PS Laboratory Measurement of Mudrock on Porosity, Pore and Pore Throat Size Distribution, and Permeability: Learnings from Comparison of Techniques*

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Abstract

Porosity, pore or pore throat size distribution, and permeability are three key petrophysical properties in mudrocks and whose measurements remain challenging. Although many techniques can be applied for measurement of these parameters for mudrocks, results from different techniques are often not consistent, leading to a lack of standard methods or procedures for such measurements. In this study, porosity was measured using helium pycnometer, nitrogen adsorption, and mercury intrusion capillary pressure (MICP). The latter two techniques were also used for measurement of pore and pore throat size distribution, respectively. Permeability was measured using a modified gas-expansion method (MGE) for plug samples and the traditional Gas Research Institute (GRI) method for crushed samples with different plug/particle sizes. Comparison of helium pycnometer and nitrogen adsorption porosity measurements shows that the values are consistent for samples with insignificant amounts of pores larger than 200 nm, but that nitrogen adsorption measurements underestimate porosity for samples having substantial larger than 200 nm pores. Conformance and compression are two important sources of error for MICP analysis that lead to overestimation of porosity.

A new conformance and compression correction method were developed. A previously published method was also applied for conformance and intrusion corrections. MICP porosity values after conformance and compression corrections are consistent with that from helium pycnometer. Apparently inconsistent pore size distribution from nitrogen adsorption and pore throat size distribution from MICP produce similar results in pore volume. Combination of these two size distributions reveals interesting findings on pore size composition of the pore network that cannot be seen in individual pore or pore throat size distribution. Permeability values from plug samples were found several orders of magnitude larger than that from crushed-rock samples. Permeability from both plug and crushed-rock samples shows a scaling relation to plug/particle size. This scaling behavior can be caused by inevitable sample damages and artifacts formed during sample preparation. The inability to catch the pressure decay from higher permeable layers in GRI method can also contributed to the scaling behavior of permeability.

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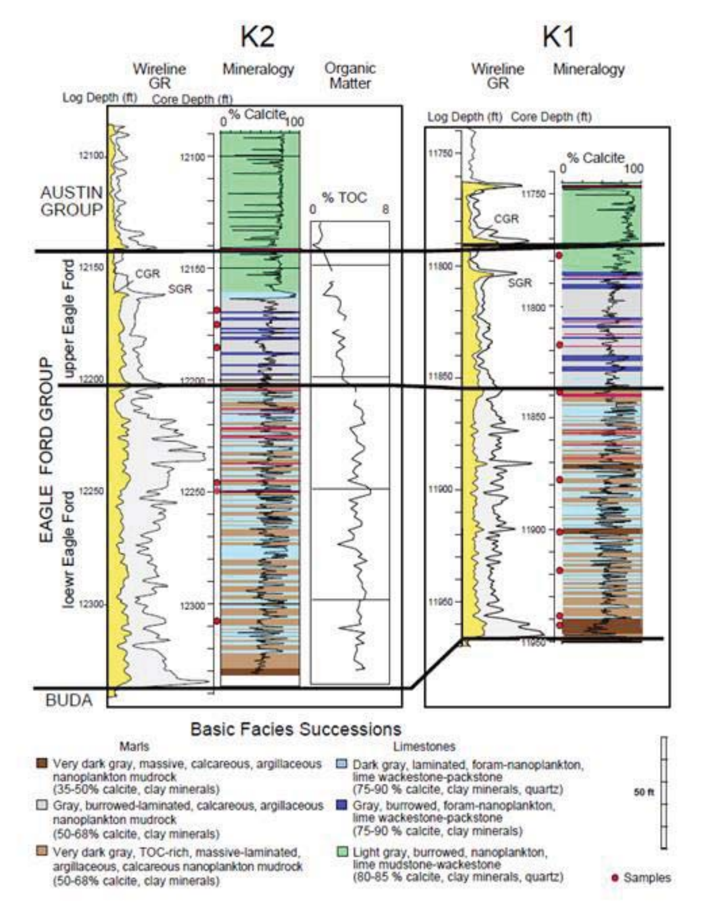
Problem Statement and Objectives

- Porosity, pore or pore throat size distribution, and permeability are three key petrophysical properties in mudrocks
- Many techniques can be applied for measurement of these parameters, but there is a lack of consistency between results from different techniques
- In this study, porosity was measured using helium pycnometer, nitrogen adsorption, and mercury intrusion capillary pressure (MICP)
- Nitrogen adsorption and MICP were also used for measurement of pore and pore throat size distribution, respectively
- Permeability was measured using a modified gas-expansion method (MGE) for plug samples and the traditional Gas Research Institute (GRI) method for crushed samples with different plug/particle sizes

Sample Description

- 14 Eagle Ford Shale samples and 25 lacustrine samples for helium pycnometer and nitrogen adsorption measurement
- Five samples for MICP measurement
- Six samples for permeability measurement



Lithologies and locations of samples studied in the K1 and K2 cored Eagle Ford wells

Sample	TOC (%)	Quartz (%)	Calcite (%)	Clay (%)
K1-11778	1.71	11.2	76.2	6.1
K111818	2.85	14.7	67.7	9.5
K1-11838	4.05	15.3	57.5	19.5
K1-11878	5.5	19.3	60.7	11.1
K1-11901	2.56	33.8	31.4	20.2
K1-11918	4.92	17	62.2	13.6
K1-11939	3.72	9.8	66.6	19.6
K1-11943	3.83	17	35.9	37.4
K2-12169	3.39	15.7	59	13.7
K2-12174	3.17	15.1	65.1	10.5
K2-12185	3.33	15.4	63.1	11.6
K2-12246	5.06	22	58.6	11.8
K2-12247	1.14	5.9	89	3.2
K2-12308	3.29	17.5	64.9	8.1

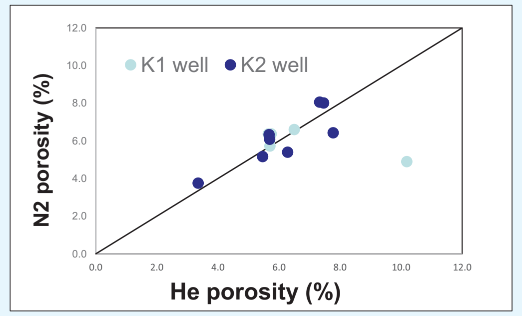
TOC and mineralogy of the 14 samples.

Brief Technique Review

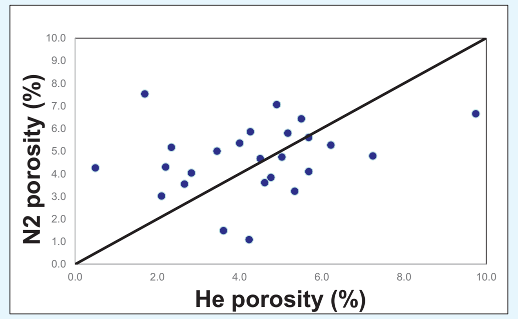
	Principle	Advantages	Disadvantages
He	Gas expansion; Boyle's law for porosity Pressure decay for permeability	Faster; wider pore size coverage; permeability	Crushed sample related errors
MICP	Mercury intrusion under pressure; Washburn's Equation	Pore throat size distribution	Destructive; not fast; <3 nm no coverage; High pressure related issue
N_2	Multi-layer adsorption and capillary condensation	Pore size distribution	Slow; small amount of sample; limited pore size coverage
WI	Water saturation under pressure	wider pore size coverage	Slow; Can be destructive; No other parameter
SEM	Direct measurement based on pore images	Pore type, geometry	Limitation on resolution and problems w/ representative area

Porosity Results

He vs. N_2 porosity



Eagle Ford Shale samples: similar results for most samples

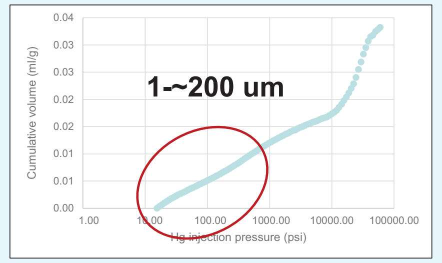


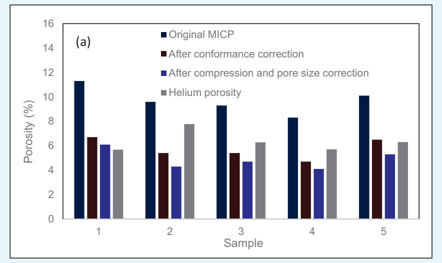
Lacustrine shale samples: not consistent

- >200 nm pores cannot be effectively measured in N_2 adsorption
- Small amount of sample used in N_2 adsorption experiment (~0.5 g)

MICP porosity

- Conformance
  - Voids between crushed grains
  - Irregular surface of crushed grains
- Compression
  - Low density features (organic matters)
  - Pores and microfractures

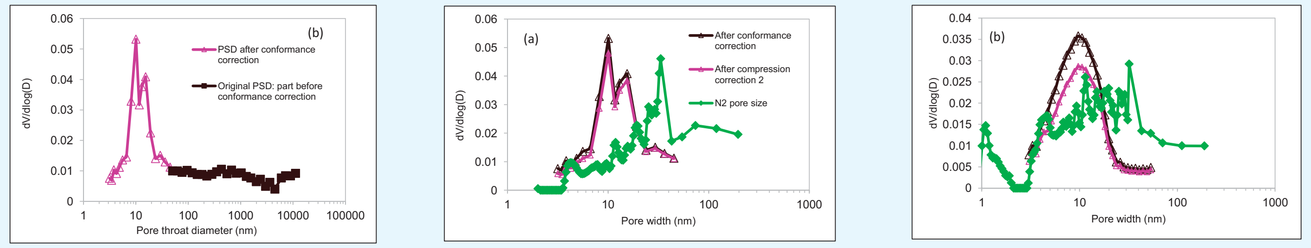




Results of MICP porosity after conformance and compression correction

- MICP overestimates porosity without corrections
- Comparable with He porosity if no <3.0 nm pores after corrections
- Underestimate porosity with <3.0 nm pores

Pore and Pore Throat Size Distribution



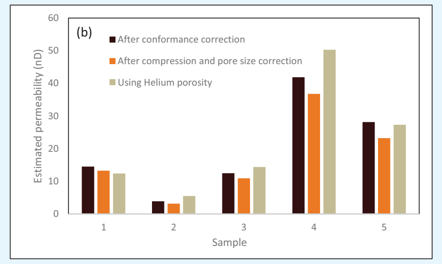
Results of MICP pore throat size distribution after conformance and compression correction, and comparison with N2 adsorption pore size distribution

- MICP pore throat size distribution: conformance correction is more important than compression correction
- Dominant pore throat size after compression correction is similar to that before correction
- Pore throat size distribution is consistent with pore size distribution in respect of pore volume

Permeability Estimation after Conformance and Compression Corrections

$$logk = -1.92 + 0.949logPhi + 2.18logr_{10}$$

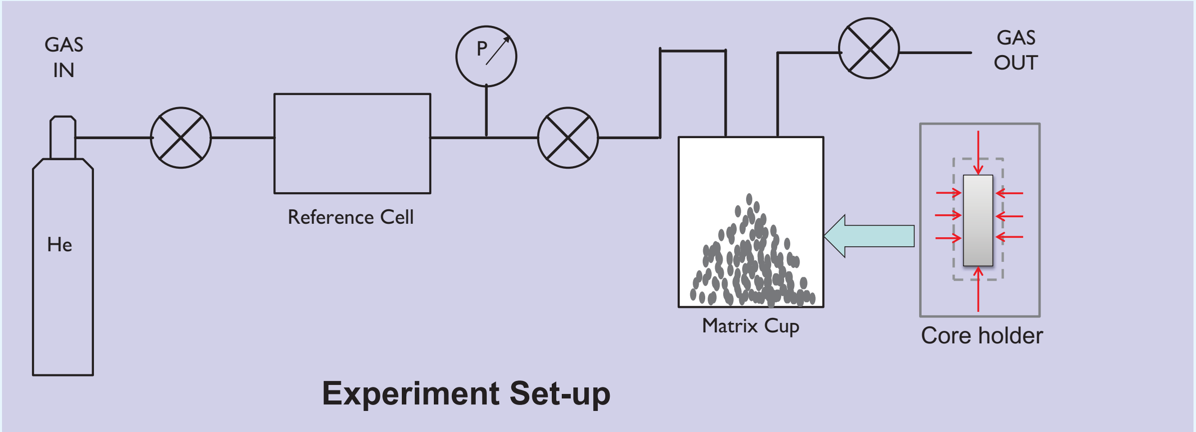
where k is the dry gas permeability (mD), Phi is porosity (%), r_{10} is the pore throat radius (μm) corresponding to 10% of the mercury saturation (Razaee et al., 2012)

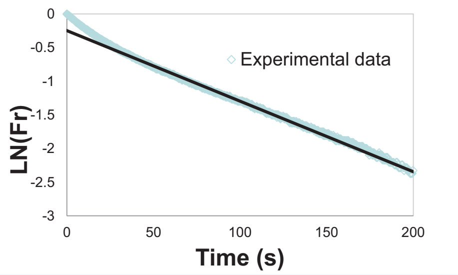


- Without conformance correction: k > 200 mD
- After conformance correction: k = 2-50 nD
- Compression correction: influence not significant

Permeability Measurement

- **GRI Method (Crushed sample)**





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

$$Ln(F_R) = f_0 - s_1 t$$

$$F_R = 1 - \frac{(K_c + 1)(P_i - P)}{P_i - P_0}$$

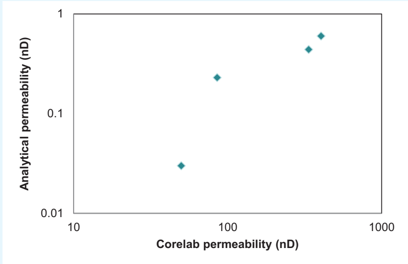
Cui et al., 2009

Typical pressure decay data

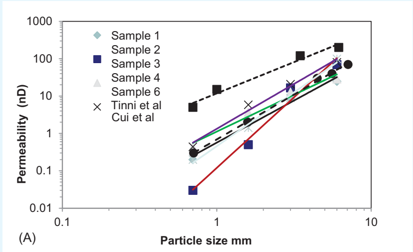
Permeability proportional to slope

- **GRI Method: Influencing Factors**

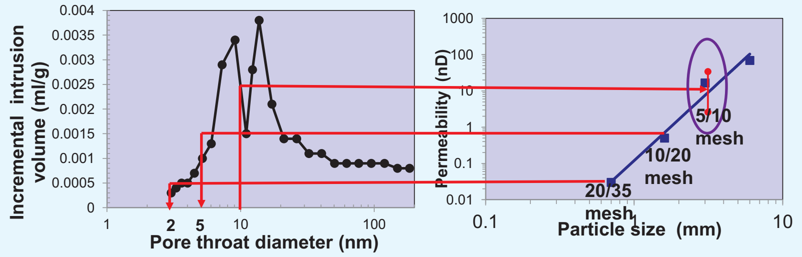
- Different analytical procedures
  - Commercial laboratory: calculation algorithm kept proprietary
  - Orders-of-magnitude-difference between results from the Cui (2009) analytical solution and that from the commercial laboratory software



- Particle size effect
  - Measured permeability values increase with increasing sample particle size
  - Can result in orders-of-magnitude difference
  - Using 20/35 mesh-sized sample may underestimate matrix permeability



- Reasons for particle size effect
  - Sample with small particle size cannot measure the high-permeable part
  - Micro-fracture can cause overestimation for sample with larger particle size
  - Suitable particle size is sample pore throat size dependent



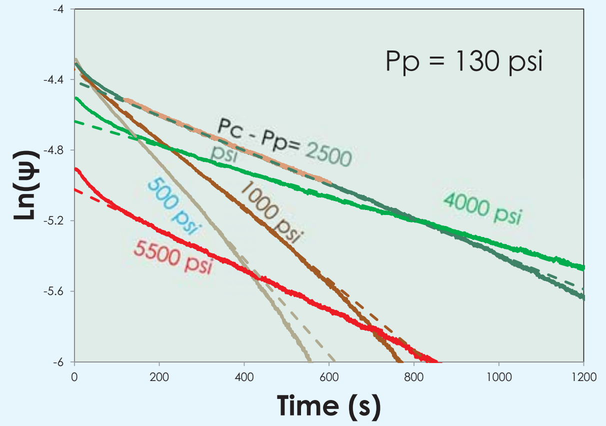
- **A Modified Gas Expansion Method (Plug Sample)**

- Basically the same as GRI method
- But with a core holder → confining pressure
- Using plug sample, instead of crushed sample
- Comparing to pulse decay method: easier and faster

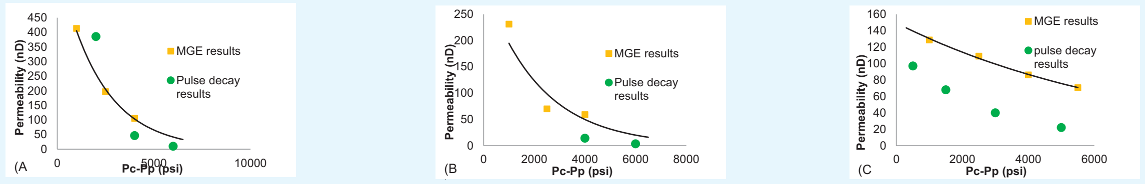
$$Ln(\psi) = f - st$$
$$s = K\beta^2$$
$$K = \frac{k}{\phi\mu/P}$$

k: permeability

Carslaw and Jaeger, 1947, Condution of heat in solids

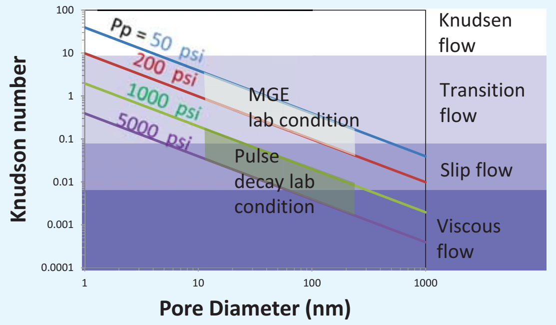


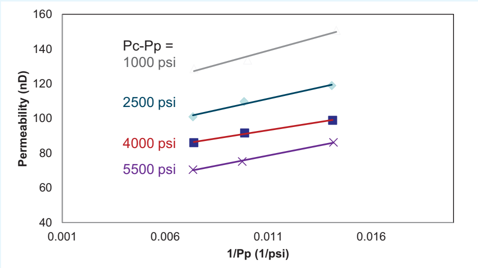
- **Measurement Results**



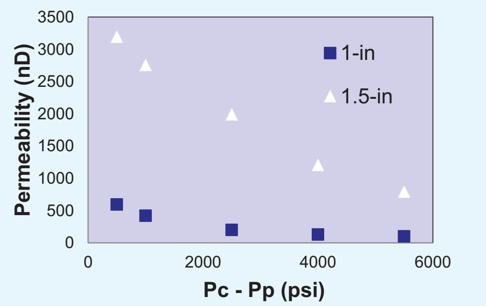
- Decreasing permeability with increasing confining stress
- Fair agreement with results from pulse decay measurements (from Bhandari et al., 2015)

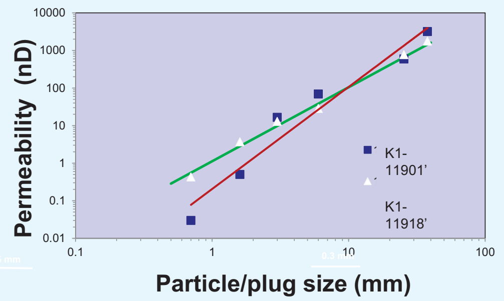
- **Influencing Factors**





- Confining pressure and pore pressure
  - Measured permeability values decrease with increasing confining pressure
  - Measured permeability values increase with decreasing pore pressure
    - Klinkenberg correction is needed





- Plug size effect
  - Measured permeability values increase with increasing plug size
  - The difference decreases with increasing confining pressure, but not completely removed
  - Plug permeability can be orders-of-magnitude larger than GRI permeability

- **Comparison of the measured permeability**

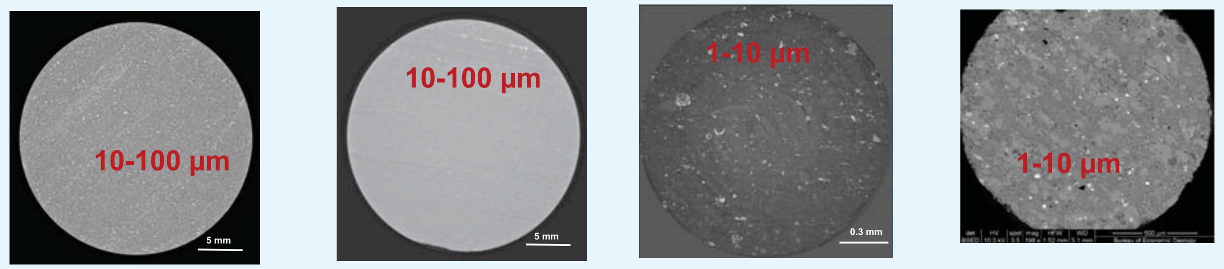
GRI – Crushed sample ⇒
- What's the particle size?
- What's the pore pressure?
- How the data is analyzed?

Plug sample ⇒
- What's the plug diameter?
- What's the pore and confining pressure?

- All the experimental conditions need to be the same for a valid comparison
- Sample pre-treatment needs to be the same as well

- **Challenges for laboratory measurement of matrix permeability**

- Existence of micro-fractures induced by pressure release and other processes in sample retrieval



- High confining pressure (up to 5000 psi effective stress) can reduce the influence
- It is unclear if higher pressure (up to 9000 psi) can totally remove the influence
- It is unclear how the micro-fracture will recover at lower confining pressures
- Some of the micro-fractures may be real

Summary

- Porosity measurement
  - Helium pycnometer is used as a standard porosity value for comparison
  - N_2 adsorption can measure porosity for samples with pores <200 nm.
  - Conformance and compression corrections are needed for MICP porosity
- Pore size and pore throat size distribution
  - N_2 adsorption can provide pore size distribution at the range of ~0.3 to 200 nm
  - Conformance correction is more important than compression correction for MICP pore throat size distribution
  - N_2 adsorption pore size distribution and corrected MICP pore throat size distribution are consistent in respect to pore volume
  - Corrected MICP data can be used for matrix permeability estimation
- Permeability measurement
  - Many factors can affect GRI permeability measurement results
    - Analytical procedure
    - Sample particle size
  - Influencing factors in plug permeability measurement
    - Plug size
    - Confining and pore pressures
    - Existence of micro-fracture
  - All the experimental conditions and sample pre-treatment need to be the same for comparison
  - Direct measurement of matrix permeability is still a challenge

Acknowledgements

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