

Transport properties of the Cobourg Limestone: A benchmark investigation

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Abstract. Cobourg limestone formation is proposed as a possible repository site for nuclear wastes in Canada. This limestone displays significant heterogeneity, characterized by light grey calcite nodular regions, interspersed with dark grey calcite-dolomite-quartz partings containing a clay component. Mineral composition is dominated by calcite, with some minor amounts of ankerite, illite/muscovite/I-S and quartz. Analysis of the pore structure show that Cobourg limestone is extremely tight with porosities between 0.33 and 2.51%.

This paper aims at both, a comprehensive description of the transport properties of the Cobourg limestone on the nm- to cm-range, and the comparison of different experimental techniques: gas measurement using decay, quasi-static, or steady-state methods. In total four labs measured permeability, porosity and analysed the pore system by various methods (micro-CT, BIB/SEM). In all flow experiments, slip flow was accounted for by means of the Klinkenberg correction.

Effects of pore pressure, confining pressure, sample size and coring orientation are studied. For all laboratories, results range from 100 microDarcy to 1 nanoDarcy. Even for a given laboratory, results are comprised in a broad range, with several orders of magnitude differences depending on coring direction, confining pressure and sample size. Flow occurs through slit shaped pores/fractures, which are orientated along heterogeneities. Upon loading, these natural and/or artificial pores successively close, resulting in a reduced permeability and stress sensitivity.

Results are dominated by heterogeneity and anisotropy of the Cobourg limestone, so that it is delicate to select one method over another. Rather, each brings useful information to better understand this low permeability and low porosity natural material.

1 Introduction

1.1 Background

The Cobourg limestone formation is located in the eastern shores of Lake Huron in southern Ontario, Canada. The interest in the limestone stems from its potential suitability as a host rock of a Deep Geologic Repository (DGR) for storing low- and intermediate-level radioactive waste. This choice is enhanced by the proximity of the DGR to the Bruce Nuclear Facility, in Ontario, Canada. The planned depth of the DGR is approximately 680 m below ground level, within the Cobourg limestone formation of the Paleozoic sedimentary sequence that rests on a Pre-Cambrian granitic gneiss basement rock. The Cobourg limestone host rock is overlain by the upper Ordovician siltstone and grey shale extending to a thickness of approximately 200 m and underlain by argillaceous limestone and grey shale, about 150 m thick, resting on the granitic basement rock of the Canadian Shield. Despite its low clay content, the Cobourg limestone is nominally referred to as an *argillaceous*

limestone. It shows fabric heterogeneity, consisting of *lighter* nodular regions of calcite and dolomite separated by *darker* argillaceous partings of a similar composition with additional quartz and low clay content (Figure 1). In the literature, the consensus is that the rock displays some nominal evidence of “stratification” resulting from the nodular fabric (the *light grey rock*) that contains calcite and dolomite interspersed by the partings (the *dark grey rock*) that contains calcite, dolomite and a clay fraction. The lighter calcite-dolomite nodular regions can have dimensions in the range of 25 mm, which places a restriction on the sample size that can capture a representative volume element (i.e. a cross-sectional area and length of flow path; Figure 1).

Previous researches on permeability testing of Cobourg limestone have been conducted by several investigators using both *in situ* packer testing and laboratory testing. In the former types of tests, the influence of scale is addressed indirectly through the selection of a packer length and borehole diameter that can capture a representative surface area. In the latter

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category of tests, the sample dimensions, notably the cross-sectional area of cylindrical samples is chosen to capture a representative area fraction. The length of the flow path is selected to minimize the time needed to establish a steady state.



Figure 1. The visual heterogeneity of the Cobourg limestone (top) cuboid measuring 300 mm (bottom) modular regions in a zone measuring 100 mm x 130 mm

The research reported by Vilks and Miller [1], Selvadurai et al. [2], Selvadurai and Jenner [3] and Selvadurai and Najari [4,5] present results of laboratory research of one-dimensional flow conducted on specimens with diameters ranging from 50 mm to 100 mm and flow paths ranging from 5 mm to 200 mm. The measured permeabilities ranged from 10^{-23} m^2 to 10^{-19} m^2 . The work of Selvadurai and Glowacki [6] deals with the measurement of the local permeability of the heterogeneous Cobourg limestone.

1.2 Aims and scopes

The determination of permeability in the nanoDarcy-range is always challenging due to the small flowrates. So far, there is no standard procedure or reference samples. Different research laboratories follow different approaches and comparing the results therefore is difficult. Differences might be induced by the use of different fluids, the flowrate measurement technique itself, sample size, sample confinement, and loading history.

This research focuses on the testing of suitable sample sizes that can satisfy the constraints imposed by the heterogeneity of the Cobourg limestone and accomplish the testing at a reasonable time duration.

This benchmark was launched by the department of Civil Engineering at McGill University who provided samples to all the involved laboratories.

A similar study was published by Profice [7] with the conclusion of good agreement between the different methods and laboratories, but that study was conducted

on pyrophyllite, a very homogeneous rock with liquid permeability in the range of 100 nanoDarcy.

In the following, the methodologies for measuring permeability used by four different laboratories are presented: gas pressure decay, quasi-static, step decay or steady-state methods. We always performed the Klinkenberg correction to derive permeabilities independent of fluid nature (nitrogen, helium or brine). Further, porosity measured at ambient conditions and under stress, and permeability results are presented. Porosity and permeability results are presented and discussed with respect to the effects of pore pressure, confining pressure, sample size and coring orientation. The results are also compared to literature data, especially the liquid permeability measurements performed by McGill.

2 Materials and methods

2.1 Materials

The samples received by all participants are cored from blocks of the Cobourg limestone, that have been obtained from the Saint Mary's Quarry, located in Bowmanville Ontario, Canada. The original block samples measured approximately 2 cubic meters.

Whenever requested by the participants (McGill and RWTH Aachen University), the samples are cored out from cubical samples (measuring 100mm, 150mm, or 200mm) of the Cobourg Limestone. Each cubic sample is in an air-dry condition.

The operating speed of the corer is 600 rpm. The feed rate is at 0.127 mm per revolution. For the machining of the samples to the requested dimensions, we use a lathe with a diamond tip cutter. The lathe is operated at 550 rpm and the feed speed is at 30 mm/min. The samples are mostly cored perpendicular to the nominal bedding plane. Some participants, however, also requested samples that were cored parallel to the nominal bedding plane.

2.2 Experimental approach at Mc Gill University

As has been shown by Selvadurai [8], gas steady state tests needs very long time for equilibrium, while hydraulic pulse tests can be conducted in a short duration. In order to interpret the results of hydraulic pulse tests, the pore space of the sample needs to be completely saturated and parameters such as the porosity, the skeletal compressibility and solid material compressibility are needed to correctly estimate the hydraulic pulse decay in a pressurized region in contact with the Cobourg limestone.

Therefore, the research presented here involves performing one-dimensional steady state flow tests on cylindrical samples with a cross-sectional diameter of 150 mm and an axial flow path of 50 mm. The permeability tests are performed in an active GDS Triaxial Cell, which

is used to apply a pressure of 5 MPa to ensure impervious contact between the sample and the enclosing rubber membrane. The sample (with stainless steel porous discs on the plane ends) is encased within the nitrile rubber sleeve exposed to vacuum desaturation for a period of two weeks in the test cell. A precision pump is used to initiate a hydraulic gradient with inlet pressures ranging from 0.5 MPa to 1.5 MPa. Figure 2 shows a typical arrangement for conducting the steady state permeability test.

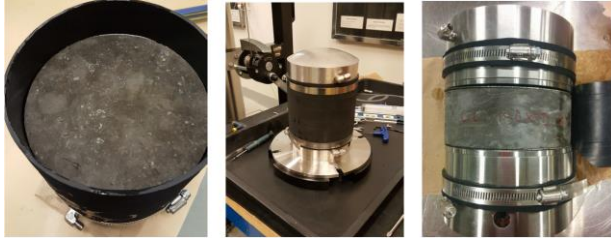


Figure 2. Steady state permeability tests conducted on the Cobourg limestone at Mc Gill University

The flow rate is accurately determined by measuring the volume of fluid injected for a given time.

2.3 Experimental approach at RWTH Aachen University

2.3.1 Materials

For sample preparation, RWTH-Aachen University received a block of about $10 \times 5 \times 5 \text{ cm}^3$ from McGill University. Three cylindrical sample plugs of 38 mm in diameter (samples C, D and E) were drilled in this block. Additionally, we received another sample, which had been already trimmed to the required dimensions (sample B, provided by the Structural Engineering Laboratories of McGill University). The height of samples B, C, D and E are 26.48, 44.56, 42.43 and 35.04 mm, respectively

2.3.2 Gas transport experiments

Samples were dried at 105°C in a vacuum oven for at least 24 h. Subsequently, porosity and gas permeability were measured with gas. Tests are run at different confining pressure levels up to 20 MPa. In most cases, experiments are conducted on the second loading and unloading cycle. Helium is used as the gas phase.

Gas permeability is determined by the constant downstream pressure procedure, where the volume flow rate is calculated from the pressure decay in the upstream compartment (Figure 3). Due to the simultaneous decrease in mean pressure, slip flow can be accounted for by means of the Klinkenberg correction from only one pressure decay curve. Details about the experimental method are described in [9-10].

2.3.3 Additional analysis

Qualitative XRD analysis demonstrates that the Cobourg limestone is dominated by calcite (79.05%), ankerite

(9.31%), illite/muscovite/I-S (5.12%) and quartz (4.81%) (Table 1).

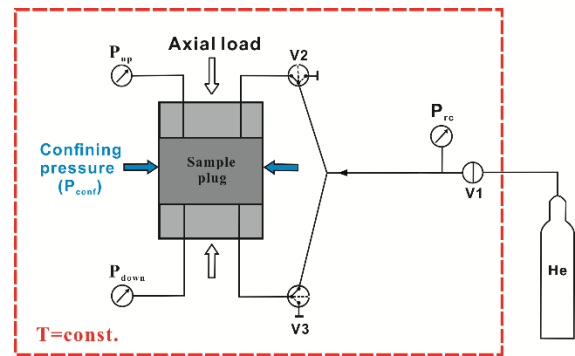


Figure 3. System sketch used for both pore volume and permeability measurements under defined confining pressures (The reference cell consists of the volume of the lines between valves 1, 2 and 3. The pressures in reference cell and top and bottom compartments (P_{rc} , P_{up} and P_{down}) are recorded by PMP 4070 pressure transducers)

Table 1. XRD result of the Cobourg limestone

Calcite (%)	Ankerite (%)	Illite/muscovite/I-S (%)	Quartz (%)	Kaolinite (%)	Pyrite (%)
79.05	9.31	5.12	4.81	1.22	0.48

Analyses of the pore structure by micro-CT and BIB-SEM analysis show that the Cobourg limestone is extremely tight. Isolated, irregularly shaped pores up to 10^{th} -nm to several μm -range are observed in calcite grains. In the clay matrix, slit-shaped pores of about 100 nm in width appear to be interconnected. Micro-CT imaging indicates open discontinuities in sample B, which is further identified as stylolites containing a higher percentage of clay minerals in BIB-SEM.

He-pycnometry under unconfined conditions shows that porosity values of all plugs are below 3% (Table 2). There is no measurement of porosities under stress.

Table 2. Porosity values of samples under unconfined conditions

Sample	B	C	D	E
Porosity (%)	1.02	0.98	2.51	1.83

2.4 Experimental approach at Centrale Lille

The initial cube (measuring 150mm) provided by Mc Gill University (reference S6-13) is labelled with a letter on each face (U and T, or B and D, or C and A, on parallel faces respectively). It is cored at Centrale Lille along the cube three main axis (labelled respectively U-T, B-D and C-A). This provides cylindrical samples of 65mm diameter, which are cut to a given height, comprised between 18.9 and 36.4mm (Figure 4). This location is chosen to be in the calcite-dolomite nodular regions, which can have dimensions in the range of 25 mm, or slightly bigger. No bigger sample was tested, in order to limit the duration of permeability experiments.



Figure 4. Sample preparation from a cube provided by Mc Gill University to cylinders of 65 mm diameter and varying height

Table 3. Sample volume and porosity results for 15 cylindrical samples cored out of a Cobourg limestone cube.

	Sample height (mm)	Sample diameter (mm)	Porosity (%)
U1, U2, U3, U4, U5	20.6, 23.7, 17.0, 25.0, 26.9	65.2	0.93, 0.67, 0.69, 0.53, 0.33
D1, D2, D3, D4, D5	18.7, 36.4, 17.8, 23.6, 31.3	65.2	0.99, 0.55, 0.61, 0.56, 0.33
C1, C2, C3, C4, C5	18.5, 18.8, 32.9, 13.3, 25.6	65.3	1.21, 0.44, 0.59, 0.74, 0.43

As suggested by Mc Gill University, the final characteristics of the samples, i.e. their water-saturated mass, their dry mass and volume, are recorded prior to conducting the permeability tests. To this purpose, each cylinder is oven-dried at 105°C until mass stabilization.

A total of 15 samples (5 along each main axis of the initial cube) has been tested for porosity (by the water saturation

method), see Table 3, out of which 4 have been tested for gas permeability. Bulk volume is determined from length and diameter of the cylinders. Standard accuracy of 1 PU is assumed for porosity.

Samples D1, D2, C3 and U5 have been tested for gas permeability. To this purpose, each sample is placed in a triaxial cell and tested at successive confining pressures of 5, 10 and 15 MPa and gas pressures of 0.5, 1.0 and 1.5 MPa (with Argon). Gas pressure is varied to quantify Klinkenberg corrected. Gas permeability is measured in the quasi-static state using either the upstream or the downstream pressure, measured just before or just after the sample (see Figure 5). The upstream gas pressure is used to analyse permeabilities down to 10^{-18} m² (i.e. 1 μD), whereas downstream pressure is for lower permeabilities, down to $10^{-20}/10^{-21}$ m² (i.e. 1-10 nD).

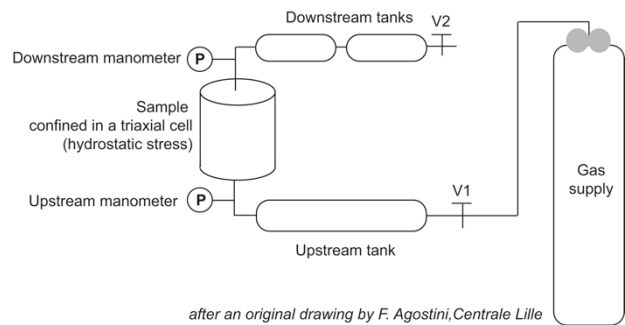


Figure 5. Quasi-static gas permeability set-up available at Centrale Lille, using either the upstream or downstream gas tanks, depending on the expected order of magnitude of permeability

The effect of sample direction and height is analysed, as well as the effect of sample confinement (hydrostatic loading) and Klinkenberg effect [11-12].

2.5 Experimental approach at Cydarex

A unique feature of our approach is to use small samples in order to minimize the contribution of large-scale core fractures and to reduce the durations of equilibrium before measurements and duration of measurement since all these durations are proportional to the square of the length of the samples. The principle is described in a previous SCA paper [13].

We performed two types of measurements: a measurement at different pore pressures to determine the Klinkenberg corrected permeability and the determination of a permeability profile over 7 cm to quantify the heterogeneity at the scale of 5mm.

2.5.1 Manufacturing of resin disks

For the profile measurement, a cylindrical sample with diameter of 12.5 mm was embedded in resin. Once embedded in resin, several adjacent thin slices were cut parallel to the bedding plane, with thicknesses 3.4 mm (figure 6). After polishing, the slices were then put

between two steel end-pieces (figure 7) and mounted in a hydraulic press (Figure 8).



Figure 6- Resin disk sample

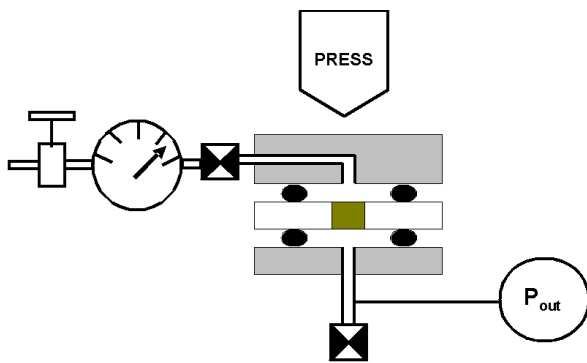


Figure 7- Resin disc: experimental setup with constant pressure injection (injection with a constant volume can also be used)

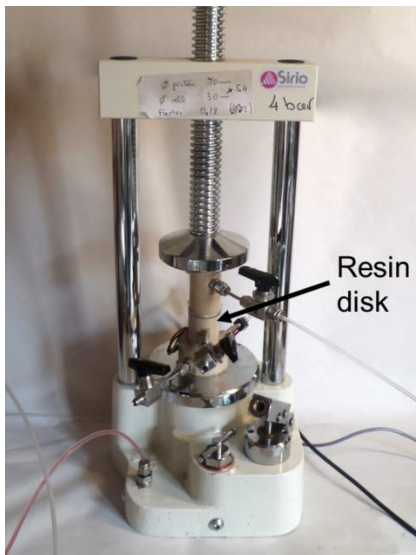


Figure 8 – The cell under the press

2.5.2 Principle of permeability measurements

Because the original sample is cut parallel to the Cobourg limestone bedding planes, gas flow in the disc will be perpendicular to the bedding (vertical flow in the reservoir).

Measurements are performed with nitrogen at room temperature. Vertical pressure applied by the press acts on both the resin and the rock. Assuming that the resin is "soft" compared to the rock, the stress on the sample is around 190 bars (19 MPa).

The entry can be connected to several vessels of different volumes. The outlet is closed on a small volume. Inlet and outlet pressures are measured. By measuring the pressure at the outlet, any leakage on the high-pressure part of the apparatus is irrelevant, since the real inlet pressure is recorded and used in the numerical simulation.

The main error comes from the estimate of the surface area of the embedded sample when the shape is not circular. The overall error is estimated to be less than 10% of the measured value.

2.5.3 Numerical simulations

Interpretations of the results are performed using the commercial software CYDAR. The simulation is in one dimension and assumes perfect gas with Klinkenberg and Forchheimer corrections. The numerical scheme is "implicit" and the system is solved using a Newton-Raphson algorithm. Parameters such as permeability, Klinkenberg coefficient β can be optimized using a nonlinear least-squares minimization algorithm (Levenberg-Marquardt). For the experiments performed on low permeability samples, the cost function is calculated only on outlet pressures.

2.5.4 Porosity measurement

The solid volume is obtained from gas expansion and pore volume is given by the difference in weight between brine saturated and dry sample. Accuracy is around 1 P.U

3 Accuracy of the results

In this domain of very low permeabilities, there is no reference sample that can be used to calibrate the apparatus. Accuracy must be estimated from the instruments and the procedure.

Standard error calculation accounts for errors on the size of the sample, on pressure sensors, on fluid viscosity linked to the temperature, on atmospheric pressure (not always measured). The different laboratories estimate the relative error due to the instruments and fluid properties to be less than 10% of the measured permeability values.

Another source of error is the leaks. The leaks from the upstream circuit to the atmosphere are determined by using an impermeable sample. More difficult to estimate is the leaks between the sample and the rubber sleeve. For each equipment, a minimum confining pressure is always determined.

For gas measurements, the determination of the Klinkenberg correction is always a source of error since it is based on an optimisation process or an extrapolation at origin in the Klinkenberg plot. For Cydarex, the measurements are performed with three points at different pressures and the correlation coefficient is always larger than 0.98. For the measurement reported in the next section, the extrapolated Klinkenberg corrected permeability is 0.12 nD +/- 0.01 nD. The range is determined using the extreme values calculated with only 2 points. We can consider that the Klinkenberg correction adds 10% in the error.

4 Results

4.1 Permeability at Mc Gill

Steady state tests were conducted on cylindrical samples of the Cobourg. Transverse permeability (K_T) is determined with bedding plane along the axis of the sample and normal permeability (K_N) with bedding planes perpendicular to the axis of the sample

The measured flow rates range from 4.02×10^{-5} ml/min to 6.83×10^{-3} ml/min. The experimental results gave the following:

$$K_N = (2.0 \text{ to } 3.9)10^{-21} \text{ m}^2$$

and

$$K_T = (2.2 \text{ to } 4.2)10^{-19} \text{ m}^2,$$

with the observation that the lower limit of permeability is obtained for the higher inlet pressure (highest pore pressure).

4.2 Porosity and permeability experiments under defined confining pressure conditions at RWTH Aachen University

4.2.1 Porosity

Porosity of the loaded samples was determined by He-expansion from a calibrated reference volume into the flow cell. Volume of solid is derived from the mass and the grain density. Error is around 1 PU. Porosity is ranging between 0.6 and 2.1% and decreases with increasing confining pressure. Loading from 4 MPa to 19 MPa leads to a reduction by 8-13% (Figure 8). The exponential stress sensitivity coefficient of porosity varies in between 0.004 - 0.010 MPa^{-1} . The porosity values on the first unloading path are about 92% to 100% of the corresponding values on the first loading path (Figure 9).

Klinkenberg-corrected permeabilities at 5 MPa confining pressure range in between 5×10^{-20} and $1 \times 10^{-17} \text{ m}^2$ (Table 4). Sample B has the highest Klinkenberg-corrected permeability, which is related to the open discontinuities identified by micro-CT. During the first loading path from 5 MPa to 20 MPa, the Klinkenberg-

corrected permeability decreases by 49-67%. The exponential stress sensitivity coefficients for permeability is much larger than those for porosity, ranging in between 0.030 and 0.071 MPa^{-1} .

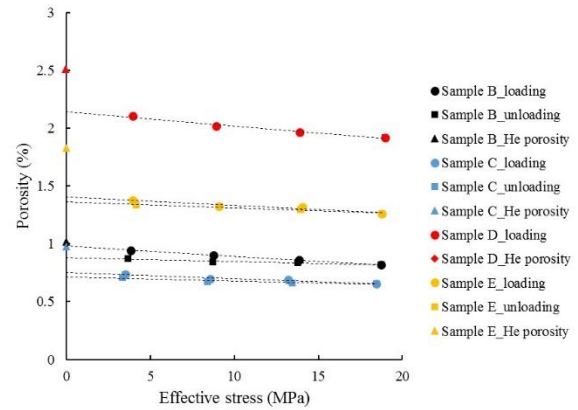


Figure 9. Relationship between porosity and effective stress

4.2.2 Gas permeability

Table 4. Klinkenberg-corrected permeabilities (m^2) at different confining pressures in one load-unload cycle

Confining pressure (MPa)	B	C	D	E
5	1.45×10^{-17}	4.64×10^{-20}	2.29×10^{-19}	1.17×10^{-19}
10	9.46×10^{-18}	2.69×10^{-20}	1.81×10^{-19}	8.43×10^{-20}
15	7.42×10^{-18}	2.30×10^{-20}	1.41×10^{-19}	7.23×10^{-20}
20	6.19×10^{-18}	1.95×10^{-20}	1.18×10^{-19}	3.84×10^{-20}
15	6.81×10^{-18}	2.03×10^{-20}	-	3.86×10^{-20}
10	8.27×10^{-18}	2.19×10^{-20}	-	4.68×10^{-20}
5	1.30×10^{-17}	3.15×10^{-20}	-	9.83×10^{-19}

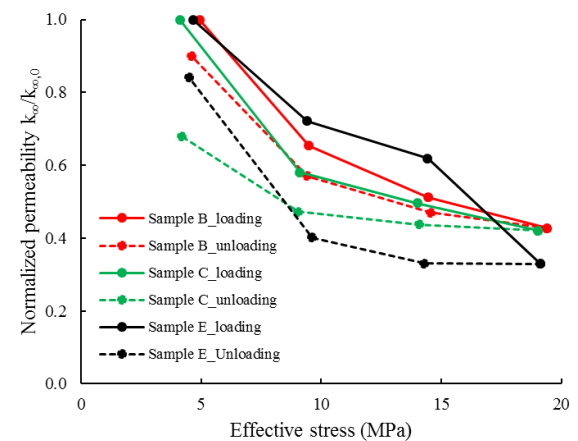


Figure 10. Normalized Klinkenberg corrected permeability (permeability at certain effective stress divided by permeability at initial effective stress) versus effective stress for both loading and unloading process

In view of the observed difference between permeability on the first loading path and first unloading path (Figure 10), we conclude to a large hysteresis effect.

4.3 Permeability experiments at Centrale Lille

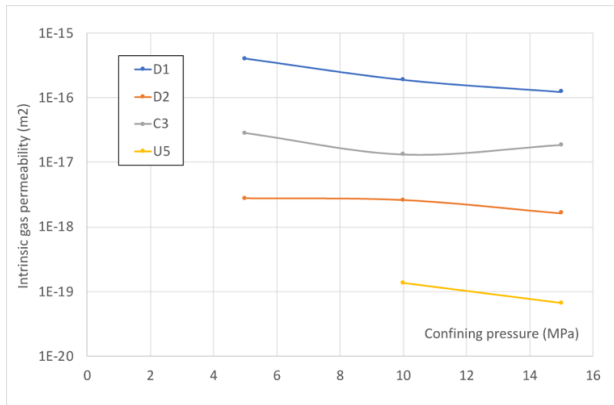


Figure 11. Klinkenberg corrected gas permeability (in m²) for all four Cobourg limestone samples tested, as a function of confining pressure P_c (in MPa)

Klinkenberg corrected gas permeability results and associated Klinkenberg coefficient β are given for the three main axis of the original cube provided by McGill, which correspond to directions along or perpendicular to bedding planes of Cobourg limestone, for three given values of confining pressure (Table 5 and Figure 11).

As expected, gas permeability decreases with increasing confinement, meaning that the pore system is sensitive to hydrostatic stress changes. This is frequently observed for slit-like pores (or cracked materials), rather than for more rounded pore systems. In accordance, Klinkenberg coefficient β , which reputedly is proportional to the inverse of a mean pore radius, also increases with increasing confinement.

More interestingly, for the three different sample orientations, highly variable permeability orders of magnitude are obtained. The significantly smaller permeability of U5 sample compared to those in the two other directions is normal for a sample oriented perpendicularly to the bedding planes. For the two samples oriented along the same D axis, i.e. along an axis parallel to the bedding planes, a large range of permeability values were observed gas permeability changes: two orders of magnitude (between 10⁻¹⁶ and 10⁻¹⁸ m², i.e. 1 to 100 μ D). This is attributed to the sample size (with a height ranging from 18.7 to 36.4 mm). It is below the biggest size of calcite-dolomite nodular regions for sample D1. The large heterogeneity of Cobourg limestone is clearly identified.

Table 5. Klinkenberg corrected gas permeability (in m²) and Klinkenberg coefficient β (in MPa) of four cylindrical samples of Cobourg limestone, tested at three successive confining pressures

K _{int} at given confining pressure (MPa)	5	4.0.10 ⁻¹⁶	2.8.10 ⁻¹⁸	2.8.10 ⁻¹⁷	N/A
	10	1.9.10 ⁻¹⁶	2.6.10 ⁻¹⁸	1.3.10 ⁻¹⁷	14.10 ⁻²⁰
	15	1.2.10 ⁻¹⁶	1.6.10 ⁻¹⁸	1.8.10 ⁻¹⁷	6.6.10 ⁻²⁰
β at given confining pressure (MPa)	5	3.2	1.1	0.9	N/A
	10	4.4	2.7	1.3	2.2
	15	4.8	1.2	N/A	2.5

4.4 Results from Cydarex

4.4.1 Porosity measurement

For four samples, porosities without confining pressure range between 2 and 3%. Accuracy of the porosity measurement is estimated to 1 PU.

4.4.2 Permeability

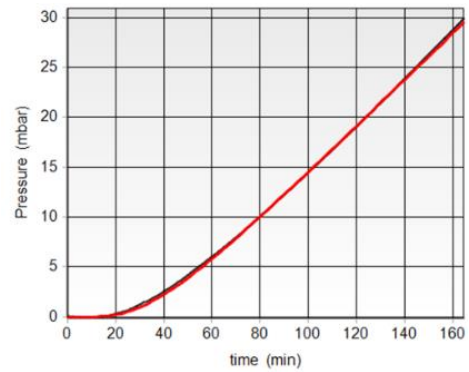


Figure 12. Pressure response in the downstream volume as function of time. The experiment in black and the numerical simulation in red are nearly superimposed. The transient below 60 minutes is fitted with a porosity of 1.7%.

Figure 12 shows the record of pressure in the downstream volume when a gas pressure is applied upstream. The outlet pressure shows a delay before starting to increase, due to the accumulation of gas inside the sample. The transient below 60 minutes is fitted with a porosity of 1.7%, in agreement with porosity measurement.

Measurements at different pressures are shown in figure 13. The result is a Klinkenberg-corrected permeability of 0.12 nanoDarcy (0.12 10⁻²¹ m²) and β = 20 bar (= 2 MPa).

Sample	D1	D2	C3	U5
Height (mm)	18.7	36.4	32.9	26.9
Porosity (%)	0.99	0.55	0.59	0.33

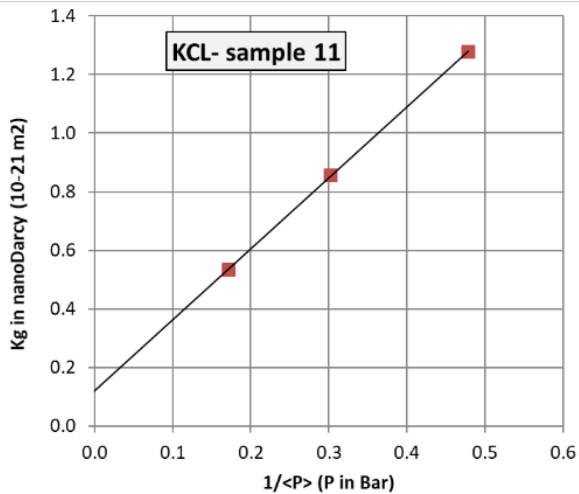


Figure 13- Sample 11: determination of the Klinkenberg corrected permeability (0.12 nanoDarcy or $0.12 \cdot 10^{-21} \text{ m}^2$) from the gas permeability measured at 3 different pore pressures. $\langle P \rangle$ is the average pressure in the sample.

4.4.3 Measurement of a permeability profile

In order to determine the heterogeneity of the sample, we have measured the permeability profile on a plug, using the method of resin disks described previously with a resolution of 5 mm.

Measurements are performed with N_2 at room temperature and stress around 19 MPa. Injection pressure is around 10 bar (1 MPa), and the Klinkenberg correction is applied using $\beta = 20$ bar, as determined experimentally on sample 11.

The profile shows a spreading between 0.5 and 3 nD (fig. 14). The conclusion is that this sample is quite homogeneous at the scale of 5 mm. A factor 6 is often observed in samples used for core analysis.

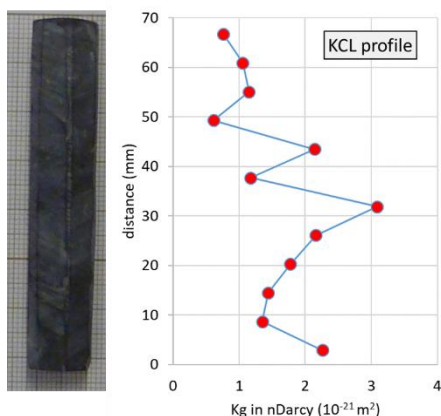


Figure 14- Sample and gas permeability profile measured on slices of 5mm.

5 Discussion - Conclusions

Performing a benchmark study on Cobourg limestone has shown that the involved teams need to possess a wide range of experimental means, enabling them to handle permeabilities from 0.1nD to $100\mu\text{D}$, and several solutions are described to perform these measurements. Despite this wide range of permeability systems, a consistency in the results of the different partners has been found, as follows.

This benchmark study has clearly evidenced that it is a very tight rock, with porosities ranging between 0.33 and 2.51%. This porosity corresponds to a pore system sensitive to changes in hydrostatic stress, i.e. it is made of slit-like pores (similar to cracks).

The significant anisotropy of Cobourg limestone is quantified through gas permeability measurements, with values varying between 10^{-16} and 10^{-21} m^2 (i.e. between 1nD and $100\mu\text{D}$) depending on the spatial direction considered.

The 1 nD order of magnitude is obtained with flow direction perpendicular to the bedding planes of Cobourg limestone.

A significant variability of Klinkenberg corrected gas permeability is observed for flow parallel to the bedding planes, with values ranging between $0.1\mu\text{D}$ (10^{-19} m^2) and $10\mu\text{D}$ (10^{-17} m^2). This range of variation over several decades cannot be explained by the error bar that we have estimated at around 20% (10% for the instruments and 10% for the Klinkenberg correction). The effect of stress is around a factor 2 for RWTH Aachen University (Figure 10) and 4 for Centrale Lille (Table 5); and cannot explain the differences.

This large range of permeability is attributed to the effect of heterogeneities and sample size. However, we did not found correlations with the measured permeabilities and sample size.

Coming back to the main purpose of the KCL study, that was the characterization of a nuclear repository site, the conclusion is that laboratory measurements are not at the same scale and can give results very far from field measurements with packers in the wells (that have also their own limitations).

If we want to draw a conclusion on the benchmark itself, with the purpose to compare and evaluate the equipments and interpretations of the four laboratories, it is obvious that the Cobourg limestone is too much heterogeneous and anisotropic to be a good material. The profession still needs this kind of benchmark. But it is difficult to find a good material.

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