1 **Permeability Measurements in Mudrocks**

2 **Using Gas-Expansion Methods on Plug and Crushed-Rock Samples**

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Abstract: Permeability is an important parameter relative to the production of hydrocarbons in shale oil/gas plays; however, the measurement of permeability in these nano- to microdarcy rocks remains a challenge. Results from different methods or from different laboratories are not consistent, and reasons are not fully understood. In the present study, permeability is measured for both plug and crushed-rock samples with different plug diameter or crushed-sample particle size to systematically investigate the permeability measurement to better understand and apply the measured results. A modified gas-expansion (MGE) method, which can measure permeability for plug samples under confining pressures, was established and applied to several Eagle Ford and Barnett Shale (mudrock) samples. Permeability results from this method are in fair agreement with those from the pulse-decay method. The traditional Gas Research Institute (GRI) method was applied to crushed-rock Eagle Ford Shale samples. The results were comparable to reported permeability for an Eagle Ford Shale sample. Particle or plug size has significant influence on permeability measurement. In general, permeability increases with increasing particle or plug size. For crushed sample with GRI method, the reason of increasing permeability is related to the limitation of the GRI technique and the data analysis method. Estimate of the permeability based on Kozeny-Carman Equation was conducted, and the results were used to evaluate the GRI permeability measurement. Particle size of 2-4 mm (5-10 meshes) is considered as an appropriate size for GRI permeability measurement. For plug sample, larger permeability with larger plug diameter is most likely caused by the artificial fractures. Higher confining pressure can reduce the influence of the fractures, but cannot fully remove it. A range of permeability, defined by the GRI permeability with 2-4 mm particles as the lower boundary and permeability of 1-in plug under high confining pressure (>5000 psi) as the upper boundary,

can be a more reliable measures to represent the shale matrix permeability. The range of the permeability also highlights the uncertainty in matrix permeability measurement for shale.

Keywords: Shale/mudrock, matrix permeability, size effect

1. Introduction

Shale gas and oil have changed the energy outlook around the world due to their enormous production potential. Permeability is one of the most critical parameters of this type of unconventional reservoir, controlling the production capability and hence determining the economic viability of the unconventional play. Various methods and techniques—such as the Gas Research Institute (GRI) technique (e.g., Luffel and Hopkins, 1993; Guidry et al., 1995; Cui et al., 2009; Tinni et al., 2012) and pulse-decay method (Brace et al., 1968; Jones, 1997; Bustin et al., 2008; Heller et al., 2014)—have been applied to permeability measurement in low-permeability mudrock (or shale for simplicity) samples. A steady-state method calibrated with low-permeability standards was also applied to mudrocks by Sinha et al. (2012). However, there are many inconsistencies with the results between different laboratories using the same technique (e.g., Passey et al., 2010; Sondergeld et al., 2010) and between different methods (e.g., Sinha et al., 2012; Heller et al., 2014). Difference up to two-to-three order of magnitude for a same sample has been reported (Sondergeld et al., 2010; Heller et al., 2014). This inconsistency makes it difficult to characterize permeability of the mudrock samples precisely. For a more in-depth understanding of these inconsistencies and the measurement results, we have conducted systematical investigation and comparison using the same set of sample with two different measurement techniques, i.e., GRI method for crushed samples and a modified gas expansion method (GEM) for plug samples. An independently-measured permeability results from pulse

decay method for the same samples were also compared. Along with the differences in sample preparation and experimental conditions (i.e., pore and confining pressures) of different methods, sample size is another factor that affects measurement outcome. The influence of sample particle size on GRI permeability has been noted by several researchers (e.g., Cui et al., 2009; Tinni et al., 2012); however, the influence of plug diameter on pulse-decay measurement has not been shown. In this paper, both particle and plug size effect and its implication on permeability measurement are examined and discussed.

Section 2 includes brief reviews on the measurement techniques. Samples used in the present study are described in Section 3. MGE method is introduced in Section 4. In Section 5, GRI permeability calculation using a modified analytical method from literature (Cui et al., 2009) is conducted. Software from Corelab (Tulsa, Oklahoma) was also applied to the measured pressure-drop data to obtain permeability for comparison. Section 6 compares permeability measurement between crushed and plug samples with different particle/plug sizes, using GRI and MGE methods, respectively. Section 7 discusses the possible causes of the scaling relation between permeability and sample size. Section 8 further discusses the implications of sample-size effect and other permeability-measurement issues. Section 9 presents the conclusions from this study.

2. Brief Technique Review

The GRI permeability method has been increasingly applied because it has a simple experimental setup and is considerably faster than the traditional steady-state or pulse-decay methods. No requirement for intact plug sample is also an advantage in many cases. However, its shortcomings include the lack of confining pressure and the fact that the measurement is

commonly conducted under a pore pressure that is lower than reservoir pressures; in addition, the Klinkenberg effect is ignored. These limitations make the GRI permeability method inappropriate for indicating permeability under in-situ conditions. There is also a lack of standard procedures for data interpretation of the GRI method (Sinha et al., 2012), which is one of the reasons for inconsistent results from different laboratories.

The pulse-decay method can measure permeability under high confining and pore pressures; however, the experimental setup is more complicated and expensive, and the test is considerably more time-consuming. A gas-booster or pump is required for high pore pressure (>~1000 psi). It can take several months to measure permeability at different pore/confining pressure levels (e.g., Heller et al., 2014). Therefore, there is strong motivation to develop a method that is not only relatively easier and faster, but that also incorporates in-situ confining pressure and considers Klinkenberg effect.

A modified gas-expansion (MGE) method is introduced in this paper. Cui et al. (2010) firstly developed this method but it has not been applied and evaluated broadly. This method is modified based on the original GRI method, a type of gas expansion method, and is applied for permeability measurement of mudrocks under high confining stress and different pore pressures. Comparisons are made between the MGE permeability and those from pulse-decay method which were conducted independently (with Barnett sample data published in Bhandari et al. 2015, and Eagle Ford sample data unpublished) for the identical samples.

3. Sample Description and Preparation

Upper Cretaceous Eagle Ford samples from wells of K1 and K2 (Table 1) and a Mississippian Barnett sample were used in this study. All samples were analyzed “as-received”, with only a pretreatment of oven drying at 50°C (designed to avoid affecting clay minerals) for 7 days to remove residual moisture before measuring permeability. Two Eagle Ford samples (Sample #2 and Sample #5, Table 1) from oil-producing wells and the Barnett sample (6H1) from a gas-producing well were selected for the plug-permeability measurement using the MGE method under in-situ confining pressures (more details in Section 4). Plug diameter for the Eagle Ford samples is one inch (2.54 cm), and for the Barnett sample, 1.5 inch (3.81 cm). Permeability was also independently measured for these same plugs, using the pulse-decay method (Bhandari et al., 2015). Plugs with 1.5-inch diameter were obtained from Eagle Ford Samples #2 and #3 and measured using MGE method to investigate the plug size effect on permeability.

Ten other plugs from five Eagle Ford samples were taken from different depths of the K1 and K2 cores, representing four lithofacies ranging from calcareous-nanoplankton argillaceous lime mudstone to gray calcareous–foram-nanoplankton lime mudstone–grainstone. Two plugs were taken from each sample. Table 1 lists the samples, depths, and facies descriptions. The bulk volume of each plug was measured using a laser scanner (NextEngine 3D Scanner) with a resolution of 0.1 mm and a measurement error of less than 0.1% for a standard volume of 51.48 cm³. Bulk volume measured in such a way is more accurate than that calculated from the geometry since it can capture irregular surfaces related to imperfect plug drilling and cutting. Bulk density was then calculated with oven-dry mass and bulk volume. The bulk density, along with grain density measured through the GRI analysis, was then used to calculate porosity.

The plugs were then crushed and sieved to obtain approximately 30 g of 4–8 mm particles for each sample. Permeability measurement was conducted for the crushed samples

before being crushed again to obtain smaller samples using the GRI method. In this way, all the different sizes of samples are obtained from the same set of plug samples. Although by crushing the plug samples, mineralogical composition of the plugs may be different from that of the crushed samples, and so is the measured bulk density, the difference is considered minimum here. Samples with four different particle sizes of approximately 30 g with size ranges of 4–8 mm, 2–4 mm, 0.85–2 mm, and 0.5–0.85 mm were eventually obtained. The samples were oven dried at 50°C under vacuum for 7 days before the measurement.

Micro-computed tomography (micro-CT) scanning was conducted for 1-in plugs before crushing. Mini-plugs (diameter of 1-2 mm) were obtained for selected samples to detect smaller-scale features (e.g., micro-features) using micro-CT or scanning electrical microscopy (SEM). More details on the micro-CT scanning conditions and tomographic volume reconstruction can be seen in Peng et al. (2015).

4. Modified Gas-Expansion Method

4.1 Experimental setup and data analysis

Figure 1 shows the schematic experimental setup of the MGE method. The setup is basically the same as that for the GRI method, which is essentially a gas-expansion apparatus (Luffel et al., 1993). The only difference is that in the MGE method, a plug sample is loaded within a core holder in which confining pressure can be applied, whereas in the GRI method, crushed samples are loaded under no confining pressure. The plug is inserted in a rubber sleeve and hydraulic oil is filled between the core holder and the sleeve, then pressurized to provide confining pressure. Two stainless steel gas distributors were set tightly against the plug at the

two ends, respectively. Thus, an equilibrated stress with the confining pressure is expected in the axial direction. This experimental setup was designed independently but is similar to that in Cui et al. (2010). Helium is provided with a high-pressure gas tank and is directed to the reference cell. The valve between the tank and reference cell is closed when the gas pressure approaches the target pressure, and generally the gas pressure stabilizes in the reference cell within 5-10 minutes; helium is then introduced to the core holder by opening the connecting valve. At first, helium occupies the dead volume in the core holder and subsequently penetrates into the shale plug from the top surface. A high-accuracy pressure transducer ($\pm 0.1\%$ of full scale) is used to record the pressure decay in the system. The full-scale range of the pressure transducer is from 0 to 210 psi. The whole system except the gas tank (Figure 1) is included in a heating box in which temperature is set to be stable at 22.7 ± 0.2 °C, slightly higher than the room temperature. After the gas-expansion experiment, initial pressure and equilibrium pressure in the core holder are used to calculate the porosity based on Boyle's law, as introduced in Cui et al. (2009). Pressure-decay history measured from the experiment is used to calculate permeability.

Gas flow in a cylindrical porous medium can be described by a 3D model under cylindrical coordinates in the form of:

$$\phi \frac{\partial \rho}{\partial t} = \frac{1}{r} \frac{k}{\mu} \left[\frac{\partial}{\partial r} \left(r \rho \frac{\partial P}{\partial r} \right) + \frac{\partial}{\partial \theta} \left(\frac{1}{r} \rho \frac{\partial P}{\partial \theta} \right) + \frac{\partial}{\partial z} \left(r \rho \frac{\partial P}{\partial z} \right) \right] \quad (1)$$

where ϕ is porosity, ρ is gas density, r is radial distance from the center of the plug, μ is viscosity, and P is pressure. k actually is apparent permeability because Equation 1 is a continuum-flow equation and does not explicitly include Knudsen diffusion in nano-scale pore and pore throat; slip boundary condition was not considered in Equation 1 either. However, for

simplicity, k is still named as permeability in this paper. If assuming that gas pressure and density are only dependent on depth, (1) can be simplified to a one-dimensional equation:

$$\phi \frac{\partial \rho}{\partial t} = \frac{k}{\mu} \frac{\partial}{\partial z} \left(\rho \frac{\partial P}{\partial z} \right) \quad (2)$$

In (1) and (2), permeability is assumed to be uniform in the radius and depth directions. In other words, permeability based on (1) and (2) is an averaged apparent permeability for the whole volume of the plug. Given:

$$\rho = \frac{P}{ZRT} \quad (3)$$

where Z is compressibility factor which can be approximated as one in low pressure range (0-200 psi), R is ideal gas constant, and T is temperature, (2) can be transformed to:

$$\phi \frac{1}{P} \frac{\partial P^2}{\partial t} = \frac{k}{\mu} \frac{\partial^2 P^2}{\partial z^2} \quad (4)$$

Initial and boundary conditions of the studied system are as below:

$$P = P_0, 0 \leq z < L, t = 0 \quad (5a)$$

$$P = P_i, z = L, t = 0 \quad (5b)$$

$$-\pi R_0^2 \frac{k}{\mu} \frac{\partial P^2}{\partial z} = (V_r + V_d) \frac{\partial P^2}{\partial t}, z = L \quad (5c)$$

in which P_0 is initial pressure in the core holder, which is ambient pressure; P_i is initial pressure pulse after the gas introduction from the reference cell into the core holder, but before the penetration into the plug; P_i is determined based on P_0 , introduced gas pressure (generally around 200 psi), and the volume of reference cell and dead volume in the system (V_r and V_d , respectively); and L and R_0 are the length and radius of the plug, respectively. In order to obtain the analytic solution, the term $1/P$ in (4) is assumed to be constant, which should be valid for

such a system where the pressure change in the studied domain is small. An example P_e/P_i is about 93.5 psi/94.5 psi. Using either P_e or P_i to calculate I/P will not cause big difference. According to Carslaw and Jaeger (1947), the analytic solution for $P(z=L)$ is in the form below:

$$P^2 - P_0^2 = \frac{P_i^2 - P_0^2}{1 + Lh} + \sum_{n=1}^{\infty} \frac{2h(P_i^2 - P_0^2)\exp(-K\beta_n^2 t)}{L(\beta_n^2 + h^2) + h} \quad (6)$$

where β_n are the roots of:

$$\beta \cot(\beta L) + h = 0 \quad (7a)$$

and

$$h = \frac{\pi R_0^2 \phi}{V_r + V_d} \quad (7b)$$

When t is large enough, (6) can be simplified to:

$$\psi = (P^2 - P_0^2) - \left(\frac{P_i^2 - P_0^2}{1 + Lh} \right) = \frac{2h(P_i^2 - P_0^2)\exp(-K\beta_1^2 t)}{L(\beta_1^2 + h^2) + h} \quad (8)$$

(8) can be further simplified to:

$$\ln(\psi) = f - st \quad (9)$$

and

$$s = K\beta_1^2 \quad (10a)$$

$$K = \frac{k}{\phi\mu/P} \quad (10b)$$

β_1 is the 1st root of (7a). f is the intercept of the linear fitting of $\ln(\psi)$ vs. time. Therefore, permeability can be obtained through (9-10) after obtaining the slope (s) by plotting $\ln(\psi)$ vs. time. Equilibrium pressure P_e is used for calculation of I/P in Equation 10b. All the calculation is based on standard metric unit and permeability is reported in unit of nD after conversion.

Calculation of permeability in Cui et al (2010) is basically the same as the steps described herein. One difference is the gas flow model herein is based on P^2 for gas (Equation 4), while it is based on P although the model is not explicitly presented in Cui et al (2010).

Permeability was measured under pore pressures (P_p) ranging from 50 to 200 psi and confining pressures (P_c) to produce net stress ($P_c - P_p$) from 500 to 5500 psi. Plugs were loaded in the core holder and prestressed for 24–72 hrs to allow elastic deformation to reach equilibrium before the final permeability measurements. Measurement repeatability was examined for selected samples, with standard deviation of $< \pm 10\%$.

4.2 Measurement results from MGE method

Figure 2 shows examples of the linear relationship between $\ln(\psi)$ and time resulted from experimental measurements for the Eagle Ford Sample 2 plug under different confining pressures. Pore pressure in these measurements is approximately 130 psi, and the value of net stress ($P_c - P_p$) ranges from 500 to 5500 psi. Generally, the steeper the slope, the larger value of permeability that will be obtained. For example, the curve at a net stress of 500 psi has the largest slope and leads to the largest permeability value (596 nD); 4000 psi and 5500 psi have the smallest slopes and thus the smallest permeability values (128 and 101 nD). Note that permeability is not only proportional to the slope but also to porosity. Although the slope is slightly higher at net stress of 5500 psi than at 4000 psi, porosity at net stress of 5500 psi is smaller. This led to smaller permeability under 5500 psi. The pressure-decay curve for Sample 2 under net stress of 500 psi is included in Figure A-1 in Appendix. Calculated permeability is presented in Figure 3 for the three samples: Eagle Ford Samples #2 and #5, and Barnett 6H1. The two Eagle Ford samples have permeabilities larger than that of the Barnett sample. For all the samples, permeability decreases exponentially as confining pressure increases, and the

decrease tends to vanish at high confining pressures of >5000 psi. There is an approximate four- to six-fold total decrease of permeability from net stress 500 psi to 5500 psi for the two Eagle Ford samples, and about a two-fold decrease for the Barnett sample. In the same time, porosity is also decreasing relative to the increase of confining pressure (Figure 3). The porosity is reduced from ~7% to 3% for the two Eagle Ford samples and from 5.5% to 4.3% for the Barnett sample. In porosity calculation for the plug sample under confining pressure, initial bulk volume measured at ambient condition was subtracted by the amount of pore volume reduction under the specific confining pressure from the initial state. The assumption behind is all the bulk volume reduction is caused by the reduction of the pore volume. The ductile components in the sample, i.e., clay minerals and organic matters, can be compressed under pressure; however, on one hand, it is difficult to measure such compression, on the other, such compression is speculated to be relatively smaller compared to that of the pores.

4.3 Klinkenberg correction and comparison with pulse-decay-method results

Permeability measurement with the pulse-decay method was done under higher pore pressures using Argon. The pore pressures is 2000 psi for the Eagle Ford samples and 1000 psi for the Barnett sample, both higher than pressure for the MGE method of 130 psi. A Quizix Q5000 gas pump was used to provide high pore pressure (Bhandari et al., 2015). The pore diameter is generally in the range of <10 nm to a few hundred nanometers for Eagle Ford and Barnett mudrocks (Loucks et al., 2009; Reed et al., 2012), whereas the dominant pore throat diameter is ~7-12 nm for many Eagle Ford samples according to mercury intrusion (Shultz, 2015). For mudrocks with nanoscale pores or flow channels, different pore pressure means a different extent of molecule–pore wall interaction and different flow regimes that can be defined according to Knudson number, which can be calculated as:

$$K_n = \frac{\lambda}{d} \quad (11)$$

and

$$\lambda = \frac{K_B T}{\sqrt{2} \pi \sigma^2 P_p} \quad (12)$$

where λ is the molecular mean free path and d is the pore diameter. K_B is the Boltzmann constant, T is the temperature, σ is the kinetic diameter of the gas molecule, and 0.26 nm is the value for helium. Gas flow under pore pressures of 50–200 psi, which is the condition in the MGE method, is within slip and transition flow regimes, whereas under the condition of the pulse-decay method with high pore pressures of 1000–5000 psi, there are more slip and viscous flow being involved (Figure 4). Therefore, direct comparison of these two results is not appropriate, and the Klinkenberg correction is needed for the low-pore-pressure permeability.

Permeability was measured under three low pore pressures (around 130, 100, and 80 psi) at different confining pressures for the three samples. Figure 5 shows the results. It can be seen that permeability increases with decreasing pore pressure (or increasing $1/P_p$) related to the more significant molecule-wall collision at lower pore pressures. The linear relationship between permeability and $1/P_p$ suggests that this increase can be well described by the Klinkenberg equation. Therefore, permeability at higher pressures can be extrapolated based on the Klinkenberg correction. Figure 6 shows the resulting permeability at pore pressure of 2000 psi for the two Eagle Ford samples and 1000 psi for the Barnett sample. The solid curves are exponential fittings of the measured data.

The results from the pulse-decay method are also shown in Figure 6 as the dotted data points. It can be seen that estimated permeability based on our MGE method is in fair agreement with that measured from the pulse-decay method even though different gases were used in these

two methods. The MGE permeability generally is 2-4 times larger than the pulse decay permeability, with one MGE permeability ~25% lower than the pulse decay permeability. This is in contrast with the result from Cui et al. (2010) using the same experimental setup. In Cui et al. (2010), this MGE type method produced permeability of approximately two-order-of-magnitude lower than that from the pulse decay method. The reason is not clear; however, there are several differences between the two methods. For example, they used P instead of P^2 in the governing equation; they also made direct comparison of permeability without considering Klinkenberg correction. While Klinkenberg correction will produce even smaller permeability at higher pore pressure, the difference of P vs. P^2 will lead to underestimation of permeability based on our tentative calculation by a factor of 2-3. Based on our results, this MGE method, which can be easily set up by adding a core holder in the GRI permeability system, can be used as an easier and faster alternative for permeability measurement under confining pressures.

5. Crushed-Sample Permeability and Analytic Solutions

Gas-expansion experimental setup for crushed sample is basically the same as that in Figure 1, except that a matrix cup instead of a core holder is used. This method is known as GRI method. Crushed samples are loaded in the matrix cup under ambient pressure conditions. Two typical pressure-decay curves for samples with different particle sizes are shown in Figure A-1 in the Appendix. Porosity is obtained with initial and equilibrium gas pressures similar to those described in Section 3. A data analysis procedure for GRI permeability from Cui et al. (2009) is modified by considering that density is a function of pressure, and the gas flow model, Equation (2) in Cui et al. (2009) becomes:

$$\phi \frac{1}{P} \frac{\partial P^2}{\partial t} = \frac{k}{\mu} \frac{1}{r^2} \frac{\partial}{\partial r} \left(r^2 \frac{\partial P^2}{\partial r} \right) \quad (13)$$

The analytic solution given in Cui et al. (2009) was then modified correspondingly, with P being replaced by P^2 . Eventually, a relative term of pressure is in a semilog linear relationship with time as the form of:

$$\ln(F_R) = f_0 - s_1 t \quad (14a)$$

$$F_R = 1 - \frac{(K_c+1)(P_i^2 - P^2)}{P_i^2 - P_0^2} \quad (14b)$$

$$K_c = \frac{V_r + V_d}{\phi V_s} \quad (14c)$$

where V_s is the bulk volume of the sample, f_0 is the intercept term. Permeability k is then calculated through the slope s_1 using the late-time technique and r_o^2 , where r_o is the particle radius (as in Cui et al., 2009).

Equations 14a-c were used for the crushed-sample data for GRI permeability calculation. Figure 7 presents the curve fitting with the analytic solution for Eagle Ford Sample #2, 2–4-mm sample. Good linearity was found for the $\ln(F_R)$ vs. time data. The same samples were also measured using Corelab's shale matrix permeameter (SMP-200) with a proprietary software. Two typical pressure decay curves in such experiment are presented in the Appendix (Figure A-2). The resulting permeability is found to be two-to-three order of magnitude higher than that from the modified Cui's solution (Figure A-3). By contrast, another set of measured permeability from literature on an Eagle Ford Shale sample (Tinni et al., 2012) is in good agreement with our results using the modified Cui's solution (Figure 8A). Based on these two comparisons and the fact that it is difficult to evaluate the reliability of Corelab's data-analysis procedure because it is

kept proprietary, only GRI permeability from the modified Cui's solution is used in the following discussions.

6. Permeability Measured for Crushed and Plug Samples with Different Particle/Plug Sizes

Crushed samples is used to avoid fracture's influence since fractures in such samples are expected to be destroyed so the permeability is mostly contributed by matrix pores. Permeability of intact plugs is sometimes also regarded as "matrix" permeability and is compared directly with the GRI crushed-sample permeability. However, as we can see from the results, permeability of GRI crushed samples of 20/35 mesh is on the level of 0.01-1 nD, under no confining pressure (Figure A-3); permeability for the 1-in plug sample is on the level of 50–500 nD (Figure 6). There is a large gap between these two permeability values. Even though the gap is reduced at higher confining pressure, it is not totally removed.

Permeability of different particle sizes of the five crushed Eagle Ford samples is plotted in Figure 8A. Two other data sets from the literature are also included: an Eagle Ford shale sample from Tinni et al. (2012), and an unknown shale sample from Cui et al. (2009). All data show a linear increase of permeability with sample particle size on a log scale, and the increase scales with particle size and can be described by power-law equations.

Permeability for plug samples of Eagle Ford Samples 2 and 3 with a 1-inch and 1.5-inch diameter was also measured under net stress of 500 psi and equilibrium pore pressure of ~130 psi. Permeability for a 1.5-inch plug is significantly larger than that for a 1-inch plug. For example, 1-inch plug permeability is 595 nD for Eagle Ford Sample 2, and 1.5-inch plug

permeability is 3194 nD (Figure 8B). As shown in Figure 8B, with the addition of plug permeability, the scaling relation between permeability and sample size continues to exist.

7. Discussions on Why Permeability Increases with Sample Size

7.1 System measurement error of GRI method with crushed samples

As noted by Suarez-Rivera et al. (2012), the measurement capacity of the GRI method has some limitations. Specifically, for a crushed sample with small particle size, the higher-permeability part of the sample may not be detected by this method because 1) quick pressure drop related to this high permeability, and 2) the quick pressure drop occurs in the early phase and is not used in the calculation with late-time technique (Cui et al., 2009). In other words, permeability measured using GRI and the late-time data analysis method only represent the permeability for the lower permeable part of the sample, and thus underestimates the real permeability. Early-time data analysis may be used for permeability calculation; however, early-time method generates larger error (Cui et al., 2009) and thus not included in our discussion here. As an example, the permeability measured for the Eagle Ford 0.5–0.85 mm samples has a permeability range of 0.01 to 1 nD. These values can be lower than the actual permeability; however, these values do represent the permeability of the part of the sample that has small pore throats and limited connections.

Kozeny-Carman (K-C) Equation is often used to predict permeability for a porous media, which has the form of:

$$K = \frac{\phi r_h^2}{\beta_s \Gamma} \quad (15)$$

where ϕ is porosity, r_h is hydraulic radius, β_s is a shape factor that accounts for the influence of different shape of the cross-sectional area, and Γ is tortuosity factor $(L_{eh}/L_m)^2$, with L_{eh} being effective hydraulic path length and L_m minimum length along the flow path. The shape factor β_s has a range of 2 to 3 (Van Brakel, 1975). The β_s value of 2 is for circular pore shape, and is used here for permeability estimation. Hydraulic radius can be estimated through porosity and specific surface area (Ben Clennell, 1997). Since no specific surface area data available, instead, the dominant pore throat radius obtained from mercury intrusion was used in Equation 15. Tortuosity is difficult to measure experimentally, but can be estimated based on formation factor (F_m) and Archie's law:

$$F_m = \phi^{-m} \quad (16a)$$

$$\Gamma = F_m \phi \quad (16b)$$

Therefore,

$$\Gamma = \phi^{1-m} \quad (16c)$$

where m , sometimes known as cementation exponent, generally lies between 1.3 to 3 (Ben Clennell, 1997). Value of m is related to the importance of a random pore on pore connection. Higher values of m mean greater importance of a random pore on pore connection, whereas lower values of m indicate less importance, such as dead-end pores (Dullien, 1992). An m value of 2 indicates equal importance of random pores. For Mudrock with low porosity ($< \sim 5\%$), every pore or major part of the pores should be important in pore connection. Therefore, the m value of 2 to 3 is used for permeability estimation.

Figure 9 shows the pore throat size distribution for Eagle Ford Sample 2 from mercury intrusion (Schultz, 2015). A dominant pore throat diameter of 10 nm (radius of 5 nm) is

determined based on the peak of incremental pore volume. Mercury-intrusion-measured porosity of 6% (Schultz, 2015) is used in Equations 15 and 16a-c. With m value of 2 to 3, the estimated permeability is 2.7 to 45 nD. The range of 2.7 to 45 nD is in line with the measured result of 16.6 nD from crushed sample with particle size of 2-4 mm. Mercury intrusion analysis was not conducted in Schultz (2015) for the other samples used in this study; instead, three different samples from the same wells (K1 and K2) but from nearby depths (11939-ft, 12169-ft, and 12247-ft) were measured with mercury intrusion (Schultz, 2015). These three samples, although not the same as samples used in this study, all have dominant pore throat diameter of 7-10 nm. Calculated with Equations 15 and 16a-c, the estimated permeability has a range of 2.25-36 nD for these samples. This range is in line with the measured results of 8.8-20.8 nD for the other samples with particle size of 2-4 mm (Figure 8A).

It is worth to note that using dominant pore throat diameter in Equation 15 may underestimate the actual permeability. It is pore throat that constrains the flow; however, pore body plays a role too. Chaudhary et al. studied the effect of pore geometry on permeability (2013). They found the permeability for pores with larger pore body size but with the same pore throat size can be larger by a factor up to 10 (Figure 5 in Chaudhary, 2013). In addition, K-C Equation does not consider Knudson diffusion or slip-flow boundary; therefore, estimation based on K-C Equation should be smaller than the actual permeability. Therefore, measured GRI permeability from crushed sample with particle size of 2-4 mm, or 5-10 meshes, which have similar results with the permeability estimated based on K-C equation using dominant pore throat size, may still underestimate the permeability. Nevertheless, since it is difficult to evaluate such underestimation more quantitatively, the GRI permeability from particle size of 2-4 mm can be used as a lower boundary of the real permeability. Some commercial laboratories (e.g., Core

Laboratory and Weatherford Laboratory) use the size of 20-35 meshes (0.5-0.85 mm) as the best particle size for matrix permeability measurement. The reason behind is because the presence of fractures or artifacts is supposed to decrease when the sample is crushed into smaller particles, and the smaller the sample particles, the less influence artifacts will have. From the discussion above, the size of 0.5-0.85 mm will very likely underestimate the real permeability in a larger degree.

For Sample 2, the measured GRI permeability using samples of 0.5-0.85 is 0.03 nD. This corresponds to permeability contributed by part of the pore network with pore throat diameter of less than 2 nm based on calculations from Equation 15 and 16a-c. The measured GRI permeability using samples of 0.85-2 mm (10-20 meshes) is 0.5 nD, corresponding to a pore throat diameter of ~5 nm. Therefore, samples with particle size smaller than 2-4 mm only measure part of the permeability from pores with certain pore or pore throat size using GRI method. This fact also explains the log linear relationship between permeability and particle size as shown in Figure 8A, given the power-law-equation relationship between permeability and pore throat size (K-C Equation).

7.2 Artificial fractures

For plug samples or crushed samples with larger particle size, the increased permeability is most likely caused by the sample artifacts or fractures. GRI method will underestimate the permeability for samples with smaller particle size, as discussed earlier, however, either GRI or MGE method will not overestimate the permeability. In the plug sample or crushed samples with a larger particle size, if there are no higher permeable layers/parts in the sample, permeability will not keep increasing with sample size. In other words, higher permeability in the larger

samples is not a measurement error but, rather, is caused by the inclusion of higher permeable layers/parts that are most likely sample damage or artifacts.

In the time from the in situ subsurface condition to the laboratory bench, many processes can cause sample damages or artifacts. Horizontal cracks created by pressure release is one of most common artifacts. Figure 10 shows several example SEM and micro-CT images with artificial fractures caused by pressure release in 1-in plug and 1.5-mm mini-plug for Eagle Ford Samples 2 and 6. Observation of these images reveals that larger samples include larger fractures, whereas smaller samples have smaller fracture (both pointed by red arrows). Specifically, plug samples with 1-in diameter contain fractures with aperture of 1-20 μm (Figures 10a, c), and samples with 1.5 mm diameter contain fractures with aperture of 0.1-1 μm (Figures 10b, d). There should be smaller fractures in the 1-in plug as well; however, resolution to obtain the whole plug is too coarse to detect such smaller fractures. High confining pressures can be applied to reduce the influence of the artificial fractures. Figure 11 shows the measured permeability under increasing confining pressure for Eagle Ford Sample 2 plug with diameter of 1- and 1.5-in. Permeability under confining pressure of 5500 psi for the 1.5-in plug sample is 764 nD, six-fold larger than that for the 1-in plug, which is 101 nD. This indicates that there must be larger or denser fractures in 1.5-in plug than in 1-in plug, and the influence of the fractures persists even under very high confining pressure because of hysteresis. In addition, compression or closure of the fractures under confining pressure is more effective and faster for smaller plugs than larger plugs. Therefore, 1-in plug should be better for permeability measurement. Currently most core holders used in permeability measurement, either with technique of MGE or pulse decay, have minimum diameter of 1-in. It will be helpful if smaller core holder, for example, 0.5-in, can be made and used for 0.5-in plug permeability measurement.

8. Further Discussions on Matrix permeability and Measurement

8.1 Matrix permeability

Based on the discussions in Section 7, we understand that GRI method with crushed samples may underestimate the permeability because of its inability in measuring the higher permeable layers/parts in the sample, whereas the MGE method with plug samples may overestimate the permeability because of the influence of artificial fractures. Therefore, instead of using a single value of permeability to define the mudrock matrix permeability, a range of permeability is a more reliable measures. As discussed earlier, GRI permeability from crushed samples with particle size of 2-4 mm can be used as a lower boundary, and permeability from 1-in plug under high confining pressure as upper boundary. For example, permeability range for Eagle Ford Samples 2 and 5 is 16.6-101 nD and 12.9-89.7 nD, respectively. These ranges, still spanning a factor of 6-7, highlight the measurement uncertainty on the matrix permeability for mudrocks.

According to many inversely calculated matrix permeabilities based on history matching with production curves, a typical matrix permeability value needs to be in the range of 100–1000 nD to meet producing capacity (e.g., Mattar et al., 2008), which is approximately one to two orders of magnitude larger than the measured matrix permeability range of ~10–100 nD for the samples studied herein. This discrepancy can be an indication of the presence of microfractures created during hydraulic fracturing, for which direct evidence is difficult to obtain. These microfractures can be created at weak interfaces when the growth of hydraulic fractures blunted, or offset at the layered interface that has weaker binding (Fisher and Warpinski, 2012).

8.2 Comparison of measured permeability

It is already well understood that pore and confining pressures are important to the comparison of measured permeabilities. This present study shows that sample size, either particle size or plug diameter, has significant influence on permeability measurements, as well. Therefore, for the purpose of reservoir evaluation, comparison of permeability should be made with identical conditions. These conditions include sample size, pore and confining pressure, analytic procedure, and sample pretreatment.

9. Conclusions

A modified gas-expansion (MGE) method for permeability measurement under in-situ confining pressure and low pore pressures was applied to several Eagle Ford and Barnett mudstone plug samples. Both the permeability and the porosity of these samples decrease with increasing net stress. With the Klinkenberg correction, permeability can be estimated for high pore pressures, with results in fair proximity to those from the pulse-decay method. The MGE method, with its advantages of relatively easier experimental setup and shorter test time, may serve as an alternative to permeability measurement for plug samples under in situ confining stress. The GRI method was also applied to crushed samples. An analytic solution from the literature (Cui et al., 2009) was modified and employed for GRI permeability calculations. The results were in good agreement with that from literature for the Eagle Ford samples. By contrast, GRI permeability using commercial software is two-to-three orders of magnitude higher.

Sample particle/plug size was found to have significant influence on the measured permeability values, for crushed samples as well as plug samples. Measurement of larger samples, i.e., crushed samples with larger particle size or plug sample with larger diameter, resulted in higher permeability. The reasons behind, however, may be different. For GRI method

with crushed sample, the log-linear increase of permeability with particle size is associated with the measurement limitation of this method. GRI method and the late-phase data analysis enable only part of the permeability of the sample to be measured. For larger-particle-size crushed sample and plug sample, the increased permeability is most likely caused by the artificial fractures that were created during the course of core sampling from subsurface and sample preparation in the laboratory.

GRI method will underestimate the matrix permeability because of its inability in measuring the higher-permeable parts of the sample, whereas MGE method will overestimate the matrix permeability for plug sample because of the persistent existence of the artificial fractures. Therefore, a range of permeability obtained from GRI measurement for crushed samples with 5-10 mesh-size (2-4 mm) particles and from 1-in plug measurements under high confining pressure is deemed as a more reliable definition of matrix permeability for shale samples. The range spanning a factor of 6-7 highlights the uncertainties in matrix permeability measurement for shales.

Nomenclature

α	total-volume effective-stress coefficient
β_n	roots of Equation (7a)
β_s	shape factor
μ	gas viscosity
ρ	gas density
λ	molecular mean free path
σ	kinetic diameter of the gas molecule
ϕ	porosity

526	Γ	tortuosity factor
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528	d	pore diameter
529	f	intercept term of Equation (9)
530	f_0	intercept term of Equation (14a)
531	F_m	formation factor
532	k	permeability
533	K_B	the Boltzmann constant
534	K_n	Knudson number
535	L	length of the plug
536	P	pressure
537	P_0	initial pressure
538	P_c	confining pressures
539	P_e	equilibrium pressure
540	P_i	initial pressure pulse
541	P_p	pore pressures
542	R	ideal gas constant
543	r	radial distance from the center of the plug
544	R_0	radius of the plug
545	r_0	particle radius
546	s	slope term of Equation (9)
547	s_I	slope of Equation (14a)
548	t	time
549	T	temperature
550	V_d	dead volume
551	V_r	volume of reference cell
552	V_s	bulk volume of the sample

Z compressibility factor

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638

639 Table 1. Eagle Ford samples and lithofacies description.

Sample #	Well	Depth (ft/m)	Facies description
1	K1	11,778/3,590	Dark gray, massive, calcareous nanoplankton lime mudstone
2	K1	11,901/3,627	Black, massive, high-TOC, calcareous, argillaceous nanoplankton mudstone
3	K1	11,918/3,633	Black, massive-laminate, high-TOC, calcareous nanoplankton mudstone
4, 5	K2	12,185/3,714, 12,174/3,711	Black, massive, calcareous, argillaceous nanoplankton mudstone
6	K2	12,246/3,733	Black, massive-laminate, high-TOC, calcareous nanoplankton mudstone

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Figure 1. Schematic experimental setup for the modified gas-expansion (MGE) method for plug samples under in situ confining pressure.

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672 Figure 2. Linear relationship between $\ln(\psi)$ and time resulted from permeability measurements
 673 for the Eagle Ford Sample 2 plug at different confining pressures and a constant pore pressure of
 674 approximately 130 psi. Solid curves are measured data, and dashed lines show slopes.

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Figure 3. Permeability measured with the MGE method under confining pressure at a pore pressure of approximately 130 psi for (A) Eagle Ford Sample 2, (B) Eagle Ford Sample 5, and (C) Barnett 6H1.

Figure 4. Gas-flow regimes under different pore pressures, based on the Knudson number for mudrock. Shaded areas represent MGE-method and pulse-decay-method laboratory conditions, respectively.

Figure 5. Klinkenberg plots for permeability vs. $1/P_p$ at different confining pressures for the (A) Eagle Ford Sample 2, (B) Eagle Ford Sample 5, and (C) Barnett 6H1.

Figure 6. MGE permeability after Klinkenberg correction and pulse decay permeability for (A) Eagle Ford Sample 2, (B) Eagle Ford Sample 5, and (C) Barnett 6H1.

Figure 7. $\ln(Fr)$ vs. time for GRI permeability analysis with the modified Cui's analytic solution for Eagle Ford Sample 2, 4-8-mm crushed sample. Solid curves are fitted to the slope of late-phase data (>50 sec).

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721 Figure 8. (A) Size effect on permeability for crushed Eagle Ford samples. Solid lines are fittings
 722 with power-law equations. Data for an Eagle Ford Shale sample from Tinni et al. (2012) and an
 723 unknown shale sample from Cui et al. (2009) are also included for comparison. (B) Scaling of
 724 permeability with sample particle size or plug diameter for two Eagle Ford samples. Data in the

725 first interval between the two vertical lines are for crushed samples; data in the second interval
726 are for plugs.

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729 Figure 9. Mercury intrusion pore throat diameter distribution for Eagle Ford Sample 2. The
730 dominant pore throat diameter is determined as 10 nm, the center of the two peaks.

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735 Figure 10. Horizontal cracks caused by pressure release observed through micro-CT imaging at
 736 different resolutions and SEM image. A and B are images for Eagle Ford Sample 2 with plug
 737 diameter of 1-in and 1.5-mm, respectively. C and D are images for Eagle Ford Sample 6 with
 738 plug diameter of 1-in and 1.5-mm. Left micro-CT images (A and C) have pixel resolution of 27
 739 μm . Micro-CT image C has pixel resolution of 0.74 μm . Figure D is a SEM image.

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Figure 11. Decrease of permeability measured with MGE under increasing confining pressure for 1- and 1.5-in plugs of Eagle Ford Sample 2.

Appendix

Typical pressure decay curves for measurements with crushed samples and plug are shown in Figure A-1. The gas expansion experimental setup was connected with the helium tank through a coiled copper tubing of approximately 20-ft. The long tubing allows the helium gas temperature to equilibrate with the room temperature before it enters the reference cell. This will shorten the time for stable temperature and pressure in the reference cell, and will reduce the Joule–Thomson effect that causes the temperature fluctuation. Major part of our experiments did not show such temperature fluctuation when the connecting valve between the reference and sample cells was opened; only a few experiments showed certain temperature fluctuation (Figure A-1C and Figure A-2A).

Typical pressure decay curves from Corelab's Shale Matrix Permeameter (SMP-200) and the fitting curves using Corelab's proprietary software are shown in Figure A-2. The fitting was conducted based on the later phase of the data that is between ~20 sec and 100 sec. It is important to note that the Corelab's data-fitting software should only be used for crushed sample with particle size of 0.5-0.85 mm (20-35 meshes). Permeability was measured using SMP-200 for Eagle Ford Samples 2-5 with particle size of 0.5-0.85 mm. Permeability obtained from SMP-200 and its fitting software ranges from 50 to 400 nD, which is 2-3 order-of-magnitude higher than that from the modified Cui's solution (Figure A-3).

Figure A-1. Typical pressure decay curves for (A) Eagle Ford Sample 2, 0.5-0.85 mm; (B) Eagle Ford Sample 2, 2-4 mm; and (C) Eagle Ford Sample 2, 1-in plug at confining pressure of 500 psi.

Figure A-2. Typical pressure decay curves from Corelab SMP-200. (A) Eagle Ford Sample 2 and (B) Eagle Ford Sample 5. Blue stars are measured data. Red solid curve are fitting curves using Corelab's proprietary software based on pressure decay data between ~20 sec and 100 sec. Resultant permeability is 50 and 336 nD for Samples 2 and 5, respectively.

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780 Figure A-3. Comparison between permeability obtained from SMP-200 using its fitting software
781 (Corelab) and that from Cui's solution. Corelab's permeability is 2-3 orders-of-magnitude higher
782 than that from Cui's analytical solution (Cui et al. 2009).

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