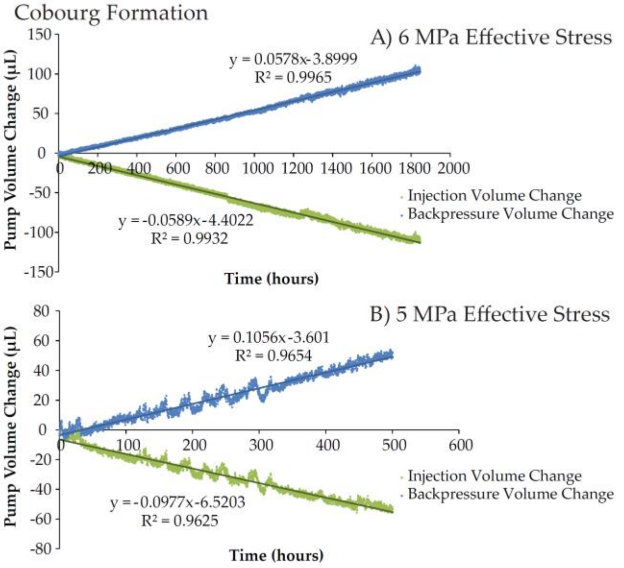

Fig. 4. Volume change (μL) against time (hours) in the Cobourg Formation sample for an effective stress of A) 6 MPa (4 MPa differential pressure) and B) 5 MPa (6 MPa differential pressure).

IV. DISCUSSION

The ultra-low steady-state permeability of these two prepared rock core samples is consistent with *in-situ* testing and formation scale estimates obtained during the site characterisation program conducted at the Bruce nuclear site by Intera Engineering Ltd[2]. The permeabilities measured in the Intera study were measured using a pulse-decay permeameter and represent the transient method of estimating permeability originally described by Brace and co-workers[9], whereby the characteristics of the decay profile of a temporary pressure pulse across the sample allows the calculation of the permeability. Steady-state measurements have previously been difficult to obtain because of their long duration and the requirements for accuracy when measuring microscopic flowrates[10]. However, this is no longer the case with high precision syringe pumps providing a means to accurately measure very small volumetric flow rates (See Section II.D). By measuring both in- and outflow and establishing a well-defined steady-state, the hydraulic properties of the material can be accurately measured. Importantly, by imposing a constant boundary condition, and defining the permeability at steady-state, the stress/mechanical sensitivity of these materials can be assessed and accurately quantified. In addition, steady-state approaches test a much larger pore volume than can be achieved through transient methods, thereby reducing bias introduced from localised features (such as sampling damage, micro-fractures, localised bioturbation etc.) which in turn can provide a more accurate representation of the bulk permeability.

The hydraulic conductivities and permeabilities of the Silurian and Ordovician sediments at the Bruce nuclear site have been measured on a number of different scales as part of the repository site characterisation programme, from the small scale (rock cores on the order of centimetres tested by Intera[2]) through *in-situ* borehole testing (tens of metres[11]) to the large scale (natural analogues; hundreds of metres[12]). Intera[2] conducted permeability tests on “as received” saturated core samples from DGR-1, DGR-2 and DGR-3 boreholes; the permeability of the Queenston Formation (DGR-3) was between 10^{-20} m^2 and 10^{-21} m^2, whilst permeability measurements from the Lower Member of the Cobourg Formation (DGR-1 and DGR-2) and lie in the range 10^{-18} m^2 to 10^{-20} m^{22}. As discussed in Intera’s study[2], the permeabilities observed were higher than expected and the lower limit on the measured permeability ($5x10^{-21}$ m^2) was not low enough to accurately measure the permeability of the samples. The transient permeabilities measured[2] are therefore at least 1-2 orders of magnitude higher than the steady-state measurements presented in this study. For “as received” core tests, the state of saturation is unknown and the results may thus be affected by hydration of the core. Unless multiple repeat transient tests are performed on the same core plug, it would not be possible to differentiate between flow induced by resaturation of the core from that which occurred as a direct result of the transient hydraulic gradient under fully saturated conditions. Either way, the coupling between permeability and stress often observed in clay-based low permeability materials[13,14,15,16,17,18,19,20] is likely to affect the results as is the total pore volume tested.

In-situ measurements made by straddle-packer hydraulic testing[11] give estimated horizontal hydraulic conductivities for the Upper Ordovician sediments (including the Cobourg Formation) that average 10^{-14} m s^{-1} or less, equating to permeabilities on the order of 10^{-21} m^2 or less. The measured horizontal hydraulic conductivities for the Lower Member (repository host rock) were in the range $3E^{-14}$ m s^{-1} to $4E^{-15}$ m s^{-1} (Ref. 11); the average hydraulic conductivity range of the Lower Member measured in this study was $1.2E^{-15}$ m s^{-1} to $1.4E^{-15}$ m s^{-1}. The *in-situ* measured values are consistent with the results presented in this study and give additional confidence to both our measurements and the steady-state method. Additionally, these values are also in agreement with formation-scale simulations of the subsurface hydrological properties of the Michigan Basin (encompassing the Bruce nuclear site) based on observed underpressures[12]. Neuzil and Provost[12] used the fluid pressures in the low permeability horizons to inversely estimate vertical site-scale hydraulic conductivities. The values they obtained were in the range 10^{-14} m s^{-1} to 10^{-15} m s^{-1} for the Cobourg Formation. Importantly, the results presented here demonstrate that reliable steady-state measurements can be made on ultra-low permeability rock samples, provided that an appropriate methodology is followed.

The fluid composition used in the study was matched to the groundwater measured from the same borehole as the samples were derived from[2], although the salinity of the fluid used in the testing (3.8 M) was lower than the measured salinity of the downhole brine water. It is important that the fluid chemistry is in equilibrium with the rock sample because disequilibrium will induce chemical reactions between the fluid and the rock. This could affect the fluid pathways and alter the permeability of the sample. However, the linear responses observed in Figures 3 and 4 suggest no underlying long-term changes in permeability are occurring, indicating the test fluid used in this study has not had a deleterious effect on the core.

In both experiments, the sample was manufactured so that the orientation of the bedding relative to the flow direction was perpendicular. The anisotropy of shales can be examined by diffusion testing[21], although the degree of anisotropy observed can be variable. Xiang and co-workers[21] found that the water accessible porosity of the Queenston Formation was in the range 5.8-10.9% both normal (average 8.6%) and parallel (average 8.5%) to bedding. The diffusion accessible porosity was found to range between 4.4 and 9.4% (average 5.7%) normal to bedding and 4.2-7.5% (average 5.7%) parallel to bedding. The average values suggest that the amount of anisotropy normal to and parallel to bedding is low. For the Cobourg Formation the water accessible porosity was lower both normal and parallel to bedding (0.7-2.1%, average 1.28%) with the diffusion accessible porosity in a similar range (0.4-2.5% parallel to bedding (average 1.47%), 0.5-1.8% normal to bedding (average 1.27%)). While these values seem somewhat counterintuitive, Xiang et al. cite anion exclusion as a possible mechanism to explain the observed differences. The presence of small-scale fossils and bioturbation in the Cobourg Formation (crinoids, shell fragments and brachiopods have all been documented[7]), introduce some degree of heterogeneity and possibly increase the flow path of fluids in or around areas of bioturbation, illustrated by the range of values presented by Xiang and co-workers[21]. The Queenston Formation sample had bedding structures that could be observed within the sample. As such, the permeability calculated perpendicular to the bedding will constitute an average permeability of the different layers through which the fluid passes. With the sample orientation parallel to bedding, it is likely the fluid will flow more quickly and give a slightly higher value of permeability for the same rock type, depending on interconnectivity. However, manufacture of a sample in this orientation can be difficult because the rock tends to fracture along planes of weakness parallel to the bedding. Manufacturing the right sample size is a trade-off between making measurements on a reasonably short timescale (months) but with a low enough hydraulic gradient to prevent damage to the sample and also capture its true properties. A sample with a length and diameter of about 5 cm achieves these aims, based on a comparison with field estimates. To remove uncertainty, future studies should investigate the effect of the flow orientation relative to bedding on the permeability. In addition, good sample preservation and preparation is essential to ensure good measurement reliability.

These ultra-low permeabilities provide further evidence of the applicability of steady-state experimental methods to obtain reliable estimates of extremely low rock matrix permeabilities from rock core samples subject to an *in-situ* stress.

V. CONCLUSIONS

Measurements of hydraulic conductivity and intrinsic permeability for two Upper Ordovician bedrock formations within a sedimentary sequence beneath the Bruce nuclear site in Ontario, Canada, have been successfully completed using the steady-state method. The Queenston Formation (Shale) and the Cobourg Formation (argillaceous limestone) were found to have extremely low permeabilities, on the order of 10^{-22} m^2 or 0.1 nD. Along with their low porosities, this low permeability renders them an effective barrier to groundwater flow and mass transport for the purpose of geological disposal.

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REFERENCES

1. D. K. ARMSTRONG and T. R. CARTER, “The Subsurface Paleozoic Stratigraphy of Southern Ontario,” Ontario Geological Survey, Special Volume 7, (2010).
2. INTERA, “OPG’s Deep Geological Repository for Low and Intermediate Level Waste. Descriptive Geosphere Site Model,” Intera Engineering Ltd, NWMO DGR-TR-2011-24 (2011).
3. D. K. ARMSTRONG and J. E. P. DODGE, “Palaeozoic Geology of Southern Ontario,” Ontario Geological Survey (2007).
4. M. E. BROOKFIELD, “A mid-Ordovician temperate carbonate shelf – the Black River and Trenton Limestone Groups of southern Ontario, Canada,” *Sedimentary Geology*, **60**, 137-153 (1988).
5. G. A. COOPER “Early Middle Ordovician of the United States.” In *The Ordovician System,* M. G. BASSETT, Ed., University of Wales Press, Cardiff (1976).
6. D. K. ARMSTRONG and T. R. CARTER, “An updated guide to the subsurface Paleozoic stratigraphy of southern Ontario,” Ontario Geological Survey (2006).
7. NWMO, “OPG’s Deep Geologic Repository for Low and Intermediate Level Waste: Geosynthesis”, DGR-TR-2011-11, (2011).
8. J. F. HARRINGTON and S. T. HORSEMAN, “Gas transport properties of clays and mudrocks,” *Geological Society of London, Special Publication*, **158**, 107-124 (1999).
9. W. F. BRACE and J. B. WALSH and W. T. FRANGOS, “Permeability of granite under high pressure,” *Journal of Geophysical Research*, **73** (6), 2225-2236 (1968).
10. S. FINSTERLE and P. PERSOFF, “Determining permeability of tight rock samples using inverse modeling,” *Water Resources Research,* **33** (8), 1803–1811 (1997).
11. R. L. BEAUHEIM, R. M. ROBERTS and J. D. AVIS. “Hydraulic testing of low-permeability Silurian and Ordovician strata, Michigan Basin, southwest Ontario,” *Journal of Hydrology,* **509**, 163-178 (2014).

12. C. E. NEUZIL and A. M. PROVOST. "Ice sheet load cycling and fluid underpressures in the Eastern Michigan Basin, Ontario, Canada," *Journal of Geophysical Research Solid Earth*, **119**, 1-22 (2014).
13. C. E. NEUZIL, "How permeable are clays and shales?" *Water Resources Research*, **30** (2), 145-150 (1994).
14. J. F. HARRINGTON and S. T. HORSEMAN, "Migration of repository gases in an overconsolidated clay", *British Geological Survey Commissioned Report*, WE/94/7 (1994).
15. J. F. HARRINGTON and S. T. HORSEMAN, "Projects on the effects of gas in underground storage facilities for radioactive waste (Pegasus project): Gas Migration in Clay", Proceedings of a progress meeting held in Mol, Belgium, 28-29 May, 1997, European Science and Technology Series (1998) EUR 18167 EN, pp153-173, ISBN 92-828-2757-7 (1997).
16. D. N. DEWHURST, A. C. APLIN, J.-P. SARDA, and Y. YANG. "Compaction-driven evolution of porosity and permeability in natural mudstones: An experimental study", *Journal of Geophysical Research*, **103**, 651-661 (1998).
17. D. J. BIRCHALL, J. F. HARRINGTON, D. J. NOY and R. J. CUSS, "Laboratory study examining permeability evolution along an isotropic unloading stress path", British Geological Survey Commissioned Report, CR/07/016 (2007).
18. R. M. BUSTIN, A. M. M. BUSTIN, A. CUI, D. ROSS and V. M. PATHI, "Impact of Shale Properties on Pore Structure and Storage Characteristics", *Society of Petroleum Engineers*, doi:10.2118/119892-MS (2008).
19. M. GUTIERREZ, D. KATSUKI, A. TUTUNCU, "Determination of the continuous stress-dependent permeability, compressibility and poroelasticity of shale," *Marine and Petroleum Geology*, **68**, 614 (2015).
20. P. J. ARMITAGE, R. H. WORDEN, D. R. FAULKNER, A. R. BUTCHER and A. A. ESPIE, "Permeability of the Mercia Mudstone: suitability as caprock to carbon capture and storage sites," *Geofluids*, **16**, (1), 26-42 (2016).
21. Y. XIANG, T. AL, L. SCOTT, and D. LOOMER, "Diffusive anisotropy in low-permeability Ordovician sedimentary rocks from the Michigan Basin in southwest Ontario," *Journal of Contaminant Hydrology*, **155**, 31-45 (2013).