



## Research paper

# Quantitative validation of pore structure characterization using gas slippage measurements by comparison with predictions from bundle of capillaries models



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## ABSTRACT

The use of gas slippage measurements as a tool for quantitative pore structure geometry characterization was investigated by comparing pore sizes estimated from gas slippage measurements to pore sizes predicted by bundle of capillaries models. A large data set of gas slippage measurements was generated for the study by combining measurements made in our laboratory with measurements from seven previously published studies. Pore size was estimated from the gas slippage measurements using two models, one assuming a circular cross sectional pore geometry and the other slot shaped. When using a porosity function generated for the data set using experimental data and a theoretically derived tortuosity function, pore sizes estimated from bundle of capillaries models yielded a good match to the pore sizes independently estimated from gas slippage data. Better agreement between the models exists when pores in high permeability rocks are modelled as having slot shaped cross sectional geometry and pores in low permeability rocks circular cross sectional geometry, suggesting a systematic shift in pore morphology with changing permeability.

## 1. Introduction

Characterizing pore structure geometry is fundamental to understanding and predicting many aspects of porous media behavior. Fully characterizing reservoir rock pore structure is expensive and technically challenging due to the geometrical complexity and small length scale of these media, and is impossible for low permeability reservoir rocks with pore structure length scales below the resolution limits of even the most advanced imaging techniques. Techniques based on simplified models are therefore required for practical applications. Here we investigate the quantitative validity of characterizing pore structure geometry using gas slippage measurements. Compared to other pore structure characterization techniques, such as mercury intrusion porosimetry and CO<sub>2</sub> and N<sub>2</sub> gas adsorption, the gas slippage technique has the advantageous attribute of being applicable to samples confined at in situ reservoir stress conditions (Letham and Bustin, 2016). This attribute is important because reservoir rock pore structures show varying degrees of stress sensitivity (McLatchie et al., 1958; Vairogs et al., 1971), and pores and pore throats can be significantly smaller at reservoir stress conditions than at ambient surface conditions. Using pore structure characterizations determined at ambient surface stress conditions as inputs for subsurface reservoir models can hence lead to inaccurate

predictions of reservoir behavior. It is therefore desirable to use pore structure characterizations determined at subsurface reservoir stress conditions as model inputs.

In this study we quantitatively validate the gas slippage technique, which is capable of pore structure characterizations at in situ stress, by comparing pore sizes estimated from gas slippage measurements with pore sizes independently estimated using bundle of capillaries models. The good match found between the techniques provides confidence that pore structure characterizations made using gas slippage measurements can be used as model inputs to understand and predict reservoir behavior. The findings of this study are particularly significant for the successful exploitation of shale gas and shale oil reservoirs that are typically highly stress sensitive and, by definition, have small-scale pore structures that are difficult to characterize with techniques other than gas slippage measurements.

## 2. Background

## 2.1. Gas slippage

In 1941, Klinkenberg published a seminal paper that showed significant pore pressure and gas species dependent permeability variation

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**List of symbols**

$b$	Klinkenberg's slip parameter (Pa)	$P$	gas pressure (Pa)
$C$	Adzumi's constant	$P_c$	confining pressure (Pa)
$d_{kin}$	kinetic diameter (m)	$P_p$	pore pressure (Pa)
$K_a$	apparent permeability (m <sup>2</sup> )	$R$	gas constant (J mol <sup>-1</sup> K <sup>-1</sup> )
$K_n$	Knudsen number	$r$	pore radius (m)
$K_\infty$	permeability intercept of Klinkenberg plot (m <sup>2</sup> )	$T$	temperature (K)
$L_c$	characteristic length (m)	$w$	slot width (m)
$M$	molar mass (kg mol <sup>-1</sup> )	$\lambda$	mean free path (m)
$N_A$	Avagadro's constant (mol <sup>-1</sup> )	$\mu$	viscosity (Pa * s)
		$\tau$	tortuosity
		$\Phi$	porosity

in conventional reservoir rocks (Klinkenberg, 1941). By varying pore pressure to systematically change mean free path (the average distance a molecule of the probing gas used for permeability measurements travels between two collisions with other gas molecules), he showed that the observed permeability variation was the result of gas slippage; linear relationships between permeability and inverse pore pressure were observed, which were predicted by slip theory because mean free path of a gas is proportional to the inverse of gas pressure. Permeability intercepts of permeability-inverse pore pressure plots, representative of infinite pore pressure and therefore zero mean free path and no gas slippage, were in agreement with liquid permeability measurements (Klinkenberg, 1941). These permeability intercepts were referred to as true permeability in Klinkenberg's paper, and have since been termed “slip-corrected” or “Klinkenberg-corrected” permeability.  $K_\infty$  is used herein to designate permeability from extrapolation to infinite pore pressure.

Gas slippage is a significant control on flow rate when flow takes place in the slip flow regime. The boundaries of the slip flow regime can be defined using the Knudsen number. The Knudsen number is the ratio of mean free path of a gas to the characteristic length scale of the pore structure through which it is flowing (Zhang et al., 2012)

$$K_n = \frac{\lambda}{L_c} \quad (1)$$

where  $\lambda$  is mean free path and  $L_c$  is characteristic length. Characteristic length is loosely defined in the literature because of the many different possible metrics for pore size (e.g. pore diameter, pore throat diameter). For calculating  $K_n$ , the important dimension of the pore structure is the length scale of the restrictions most responsible for limiting fluid flow. Characteristic length is therefore defined as such in this work.

The slip flow regime is defined as  $0.001 < K_n < 0.1$  (Zhang et al., 2012). When flow takes place in the slip flow regime, gas molecule-gas molecule collisions are far more frequent than gas molecule-pore wall collisions. However, gas molecule-pore wall collisions are frequent enough that the non-zero flow velocity at the pore walls is significant in comparison to the mean flow velocity (Landry et al., 2016). Hence gas slippage results in significantly  $\lambda$ -dependent permeability in the slip flow regime. In contrast to the Darcy flow regime ( $K_n < 0.001$ ), permeability is not a constant property of a given pore structure in the slip flow regime, and is therefore referred to as apparent permeability,  $K_a$ .  $K_a$  of a given pore structure is controlled by  $\lambda$ , which is dependent on temperature, pressure, and the kinetic diameter of the gas molecules (Loeb, 2004)

$$\lambda = \frac{RT}{\sqrt{2} \pi d_{kin}^2 N_A P} \quad (2)$$

where  $R$  is the gas constant,  $T$  is temperature,  $d_{kin}$  is the kinetic diameter of a molecule of the gas being considered,  $N_A$  is Avogadro's constant, and  $P$  is pressure.

## 2.2. Gas slippage measurements as a pore structure characterization technique

Klinkenberg developed an equation to predict  $K_a$  as a function of pore pressure

$$K_a = K_\infty \left( 1 + \frac{b}{P_p} \right) \quad (3)$$

where  $P_p$  is average pore pressure and  $b$  is a constant usually referred to as Klinkenberg's slippage factor. Calculating  $b$  is a way of quantifying gas slippage, as it is a measure of variation of  $K_a$  with respect to  $K_\infty$ .  $b$  is dependent on temperature and gas species (both of which determine  $\lambda$ ), as well as the size of the pores through which the gas is flowing. In Klinkenberg's theoretical derivation of Equation (3), he developed the following relationship for  $b$

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

where  $c$  is Adzumi's constant (0.9; Adzumi, 1937) and  $r$  is capillary radius under the assumption that flow paths can be represented as tubular shaped capillaries.

From Equation (4), by calculating  $b$  using experimental data (Equation (3)) and knowing the variables determining  $\lambda$  (Equation (2)), it is possible to calculate  $r$ . Hence gas slippage measurements can be used as a technique for characterizing pore structures. A single  $r$  value calculated from slippage measurements clearly does not completely characterize the pore structure geometry of a reservoir rock; pore structures are composites of a distribution of different sized pores and pore throats. The calculated  $r$  represents an average of the smallest pore throats along those flow paths responsible for the bulk of the fluid flux through the porous medium. These pore throats limit the fluid flux. Change in  $K_a$  relative to  $K_\infty$  due to gas slippage when mean free path is varied, which is quantified by measuring  $b$ , is therefore controlled by gas slippage at the walls of these smallest pore throats. Hence,  $r$ , which is calculated from  $b$ , is a measure of the size of these smallest pore throats, based on a simplified model that represents pores as cylindrical capillary tubes. Pore (throat) sizes calculated from gas slippage measurements will be referred to as “dominant” pore sizes herein.

That gas slippage measurements only result in the calculation of a dominant pore size is a limitation of the gas slippage technique as compared to techniques capable of measuring a distribution of different pore sizes (e.g. mercury intrusion porosimetry, CO<sub>2</sub> and N<sub>2</sub> adsorption, and imaging with scanning electron microscopy). However, a key attribute of gas slippage measurements as a pore structure characterization technique is that, because the dominant pore sizes are derived from permeability measurements, the technique allows direct characterization of the pore sizes relevant for fluid flow. Another advantage of gas slippage measurements as a pore structure characterization technique is the ability to easily make measurements at reservoir stress conditions. This is especially important for lower permeability, fine-grained reservoir rocks, which typically have more stress sensitive permeability than higher permeability, coarse-grained rocks (McLatchie et al., 1958;

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