Determining gas permeability in tight rocks: How do we know if we are obtaining the right value?

Carlos A. Grattoni, Samuel Allshorn, Quentin J. Fisher

University of Leeds, Leeds LS2 9JT, UK

ABSTRACT

The measurement of gas permeability for core plugs of conventional reservoirs, with values larger than 1 md, is a matter of routine in laboratories around the world. The methodology is well established and there are several core analysis companies that offer check plugs for calibrating equipment and validating the methodology used. However, the measurement on tight rocks is more challenging, the results from different laboratories often do not agree, and there are large number of papers comparing different techniques. Additionally, there are a range of mechanisms that might affect these measurements such as: slippage, Knudsen flow, inertial effects, net stress, etc. The permeability of shales determined by different laboratories can vary up to three orders of magnitude. The determination of gas permeability in tight rocks is fraught with difficulties, thus inducing large uncertainty in their values and the mechanisms involved.

To reduce this uncertainty we have designed and built standards based on fundamental flow through capillaries following a concept presented by Sinha et al. [1]. We used small diameter glass and fused silica capillaries embedded in PVC cylinders of 38mm diameter and ~ 50 mm length. The permeability of these standards can be theoretically calculated using Hagen–Poiseuille equation for laminar flow. The permeability of our set of standards range from 5.9 mD to 11 nanoD. The gas permeability of these standards was measured both with steady-state and pulse-decay methods. The Klinkenberg corrected permeability agreed very well with the theoretical value within the tolerance of manufacture of the capillaries. The experimental results and theoretical model also allows us to correlate the Klinkenberg slip factor with the mean free path and the radius of the capillaries for the whole range.

Once the validity of the standards was verified a second more robust set was built that includes a pore volume. These have been successfully tested under a range of setups and methodologies. These standards are also useful to check the experimental setups are leak free or determine their lower limit of measurement.

The standards have allowed us to compare different methodologies used for tight rocks and verify the limits of our permeability apparatus reducing the measurement uncertainty. This allows us to separate sample behaviour from measurement technique and improve our understanding of the mechanisms of gas flow in tight rocks.

INTRODUCTION

The determination of gas permeability by steady state on core plugs of conventional reservoirs are part of the routine core analysis. However, their measurement on unconventional low permeability reservoirs is not so well established, and fraught with difficulties, as traditional methods such as steady state or pulse decay can take long time and are prone to larger errors. The importance of sample preparation, selecting test procedures, adequate sensors and applying the necessary corrections for compressibility, inertial effects, stress, etc. are extremely important to obtain reliable and realistic data.

Recently there have been many papers that have brought controversy on the determination of the gas permeability from low permeability rocks as porosity and gas permeability determined by different laboratories often do not agree, which makes it very difficult to compare fields and evaluate their potential. In addition to the discrepancies between laboratories, many publications have criticized industry protocols on various grounds. There are three areas of argument, namely sample preparation, measurement techniques and interpretation and gas flow mechanisms.

Comparing the different methodologies used to determine gas permeability and their interpretation is usually difficult and sample dependent. Jannot et al. [2] performed a theoretical study to determine the best conditions under which precise estimations of both Kinkenberg permeability and slip factor can be obtained by steady and unsteady state methods and gave a series of recommendations and optimal parameters. They concluded that the draw-down experiment is the optimal configuration to estimate both Klinkenberg parameters in a single test. Wang and Knabe [3] proposed a pore oscillation method for tight gas sandstones and compared it with steady state measurements on 3 sets of samples and found a good agreement. Passey et al. [4] after doing a Round Robin concluded that shale gas permeability determined by different laboratories can vary up to three orders of magnitude. Gas permeability values often vary dramatically depending upon which laboratory made the measurements and which experimental protocol was used. On the other hand, Profice et al. [5] compared the permeability from Step Decay, Pulse Decay and steady-state and the Klinkenberg slip factor from some of these methods. In homogeneous samples of pyrophyllite and shales all methods produced similar results. They also did a Round Robin and concluded that comparable results can be obtained by different laboratories using their own techniques and interpretative procedures, provided that sample preparation is carefully defined. They also stated that Darcy's law and Klinkenberg's phenomena remain valid when modelling gas flows in low permeability rocks.

To increase understanding of the causes of the discrepancies between laboratories Fisher et al [6] have also undertaken an extensive testing program on a range of shale samples including a round-robin test in which six samples were analysed by service companies. The gas permeability on core plugs showed up to two orders of magnitude difference between laboratories. Some correlations were observed between laboratories. When the core plug is modelled as a dual permeability (fracture-matrix) the fracture permeability obtained was of a similar magnitude to the measurements provided by some service companies and the matrix permeability was of a similar order of magnitude to that obtained from the crushed shale measurements. This may indicate that the values routinely provided on shale core plugs may reflect core damage and not the intrinsic properties of the shale matrix.

In general, there is a large uncertainty in gas permeability and the mechanisms involved. This is due to the lack of a standardised methodology and reference standards to determine gas permeability in the lower permeability range. In order to reduce this uncertainty we have been searching for standards with a permeability range of similar to tight rocks, and tried several materials/methods of construction. In this paper we describe a set of standards developed on basic principles and theoretical ground. We have used them to verify and calibrate experimental setups and verify the methods of interpretation.

Background

Flow through straight capillaries

The Hagen–Poiseuille or Poiseuille equation describes the laminar flow through a capillary of constant circular section. It assumes that the fluid is incompressible and Newtonian. Additionally, the capillary length, *l*, needs to be longer than its radius, *r*, as the equation does not hold close at the capillary entrance. It can be expressed as:

$$Q = \frac{\pi \, r^4 \, \Delta P}{8 \, \mu \, l} \tag{1}$$

Where Q is the volumetric flow rate, ΔP is the pressure drop along the capillary, μ is the dynamic viscosity.

For a compressible fluid the linear velocity is not constant and the flow rate is usually expressed at ambient pressure, P_a. When the temperature of the fluid is constant (isothermal flow), and when the pressure difference between ends of the capillary is small, the volumetric flow rate at the outlet is approximated by:

$$Q = \frac{\pi \, r^4}{16 \, \mu \, l} \left(\frac{{P_i}^2 - {P_o}^2}{P_a} \right) \tag{2}$$

Where, P_i is the inlet pressure, P_o is the outlet pressure. This is mainly applicable to gas flow through short capillaries.

Slippage

The Klinkenberg or slip effect is important when the tangential speed of the gas in the boundary of the wall becomes nonzero. The mean free path of the gas molecules varies with its pressure; as a result, the slippage is a function of the mean free path, λ, and the dimension of the pore, ***r***. The ratio ***λ/r*** is also known as Knudsen number, *Kn*.

When there is slippage the gas permeability of a rock is greater than its absolute permeability. Klinkenberg proposed a linear correlation between the measured gas permeability and the absolute permeability:

$$K_g = K_g{}^{\infty}\left(1 + \frac{b}{P_m}\right) \quad (3)$$

Where; $\boldsymbol{K_g}$ is the gas permeability at the mean pressure $\boldsymbol{P_m}$, $\boldsymbol{Kg}^{\infty}$ is the absolute permeability (also known as the Klinkenberg's corrected permeability), and ***b*** is the slip factor. When ***b*** = 0, the flow is Darcy's flow. The slip factor depends on the molecule's mean free path ($\boldsymbol{\lambda}$), the throat radius ($\boldsymbol{r}$), and the mean pressure ($\boldsymbol{P_m}$), [7]:

$$b = \frac{4c\,\lambda}{r} P_m \quad (4)$$

where ***c*** is a constant which is usually assumed to be close to one. Slip also occurs for gas flow through capillaries and the link between capillary flow and Darcy's permeability is described in next section.

Materials and methods

Permeability standards

The concept presented by Sinha et al. [1] is simple; if a small cylindrical capillary is created in a core plug matrix, which is non-porous and impervious, then the permeability of core plug (K) can be calculated. The reference plug permeability can be obtained by combining Poiseuille's equation and Darcy's law;

$$K = \frac{r^4 L}{8\,R^2 l} \quad (5)$$

where, R and L are the radius and length of the reference plug. For gas flow through a reference plug and a capillary, the effect of compressibility and gas slippage still need to be included.

We have designed and built standards based on Sinha's concept. The standards consist of small diameter glass or fused silica capillaries embedded in PVC cylinders of 38mm diameter and 50 mm length. The internal diameter of circular capillaries ranged from 5 to 75 microns. One rectangular cross section capillary of 50 x500 microns was also built and tested. The initial set of standards built had an extremely small pore volume and were very fragile, now we have improved the design which has a pore volume comparable with natural rocks and is more robust. The reference plug has an internal gas filled volume connected through the capillary to the inlet, which represents the pore volume of the plug. A schematic and pictures of the reference plugs are shown in Figure 1. The porosity of the plugs was determined with a traditional helium porosimeter.

Permeameters

Two gas permeability setups and interpretation methods were used to experimentally determine the gas permeability of the reference plugs:

1) CoreLabs PDP200 pulse decay permeameter with proprietary software. The 200 PDP can measure semi-automatically gas permeabilities in the range 0.1 mD to 10 nD. The equipment has core holders for 1 in and 1.5 in cores and was originally designed to operate at pore pressures of 1200 psi and confining pressures of up to 2000 psi. This set-up have been redesigned so that it can also do steady-state tests when the permeability of samples is over 0.1 mD and can operate at a confining pressure at up to 5,000 psi.

Figure 1. Photos and schematic of the reference plugs of 38mm diameter; left: first set; centre: second set; and right: schematic of the second set.

2) We have recently built and commissioned a gas permeameter (Wolfson Transient Permeameter, WTP) based on the instrument described by Cui et al. [8] with the addition of a pressure transducer in the downstream side. It is capable of making measurements using transient pressure pulses for ultralow permeability rocks and a schematic set up is shown in Figure 2. The valves of the set-up are all manual allowing great flexibility of operation and the pressures are automatically logged. It can be used with gas up to 1000 psi and confining pressure of 5000 psi. The gas permeability can be obtained from the pressures as a function of time, the upstream and downstream volumes and modelled using CYDAR or Eclipse software. The pore volume can be used as an input or obtained during the history matching of the pressures. In order to include the slip factor several tests are recommended similar to the Step Decay [9].

Methodology

Each reference plug was tested a constant confining pressure as they are not stress sensitive and a range of pore pressures in order to evaluate the slip factor and the absolute permeability. All the tests were performed with helium at ambient temperature. The reference plugs with permeability higher than 0.1 mD, first set of reference plugs, were tested using steady state in the PDP200 using helium and Omega FMA gas flow meters. The plugs with lower permeability were tested with the pulse-decay method in the PDP200 and the WPT.

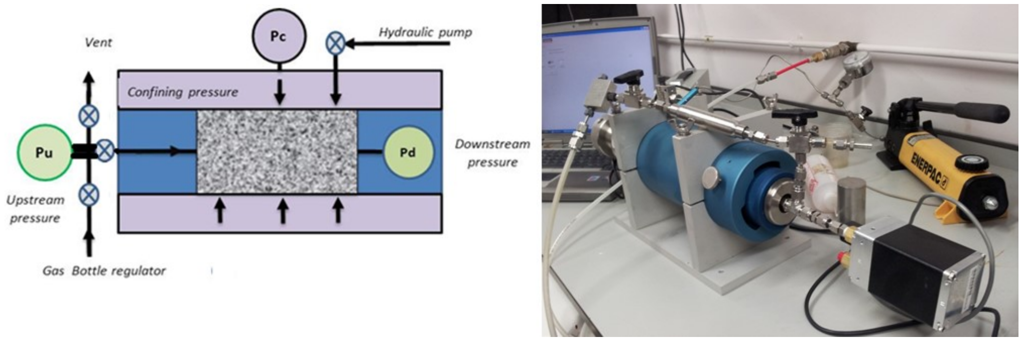

Figure 2. Schematic and Photo of the WPT set-up.

RESULTS

An example of gas permeability results from the first set of reference plugs measured under steady-state (75 microns) and pulse-decay (25 microns) are shown in Figure 3.

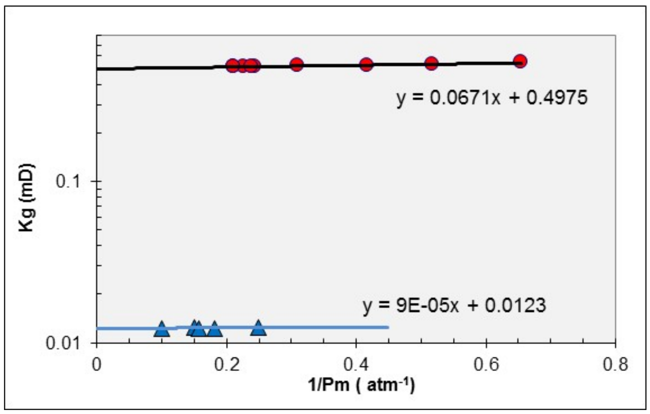

Figure 3. Gas permeability as a function of the inverse of mean pressure to obtain the absolute permeability and slip factor. The highest permeability corresponds to a reference plug with a 75 micron capillary and the lowest is for a 25 microns capillary.

The Klinkenberg corrected permeability, determined as shown in Figure 3, was compared with the theoretical value for incompressible flow and for both sets of plugs they agree extremely well, as shown in Figure 4. The small discrepancies are within tolerances of the capillaries and errors induced by the extrapolation to infinite pressure. The point that appears as an outlier in the left plot is a capillary with rectangular cross sectional area (50*500 microns) and its equivalent diameter (91 microns) was calculated with the hydraulic radius approximation. The agreement between measured and theoretical permeability for this capillary is extremely good even if does not follow the trend for circular capillaries. The absolute permeabilities obtained using different experimental setups showed a good agreement between them and with the theoretical values, validating not only the setups but also the methodology for obtaining the permeability from pressures vs. time.

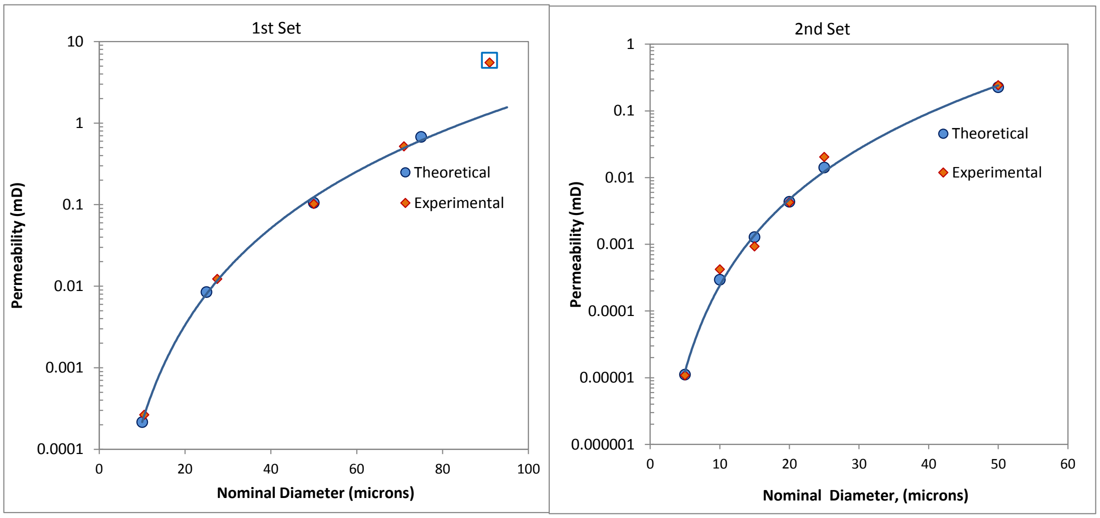

Figure 4. Theoretical and measured absolute permeability as a function of the diameter of the capillary. The solid line is the best fit through the experimental data using a power law function. The graph on the left presents a capillary that appears as an outlier because it has non-circular cross sectional area.

All the characteristics of the second, improved, set of reference plugs and their theoretical absolute permeability are shown in Table 1. The values of the experimentally obtained slip factors (*b*) seem not to have a correlation with the permeability. Based on its definition the slip factor should be inversely proportional to capillary size. Therefore, the link between mean free path, capillary radius and mean pressure was studied in more detail.

Table 1. Characteristics of the improved set of reference plugs, including their experimental and theoretical absolute permeability.

EXPERIMENTAL						**THEORY**		
ID	Diameter cm	Length cm	Porosity %	K klink.	b (1/psia)	Nominal size microns	Tolerance in size+/- microns	K Plug mD
50	3.78	5.13	2.50	0.242	17.89	50	5	*0.225*
25	3.80	5.11	2.94	0.020	6.22	25	5	*0.014*
20	3.78	5.14	2.49	0.0043	7.18	20	2	*0.0043*
15	3.79	5.14	2.81	0.00093	8.21	15	2	*0.00128*
10	3.78	5.14	2.82	0.00042	9.58	10	2	*0.00029*
5	3.78	5.15	2.31	0.000011	14.37	5	2	*0.000011*

The mean free path for helium was calculated using a spreadsheet available, to download, from the web from Prince George's Community College [10]. The values of mean free path for helium agree well with data from literature. Then for a given capillary diameter the absolute permeability can be calculated with Eqn. 5 and slip factor and apparent permeability from Eqn. 4 and 3 respectively, thus the only variable is the mean pressure which controls the mean free path. According to Klinkenberg the value of the constant C of Equation 4 should be slightly less than one [11]. However, it became immediately apparent that *C* is not a general constant and could not be unity as normally assumed. An example of the experimental and theoretical permeability for a 50 microns capillary with *C*=1 is shown in Figure 5. For all the capillaries the apparent calculated permeability at different mean pressures with *C*=1 underestimates the pressure dependence.

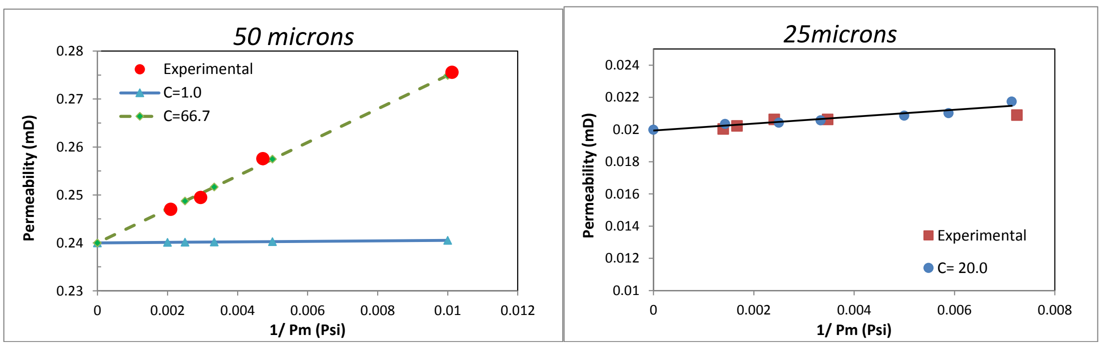

Figure 5. Gas permeability as a function of inverse of mean pressure. Experimental values and theoretical using different values of *C* in Equation 4.

If the value of C is used as the only adjustable parameter to fit the experimental data it was observed that its value of is proportional to the size of the capillary. The figure 5 shows the best fit of the experimental data for capillaries of 50 and 25 microns. A summary of the results for all the capillaries are plotted in Figure 6. For the capillary of 5 microns the value of C becomes 2.5, indicating that it may become unity for a diameter of 1-2 microns.

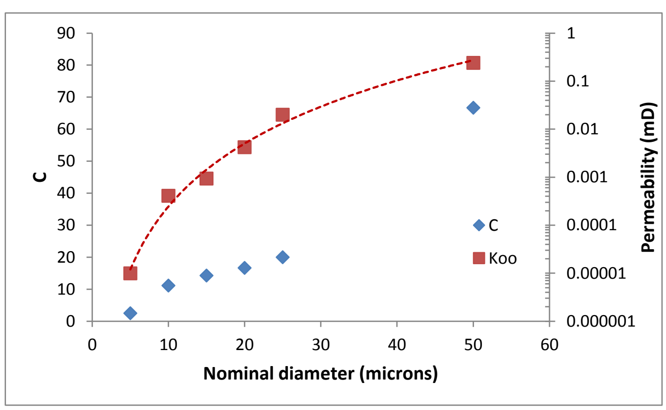

Figure 6. Results of the absolute permeability and constant C that produces the best match between theoretical and experimental data. The dotted line is the best fit through the permeability data using a power law function.

SUMMARY

In order to reduce the uncertainty of gas permeability measurements of low permeability rocks we have designed and built reference plugs based on fundamental flow through capillaries. These plugs also include an internal pore volume which plays an important role in the interpretation of transient methods. The reference plugs have been successfully tested under a range of pressures using different setups and methodologies. These standards are also useful for regularly checking whether the experimental setups are leak free or to determine their lower limit of measurement.

The Klinkenberg corrected permeability for our reference plugs ranges from 5.9 mDarcy to 11 nanoDarcy. The gas permeability of these standards was measured both with steady-state and transient pressure methods. Measurements were made at various gas pressures and the results highlight the importance of properly accounting for gas slippage.

The theoretical permeability of these standards can be calculated using Hagen–Poiseuille equation for laminar flow. The Klinkenberg or theoretical absolute permeability agrees very well within the tolerance of manufacture of the capillaries.

The experimental results and theoretical model also allows to correlate the Klinkenberg slip factor with the mean free path and the radius of the capillaries for the whole range of capillaries used. It was found that in order to match the theoretical slip factor the value of the constant C must be a function of the capillary size and not unity as normally assumed.

The standards have allowed us to verify our permeability apparatus significantly reducing measurement uncertainty. This allows us to separate sample behaviour from measurement technique and improve our understanding of the mechanisms of gas flow in tight rocks. In most cases the gas permeability obtained in the Laboratory is not representative of the reservoir due to sample preparation, damage (fractures and cracks), small scale heterogeneities or stress effects.

ACKNOWLEDGMENTS

We would like to thank the sponsors of PETGAS and SHAPE for funding the Wolfson Multiphase Flow Laboratory of the University of Leeds.

REFERENCES

1. Sinha S., Braun E.M., Passey Q.R., Leonardi S.A., Wood A.C, Zirkle T., Boros J.A., Kudva R.A. ”Advances in measurement standards and flow properties measurements for tight rocks such as shales” (2012) SPE 152257. SPE/EAGE European Unconventional Resources Conference and exhibition, Vienna, Austria.

2. Jannot Y., Lasseux D., Vizé G., Hamon G.,"A detailed analysis of permeability and Klinkenberg coefficient estimations from unsteady-state, pulse-decay or draw-down experiments" (2007). SCA2007-08, International Symposium of the Society of Core Analysts held in Calgary, Canada.

3. Wang Y, Knabe R.J. "Permeability characterization of tight gas samples using pore pressure oscillation method" (2010), SCA2010-30, International Symposium of the Society of Core Analysts, Halifax, Nova Scotia, Canada.

4. Passey, Q.R., Bohacs, K.M., Esch, W.L., Klimentidas, R. and Sinha, S., "From oil-prone source rock to gas producing reservoir – Geologic and petrophysical characterization of unconventional shale-gas reservoirs" (2010) Paper SPE-131350, International Oil and Gas Conference and Exhibition, Beijing, China.

5. Profice S., Hamon G., Nicot B.,"Low permeability: Insights" (2015) SCA2015-021 International Symposium of the Society of Core Analysts, St. Johns Newfoundland and Labrador, Canada.

6. Fisher Q.J., Grattoni C.A., Rybalcenko K., Lorinczi P., Leeftink T.," Laboratory measurements of porosity and permeability of shale" (2016), Mo SWS 01, Fifth EAGE Shale Workshop, Catania, Italy.

7. Javadpour, F., Fisher, D., Unsworth, M., "Nanoscale gas flow in shale gas sediments" J. Can. Petrol. Technol. (2007), **46**(10), 55–61.

8. Cui X. A., Brezovski R., Nassichuk B., Glover K., Pathi V.," A new method to simultaneously measure In-Situ permeability and porosity under reservoir conditions: Implications for characterization of unconventional gas reservoirs" (2010), CSUG/SPE-138148-MS, Canadian Unconventional Resources and International Petroleum Conference, Calgary, Canada.

9. Lasseux, D., Jannot, Y., Profice, S., Mallet, M., Hamon, G., "The 'Step Decay': A new transient method for the simultaneous determination of intrinsic permeability, Klinkenberg coefficient and porosity on very tight rocks" (2012), SCA2012-25, International Symposium of the Society of Core Analysts, Aberdeen, Scotland.

10. Prince George's Community College, Department of Physical Sciences and Engineering, Mean free path calculator (March 2016): www.Academic.pgcc.edu/~ssinex/excelets/mean_free_path.xls

11. Klinkenberg L.J. "The permeability of porous media to liquids and gases" (1941) API Drilling and Production Practice, 200 213.