



Society of Petroleum Engineers

**SPE-184042-MS**

## **Accurate Evaluation of Marcellus Shale Petrophysical Properties**

M. Elsaig, K. Aminian, S. Ameri, and M. Zamirian, West Virginia University

Copyright 2016, Society of Petroleum Engineers

This paper was prepared for presentation at the SPE Eastern Regional Meeting held in Canton, Ohio, USA, 13-15 September 2016.

This paper was selected for presentation by an SPE program committee following review of information contained in an abstract submitted by the author(s). Contents of the paper have not been reviewed by the Society of Petroleum Engineers and are subject to correction by the author(s). The material does not necessarily reflect any position of the Society of Petroleum Engineers, its officers, or members. Electronic reproduction, distribution, or storage of any part of this paper without the written consent of the Society of Petroleum Engineers is prohibited. Permission to reproduce in print is restricted to an abstract of not more than 300 words; illustrations may not be copied. The abstract must contain conspicuous acknowledgment of SPE copyright.

---

### **Abstract**

The advances in hydraulic fracturing and horizontal well technology have unlocked considerable natural gas reserves contained in the shale formations. Reliable values of the shale key petrophysical properties including permeability and porosity are necessary to estimate the original gas-in-place, predict the production rates, and optimize the hydraulic fracturing treatments. The quantification of the key shale petrophysical properties however remain challenging due to complex nature of the shale formations. Unsteady state techniques are commonly used to estimate permeability of the shale samples because the shales typically have permeability values in nano-Darcy range. The measured permeability values by these techniques however suffer from a large margin of uncertainty and reproducibility problems. Furthermore, the unsteady state measurements cannot be performed under the reservoir stress and temperature conditions.

In this study, a fully automated laboratory set-up, which has been designed and constructed for the evaluation of the ultra-low permeability petrophysical properties under the reservoir conditions, was utilized to measure the porosity and permeability of the Marcellus shale core plugs. The core plugs were obtained from a vertical well drilled specifically for the laboratory research and other scientific purposes (science well) on the site of the Marcellus Shale Energy and Environment Laboratory (MSEEL). MSEEL is a field site and dedicated laboratory in the Marcellus Shale unconventional production region of north-central West Virginia. The field site is owned and operated by Northeast Natural Energy, LLC and contains several horizontal Marcellus Shale wells. MSEEL provides a unique opportunity to undertake field and laboratory research to advance and demonstrate new subsurface technologies and to enable surface environmental studies related to unconventional energy development.

One of the core plugs obtained from the science well was used in this study for the evaluation of reliable Marcellus Shale petrophysical properties. The permeability of the core plug was measured under different gas pressures at constant net stress. The absolute permeability was then determined by applying the appropriate gas slippage correction. The porosity and the permeability of the core plug were then measured under a wide range of net stress. The measured porosity and permeability values were found to be sensitive to the stress. The permeability measurement results exhibited two distinctive behaviors with respect to the net stress that can be attributed to the natural fracture and matrix properties. The experimental results were then utilized to determine the natural fracture closure stress. The measurements also revealed that gas adsorption, when an adsorbent gas was used for the measurements, resulted in a reduction in the absolute permeability of the sample.

## Introduction

Marcellus Shale, a Devonian black shale, spans the majority of the Appalachian Basin from New York through Pennsylvania, West Virginia and also extends into Ohio and Maryland (Bartuska, et al. 2012). It is prolific in size and is located strategically in regards to markets in the Northeastern areas, Eastern Seaboard, and Great Lakes region of the United States. Located 4,500 feet to 8,500 feet underground, the natural gas in the Marcellus Shale is produced most efficiently by using innovative horizontal drilling practices in combination with massive hydraulic fracturing stimulation treatments. Even though advances in technology have unlocked considerable reserves of hydrocarbon, the emergence of the ultra-low permeability formations, such as Marcellus Shale, as a target of exploration and development has created new challenges for resource development. In order to estimate the original gas-in-place, predict the production rates, and optimize the hydraulic fracturing treatments, reliable values of the shale key petrophysical properties including permeability and porosity are necessary. The quantification of the shale petrophysical properties however is challenging due to complex nature of the shale. Shale is an organic-rich, naturally fractured formation with ultra-low matrix permeability. The gas is stored in the limited pore space of the shale matrix and also adsorbed on the organic material.

In this experimental study, the Marcellus shale petrophysical properties were measured using a laboratory set-up that was designed and assembled for fast and robust shale core plug porosity and permeability measurements under steady-state conditions. This laboratory set-up has a resolution of one millionth standard cubic centimeters per second for gas flow rate and one hundredth cubic centimeters for pore volume measurement. The automated temperature control insures that the measurements are performed under isothermal conditions. The permeability and porosity can be measured under a wide range of net stress by applying confining pressure on the shale core plug.

## Background

Shale, an organic-rich formation, is the source rock as well as the reservoir. The gas is stored in the limited pore space of these rocks and a fraction of the gas in place may be adsorbed on the organic material (Cipolla, et al. 2009). The shale formations typically have permeability values in nano-Darcy range. This ultra-low permeability is the consequence of the shale pore structure which are only a few nanometers in diameter. It is not practical to measure the permeability of the shale samples by the conventional steady-state laboratory technique due to extremely low flow rates and the length time required for establishing steady-state conditions. Consequently, unsteady-state laboratory techniques such as GRI crushed sample and pressure pulse decay have been commonly used for measuring the extremely low permeability of shale samples. However, the measured permeability values by these techniques often suffer from large margin of uncertainty and non-uniqueness.

The limitations of the unsteady-state laboratory techniques have been discussed by different authors (Sondergeld et al. 2010; Tinni et al., 2012; Zamirian et al. 2014). Key among these limitations is the inability to apply the gas slippage correction. Klinkenberg (1941) showed that the measured permeability of a rock sample by gas flow, decrease as the gas pressure increases. Klinkenberg postulated that at low pressures the slippage of the gas molecules at the surface of porous media leads to the higher gas rates. Klinkenberg (1941) demonstrated that under the slip flow regime the permeability to a gas is a linear function of the inverse of the gas pressure. However, the flow regime in the shales which have average pore sizes smaller than 10 nm (Akkutlu and Fathi 2011, Adesida et. al, 2011), can be best be described by Transition flow. Fathi et al (2012) have concluded that the permeability values measured under the Transition flow are higher than those predicted by Klinkenberg theory and the absolute permeability must be determined from the plot of the gas permeability versus the inverse of the gas pressure-squared (double-slippage correction). More recently, Mathur et al (2016) concluded from experimental studies that the Transition flow is prevalent in the shale core plugs at low pore pressures (below 250 psia) while the Slip flow is prevalent at higher

pressures (above 1000 psia). However, they did not provide any measurement results in the intermediate pore pressure range (250-1000 psia).

More recently, a laboratory set-up, referred to as PPAL (Precision Petrophysical Analysis Laboratory) here, has been shown to provide accurate and repeatable measurements of the shale petrophysical properties. PPAL measures the permeability of shale core plug under steady-state conditions (Zamirian et al., 2014a, 2014b, 2015). PPAL is inspired by Computer Operated Rock Analysis Laboratory (CORAL) designed at the Institute of Gas Technology where it was originally used to measure porosity and permeability of tight sandstones of Mesaverde (Randolph 1983) and later was adopted for shale permeability measurements (Soeder, 1988). The PPAL measurements are performed under steady-state flow conditions and the analysis of the results do not require complicated interpretations such as those for pulse-decay or GRI methods. The flow of gas passing through the core sample is measured with the extremely accurate differential-pressure transducers and as a result the permeability measurements can be performed in minutes up to hours. This allows the permeability measurements to be performed as many time as needed to produce repeatable results. The extremely accurate transducers also provide accurate porosity measurements. The measurements are performed under isothermal conditions and confining pressure. The laboratory set-up is fully automated to eliminate any human error and more importantly maintains the temperature stable within the enclosed unit. The flow rate is continuously is monitored throughout the experiment to determine when the sample is fully saturated (adsorbed or desorbed) when adsorbent gases such as  $N_2$ ,  $CH_4$  or  $CO_2$  are used for the measurements. Therefore, there is no need for gas sorption correction. The detailed description of the apparatus and its operating principal as well as the detailed experimental protocols have been presented previously (Zamirian et al., 2014a, 2014b, 2015).

One of the key advantages of the PPAL is the capability to measure the permeability and porosity of the shale core plug under a wide range of net stress by adjusting the confining and pore pressures. Shale is a naturally fractured formation. The differences in the compressibility of the natural fractures and the matrix result in a non-linear responses for permeability and porosity relative to stress. At low stress conditions, the fractures and matrix both contribute to the permeability. As stress increases, the fractures which are more compressible begin to close down resulting in a major reduction in the total permeability. At higher stress conditions, the fractures would be completely closed and the matrix would become the only contributor to total permeability. Walsh suggested that a linear relationship exists between  $(k/k_o)^{1/3}$  and  $\ln(P/P_o)$  where  $k$  is the permeability measured at a specific stress ( $P$ ), and  $k_o$  is the permeability measured at the lowest stress ( $P_o$ ). When more than one porous systems are present in the rock, the Walsh plot yield more than one straight line with different slopes. Therefore, the Walsh plot can be used to detect the presence of different media and to determine the closure stress at which the more compressible medium (fracture) is completely closed.

## Objectives and Methodology

The objectives of this study were:

1. To investigate the impact of the gas (pore) pressure on the measured permeability of the shale core plug.
2. To determine the absolute permeability of the shale core plug by applying the appropriate gas correction (slippage or double-slippage).
3. To investigate the impact type of the gas used for the permeability measurements.
4. To investigate the impact of stress on the on the measured porosity and permeability values and determine the fracture closure stress.

Precision Petrophysical Analysis Laboratory (PPAL) was utilized to measure the petrophysical properties of a Marcellus shale core plug, obtained at the depth of 7547 feet from the science well. Three sets of the experiments were performed during this study were as follows:

1. The porosity and permeability of the core plug were measured using Helium. The gas (pore) pressure was increased stepwise and then was decreased stepwise to evaluate the impact of the gas adsorption. During this set of measurements, the confining pressure was adjusted to main the net stress constant.
2. The porosity and permeability measurements under constant net stress were repeated with Nitrogen. To insure the measurments were not influenced by gas adsorption, the permeability was evaluated after the gas flow rate stabilized. It should be noted when the gas pressure is increased, the gas leaviving the core plug is less than incoming gas due to adsorption of the gas. As a result, the outgoing gas rate increases as the adsorption diminishes. Consequently, the outgoing flow rate stabilizes (reaches a maximum) when the sample is saturated with the gas at the pore pressure. The process is reversed when the gas pressure is decreaed. The outgoing gas rate decreases with diminishing desorption. Similarly, the outgoing flow rate stabilizes (reaches a minimum) when the sample is saturated with the gas at the pore pressure. The contious montoring of the flow rate throughtout the experiment allows the permability to be determined when the sample is fully saturated at each gas pressure.
3. The porosity and permeability of the core plug were measured at 7 different net stress values from 1300 psig to 7000 psig. At each net stress value, the permeability was measured at 4 different gas (pore) pressures ranging from 200 to 500 psig to evaluate the absolute permeability.

## Results and Discussion

Figure 1 illustrate the impact of gas pressure on the measured permeability values with Helium at constant net stress. As can be seen observed from Figure 1, the measured permability values were not altered by the direction of the pressure change (increasing vs. decreasing). This suggests that measurements were not impacted by gas adsorption which was expected as Helium is a non-adsorbent gas. Furthermore, it is clear from Figure 1 that the measured permeability values are impacted by the gas pressure. Therefore, it is necessary to evaluate the absolute permeability of the sample by application of the gas correction. Figures 2 and 3 illustrate the application of the gas-slippage (Klinkenberg) and double-slippage corrections to the measured permeability values at different gas pressures. As Figure 2 illustrates, the application of the gas-slippage correction results in a negative value for the absolute permeability. However, the application of the double-slippage correction provides a plausible value (124 nano-Darcy) for the absolute permeability. Therefore, it can be concluded that under the gas pressure range utilized during these experiments, the Transition flow is prevalent.

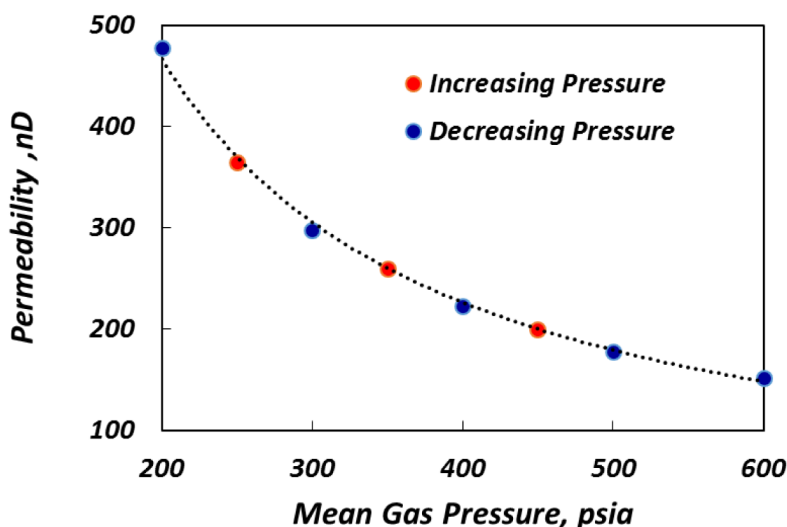


Figure 1—The Measured Permeability Values with Helium at Different Pressures under Constant Stress

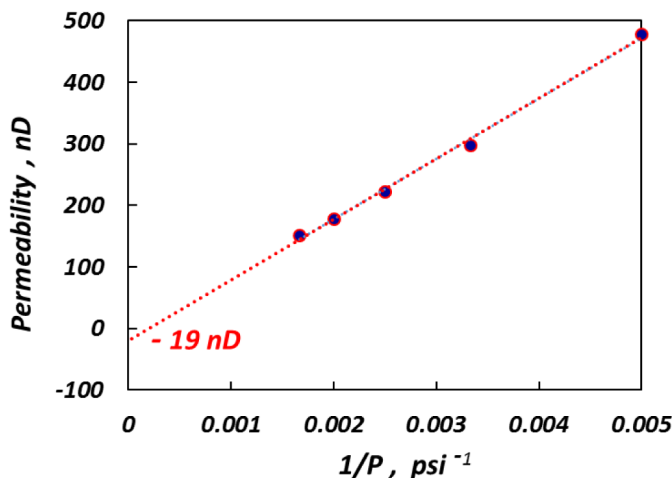


Figure 2—Absolute Permeability Evaluation by Gas-Slippage Correction

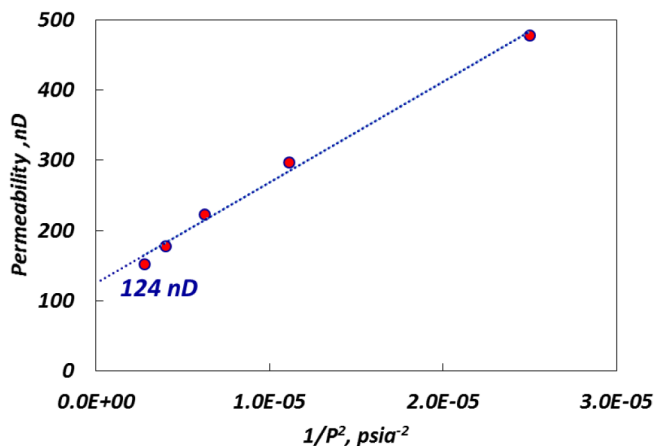


Figure 3—Absolute Permeability Evaluation by Double-Slippage Correction

Figure 4 illustrate the impact of gas pressure on the measured permeability values with Nitrogen at constant net stress. As it can be seen from Figure 4, the measurement permeability values were altered by the direction of the pressure change (increasing vs. decreasing). This suggests that measurements were impacted by Nitrogen adsorption. Similarly, the absolute permeability was determined for both sets of measurements (adsorption and desorption) by the application of the double-slippage correction as illustrated in Figure 5. The absolute permeability values, as Figure 5 illustrates, are in agreement with each other (114 nD). It is however important to note that the absolute permeability obtained from Nitrogen measurements is lower than the absolute permeability obtained from Helium measurements. This difference can be attributed to the rock expansion (swelling) due to Nitrogen adsorption which reduces the pore radius leading to a lower permeability.

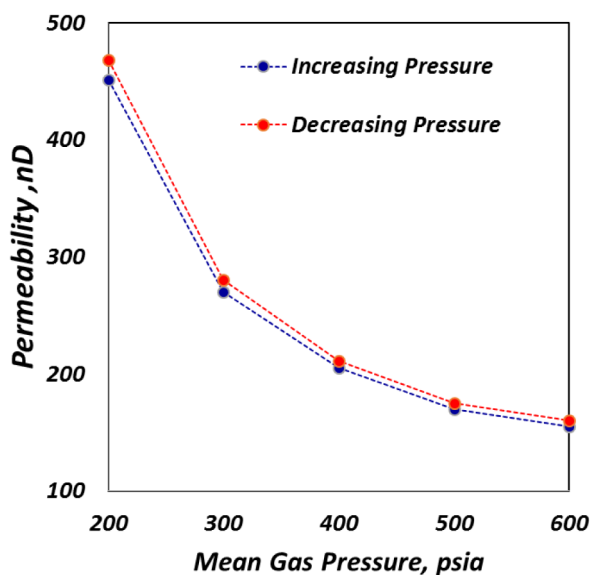


Figure 4—The Measured Permeability Values with Nitrogen at Different Pressures under Constant Stress

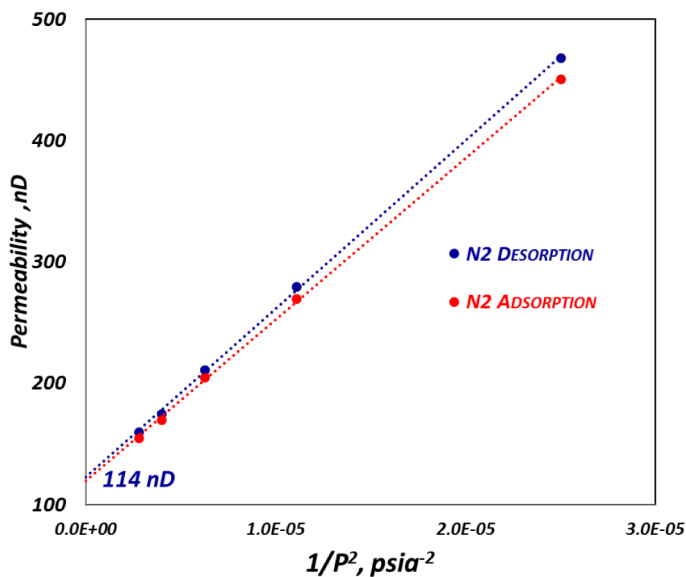


Figure 5—Absolute Permeability Evaluation by Double-Slippage Correction

The results of next set of experiments were used to determine the impact of the stress on the absolute permeability. The absolute permeability of the core plug at each stress level was determined by the application of the double-slippage correction to the measured gas permeability values at different pore pressures at that stress level. Figure 6 and 7 illustrate the impact of the net stress on the porosity and the absolute permeability. As can be clearly observed from Figure 6 and 7, the porosity and the absolute permeability response to the stress are non-linear confirming the presence of the fractures in the core plug. Figure 8 illustrate the Walsh plot for same data in Figure 7. It appears that two separate straight lines are present on Figure 8. The first straight line (blue) reflects the stress range where the permeability is dominated by the fractures. The second straight line (red) represents the stress range when the fractures are completely closed and the matrix is the only contributor to the permeability. The fracture closure stress, determined from the point where two straight lines intersect, is 4,770 psi.

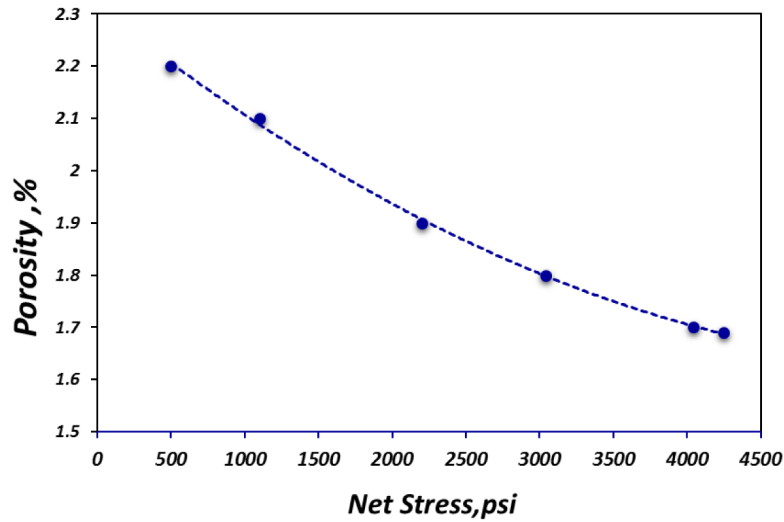


Figure 6—Impact of Net Stress on the Porosity

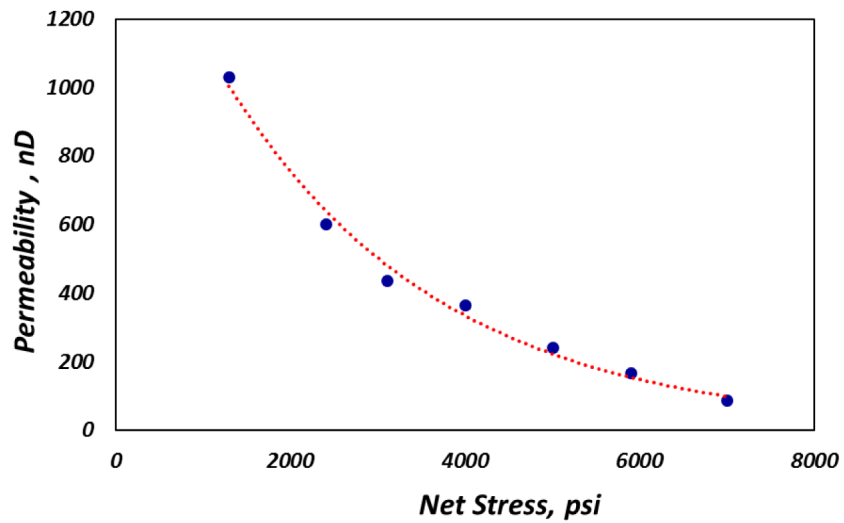


Figure 7—Impact of Net Stress on the Absolute Permeability

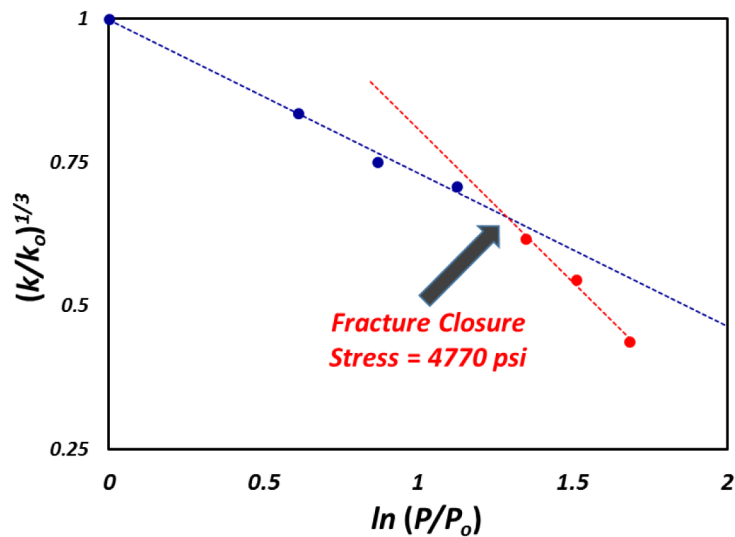


Figure 8—Walsh Plot for the Permeability and Net Stress



## Conclusions

1. The permeability and porosity of the Marcellus Shales were successfully measured under a wide range of pore and confining pressures.
2. Consistent and reliable values for the shale absolute permeability can be obtained by the application of the gas double-slippage correction to the permeability values measured at gas pressures below 500 psia.
3. The adsorption of Nitrogen to Marcellus shale sample resulted in a reduction in the absolute permeability.
4. Permeability and to a lesser extent porosity of the shale decrease with the increase in the net stress.
5. The differences in the compressibility of the fracture and matrix system present in the sample cause the permeability and porosity to exhibit non-linear responses to stress.
6. The pressure at which the fractures are fully closed was successfully determined by the Walsh plot.

## Acknowledgements

The authors with like to thank USDOE/NETL and MSEEL project for providing financial and technical support.

## References

1. Adesida, A.G., Akkutlu, I., Resasco, D., E., Rai, C. S., 2011. "Characterization of Barnett Shale Kerogen Pore Size Distribution using DFT Analysis and Grand Canonical Monte Carlo Simulations." Paper SPE 147397 presented at the SPE ATCE, Denver, Colorado.
2. Akkutlu, I. Y., and Fathi, E., 2012. "Multiscale Gas Transport in Shales With Local Kerogen Heterogeneities." *SPEJ*, **8** (4): 1002–11.
3. Bartuska, J.E., Pechiney, J.J., Leonard, R.S., & Woodroof, R.A., 2012. "Optimizing Completion Designs for Horizontal Shale Gas Wells Using Completion Diagnostics." SPE 155759, presented at the Americas Unconventional Resources Conference, Pittsburgh, Pennsylvania, 5-7 June.
4. Cipolla, C.L., Lolon, E.P., and Dzubin, B., 2009. "Evaluating Stimulation Effectiveness in Unconventional Gas Reservoirs." SPE-124843, presented at the SPE ATCE, New Orleans, Louisiana.
5. Fathi, E., Tinni, A., and Akkutlu, Y., 2012. "Shale Gas Correction to Klinkenberg Slip Theory." Paper SPE 154977 presented at the Americas Unconventional Resources Conference, Pittsburgh, Pennsylvania, 5-7 June.
6. Klinkenberg, L.J., 1941. "The Permeability of Porous Media to Liquid and Gases." Paper presented at 11th mid-year meeting, Tulsa. API Drilling and Production Practices, New York, pp 200–213.
7. Mathur, A., Sondergeld, C.H., Rai, C.S., 2016. "Comparison of Steady-State and Transient Methods for Measuring Shale Permeability." SPE Paper 180259 presented at SPE Low Perm Symposium, 5-6 May, Denver, Colorado.
8. Randolph, P.L., 1983. "Porosity and Permeability of Mesaverde Sandstone Core from the U.S. DOE Multiwell Experiment, Garfield County, Colorado." SPE 11765 Paper presented at SPE/DOE Symposium on Low Permeability, Denver, Colorado, 14-16 March.
9. Soeder, D. J., 1988. "Porosity and Permeability of Eastern Devonian Gas Shale." *SPEJ*, **3**(1): 116–124.
10. Sondergeld, C.H., Newsahm, K.E., Comisky, J.T., Rice, M.C, and Rai, C.S., 2010. "Petrophysical Considerations in Evaluating and Producing Shale Gas Reservoirs." Paper SPE 131768 presented at the SPE Unconventional Gas Conference, Pittsburgh, Pennsylvania, 23-25 February.



11. Tinni, A., Fathi, E., Agarwal, R., and Sondergeld, C., 2012. "Shale Permeability Measurement on Plugs and Crushed Samples." Paper SPE 162235 presented at the Unconventional Resources Conference, Calgary, Alberta, 30 October-1 November.
12. Walsh, J.B., 1981. "Effect of Pore Pressure and Confining Pressure on Fracture Permeability." *International Journal of Rock Mechanics and Mining Sciences & Geomechanics*, **18**(5): 429–435.
13. Zamirian, M., Aminian, K., Ameri, S., Fathi, E., 2014a. "New Steady-State Technique for Measuring Shale Core Plug Permeability." SPE 171613 Paper presented at the Unconventional Resources Conference, Calgary, Canada, 30 September-2 October.
14. Zamirian, M., Aminian, K., Ameri, S., 2014b. "A Fast and Robust Technique for Accurate Measurement of the Organic-rich Shales Characteristics under Steady-State Conditions." SPE 171018 Paper presented at the Eastern Regional Meeting, Charleston, West Virginia, 21-23 October.
15. Zamirian, M., Aminian, K., Ameri, S., 2015. "Measurement of Key Shale Petrophysical Properties." Paper SPE 174968 presented at the SPE ATCE, Houston, Texas, 28-30 Septemebr.