Low Permeability Rocks:
Validation of Routine Core Properties

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ABSTRACT

There are several industry-accepted methods to obtain gas permeability of core plugs. The most commonly used method is the steady-state method. This method allows correction for Klinkenberg gas slippage and Forchheimer inertial effects. Alternatively, gas permeability is determined by the use of unsteady-state approaches, which include pressure falloff and pulse-decay methods. As the pulse-decay method is dedicated to permeability in the low range only, it is not discussed in this paper.

When comparing steady-state and pressure falloff approaches, the pressure falloff method is of interest because it offers shorter turnaround time and the ability to determine gas slippage-corrected permeability values in a single measurement. Another advantage of the method is that porosity and permeability measurements can be executed in one single automated run using specific apparatus. As a consequence, both measurements are completed by using the same gas, which is helium. At low permeability, while the apparent permeability measured with helium is higher relative to nitrogen apparent permeability, once it is corrected for gas slippage, both helium and nitrogen Klinkenberg permeability values should converge.

In this study, a set of various outcrop samples with known porosity and Klinkenberg permeability were investigated with the different measurement methods. The reference permeabilities were determined via four-point steady-state gas permeability with nitrogen. The same samples were then tested using the pressure falloff technique with injection of helium. Based on the Thomas and Pugh acceptance criteria, the helium Klinkenberg permeability was compared to the reference nitrogen steady-state Klinkenberg permeability. If the majority of the data results showed good agreement between the two methods, the helium permeability values for low permeability samples (< 1 mD) were found to be outside of the acceptance criteria. Re-measuring the sub-set of "out-of-acceptance-criteria" samples with nitrogen, the pressure falloff Klinkenberg permeability values decreased, falling within acceptable accuracy boundaries, but always slightly larger than the nitrogen steady-state reference values. According to the observations and in the frame of understanding the permeability difference, several hypotheses for further investigations are discussed.

INTRODUCTION

Reservoir porosity and permeability are parameters of importance in petroleum engineering. While porosity describes the reservoir storage capacity, permeability quantifies the ability of a rock to allow a fluid to pass through it.

Since 1856 when Darcy [1] defined fluid conductivity of a porous material in his famous technical report, permeability has become one of the most studied rock properties. In his study, Darcy states that flow rate is directly proportional to the pressure gradient, which is described by the Stokes' equation at the pore scale. But the viscous flows or Stokes' flows are a limited case. At higher flux for instance, i.e. when the dimensionless Reynolds' number becomes greater than 1, the inertial energy dissipation induced by the molecule accelerations in the tortuous porous network leads to higher pressure gradient than the one predicted by Darcy's law, as shown by Forchheimer [2]. The Forchheimer inertial resistance is very pronounced in high-permeability samples, but almost negligible in low-permeability samples. Unlike for inertial flow, the gas slippage effect known as the Klinkenberg effect [3] occurs in samples of low permeability. This effect becomes significant when pore size is comparable to the mean free path of the flowing gas, which depends solely on pore pressure in case of isothermal gas flow. Muskat [4] was the first to report that a significant discrepancy between gas permeability and water permeability can occur during experiments on the same sample. Klinkenberg showed that this difference was due to the invalidity of Darcy's law in a certain range of pressure. This phenomenon can be more or less pronounced according to the type of gas and the type of experimental tests - steady-state or unsteady-state approach.

The primary objective of this paper was to demonstrate that Klinkenberg permeability values derived from the unsteady-state pressure falloff and the steady-state method are equivalent for samples with permeability ranging from 10,000 mD down to 0.001 mD. Using 18 core plugs of various rock types, the reference Klinkenberg permeability (determined via the four-point steady-state method using nitrogen) were compared with permeability values obtained from the pressure falloff method using helium. Helium is traditionally the preferred gas when using the pressure falloff method because pore volume can be initially determined in the same apparatus, and both porosity and permeability can be measured during a single automated run. Helium and nitrogen Klinkenberg permeability values agree to each other for samples with Klinkenberg permeability greater than 1 mD, despite use of different measurement methods. This does not apply for samples of permeability less than 1 mD. All samples with Klinkenberg permeability below 1 mD were re-measured with nitrogen using the pressure falloff technique. Results of this testing led to further ongoing investigations based on several hypotheses that are provided in this paper.

BACKGROUND

Assuming an isotropic porous medium with unidirectional and horizontal flow and isothermal condition, the original Darcy's law for compressible gas corrected for inertial effect is described by Equation 1:

$$\frac{P_1^2 - P_2^2}{2P_q L} = \frac{\mu V}{K_g} + \beta \rho V^2 \tag{1}$$

where P_1 and P_2 are the upstream and downstream pressures, respectively, P_q is the pressure at which the gas flow rate is measured, L is the length of the porous material, μ is the gas viscosity, V is the gas velocity, K_g is the gas permeability, β is the inertial factor, and ρ is the gas density.

Even when inertial effects are taken into account, the gas permeability is still dependent on the mean free path of the flowing gas, due to the gas slippage phenomenon. Klinkenberg noted that mean free path λ of gas is inversely proportional to gas pressure P:

$$\frac{4c\lambda}{r} = \frac{b}{P} \tag{2}$$

where the constant c is slightly less than 1 [2] and b is referred to as the gas slippage factor, partly a rock property and partly a gas property. Experimentally, this has an effect on the measured apparent measured permeability and leads to a relation between the Klinkenberg corrected permeability K_{inf} at high pressure, which is equivalent to the liquid permeability, and the apparent gas permeability K_g:

$$K_g = K_{inf}\left(1 + \frac{4c\lambda}{r}\right) \tag{3}$$

Combining equations (2) and (3), the Klinkenberg relationship is obtained:

$$K_g = K_{inf}\left(1 + \frac{b}{P}\right) \tag{4}$$

Klinkenberg demonstrated that the equivalent liquid permeability K_{inf} could be extrapolated by plotting gas permeability against inverse mean pressure, the intersection of the slope extrapolated to infinite mean pressure results in the gas slippage-corrected permeability. This method is known as the common steady-state method. It requires several measurements at varying mean pressure (four-points in this study). For low-permeability rocks, this test can be time consuming, as it requires long time to achieve pressure equilibrium (relaxation time is proportional to the square of the sample size).

Other unsteady-state methods based on the analysis of the transient pressure signal are also available and are known as pulse decay or pressure falloff methods. The pulse decay method is not discussed in this paper as it is dedicated to permeability range from 10 ηD to 0.1 mD only. The pressure falloff method, capable of measuring permeability in the range between 0.001 mD and 10 D, is characterized as an upstream gas reservoir that is brought to constant pressure (about 200 psi for this study). The pressured gas is then released to one end of the sample and vented to atmospheric pressure at the other end of the sample. The transient pressure signal is recorded versus time. A single pressure transducer is needed to record the pressure decrease in the upstream reservoir. The main advantages of pressure falloff over steady-state method is the considerable reduction in test duration since one single transient pressure signal is required to obtain the Klinkenberg permeability, the slippage factor b and the inertial factor β.

The equations and iterative scheme used to describe the pressure falloff method are quite complicated in comparison to the steady-state method. We refer you to the paper by Jones [6] and the Recommended Practice 40 published by the American Petroleum Institute, API RP40 [5] for a detailed description of the iterative workflow.

EXPERIMENTAL PROTOCOL

The reference measurements were performed on eighteen core plugs of different rock types. They were tested for porosity and nitrogen steady-state Klinkenberg permeability using 4-point measurements. Bulk volume was determined by the summation of ambient grain volume and pore volume obtained at 800 psi confining pressure using Boyle’s law. Porosity was calculated from the calculated bulk volume and pore volume. Finally, the steady-state nitrogen Klinkenberg permeability was measured at the same 800 psi confining pressure.

A second series of measurements was then performed on the same 18 samples using the pressure falloff system. Both pore volume and Klinkenberg permeability were measured using helium at 800 psi confining pressure. Data results were then compared to the reference measurements from the first series.

Each measurement was then validated against the experienced-based acceptance criteria from Thomas and Pugh [7] as indicated in Table 1. As there is no acceptance criteria for permeability values less than 0.01 mD, a relative error of ± 35% was used.

Table 1: Core properties and data accuracy acceptance criteria applied.

Parameter Measured	Experience-based criteria	Statistically derived criteria
Core Porosity	± 0.5 p.u.	± 0.5 p.u.
Gas Permeability (from Thomas and Pugh [7])	< 0.01 mD: N/A, ± 35% in this paper 0.01 … 0.1 mD: ± 30 % 0.1 … 1 mD: ± 25% 1 … 50 mD: ± 15% 50 … 1,000 mD: ± 15%	< 0.01 mD: N/A 0.01 … 0.1 mD: ± 21% 0.1 … 1 mD: ± 21% 1 … 50 mD: ± 13% 50 … 1,000 mD: ± 8%

Note that quality assurance and quality controls were daily performed on the equipment: these included pore volume measurement of metallic cylinders with inner holes of different diameters, and permeability measurements of sintered metallic standards covering a wide range of helium permeability. Every fifth sample was rerun for porosity and permeability repeatability. An automatic leak test was also performed while measuring pore volume and permeability to helium, as both properties are measured during a single automated run.

EXPERIMENTAL RESULTS

Porosity ϕ was first determined on eighteen samples of different rock types. Porosity values were found to be within the acceptance boundaries (Figure 1).

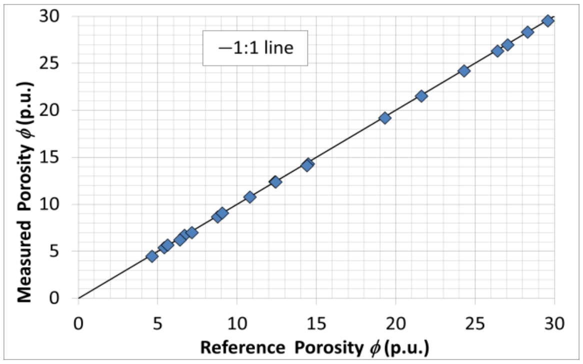

Figure 1: Cross-plots of reference data versus measured data for porosity.

Porosity data results are not discussed in the following results section: the quality of data did not raise any concerns to question further observations.

The unsteady-state pressure falloff Klinkenberg permeability was then measured on the same eighteen samples using helium at 800 psi of confining pressure.

Figure 2 presents the helium Klinkenberg permeability cross-plot between steady-state $K_{inf}[N_2\text{-ss}]$ reference values (X axis) and pressure falloff $K_{inf}[H_e\text{-pf}]$ measured values (Y axis). The Klinkenberg permeability relative errors and the acceptance criteria (Table 1) are also plotted.

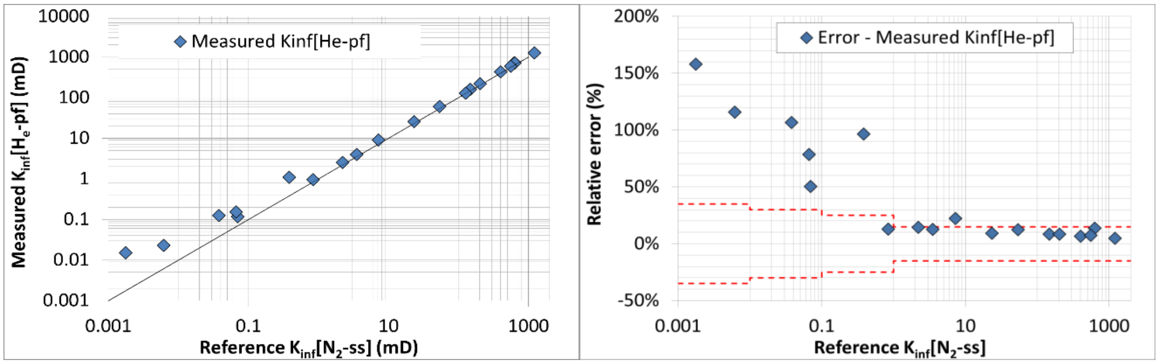

Figure 2: Cross-plots of helium Klinkenberg permeability vs. reference permeability (left) and associated plot showing data in relation to accuracy acceptance (right).

When measured with helium, the pressure falloff Klinkenberg permeability K_{inf}[He-pf] values for the majority of samples with less than 1 mD permeability fall outside of the defined accuracy limits, demonstrating higher permeability values relative to reference values. The trend shows increasing relative error as sample permeability decreases.

All out-of-acceptance-criteria samples were rerun using nitrogen. Figure 3 on the left presents the cross-plot of measured pressure falloff K_{inf}[N_2-pf]-K_{inf}[H_e-pf] (Y axis) and reference steady-state K_{inf}[N_2-ss] values (X axis). The associated Klinkenberg permeability errors based on Table 1 are also plotted on the right side of Figure 3.

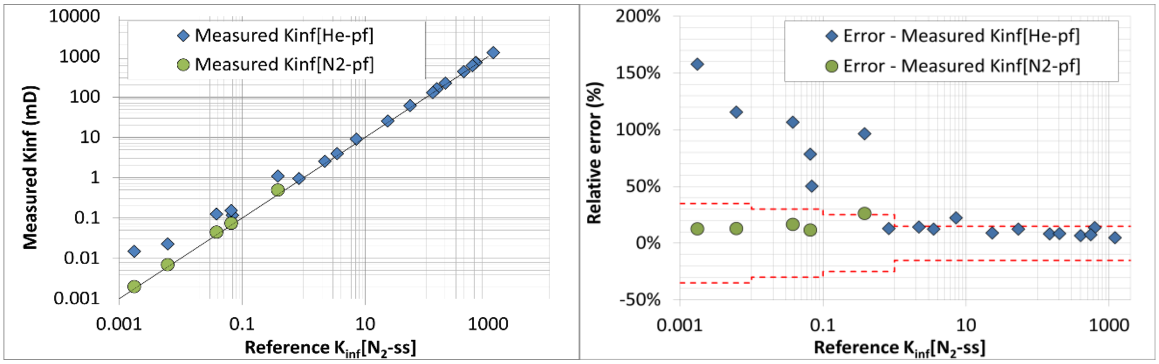

Figure 3: Klinkenberg permeability cross-plot and acceptance criteria plot (including five samples re-measured with nitrogen).

The re-measured nitrogen Klinkenberg permeability values decreased, plotting within the acceptable accuracy boundaries. These data show that pressure falloff Klinkenberg permeability is higher when using helium vs. nitrogen, especially in the range of 0.001 to 1 mD (K_{inf}[H_e-pf] > K_{inf}[N_2-pf]. Figure 3 also shows a slight but consistent overestimation of the nitrogen Klinkenberg-corrected permeability when using the pressure falloff method compared to the nitrogen Klinkenberg permeability using the steady-state approach (K_{inf}[N_2-pf] > K_{inf}[N_2-ss]).

DISCUSSION

This section is focused on summarizing the experimental observations and providing the possible reasons of the permeability difference on samples with Klinkenberg permeability less than 1 mD. The main observations from the study are:

- Observation 1, K_{inf}[N_2-pf] > K_{inf}[N_2-ss]

The use of nitrogen and the pressure falloff method to measure Klinkenberg permeability K_{inf}[N_2-pf] of low-permeability samples (<1 mD) resulted in overestimated values with respect to nitrogen steady-state permeability K_{inf}[N_2-ss]. The discrepancy increased systematically with decreasing permeability. This was also observed by Rushing *et al.* [8].

- Observation 2, K_{inf}[H_e-pf] > K_{inf}[N_2-pf]

In the low permeability samples, the use of helium in the pressure falloff method resulted in helium Klinkenberg permeability K_{inf}[He-pf] values that were higher than the nitrogen Klinkenberg permeability K_{inf}[N_2-pf] values, with the difference again increasing as permeability decreased. Rushing *et al.* [8] reported the different observation while using the steady-state approach measured with back pressure ($K_{inf}[H_e\text{-ss}] \sim K_{inf}[N_2\text{-ss}]$).

The following discussion provides hypotheses for further investigations.

- Hypothesis for $K_{inf}[N_2\text{-pf}] > K_{inf}[N_2\text{-ss}]$:

The use of two different methods and models could explain the observed permeability discrepancy. According to Rushing *et al.* [8], observation 1) could be attributed to fundamental problem with the unsteady-state methodology. Lenormand *et al.* [10] suggested that the overestimation of permeability during the transient experiments, compared to the steady-state approach, could be due to the analytical integration process and the non-linear relationship between the uniform apparent gas permeability and the average pressure.

Another idea for future investigation is to test different flow regimes and related temperature variations. If the isothermal condition is violated, the balance between gas heat capacity, thermal diffusivity, adiabatic expansion, and the Joule-Thomson effect, according to the selected gas should be investigated, especially at the low permeability range (<1 mD) while applying several hundred psi of pressure release during the pressure falloff tests.

Gas molecule adsorption is another effect potentially contributing to lower the nitrogen Klinkenberg permeability under assumed isothermal conditions. Nitrogen adsorption onto the rock surface during the long term steady-state measurement could be more pronounced compared to a short time nitrogen exposure during the pressure falloff measurements.

- Hypothesis for $K_{inf}[H_e\text{-pf}] > K_{inf}[N_2\text{-pf}]$

Below 1 mD permeability, gas slippage effect is known to be more pronounced for helium than for nitrogen when measuring the apparent gas permeability. For the intrinsic Klinkenberg permeability, this should not be an issue [8]. Nevertheless, a consistently higher helium Klinkenberg permeability during pressure falloff tests is observed in this study. A possible "leakage" was discarded since the experimental system successfully passed the leak tests before and after each measurement.

While the iterative scheme used to solve the pressure falloff permeability can perhaps lead to this deviation, the nitrogen adsorption effect can also explain the reduction of permeability with pore size reduction due to nitrogen adsorption.

CONCLUSIONS AND RECOMMENDATIONS

Eighteen core plugs were tested for porosity and Klinkenberg permeability using the pressure falloff method. Data results were then compared with reference values established via numerous measurements and the steady-state approach for Klinkenberg permeability. Laboratory errors were calculated for each parameter. The measurement error was compared with accuracy criteria. Despite porosity data falling within the acceptance criteria, helium Klinkenberg permeability showed deviations exceeding the defined criteria in the low permeability range. The samples outside of the criteria acceptance limits were re-measured for pressure falloff Klinkenberg permeability using nitrogen. All non-acceptable values significantly decreased and plotted within the defined accuracy boundaries. Nevertheless, nitrogen pressure falloff Klinkenberg permeability was found to be consistently greater than the steady-state reference values. Reasons for the discrepancies are discussed in the paper. Although a lot of experimental studies have already been performed in the past by different laboratories and research institutions, additional works need to be done to understand the permeability differences. For instance, a new gas transport model which will include an adsorption parameter is currently being tested: gas adsorption phenomenon is suspected to contribute to lower the nitrogen Klinkenberg permeability under assumed isothermal conditions especially in the low permeability range. The influence of the rock type on the observed effect will also be a subject of future investigation.

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