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EXPERIMENTAL INVESTIGATION OF MATRIX PERMEABILITY OF SHALE

A THESIS APPROVED FOR THE
MEWBOURNE SCHOOL OF PETROLEUM AND GEOLOGICAL ENGINEERING

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Dedicated to my mother

Dedicated to my mother

I would like to dedicate this work to my mother and father for always being there for me. I would like to express my sincere gratitude to Dr. Carl Sondergeld who has been my mentor and advisor during my studies at the OU. I would like to thank him for his commitment and dedication to teaching students both in and outside the classroom. I would like to thank Dr. Chandra Rax for his feedback and guidance during my entire research work. I would also like to thank Dr. Deepak Devegowda for continuous support and advice. I would like to thank Bruce Spears, Gary Stowe, Joe Flenniken and for their assistance and teaching on how to use the different equipment in the lab. I want to express gratitude to Dr. Curtis and Jeremy for their help in imaging of rocks. Special thanks to Ali Timi for the countless discussions and his incredible insight in petrophysics. Special thanks to Divyendu, Aditya, Abhinav, Saurabh, Sameer, Son, Shantam, Jyot and Aman for being my friends and helping me throughout graduate school. Finally, I would like to thank students, faculties and staffs at Mewbourne School of Petroleum and Geological Engineering for their help and friendship.

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viability of unconventional reservoirs. Laboratory measurement of shale permeability has therefore become important. Due to the inconsistencies in values reported for the pressure decay permeability measurements on crushed rock samples, measurements on core plugs have become the recent focus. An experimental apparatus has been developed in our laboratory which has the capability of performing steady state permeability measurement and transient techniques such as pressure build up and pulse decay.

Various researchers have pointed out that due to the nanometer sized pores in shale, transition region is dominant, especially at low pore pressures. In the transition flow regime, gas permeability measurement cannot be corrected with a simple Klinkenberg correction and require second order permeability corrections. An experimental investigation to understand the flow regime at different pore pressures has been carried out on two Wolfcamp shale samples. Steady state permeability of these samples was measured using helium and nitrogen at different pore pressure ranges (100 psi to 3000 psi) at a constant effective pressure of 3000 psi. It was observed that while Klinkenberg correction provided a similar value of permeability i.e. Klinkenberg and double slip, for both the gases for a pore pressure range, would be the appropriate correction for that range. It was found that when pore pressures were less than 300 psi, permeabilities required second order corrections which suggest transition flow. At pore pressures close to 1000 psi, permeabilities required the Klinkenberg correction. Interestingly, permeability measurements at pore pressures greater than 2000 psi required negligible corrections.

Abstract

Matrix permeability of shale is an important parameter needed to evaluate the economic viability of unconventional reservoirs. Laboratory measurement of shale permeability has therefore become important. Due to the inconsistencies in values reported for the pressure decay permeability measurements on crushed rock samples, measurements on core plugs have become the recent focus. An experimental apparatus has been developed in our laboratory which has the capability of performing steady state permeability measurement and transient techniques such as pressure build up and pulse decay.

Various researchers have pointed out that due to the nanometer sized pores in shale, transition regime is dominant, especially at low pore pressures. In the transition flow regime, gas permeability measurements cannot be corrected with a simple Klinkenberg correction and require second order permeability corrections. An experimental investigation to understand the flow regime at different pore pressures has been carried out on two Wolfcamp shale samples. Steady state permeability of these samples was measured using helium and nitrogen at different pore pressure ranges (100 psi to 3000 psi) at a constant effective pressure of 3000 psi. It was assumed that whichever correction provided a similar value of permeability i.e. Klinkenberg and double slip, for both the gases for a pore pressure range, would be the appropriate correction for that range. It was found that when pore pressures were less than 250 psi, permeabilities required second order corrections which suggest transition flow. At pore pressures close to 1000 psi, permeabilities required the Klinkenberg correction. Interestingly, permeability measurements at pore pressures greater than 2000 psi required negligible corrections.

Various plug permeability methods have been developed for shales which yield inconsistent result. It has therefore become important to compare them in order to standardize the permeability measurement. A round robin study for permeability measurement was conducted. Permeability of nine pyrophyllite samples was measured by four different laboratories using nitrogen (99.999% pure) as pore fluid. The laboratories used different measurement methods such as steady state, step decay and pressure build up. The confining pressure applied was 1000 psi while pore pressures were maintained below 250 psi. Second order corrections resulted in good agreement of permeability values while the Klinkenberg correction resulted in large discrepancies.

A test was performed to compare the permeability values measured on shales using the steady state method with transient methods like pulse decay and pressure build up. Permeability of five horizontal Wolfcamp shale samples as measured at an effective pressure of 3000 psi using nitrogen as pore fluid. The pore pressure ranged between 100 to 250 psi for the measurement and hence second order corrections were made. The three methods yielded similar permeabilities with differences being within 30%.

Due to growing interest in production from oil shales, it has become imperative to know whether corrected gas permeability values are comparable to liquid permeability. Comparison of permeability measurements using dodecane (liquid) and nitrogen (gas) as pore fluids was conducted. Steady state permeability measurements were made on seven horizontal Wolfcamp shales at 6000 psi confining pressure and a mean pore pressure of 3000 psi with both fluids. Corrected gas permeability values were observed to be higher than liquid permeabilities by a factor of two. NMR measurements were made before and after permeability measurements. Water saturation remained constant at 22% after

permeability measurements with either fluid. This means that the sample is not completely saturated with the flowing pore fluid during regular permeability measurements and hence relative permeability is essentially measured.

Permeability creep studies were also conducted on Wolfcamp, Eagle Ford, and Vaca Muerta shales. The steady state method was used for Vaca Muerta and Wolfcamp, while pressure build up was employed for Eagle Ford samples. Both gas, i.e. nitrogen, and liquid, i.e. dodecane, were used as pore fluids in different tests. The Vaca Muerta shale creep test was done at three different effective stresses ranging 2500 to 4500 psi while on the other samples the value of effective stress was held constant at 3000 psi. None of the shales showed any significant change in permeability with time over 30 days. This is in contrast with some published studies.

The values of effective pressure coefficient were measured for Wolfcamp and Eagle Ford shales. The steady state method was employed for Wolfcamp while pressure build up was used for Eagle Ford. Nitrogen was used as the pore fluid in both cases. The coefficient was found to be less than or equal to one which is low compared to conventional rocks. This has a positive impact during production, as effective pressure is less sensitive to changes in pore pressure.

Figure 1: Distribution of various shale plays in the US (EIA, 2011).

Chapter 1: Introduction

1.1 US shale industry

The fact that shales contain hydrocarbons is long known. But low porosity and permeability of shales had hindered their extensive commercial exploitation. Due to technological developments in the fields of horizontal drilling and hydraulic fracturing, production from shale reservoirs has become a huge success. Figure 1 shows the map of current and prospective shale plays in US. Some of the noteworthy shale formations within US are Bakken, Woodford, Eagle Ford, Wolfcamp, Marcellus, Haynesville, and Barnett shale formations.

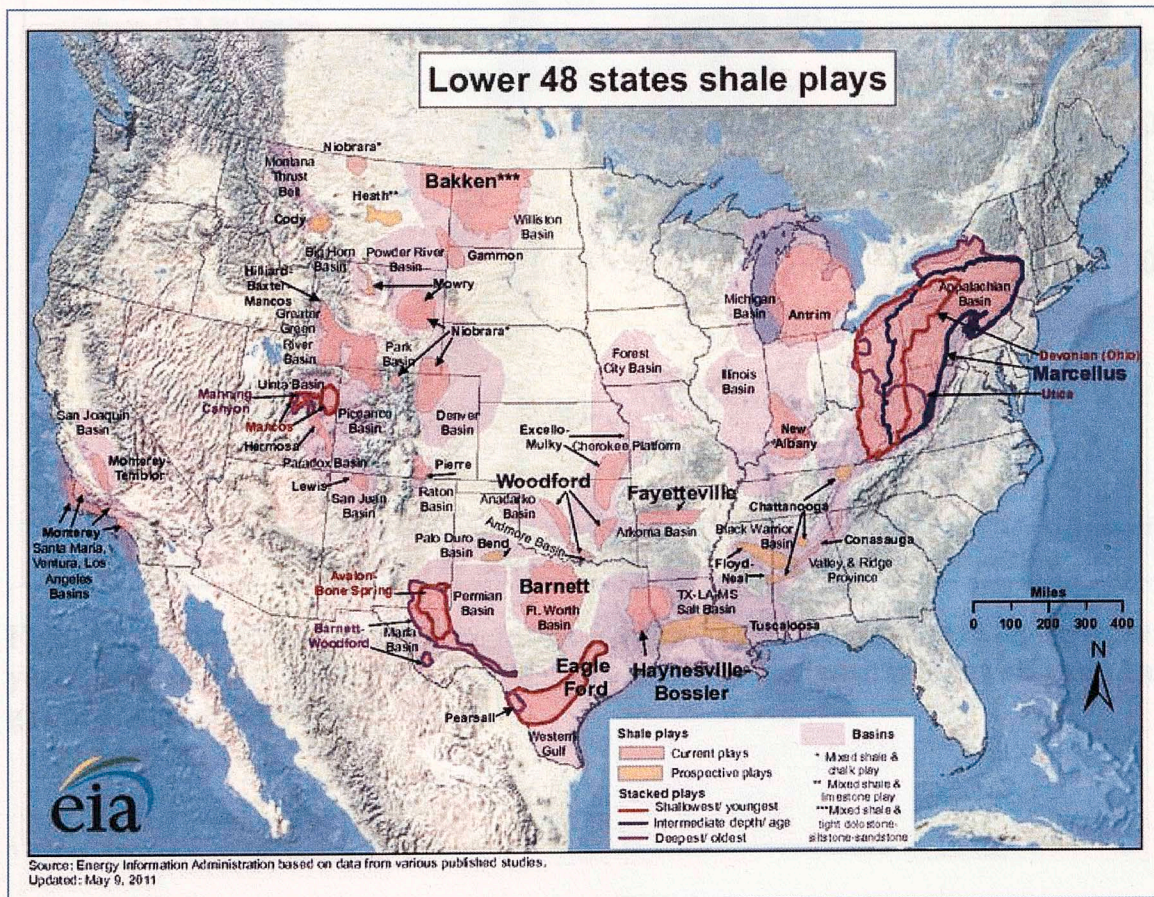


Figure 1: Distribution of various shale plays in the US (EIA, 2011).

Recently oil production from shales has gained prominence because of commercial benefits. Figure 2 provides the data of oil and gas production from shale and other tight formations. A steep increase in both graphs is seen after 2010. For tight oil production, the daily production has risen from 0.5 million BOPD in 2010 to around 3.7 million BOPD in June 2014. Most of this increase can be attributed to Eagle Ford, Bakken and Wolfcamp shale formations.

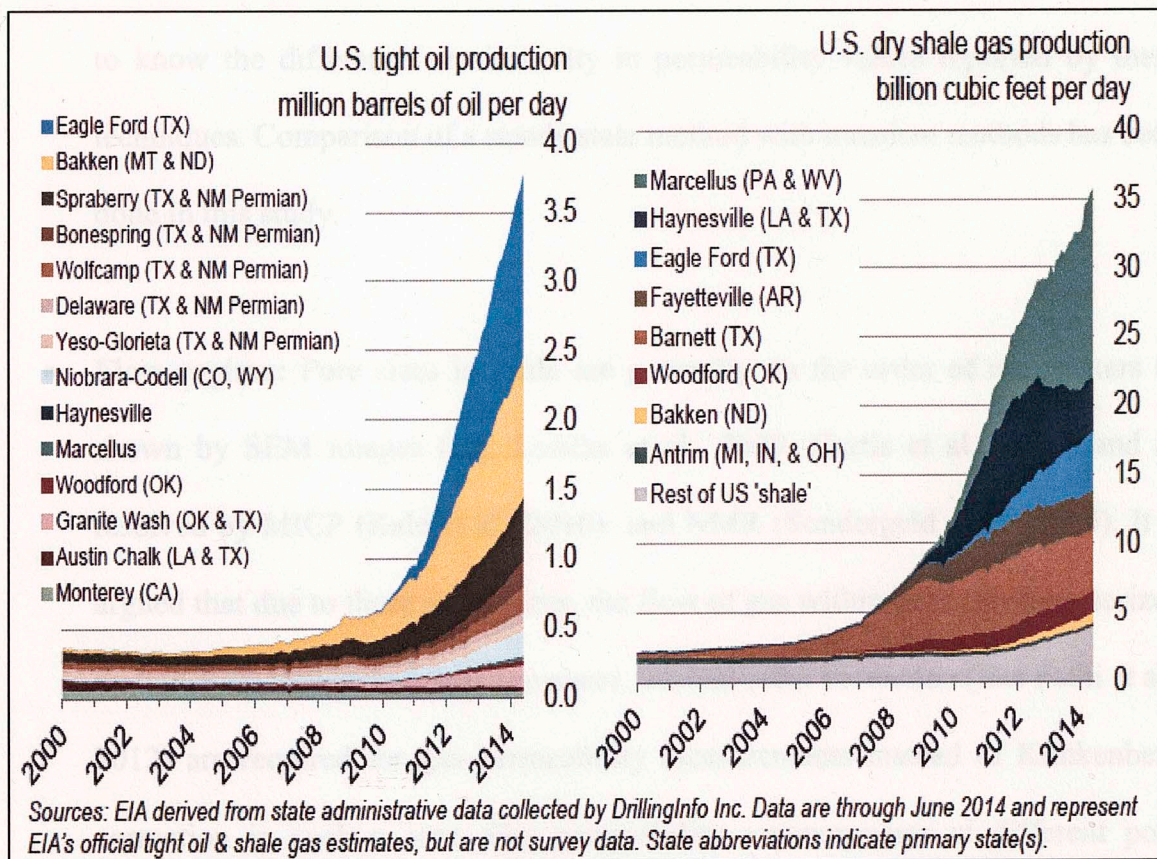


Figure 2: Estimates of gas and oil produced from tight formations ranging from years 2000 to June 2014. (EIA, 2014). Note the large increase in tight oil and shale gas production after 2010.

1.2 Objective of study

The objective for this study is to understand fundamental issues which are associated with permeability measurement in shales. These issues have been listed below.

- **Standardization of permeability measurement:** A number of methods have been developed to measure matrix permeability of shale on core plugs. These include steady state methods as well as various transient methods. It is important to know the difference or similarity in permeability values reported by these techniques. Comparison of a steady state method with transient methods has been done in this study.
- **Flow regime:** Pore sizes in shale are generally on the order of nanometers as shown by SEM images (e.g. Loucks et al., 2009, Curtis et al., 2010) and as resolved by MICP (Kale et al., 2010) and NMR (Sondergeld et al., 2010). It is argued that due to these small sizes, the flow of gas within pores is characterized by transition flow at low pore pressures. Second order correction (like Fathi et al., 2012) are required for gas permeability measurements instead of Klinkenberg correction in such a case, Gas permeability measurements at different pore pressure ranges have been conducted to know which flow regime and correction is valid for which range.
- **Influence of pore fluid:** I compared the permeability values measured using gas (nitrogen) and liquid (dodecane) pore fluids. Due to the growing interest in oil

shales, it has become important to understand whether gas permeabilities can be corrected to equivalent liquid permeabilities.

- **Permeability creep:** Permeability creep studies require permeability measurements to be conducted for extended periods of time while keeping the confining and pore pressure constant. These tests are done to observe permeability changes with time which would impact the long term production performance of the reservoir. In order to study this, permeability creep measurements have been made on Eagle Ford, Wolfcamp and Vaca Muerta shale samples.
- **Effective pressure law:** The value of the effective pressure coefficient (n) is important to decide whether confining or pore pressure has more influence on permeability. Measurements to evaluate values of effective pressure coefficient have been conducted on Eagle Ford and Wolfcamp shale.

Chapter 2: Literature Review

2.1 Methods for measuring ultra-low permeability of shale

Permeability for shales is generally found to be in the order of nanodarcies. The methods for measuring shale permeability require a sample to either be crushed or in the form of core plugs. Also the methods are either steady state or transient. Some of these methods are described below.

2.1.1 Pressure decay technique

The pressure decay technique uses crushed rock samples to measure permeability. Luffel et al. (1993) were the first to apply this technique to measure permeability of shale. They suggested samples should be crushed into small sizes close to 0.7mm; they assumed this size eliminated cracks. The experimental setup of a pressure decay measurement is similar to Boyle's law porosity configuration. It consists of a reference volume and a sample cell. The crushed rock sample is placed inside the sample cell. The pore fluid (gas) is first introduced in the reference volume. After reaching thermal equilibrium, the gas flows inside the sample cell and subsequently into the crushed rock particles which results in a slower pressure drop. The recorded pressure decay is history matched using an algorithm to estimate the permeability of the sample.

Over the years, various researchers have found problems with crushed rock technique to measure permeability in shale. Passey et al. (2010) have reported inconsistency in permeability values from three different laboratories using the pressure decay measurement. Permeability variation of more than two orders of magnitude was observed for the crushed shale samples tested in their study. Cui et al. (2009), Profice et al. (2012)

and Tinni et al. (2012) have pointed out that the size of the crushed rock sample can affect the measured permeability. These researchers report that permeability values are found to increase with increasing size of crushed rock particles, and this variation can be as high as two orders of magnitude. Tinni et al. (2012) also observed that the equilibrium pressure which is the final pressure at which decay stops, also changes the value of measured permeability. On increasing the equilibrium pressure from 55 to 260 psi, permeability variation by as high as two orders of magnitude was observed. Figure 3 shows permeability values measured by Tinni et al. (2012) as a function of equilibrium pressure and different particle sizes for a Haynesville shale sample. They have also found that crushing does not remove all the cracks from the sample. Different crushed particle sizes of 3.5 mm and 0.7mm showed microcracks of 10-20 μm when observed with a Micro CT scanner. This method does not allow the application of confining pressure to close microcracks nor does it measure a directional permeability.

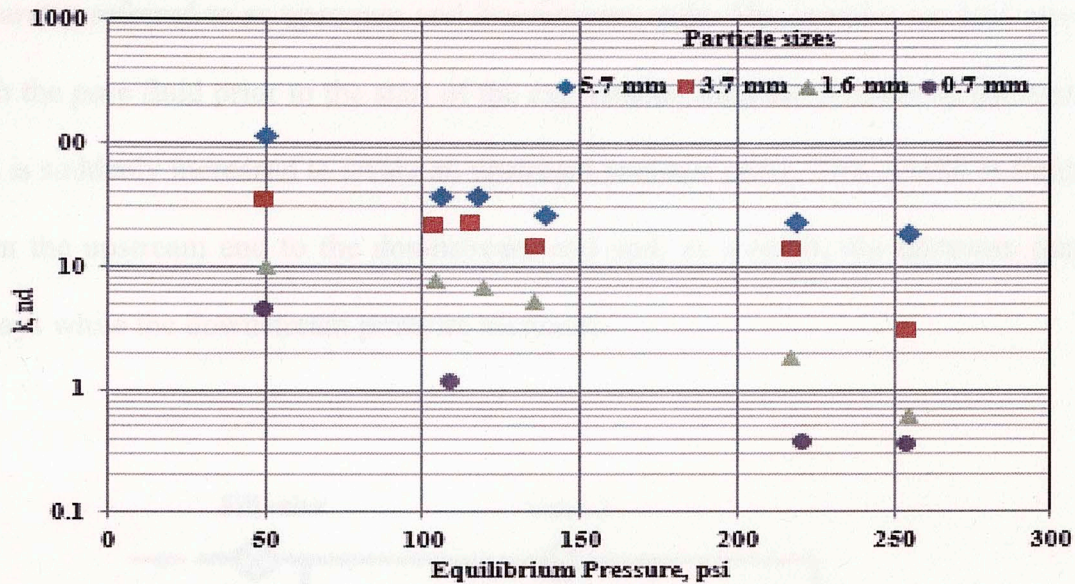


Figure 3: Permeability variation as a function of equilibrium pressure for a Haynesville shale sample (Tinni et al., 2012). Permeability decreases by more than an order of magnitude with decreasing pore size (5.7 mm to 0.7 mm) and increasing equilibrium pressure (55 to 260 psi).

2.1.2 Plug permeability measurement methods

Plug permeability measurements allow the application of confining pressure on the samples which helps in closing microcracks. A brief description of these techniques is given below. Some techniques such as pressure pulse decay, pressure build up and the steady state method have been discussed in detail in Chapter 3.

Pulse decay method is a popular method for measuring permeability of ultra-low permeability rocks such as shale. It was introduced by Brace et al. (1968) to measure permeability of Westerly granite. Figure 4 show a typical apparatus for conducting pulse decay measurement. In the experimental setup a core sample is placed inside a confining pressure cell. The pore fluid flows into the core from opposite ends of the experimental

apparatus referred to as upstream and downstream ends. The samples are first saturated with the pore fluid prior to the start of the experiment, then the pressure in the upstream end is suddenly increased to create an upstream pressure pulse. This results in fluid flow from the upstream end to the downstream end and, as a result, the upstream pressure decays while the downstream pressure increases.

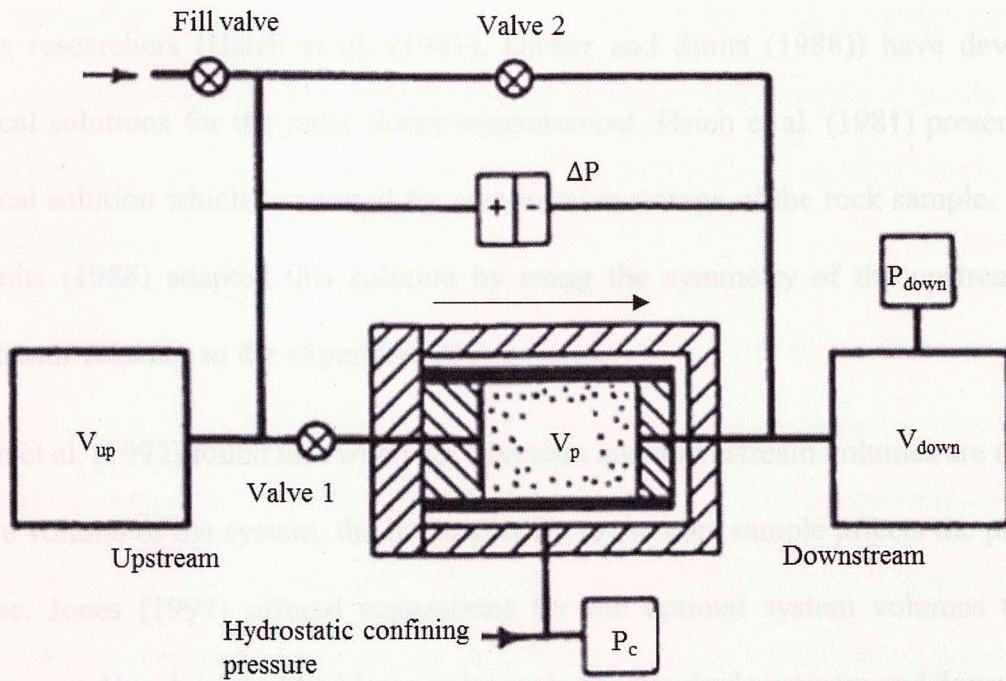


Figure 4: Typical pressure pulse decay apparatus (Jones 1997). After saturation, an upstream pressure pulse results in flow from upstream to downstream end. The resultant pressure decay in upstream is used to calculate permeability.

The solution proposed by Brace et al. (1968) uses this pressure decay to measure the permeability of the sample. This solution assumes that there is negligible compressive storage in the sample.

The compressive storage (S_s) of the rock sample is defined as:

$$S_s = (\beta_{\text{eff}} - \beta_s) + \varphi_e(\beta_f - \beta_s) \quad 2.1$$

In equation 2.1, β_{eff} is the effective compressibility of the rock, β_s is the compressibility of the solid matrix and β_f is the fluid compressibility and φ_e is the effective porosity of the sample. The assumption by Brace et al. (1968) is that the porosity of the sample is very small and the compressibility of the solid matrix and the effective compressibility of the rock are negligible.

Various researchers (Hsieh et al. (1981), Dicker and Smits (1988)) have developed analytical solutions for the pulse decay measurement. Hsieh et al. (1981) presented an analytical solution which accounted for compressive storage of the rock sample. Dicker and Smits (1988) adapted this solution by using the symmetry of the upstream and downstream volumes in the experimental apparatus.

Kamath et al. (1992) found that when the upstream and downstream volumes are close to the pore volume of the system, the heterogeneity in the core sample affects the pressure response. Jones (1997) offered suggestions for the optimal system volumes for the measurement. He advised adding large volumes to the standard upstream and downstream volumes only during the saturation stage in order to shorten the saturation time. He also proposed that the standard upstream and downstream volumes should be at least 2 to 10 times greater than the pore volume of the sample to eliminate the effect of heterogeneity on the pressure response.

Metwally and Sondergeld (2011) modified the solution developed by Hsieh et al. (1981) and Dicker and Smits (1988) to develop the pressure build up method of measuring permeability. This method was based on creating an infinite storage reservoir in the

upstream volume by maintaining its pressure constant throughout the measurement. This results in a pressure build up in the downstream volume. The recorded differential pressure data is then used to measure permeability.

Lasseux et al. (2012) have proposed another transient technique to measure permeability of shale which is called “Step Decay”. The experimental procedure is similar to a pulse decay measurement but the computation of permeability in this method is based on history matching. The upstream pressure is used as an input to a model while the downstream pressure is used to determine the permeability of the sample. Also during the experiment, the pressure in the upstream section is increased periodically which leads to the name step decay.

Clarkson et al. (2012, 2013) have proposed a transient method of measuring shale permeability using just one of the two upstream and downstream sections. This method starts with injecting gas into the core sample using a pump until a predetermined pore pressure value is reached. The pump is then disconnected from the system and pressure in the core sample is allowed to fall off as it stabilizes. After equilibration, pore fluid is then produced from the core sample. Permeability values can be obtained by finding the time for end of linear flow from the experimental data during both the falloff and production stages.

Sinha et al. (2012), Lasswell et al. (2013) and Chhatre et al. (2014) have reported some steady state permeability measurements on shale. The main principle behind these measurements is that a constant pressure gradient is applied on the sample by maintaining constant upstream and downstream pressures. Flow rates are monitored constantly throughout the measurement. On reaching steady state flow, a constant flow rate is

observed. This flow rate value is used in Darcy's Law to obtain the permeability of the sample. This, of course, is subject to the restrictions for Darcy's Law specifically laminar flow and no fluid rock interactions.

Different commercial or research laboratories use these different techniques to measure shale matrix permeability. It has hence become important to compare these methods in order to standardize the permeability measurement for shales. The steady state method has been compared with some transient techniques such as pressure build up and the pulse decay method in chapter 4.

2.2 Influence of flow regime on permeability

Knudsen number (Kn) is a dimensionless number defined as the ratio of mean free path of the gas molecule (λ) to the pore diameter (d) as given by equation 2.2.

$$Kn = \frac{\lambda}{d} \quad 2.2$$

The mean free path is inversely proportional to the pressure according to Heidemann et al., (1984):

$$\lambda = \frac{K_b \cdot T}{\sqrt{2} \cdot \pi \cdot \delta^2 \cdot P} \quad 2.3$$

In equation 2.3, P is pressure (Pa), K_b is Boltzmann constant (1.3805×10^{-23} J/K), T is temperature (K), and δ is the collision diameter of the gas molecule (nm).

Different flow regimes have been demarcated based on different values of Knudsen number. Figure 5 shows the different flow regimes based on different values of Knudsen number.

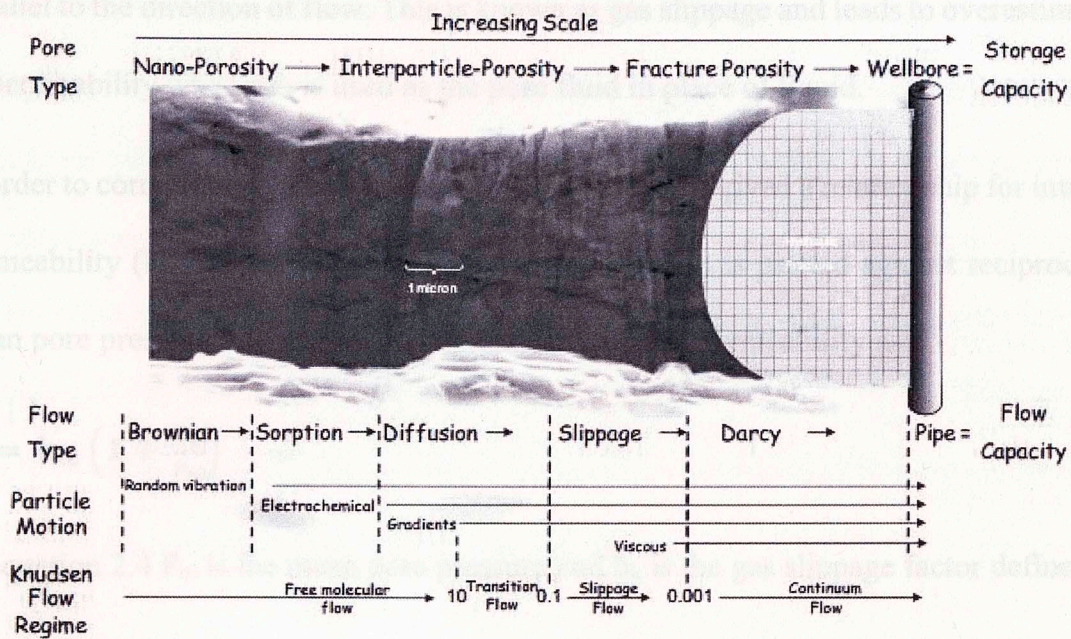


Figure 5: Relationship between Knudsen number and flow regime type (Sondergeld et al., 2010).

As Knudsen number is inversely proportional to pore size and pore pressure, it means that as the pore pressures becomes lower and the pore diameters become small, the flow regime can change from Darcy to slip and from slip to transition flow. Gas flow in slip and transition flow regimes require corrections to convert them into equivalent liquid permeabilities.

Klinkenberg (1941) explained the reason for correction in slip flow regime. From the classical theory of fluid mechanics, it can be inferred that the velocity profile in a capillary tube is parabolic with velocity of the fluid maximum at the center and zero at the walls. This is regarded as the no slip condition. Such flow is shown by liquids. However in case of gases, molecules present at the vicinity of the pore wall have a net velocity component

parallel to the direction of flow. This is known as gas slippage and leads to overestimation of permeability when gas is used as the pore fluid in place of liquid.

In order to correct for gas slippage, Klinkenberg (1941) derived a relationship for intrinsic permeability (k_{∞}). When apparent gas permeability (k_a) is plotted against reciprocal of mean pore pressure, the intercept provides the intrinsic permeability as:

$$k_a = k_{\infty} \left(1 + \frac{b_k}{P_m} \right) \quad 2.4$$

In equation 2.4 P_m is the mean pore pressure and b_k is the gas slippage factor defined as:

$$\frac{b_k}{P} = \frac{4c\lambda}{r} \quad 2.5$$

In equation 2.5 c is a constant, λ is the mean free path and r is the pore. SEM images of shales by various authors Curtis et al., (2010), Loucks et al. (2009) have showed that shales have pore sizes in the order of nanometers. Pore sizes resolved by MICP (Kale et al., 2010) and NMR (Sondergeld et al., 2010) support this observation. Transition flow can thus occur at lower pore pressures for these nanometer sized pores. Fathi et al. (2012) argued that the Klinkenberg equation ignores the momentum that the rebounding gas molecules can carry to the bulk fluid and that this factor would be important to consider for transition flow in shales.

They incorporated this effect by altering the Klinkenberg equation as shown in equation 2.6 and termed this effect as “double slippage”.

$$k_a = k_{\infty} \left[1 + \left(\frac{b_k}{P_m} \right)^2 \left(\frac{L_{KE}}{\lambda} \right) \right] \quad 2.6$$

In equation 2.6, L_{KE} is a length scale associated with kinetic energy of rebounding molecules. This equation tells us that the value of intrinsic permeability is provided by the intercept when apparent gas permeability values are plotted against reciprocal of mean pore pressure squared. As this equation involves second order of pressure it is termed a second order correction. Various second order correction equations have been provided by other authors like Beskok and Karniadaskis (1999), Civan (2010) and Sakhae-Pour (2012) for transition flow. These corrections require calculation of Knudsen number and thus require the determination of pore throat sizes.

Zamirian et al. (2014) shows the use of double slip correction at the pore pressure range of 100 to 500 psi (see figure 6). Permeability measurements are made on a Marcellus shale sample by steady state technique using both nitrogen and helium as pore fluids. The pore pressure range is 100 to 500 psi and multiple permeability measurements are made within this range. After making Klinkenberg correction there is inconsistency in the permeability values for both the fluids and the Klinkenberg corrected permeability for helium gas is negative. On the other hand using the double slip correction results in similar values of corrected permeability for both the gases. This shows the importance of second order corrections at these low pore pressures.

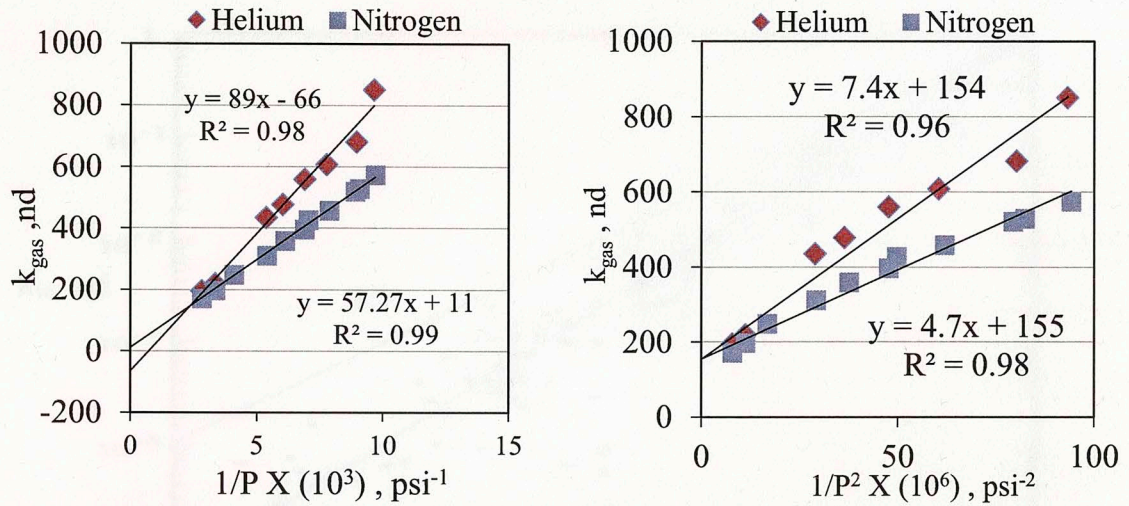


Figure 6: Klinkenberg(left) and Double(slip) correction on the same set of data measured on Marcellus shale (Zamirian et al., 2014).

2.3 Influence of pore fluid

Jones and Owens (1980) measured permeability values of 100 tight gas sand samples using brine and nitrogen as pore fluids by the steady state method. The experiments were conducted at effective pressure equal to the reservoir stress appropriate for each sample. Klinkenberg corrected gas permeability values were plotted against brine permeabilities as shown in figure 7; note the brine permeability is always less than the corrected gas permeability. They observed a relationship between the permeability values measured using the two fluids. The relationship is given by equation 2.7 where k_w is brine permeability and is k_∞ is Klinkenberg corrected permeability.

$$k_w = k_\infty^{1.32} \quad 2.7$$

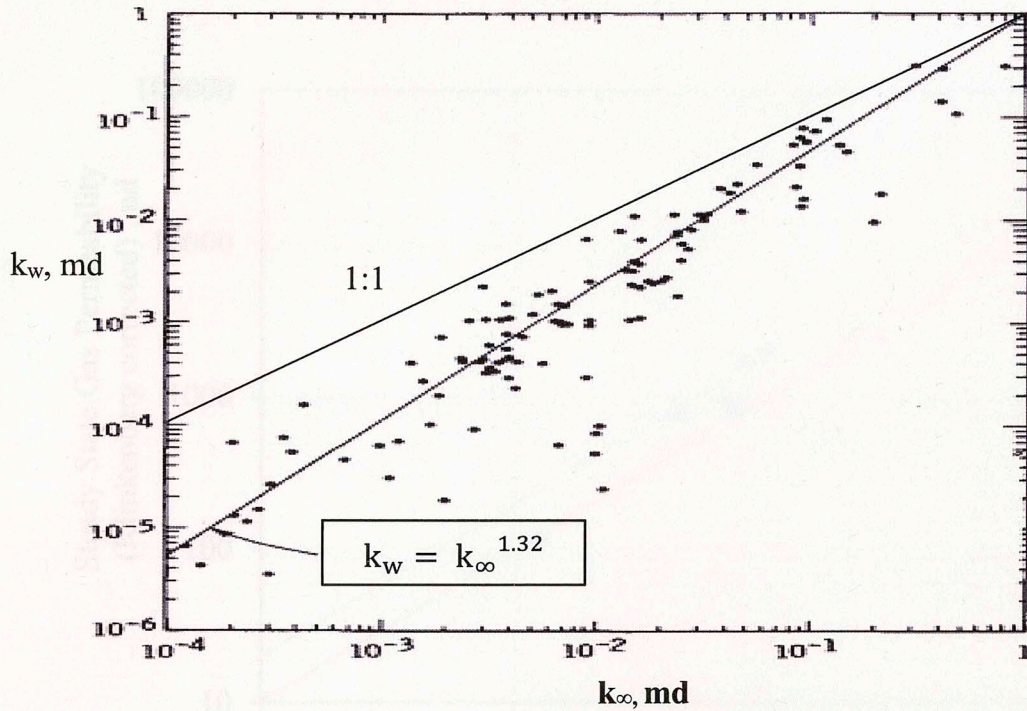


Figure 7: Brine permeability (k_w) vs. Klinkenberg corrected permeability (k_∞) for 100 tight sands (Jones and Owens, 1990). Note $k_w < k_\infty$. Permeabilities are measured using steady state technique.

This relation was developed for samples with permeabilities less than 1 md. The scatter in the data increases when brine permeability values are of the order of 10^{-4} or 10^{-5} md as seen in figure 7.

Chhatre et al. (2014) compared liquid permeability with Klinkenberg corrected gas permeability for Vaca Muerta shale as shown in figure 8. The TOC range for the samples is 0 to 12 %. Toluene or decalin are used as the liquid pore fluids for this test. A steady state method was used to measure permeability. The pore pressure used in the sample was close to reservoir pressure and the effective pressure is close to in-situ stress. As observed from figure 8, the corrected gas permeabilities are greater than the measured liquid permeability within a factor of two.

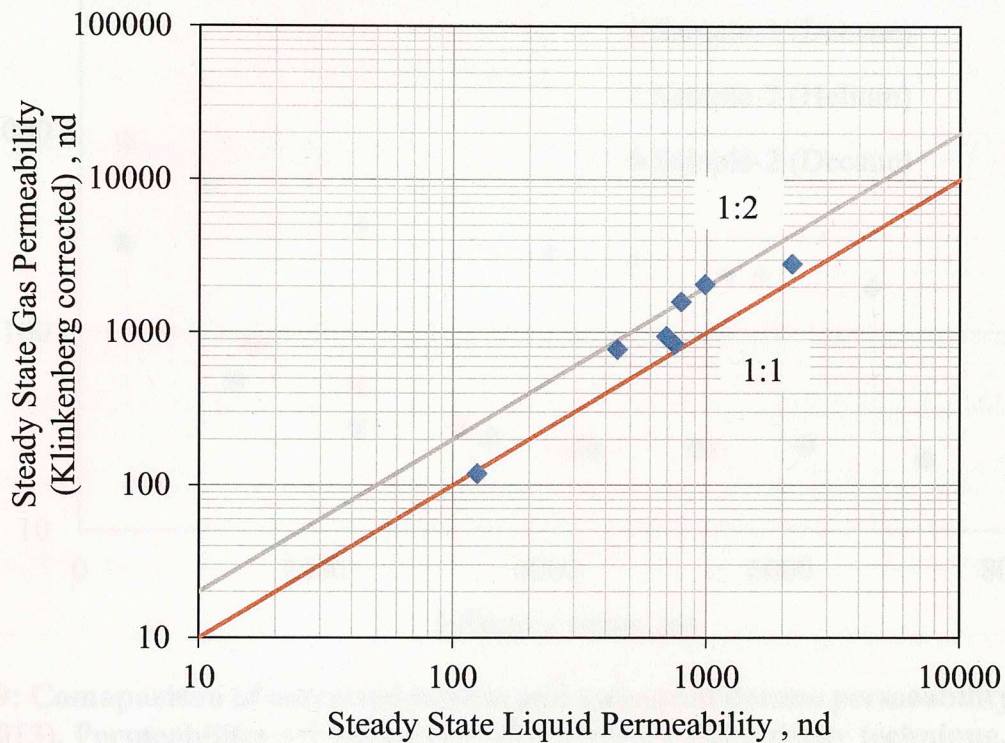


Figure 8: Steady state gas and liquid permeability comparison (Chhatre et al., 2014). Note k_{gas} is greater than k_{liquid} within a factor of 2.

Cui et al. (2013) compared the corrected helium gas permeabilities to liquid decane permeabilities for two Montney shale samples. A pressure pulse decay method was used to measure permeability. Permeability measurements are made at different effective pressures starting from 100 psi to 7200 psi. The pore pressure used in the study for helium permeability measurements was 1000 psi. Decane permeabilities are lower than corrected helium permeabilities by almost an order of magnitude.

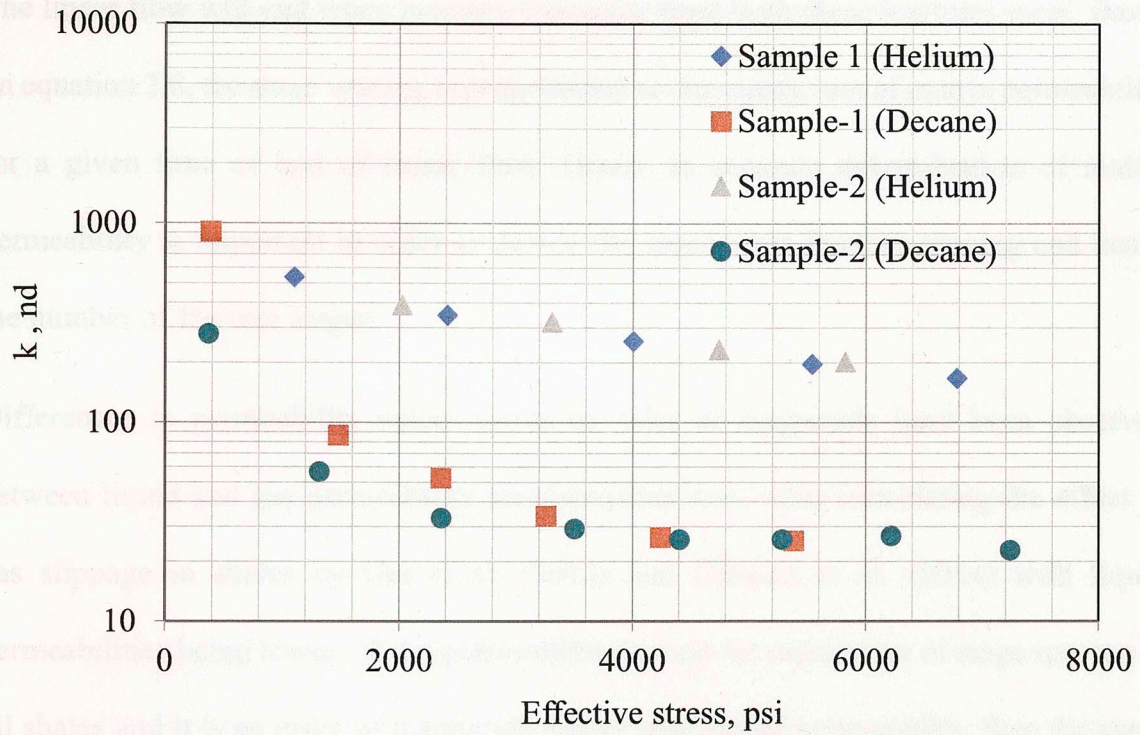


Figure 9: Comparison of corrected helium and measured decane permeability (Cui et al., 2013). Permeabilities are measured with pressure pulse decay technique. Note k_{helium} is greater than k_{decane} by almost an order of magnitude.

2.4 Permeability Concept

In shales multistage hydraulic fracturing is done to make production from these ultra-low permeability rocks economic. During the early production life from shales, transient linear flow perpendicular to these fracture stages is assumed. Before the end of linear flow only a certain volume of the stimulated volume would be drained. Wattenbarger et al., (1998) relate the distance of investigation (y) during the linear flow period to matrix permeability (k) and time (t):

$$y = a \cdot \sqrt{\frac{k \cdot t}{\phi \cdot \mu \cdot C_t}} \quad 2.8$$

Where ϕ is porosity, μ is viscosity, C_t is total compressibility and 'a' is a constant.

The linear flow will end when pressure transients from both these fractures meet. Based on equation 2.8, the stage spacing is proportional to the square root of matrix permeability for a given time of end of linear flow. Hence an accurate determination of matrix permeability is important in order to decide the appropriate fracture spacing and hence the number of fracture stages.

Differences in permeability values up to an order of magnitude have been observed between liquid and gas permeability measurements even after considering the effect of gas slippage in shales by Cui et al. (2013) and Chhatre et al. (2014) with liquid permeabilities being lower. If gas permeability is used for calculation of stage spacing in oil shales and it is an order of magnitude higher than liquid permeability, then the stage spacing will be overestimated by approximately 3.2 times.

2.4 Permeability Creep

Permeability creep measurements are done to observe permeability changes with respect to time. The confining and pore pressure in these measurements are held constant. Yashura et al. (2012) performed permeability creep measurements on Berea sandstone and fractured Horonobe mudstone. For Berea sandstone the measurement was conducted on two different samples at different effective pressures as can be seen in figure 10. The black (dark shade) curve (squares) is for the sample where effective pressure is 1100 psi (7.5 MPa) while the red (light shade) curve (circles) is for the sample when effective pressure is 2200 psi (15 MPa). Permeability was measured using a steady state method with water as pore fluid. This creep test is conducted for around 450 days. For the sample with effective pressure as 1100 psi (7.5 MPa), the permeability values trend around 10

md until 300 days. For the second sample where effective pressure is 2200 psi (15 MPa), the permeability values trend around 2 md value till 300 days. After 300 days an increase in permeability is observed in either sample. No reduction in permeability is observed in either sample.

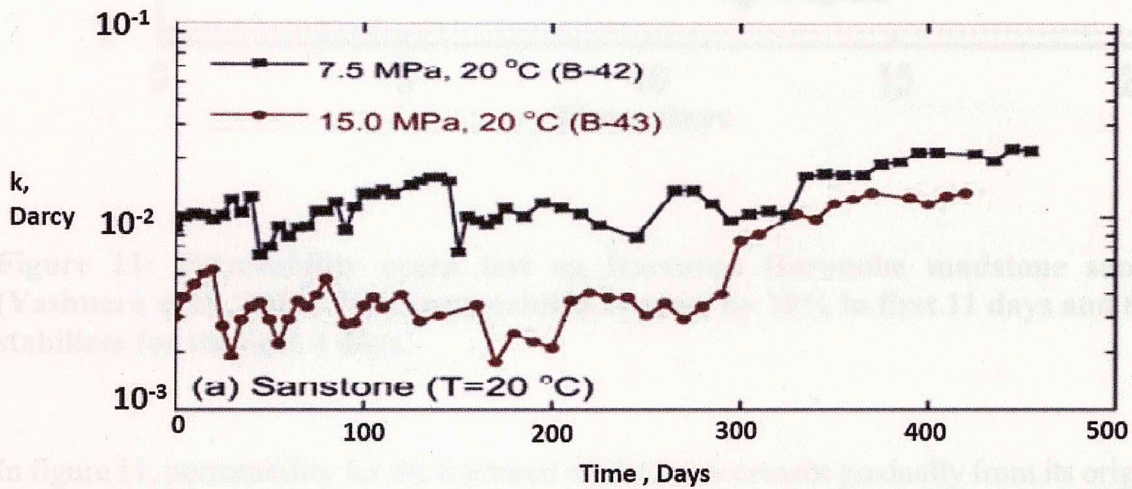


Figure 10: Permeability creep test on Berea sandstone (Yashuara et al., 2012). Note increase in permeability after 300 days.

Figure 11 shows the permeability creep measurement on a fractured sample from Horonobe mudstone formation. A steady state method is used to calculate permeability for this test. Silicon oil is used as the pore fluid. The effective stress is 450 psi.

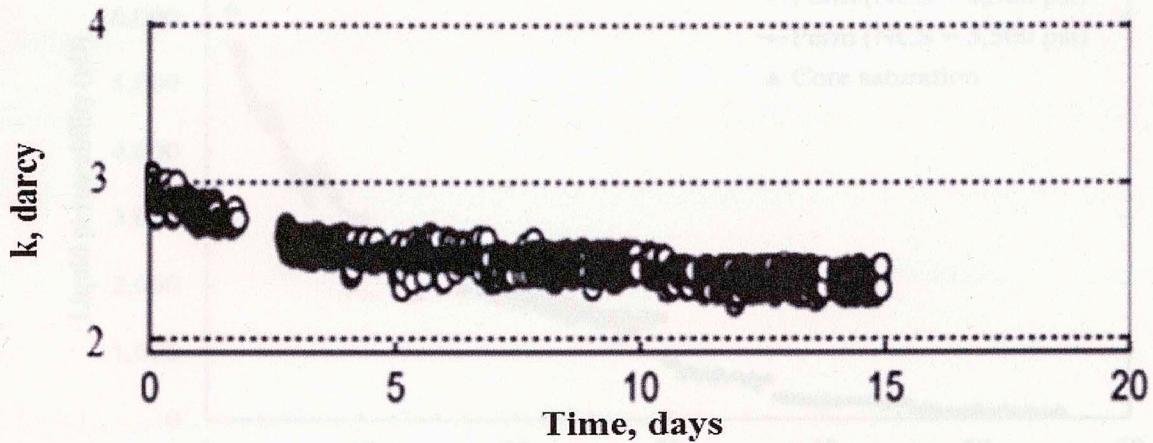


Figure 11: Permeability creep test on fractured Horonobe mudstone sample (Yashuara et al., 2012). Note permeability reduces by 20% in first 11 days and then stabilizes for the next 4 days.

In figure 11, permeability for the fractured mudstone decreases gradually from its original value of 3 darcy by about 20% within the first 11 days. After that it reaches a stable value and this value remains constant for the next 4 days.

Permeability creep measurements on Vaca Muerta shale have been reported by Chhatre et al., 2014 (see figure 12).

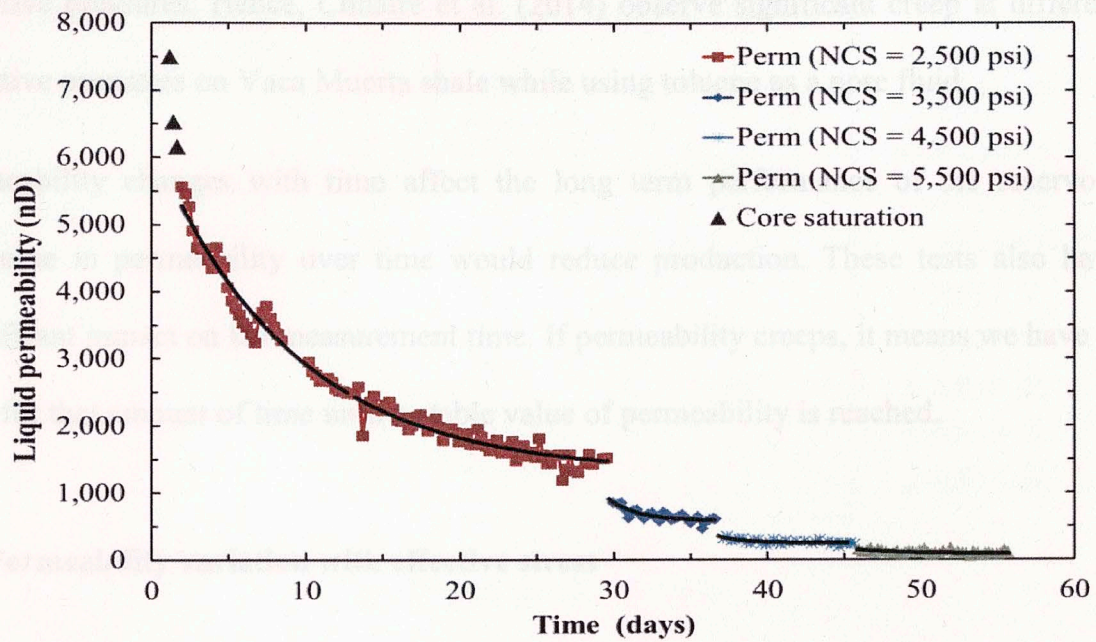


Figure 12: Permeability creep test on Vaca Muerta shale sample (Chhatre et al., 2014). Permeability decreases from 5.5 μ d to 1.5 μ d at 2500 psi effective pressure after 30 days. Permeability decrease as a function of time is also observed at 3500, 4500 and 5500 psi effective stress.

Permeability was measured using a steady state method. The pore fluid used is toluene which is a strong organic solvent. This violates the fundamental assumption of Darcy's law regarding no fluid rock interaction. Permeability creep measurements are made at different effective pressures starting from 2500 psi and increasing to 5500 psi in increments of 1000 psi. The sample is saturated until at least one pore volume of the sample flows through the sample. Average flow rates are calculated regularly to compute permeability. At 2500 psi effective pressure, the permeabilities gradually decrease from 5.5 μ d to 1.5 μ d after 30 days. The permeability values keep changing with time. After that the effective stress is increased to 3500 psi through changes in the confining pressure. As a result permeability suddenly drops to a value close to 850 nd. Then is followed by another gradual change in permeability with time. Similar response is observed at other

effective pressures. Hence, Chhatre et al. (2014) observe significant creep at different effective pressures on Vaca Muerta shale while using toluene as a pore fluid.

Permeability changes with time affect the long term performance of the reservoir. Decrease in permeability over time would reduce production. These tests also have significant impact on lab measurement time. If permeability creeps, it means we have to wait for that amount of time until a stable value of permeability is reached.

2.5 Permeability variation with effective stress

Variation of permeability as a function of effective pressure in shales have been reported by various authors e.g. Bustin et al. (2008) and Tinni et al. (2012). They have observed that permeability values decrease by orders of magnitude as a function of effective pressure. Figure 13 shows the change in permeability observed by Tinni et al. (2012) for Ordovician age shale as the confining pressure changes from 1000 to 5000 psi keeping the pore pressure constant at 100 psi. A pressure build up method is used to measure permeability in this experiment and nitrogen is used as the pore fluid. Permeability is observed to decrease by as much as 2 orders of magnitude with increasing confining pressure.

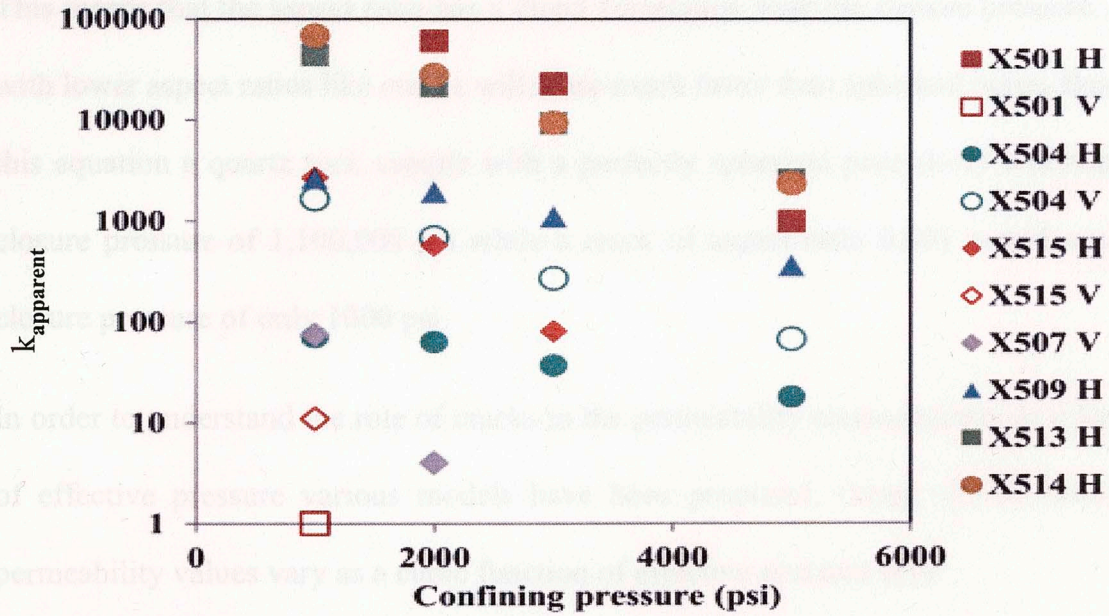


Figure 13: Apparent gas permeability as a function of confining pressure is plotted for 10 Ordovician age shale samples at a constant pore pressure of 100 psi (Tinni et al., 2012). Permeability reduction up to 2 orders of magnitude is observed as confining pressure increases from 1000 to 5000 psi. Filled symbols are horizontal cones and open symbols are vertical samples. Only samples X501, X504 and X15 have both orientation samples.

The shape of pores play an important role in understanding the change in permeability as a function of effective stress. Walsh (1965) estimated the pressure required to close a given pore is given as:

$$P_{\text{closure}} = \frac{E\lambda}{4\pi(1-\nu^2)} \quad 2.9$$

where E represents the Young's modulus, ν is the Poisson's ratio of the material and λ denotes the aspect ratio of the pore defined as:

$$\lambda = \frac{a}{c} \quad 2.10$$

In equation 2.10, c is the length and a is the width of the pore.

This means that the aspect ratio has a direct correlation with the closure pressure. Pores with lower aspect ratios like cracks will close much faster than spherical pores. Based on this equation a quartz rock sample with a perfectly spherical pore ($\lambda=1$) will require a closure pressure of 1,100,000 psi while a crack of aspect ratio 0.001 would require a closure pressure of only 1000 psi.

In order to understand the role of cracks in the permeability measurements as a function of effective pressure various models have been proposed. Gangi (1978) found that permeability values vary as a cubic function of effective pressure (P_e):

$$k = k_o \left[1 - \left(\frac{P_e}{S_A} \right)^m \right]^3 \quad 2.11$$

In equation 2.11, k_o denotes the reference permeability at zero effective pressure, S_A is an effective modulus of asperities and m is constant between 0 and 1 which characterizes the distribution of asperity heights.

Walsh (1981) proposed another fracture permeability model:

$$k = k_o \left[1 - \sqrt{2} \cdot \frac{h}{a_0} \cdot \ln \left(\frac{P_e}{P_0} \right) \right]^3 \quad 2.12$$

In equation 2.12, k_o denotes the reference permeability at reference effective pressure P_0 , h denotes the root mean square of height distribution of asperities and a_0 represents the half width of fracture aperture at effective pressure P_0 .

Walsh model is applied to the permeability data by plotting $(k/k_o)^{1/3}$ against natural logarithm of (P_e/P_0) . If a straight line is observed it represents the influence of fractures or cracks at those particular effective pressures. Therefore, to apply the Walsh model,

requires permeability variation with effective stress but information regarding asperities heights (h) is not needed. Kassis and Sondergeld (2010) provide the application of the Walsh model for an unaltered fractured shale as can be seen in figure 14. The straight line in the figure suggest that cracks control permeability for the shale at the given effective pressures ranging from 1300 to 3500 psi.

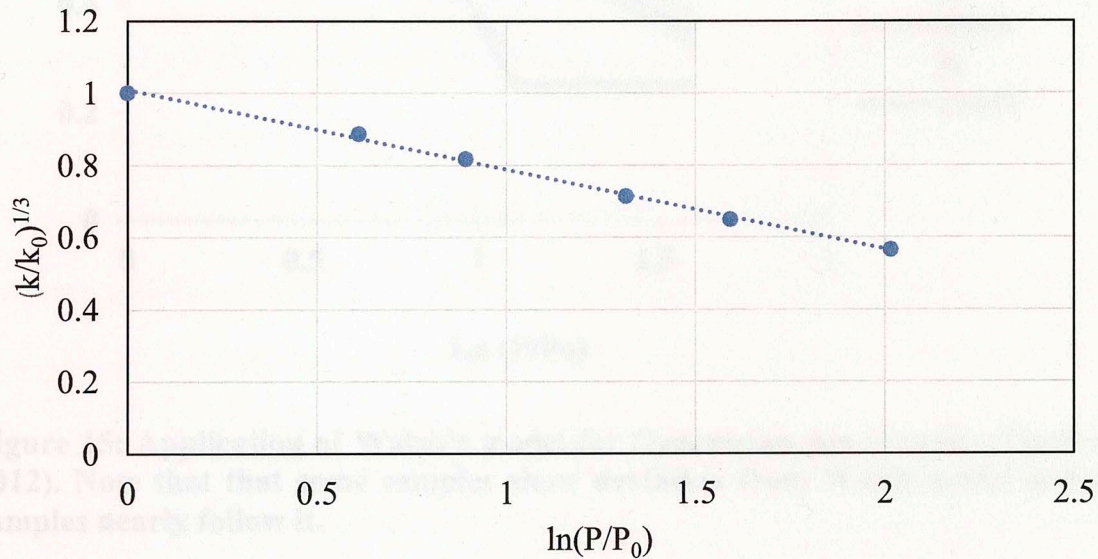


Figure 14: Application of Walsh model to unaltered fractured shale (Kassis and Sondergeld, 2010). The straight line indicates permeability values follow Walsh model (1981).

The Walsh model has also been applied to matrix permeability data in order to check for the influence of microcracks especially at lower effective stresses. Tinni et al. (2012) shows the application of Walsh model to Ordovician age shale samples as can be seen in figure 15.

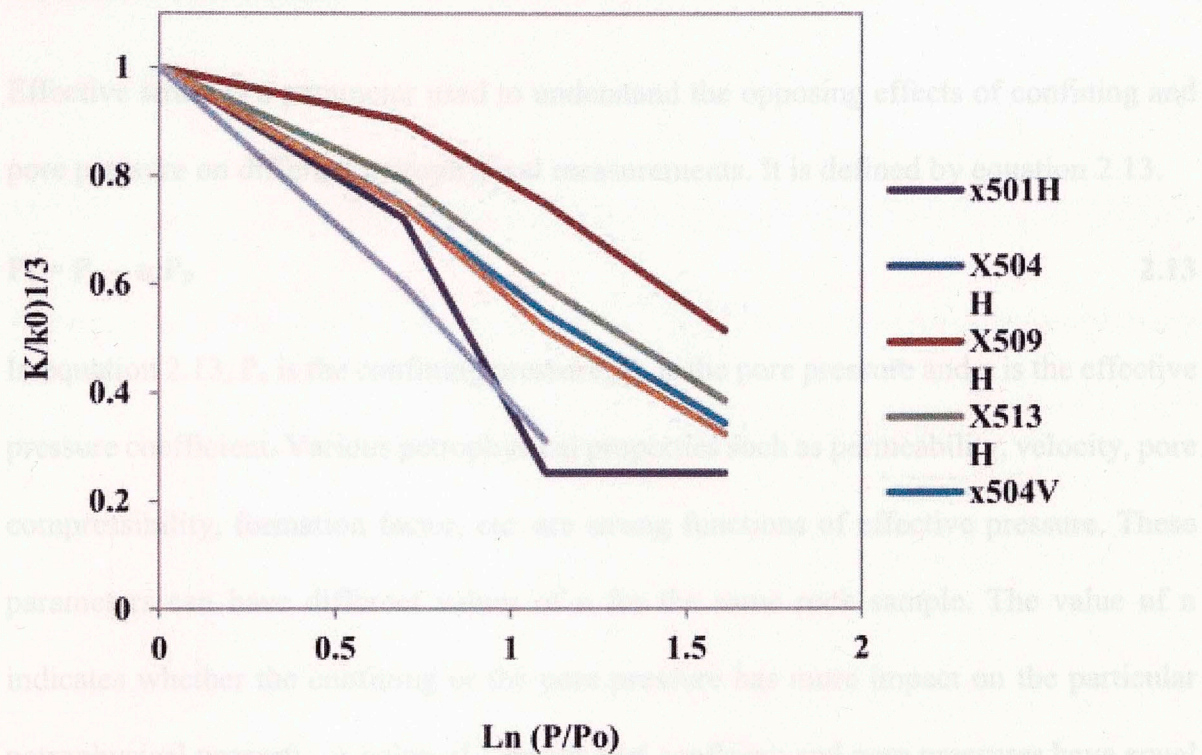


Figure 15: Application of Walsh's model for Ordovician age samples (Tinni et al., 2012). Note that that some samples show deviation from Walsh model and some samples nearly follow it.

In figure 15 some samples show deviations from Walsh model and some samples nearly follow it. If matrix permeability is assumed to be independent of cracks then it is necessary to measure permeability as a function of effective pressure. Then crack permeability models such as the Walsh model should be applied to the data to find the effective pressure point at which deviation from Walsh's model is observed. Permeability measurements made at effective pressures greater than this effective pressure would have reduced influence of microcracks.

2.6 Effective Stress Law

Effective stress is a parameter used to understand the opposing effects of confining and pore pressure on different petrophysical measurements. It is defined by equation 2.13.

$$P_e = P_c - n.P_p \quad 2.13$$

In equation 2.13, P_c is the confining pressure, P_p is the pore pressure and n is the effective pressure coefficient. Various petrophysical properties such as permeability, velocity, pore compressibility, formation factor, etc. are strong functions of effective pressure. These parameters can have different values of n for the same rock sample. The value of n indicates whether the confining or the pore pressure has more impact on the particular petrophysical property. A value of 1 means that confining and pore pressures have equal effect on the rock property. If $n > 1$, it means the pore pressure has a greater effect than the confining pressure and conversely if the value of $n < 1$, it means that the confining pressure has a greater effect than the pore pressure for that particular rock property.

In order to calculate effective pressure coefficient for a particular petrophysical property, a range of measurements have to be conducted at different confining and pore pressures. Todd and Simmons (1972) proposed equation 2.14 to obtain the value of n as a function of differential pressure (P_d). This equation has been developed for any petrophysical property and is used for permeability in this study.

$$n = 1 - \left(\frac{\left(\frac{\partial k}{\partial P_p} \right)_{P_d}}{\left(\frac{\partial k}{\partial P_d} \right)_{P_p}} \right) \quad 2.14$$

The differential pressure P_d is given by equation 2.15.

$$P_d = P_c - P_p \quad 2.15$$

The term $\left(\frac{\partial k}{\partial P_p}\right)_{P_d}$ means the change in permeability due to changes in pore pressure with

differential pressure held constant and the term $\left(\frac{\partial k}{\partial P_d}\right)_{P_p}$ means the change in permeability

due to changes in differential pressure with pore pressure held constant.

Walls (1982) proposed that the permeability can be assumed to be a linear function of confining and pore pressure as shown in equation 2.16 to calculate the effective pressure coefficient.

$$k = k_0 + aP_p - bP_c \quad 2.16$$

In equation 2.16, a and b are constants. The term k_0 is permeability at zero pore pressure and confining pressure. Experimental data was used to evaluate the the values of constants a and b . The value of a divided by b (a/b) was regarded as the value of effective pressure coefficient.

Bernabe (1987) developed another relationship to get the effective pressure coefficient by defining it as the ratio of the variation in permeability to changes in pore pressure at a particular confining pressure, to the variation in permeability with changes in confining pressure for a given pore pressure as given in equation 2.17.

$$n_k = - \frac{\left(\frac{\partial k}{\partial P_p}\right)_{P_c}}{\left(\frac{\partial k}{\partial P_c}\right)_{P_p}} \quad 2.17$$

The term $\left(\frac{\partial k}{\partial p_p}\right)_{p_c}$ means the change in permeability due to changes in pore pressure with confining pressure held constant and the term $\left(\frac{\partial k}{\partial p_c}\right)_{p_p}$ means the change in permeability due to changes in confining pressure with pore pressure held constant.

Matrix permeability of shale generally decreases with increasing effective pressure as shown in the previous section. While producing from a reservoir, the overburden stress or the confining pressure is constant but the pore pressure declines as fluids are being produced. This means that during production the effective pressure will continuously increase due to changes in the pore pressure. The measurement of effective pressure coefficient is important in calculating the changes in permeability due to changes in effective pressure as a result of the decrease in pore pressure.

Table 1 provides the values of effective pressure coefficient, n , reported by various researchers for different conventional and unconventional formations. Table 1 indicates that the effective pressure coefficient for conventional formations is greater than 1 while it is lower than or equal to 1 for shale formations. The low values of effective pressure coefficient for shales mean that changes in confining pressure has more influence on permeability than changes in pore pressure. This also means that permeability is less sensitive to changes in pore pressure than confining pressure. This finding has a positive impact during the production life of the reservoir.

Table 1 : Values of effective pressure coefficient for different formations.

S. No.	Author(s)	Permeability measurement technique	Formation/ Lithology studied	Effective pressure coefficient (n)	Method to evaluate 'n'
1	Zoback and Byerlee, 1975	Steady state	Berea sandstone	2.2 - 4	Linear model
2	Walls, 1982	Steady state	Different sandstones	1 - 7.1	Linear model
3	Bernabe, 1988	Pulse decay	Chlemsford Granite	0.6 - 1.1	Bernabe, 1987
4	Al Wardy and Zimmerman, 2004	Steady state	Stainton sandstone	5.4	Bernabe, 1987
5	Shafer et al., 2008	Steady state	GoM Lower Tertiary sandstone	2.48	Todd and Simmons, 1972
6	Kwon et al., 2001	Pulse decay	Wilcox shale	~1	Bernabe, 1987
7	Agarwal, 2012	Pressure Build Up	Haynesville shale	0.17 - 0.33	Todd and Simmons, 1972
8	Heller and Zoback, 2013	Pulse decay	Barnett shale	0.68 - 0.82	Bernabe, 1987
			Eagle Ford shale	0.4 - 0.6	
			Marcellus shale	0.15	
			Montney shale	0.85	

Chapter 3: Experimental Procedure

In this chapter I discuss the experimental set up for shale matrix permeability measurements. The process to conduct different permeability methods has been discussed in Appendix A.

3.1 Experimental setup for measuring ultra-low permeability

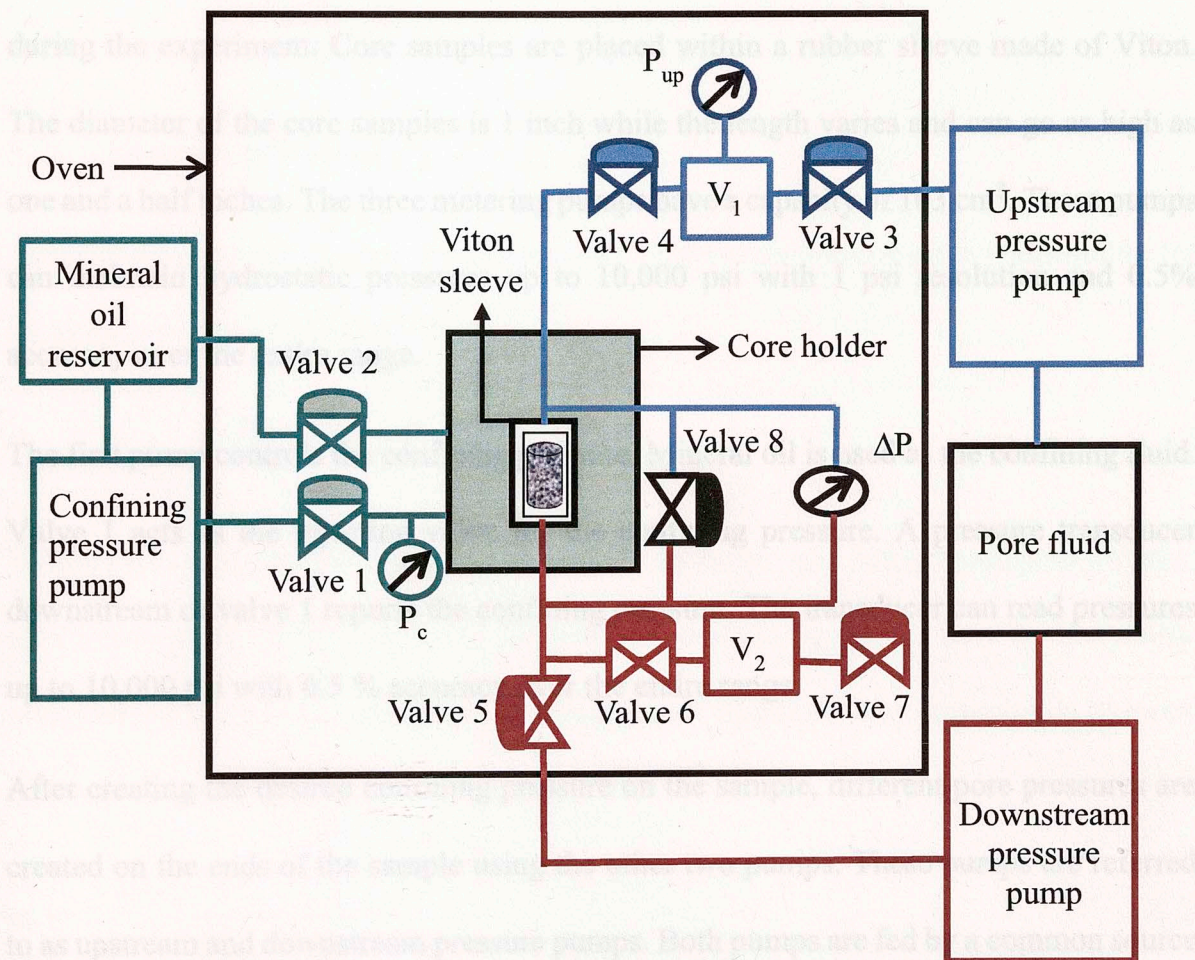


Figure 16: Experimental apparatus for permeability measurement. The core holder is placed in an oven for thermal insulation. The confining pressure pump develops overburden stress and the upstream and downstream pumps develop pore pressures on the sample. Pressures are monitored using pressure gauge in the upstream and a differential pressure gauge.

Figure 16 is a schematic of the apparatus used to measure shale plug permeability. This apparatus has been designed and built at Integrated Core Characterization Center (IC³) at University of Oklahoma.

The experimental setup primarily consists of three computer controlled metering pumps and a core holder. The core holder is a hollow stainless steel cylinder with removable caps. It is housed in a thermally isolated oven in order to maintain a constant temperature during the experiment. Core samples are placed within a rubber sleeve made of Viton. The diameter of the core samples is 1 inch while the length varies and can go as high as one and a half inches. The three metering pumps have a capacity of 103 cm³. These pumps can maintain hydrostatic pressures up to 10,000 psi with 1 psi resolution and 0.5% accuracy over the entire range.

The first pump controls the confining pressure. Mineral oil is used as the confining fluid. Valve 1 acts as the injection valve for the confining pressure. A pressure transducer downstream of valve 1 reports the confining pressure. The transducer can read pressures up to 10,000 psi with 0.5 % accuracy over the entire range.

After creating the desired confining pressure on the sample, different pore pressures are created on the ends of the sample using the other two pumps. These pumps are referred to as upstream and downstream pressure pumps. Both pumps are fed by a common source which can be either a gas or a liquid. Common pore fluids used in measurements are nitrogen and helium gases and liquid dodecane. These pumps exert different pore pressures on the sample during the permeability measurement. The two pore pressures differ by a small value generally 100 psi in order to eliminate turbulence.

The sample is initially saturated with pore fluids from both the pore pressure pumps. To ensure complete saturation of sample with pore fluid, at least two pore volumes are flowed throughout sample. During this time no permeability measurement is made. This time period can vary from hours to days depending on the permeability of the sample. After saturation, the permeability is measured.

This experimental setup can be used to measure permeability with the steady state method, pressure pulse decay and pressure build up.

3.2 Pressure pulse decay

The following equations given by Brace et al., 1968 are used to calculate permeability for the pressure pulse decay method.

$$P_{up} - P_f = \Delta P \left(\frac{V_u}{V_d} + V_d \right) \cdot e^{-mt} \quad 3.1$$

Where P_{up} is upstream pore pressure , P_f is final upstream and downstream pore pressure after decay stops, ΔP is initial pore pressure differential , V_u and V_d refer to the volume occupied by the tubing and system volumes in the upstream and downstream sections respectively and t is time. m is given by:

$$m = \left(\frac{kA}{\mu\beta L} \right) \left(\frac{1}{V_u} + \frac{1}{V_d} \right) \quad 3.2$$

Where k is permeability, A is the cross-sectional area of the measured sample, μ is the viscosity at the mean pore pressure of sample, β is the pore fluid compressibility at the mean pore pressure and L is the length of core plug. The mean pore pressure is the average of upstream and downstream pressures.

Based on equation 3.1, a plot of the natural logarithm of the difference between the upstream pore pressure and the final pore pressure $[\ln(P_{up}-P_f)]$ versus time yields a straight line. The slope of this line is m .

In figure 17, the experimental data for pulse decay permeability of a Wolfcamp shale sample, XX65 are plotted. The confining pressure applied on the sample is 3250 psi. The pore fluid used for measurement is nitrogen. The initial upstream and downstream pressure are 300 and 200 psi, respectively. Before 20 minutes, no pressure changes are observed in the downstream end of the sample and then pulse decay starts. Both the upstream and downstream pressure reach a common value (P_f) slightly greater than 250 psi at the end of the experiment.

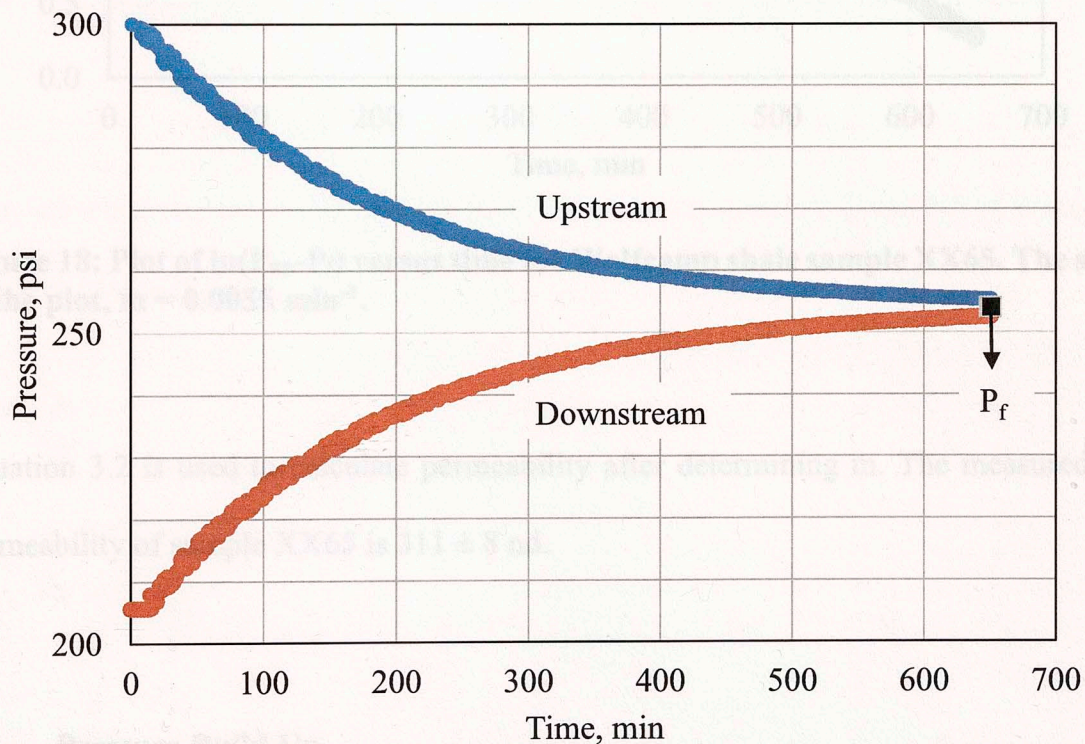


Figure 17: Plot of upstream and downstream pore pressure versus time for sample XX65 (Wolfcamp shale) using pressure pulse decay method. The upstream pressure initially decays while the downstream pressure builds up to reach a common final value close to 250 psi.

Figure 18 shows the plot $[\ln(P_{up}-P_f)]$ versus time for the Wolfcamp shale sample XX65.

The absolute value of m for this sample is 0.0055 min^{-1} .

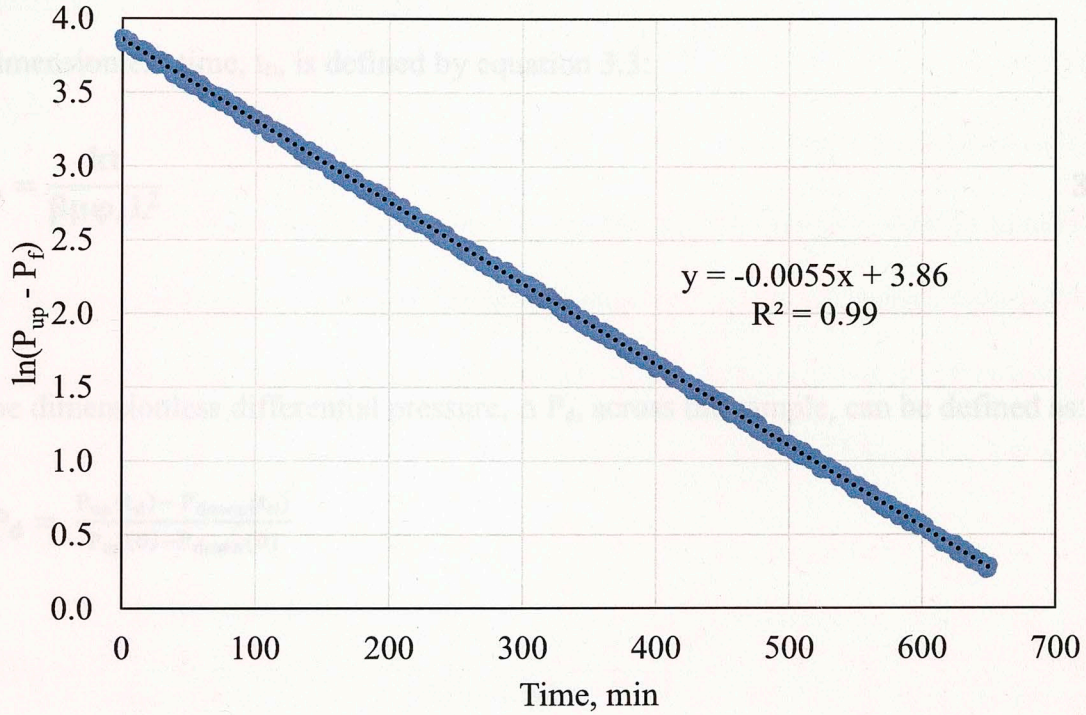


Figure 18: Plot of $\ln(P_{up}-P_f)$ versus time for Wolfcamp shale sample XX65. The slope of the plot, $m = 0.0055 \text{ min}^{-1}$.

Equation 3.2 is used to calculate permeability after determining m . The measured gas permeability of sample XX65 is $311 \pm 8 \text{ nd}$.

3.3 Pressure Build Up

The pressure build up measurement method was developed by Metwally and Sondergeld (2011). The theory behind this method is given below. Hsieh et al. (1981) presented an

analytical solution describing one dimensional flow equation through the core sample. This solution represents the dimensionless pressure difference between the upstream and downstream reservoirs as a function of time and is given in equation 3.3. In order to understand the final solution some dimensionless numbers have to be defined.

Dimensionless time, t_D , is defined by equation 3.3:

$$t_D = \frac{kt}{\beta\mu\phi_e L^2} \quad 3.3$$

The dimensionless differential pressure, ΔP_d , across the sample, can be defined as:

$$\Delta P_d = \frac{P_{up}(t_d) - P_{down}(t_d)}{P_{up}(0) - P_{down}(0)} \quad 3.4$$

Two other parameters a and b are defined below in equations 3.5 and 3.6 to normalize experimental volumes.

$$a = \frac{V_p}{V_{up}} \quad 3.5$$

$$b = \frac{V_p}{V_d} \quad 3.6$$

In equations 3.5 and 3.6, V_p , V_{up} and V_d refer to the pore volume of the sample, upstream system volume and downstream system volume, respectively.

The analytical solution as described by Dicker and Smits (1988) is given by equation 3.7

$$\Delta P_D = 2 \sum_{n=1}^{\infty} \exp(-t_D \theta_n^2) \frac{a(b^2 + \theta_n^2) - (-1)^n b[(a^2 + \theta_n^2)(b^2 + \theta_n^2)]^{0.5}}{[\theta_n^4 + \theta_n^2(a + a^2 + b + b^2) + ab((a + b + ab))]} \quad 3.7$$

where θ_n are the roots of the equation 3.8:

$$\tan \theta = \frac{(a+b)\theta}{(\theta^2-ab)} \quad 3.8$$

In the pressure build up experiment, Metwally and Sondergeld (2011) adapted this solution by applying a boundary condition where the upstream system volume (V_{up}) is made infinite. This boundary condition simplifies equations 3.7 and 3.8. The value of parameter 'a' becomes zero due to infinite upstream volume. Equations 3.9 and 3.10 reflect these changes.

$$\delta P_D = 2 \sum_{n=1}^{\infty} \exp(-t_D \theta_n^2) \frac{(-1)^{n+1} b [\theta_n^2 (b^2 + \theta_n^2)]^{0.5}}{[\theta_n^4 + \theta_n^2 (b + b^2)]} \quad 3.9$$

$$\theta \tan \theta = b = \frac{V_p}{V_d} \quad 3.10$$

The final equation to get permeability from the pressure build up experiment is shown in equation 3.11.

$$k = \frac{\beta \mu \phi_e L^2 m}{\theta^2} \quad 3.11$$

In equation 3.11, m is the slope of the straight line obtained on plotting the natural log of the measured pressure difference between upstream and downstream pressure versus

time. β is fluid compressibility, μ is viscosity at mean pore pressure, ϕ_e is the effective porosity of the sample and L is the length of the core plug. In this experiment the compressibility of the rock matrix is considered negligible in comparison to the fluid compressibility.

Experimental data for a Wolfcamp shale XX65 where permeability was measured using pressure build up method is shown in figure 19. The confining pressure is 3250 psi. Nitrogen is used as the pore fluid. The upstream pressure is 310 psi and it is maintained constant throughout the measurement. The initial downstream pressure on the sample is 210 psi. Before 30 minutes no pressure change is observed in the downstream. Thereafter, the downstream pressure increases exponentially with time; hence, the pore pressure differential also decreases exponentially with time.

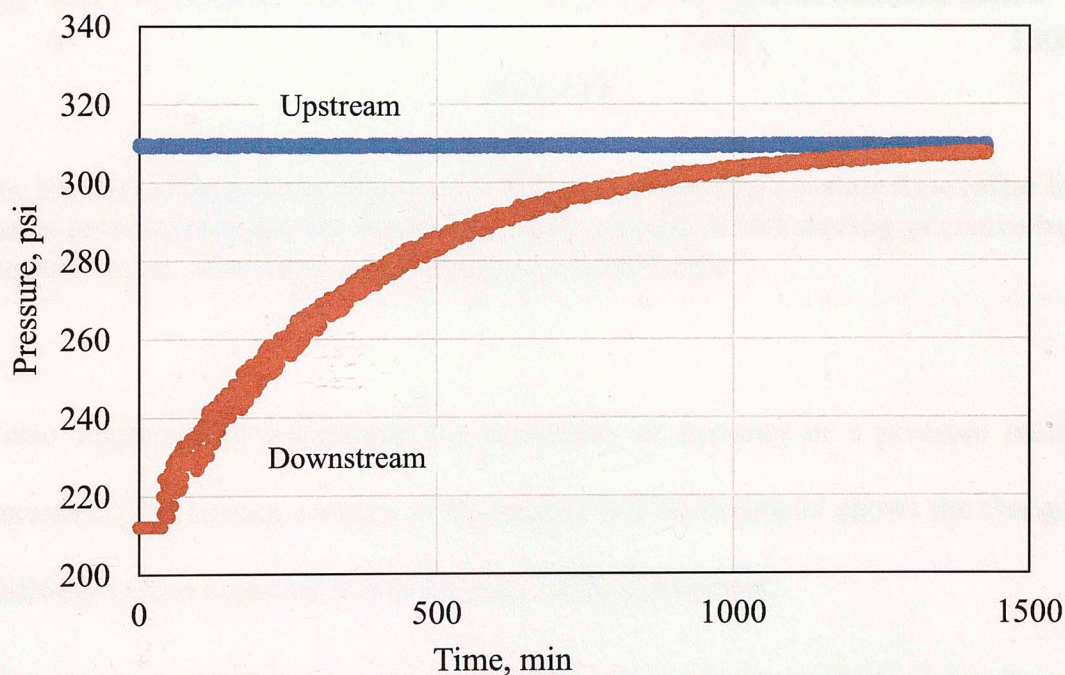


Figure 19 : Plot of upstream and downstream pressure versus time for Wolfcamp shale sample XX65 during pressure build-up measurement. Downstream pressure initially at 210 psi increases with time to finally become equal to constant upstream pressure of 310 psi.

The plot of natural logarithm of pressure differential versus time for Wolfcamp shale sample XX65 is shown in figure 3.5. The magnitude of slope, m , is 0.0037 min^{-1} . The gas permeability of sample is $306 \pm 7 \text{ nd}$.

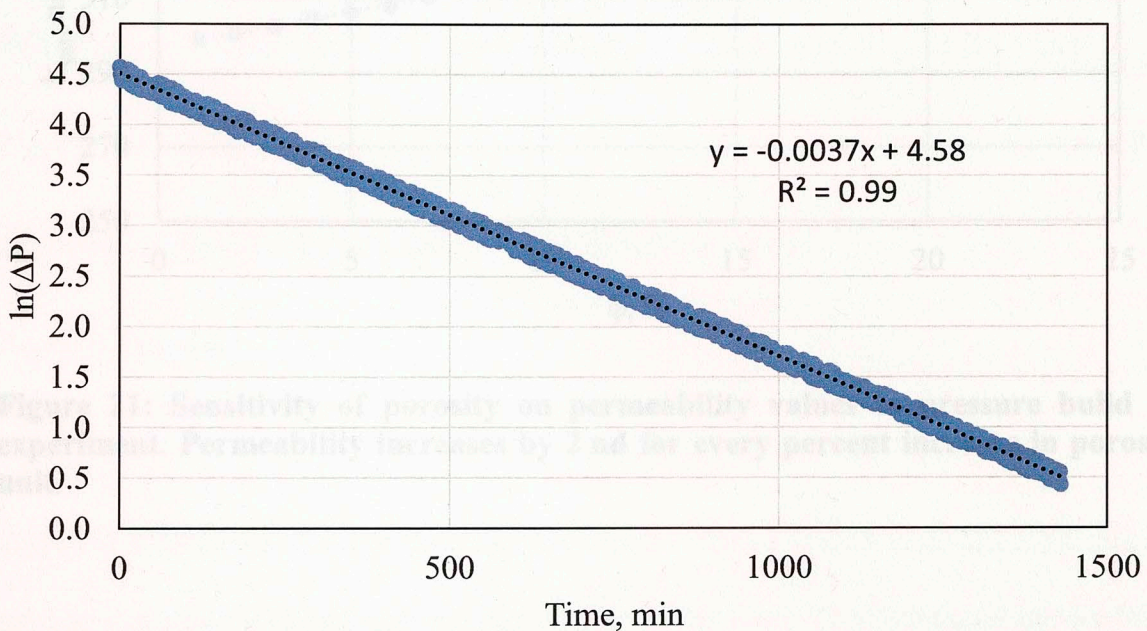


Figure 20: Plot of logarithm of pressure differential pressure versus time (after build up starts in downstream) for Wolfcamp shale sample XX65 during pressure build-up measurement. The slope m is found to be 0.0037 min^{-1} .

It is also important to understand the sensitivity of porosity in a pressure build up measurement. The helium porosity of the sample is 5 %. Figure 21 shows the changes in permeability values expected if this porosity value is incorrect.

A change in porosity value from 1 to 20 % causes change in permeability value from 300 to around 335 nd. This discrepancy is around 10% of the measured value of permeability and hence can be ignored. Permeability increases by 2 nd for every percent increase in

porosity unit. This means that permeability values produced by using pressure build up experiment are not very sensitive to porosity values used in the calculation.

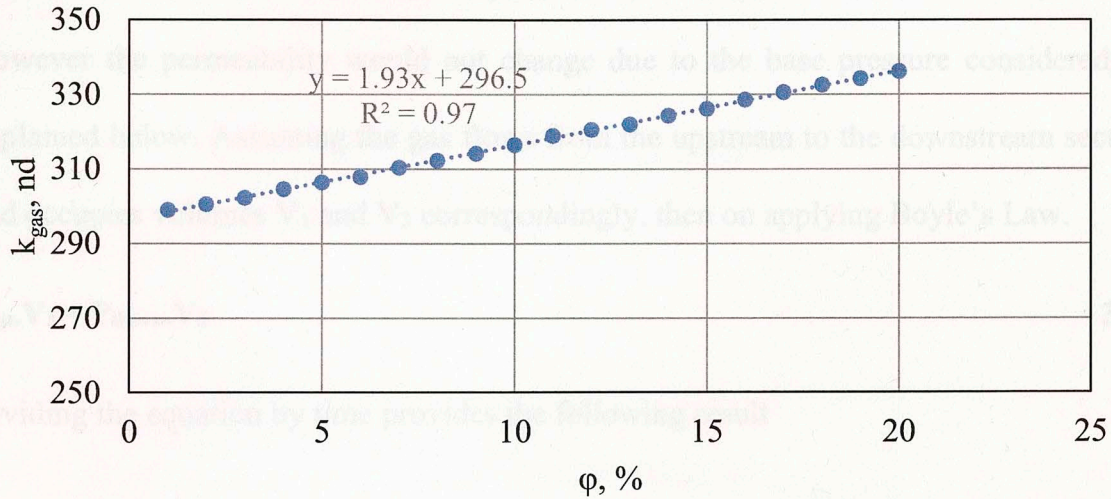


Figure 21: Sensitivity of porosity on permeability values in pressure build up experiment. Permeability increases by 2 nd for every percent increase in porosity unit.

3.4 Steady state permeability measurement

Steady state flow is reached when a constant flow rate is achieved. Darcy's Law is used to compute permeability of the sample. Equation 3.12 is the equation used to calculate permeability for gases.

$$k = \frac{2 \cdot q \cdot \mu \cdot L \cdot P_{\text{base}} \cdot Z_{\text{mean}}}{A \cdot (P_{\text{up}}^2 - P_{\text{down}}^2) \cdot Z_{\text{base}}} \quad 3.12$$

Where k is permeability in darcies, q is flow rate in cm^3/s , L is length of core plug in cm , A cross-sectional area in cm^2 , P_{base} , P_{up} and P_{down} are the base, upstream and downstream pressure respectively in atm and Z_{mean} , Z_{base} are the compressibility factors at mean pore

pressure and the base pore pressure, respectively. The mean pressure refers to the average of the upstream and downstream pressures. The base pressure is a reference pressure and in our calculations the downstream pressure is considered to be the base pressure. However the permeability would not change due to the base pressure considered, as explained below. Assuming the gas flows from the upstream to the downstream section and occupies volumes V_1 and V_2 correspondingly, then on applying Boyle's Law.

$$P_{up}.V_1 = P_{down}.V_2 \quad 3.13$$

Dividing the equation by time provides the following result

$$P_{up}.q_1 = P_{down}.q_2 \quad 3.14$$

Equation 3.14 means that the flow rate for the two pumps is different. The flow rate (q) used in equation 3.12 is based on the base pressure used. The computed permeability remains same as the product of base pressure (P_{base}) and flow rate (q) used in the equation 3.11 is always constant.

Equations 3.15 is the equation used to calculate permeability for liquids.

$$k = \frac{q.\mu.L}{A.(P_{up}-P_{down})} \quad 3.15$$

Experimental data for steady state permeability measurement is shown in figure 22. Nitrogen is used for this measurement. The upstream pressure remains constant at 300 psi and downstream pressure remain constant at 200 psi throughout the experiment. The confining stress (P_c) is 3250 psi. This results in an effective pressure of 3000 psi.

Figure 22 shows the upstream pump volume decreases with time while the downstream pump volume increases with time. This is because the upstream pressure is greater than the downstream pressure, flow occurs from the upstream to downstream section.

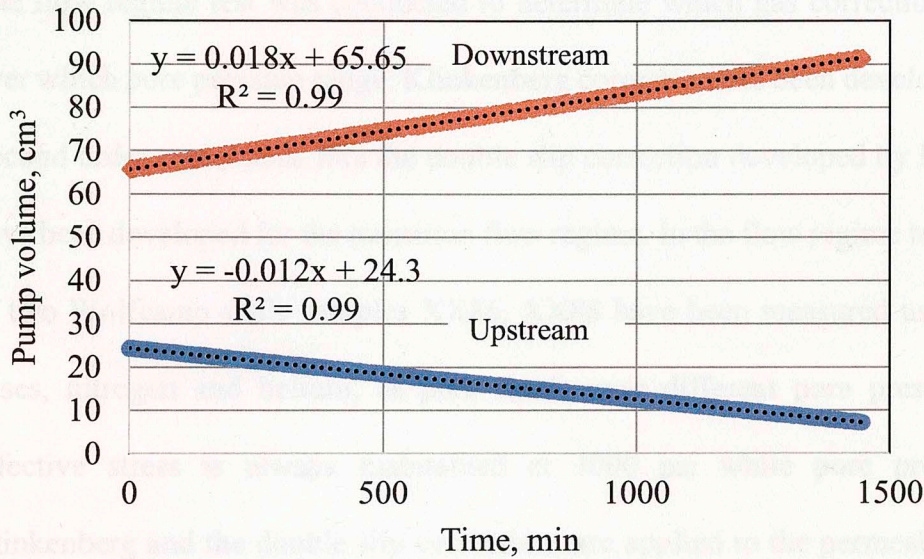


Figure 22: Flow rates of upstream and downstream pump measured as a function of time for steady state permeability measurement of sample XX65 (Wolfcamp shale). Upstream pump volume decreases while the downstream pump volume increases with time.

The flow rates of the two pumps are different. The values are 0.012 and 0.018 min^{-1} for the upstream and downstream pumps, respectively. Also the upstream and downstream pressures are different. The upstream pressure is 300 psi while the downstream pressure is 200 psi. But based on equation on 3.14, the product of flow rate and pressure is constant ($0.012 \times 300 = 3.6$ and $0.018 \times 200 = 3.6$) and hence the value of permeability measured from either of the pumps is same as. Hence the same permeability will be measured using data of either pump.

The value of flow rate is substituted in Darcy's Law (equation 3.12) along with other parameters to calculate permeability. The gas permeability of this sample is $371 \pm 8 \text{ nd}$.

Chapter 4: Results and Discussions

4.1 Flow Regime Test

The flow regime test was conducted to determine which gas correction should be used over which pore pressure range. Klinkenberg correction has been developed for slip flow. Second order corrections like the double slip correction developed by Fathi et al. (2012) have been developed for the transition flow regime. In the flow regime test, permeabilities of two Wolfcamp shale samples XX86, XX88 have been measured using two different gases, nitrogen and helium, as pore fluids over different pore pressure ranges. The effective stress is always maintained at 3000 psi while pore pressure is varied. Klinkenberg and the double slip corrections are applied to the permeability values from different pore pressure ranges for both the gases. The correction which produces similar corrected permeability for the two gases at a given pore pressure range is expected to be the appropriate correction for that range.

4.1.1 Sample Description

The FTIR mineralogy of the two Wolfcamp shale samples (XX86, XX88) is shown in figure 23. Table 2 provides the values of the helium porosity (%) and TOC content (weight %) of the two samples. Porosity is measured on crushed samples for all measurements. Illite is the dominant clay mineral.

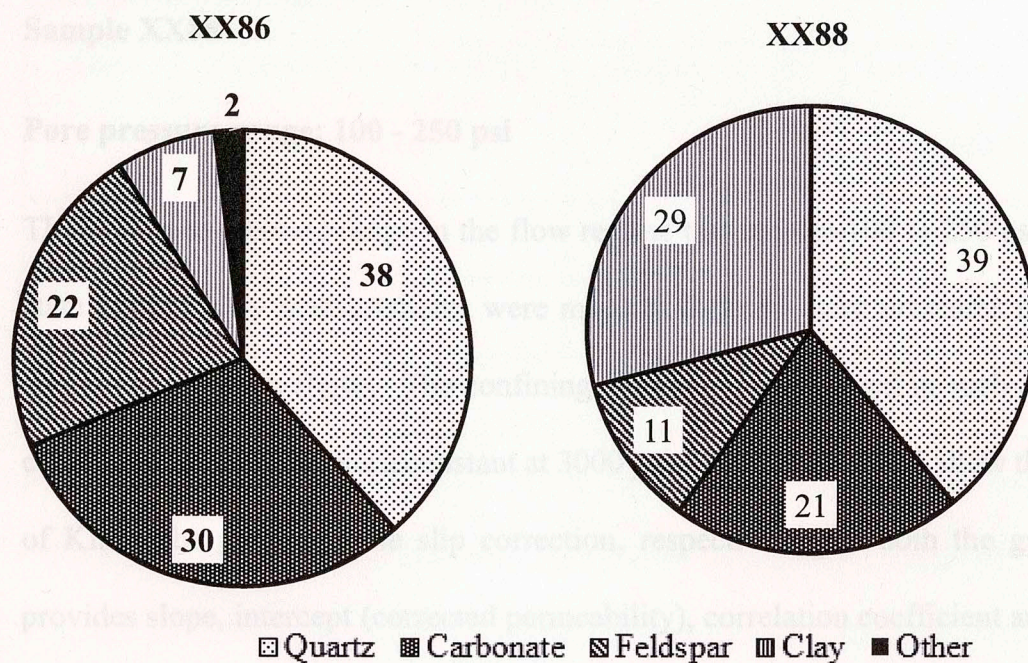


Figure 23: FTIR Mineralogy for the two Wolfcamp shale samples (XX86 and XX88) used in the flow regime test.

Table 2: Porosity and TOC values for two Wolfcamp shale samples (XX86 and XX88) used in flow regime test.

Sample	Helium porosity (%)	TOC (wgt%)
XX86	7.2	1.2
XX88	2.7	4.0

4.1.2 Test results and discussions

Sample XX88

Pore pressure range: 100 - 250 psi

The first pore pressure range in the flow regime test is from 100 to 250 psi. Five steady state permeability measurements were made at different pore pressures using nitrogen and helium as pore fluids. The confining pressure was changed in such a way that the effective pressure remains a constant at 3000 psi. Figures 24 and 25 show the application of Klinkenberg and double slip correction, respectively, for both the gases. Table 3 provides slope, intercept (corrected permeability), correlation coefficient and percentage difference in corrected permeability values for the two gases while using the Klinkenberg correction. Similar information for the double slip correction is provided in Table 4.

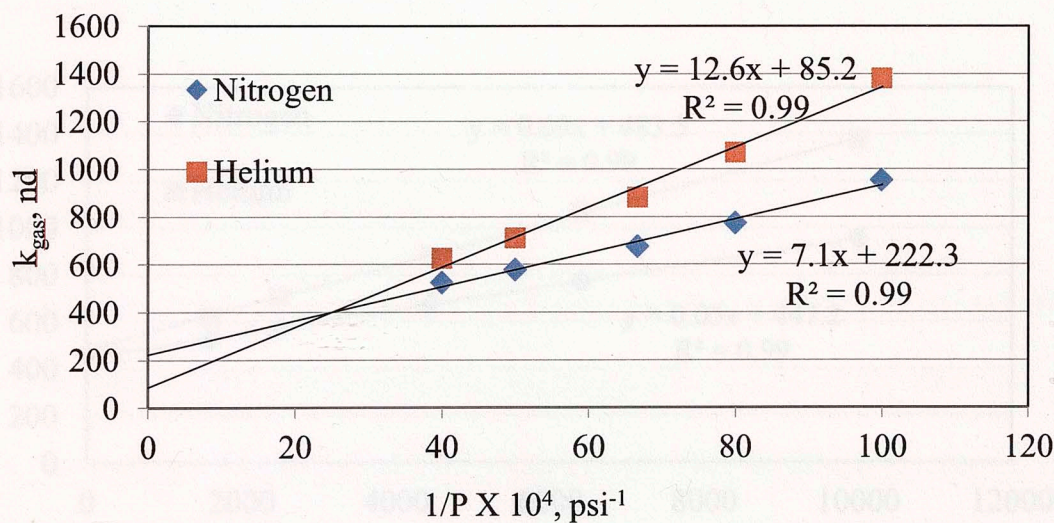


Figure 24: Klinkenberg correction for Wolfcamp sample XX88 (pore pressure range: 100 to 250 psi).

Table 3: Data for Klinkenberg corrected permeability for Wolfcamp sample XX88 (pore pressure range: 100 to 250 psi).

Gas	Corrected permeability (Intercept)	Slope	R ²	Corrected permeability ratio (k _N / k _{He})
Nitrogen	222 ± 42	7.1 ± 0.45	0.99	2.6
Helium	85 ± 31	12.6 ± 0.9	0.99	

Based on observations from figure 24 and Table 3, the Klinkenberg correction produces different values of effective permeability over the pore pressure range of 100 to 250 psi. The ratio of Klinkenberg corrected permeability of nitrogen to that of helium is 2.6. This discrepancy means Klinkenberg correction is inappropriate here.

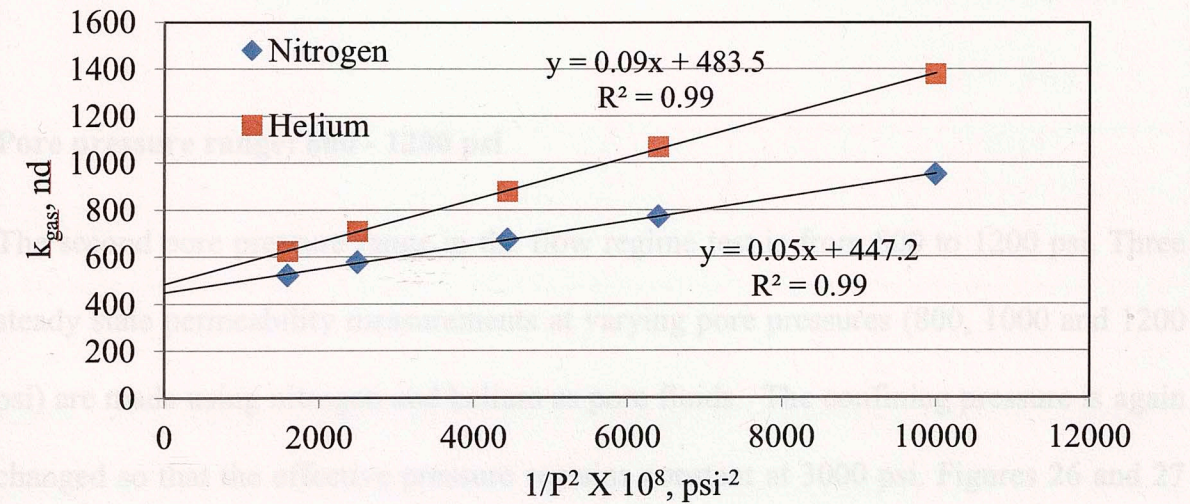


Figure 25: Double slip correction for Wolfcamp sample XX88 (pore pressure range: 100 to 250 psi).

Table 4: Data for double slip corrected permeability for Wolfcamp sample XX88. (pore pressure range: 100 to 250 psi).

Gas	Corrected permeability (Intercept)	Slope	R ²	Corrected permeability ratio (k _N / k _{He})
Nitrogen	447 ± 4	.05 ± .0006	0.99	0.93
Helium	483 ± 6	.09 ± .0009	0.99	

Based on observations from figure 25 and Table 4, the double slip correction produces similar values of permeability over the pore pressure range of 100 to 250 psi. The ratio of double slip corrected permeability of nitrogen to that of helium is 0.93. This ratio is very close to 1. This means that the double slip correction is the appropriate correction at this low pressure range of 100 to 250 psi.

Pore pressure range: 800 - 1200 psi

The second pore pressure range in the flow regime test is from 800 to 1200 psi. Three steady state permeability measurements at varying pore pressures (800, 1000 and 1200 psi) are made using nitrogen and helium as pore fluids. The confining pressure is again changed so that the effective pressure remains constant at 3000 psi. Figures 26 and 27 show the application of Klinkenberg and double slip correction, respectively, for both the gases. Statistical data for the Klinkenberg correction and the double correction plots are available in Tables 5 and 6.

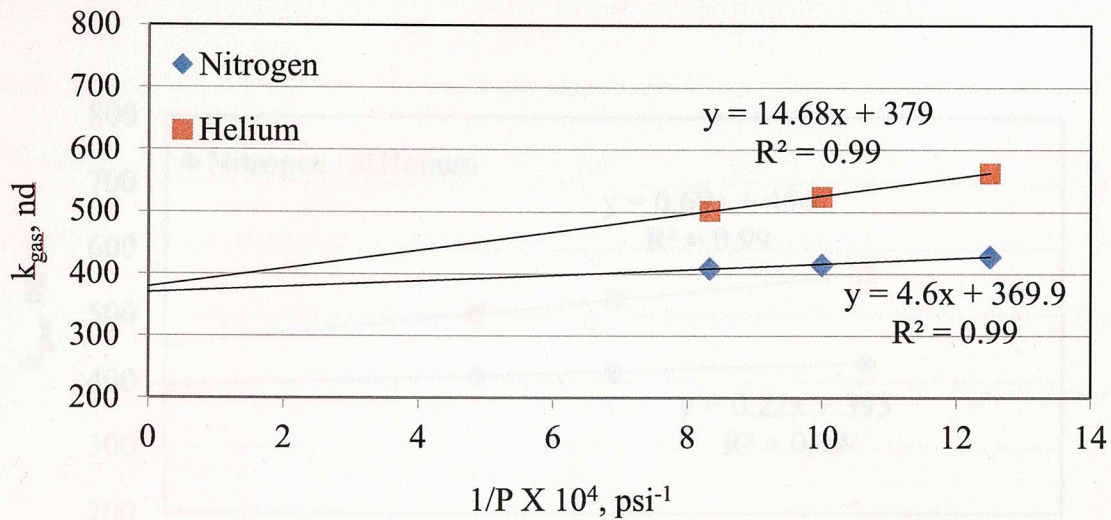


Figure 26: Klinkenberg correction for Wolfcamp sample XX88 (pore pressure range: 800 to 1200 psi).

Table 5: Data for Klinkenberg corrected permeability for Wolfcamp sample XX88 (pore pressure range: 800 to 1200 psi).

Gas	Corrected permeability (Intercept)	Slope	R ²	Corrected permeability ratio (k _N / k _{He})
Nitrogen	370 ± 5	4.6 ± 0.4	0.99	0.98
Helium	379 ± 4	14.7 ± 0.4	0.99	

Based on observations from figure 26 and Table 5, the Klinkenberg correction results in similar values of permeability over the pore pressure range of 800 to 1200 psi. The ratio of Klinkenberg corrected permeability with nitrogen to that of helium is 0.98. This similarity of permeability value means Klinkenberg correction is appropriate for the pore pressure range of 800 to 1200 psi.

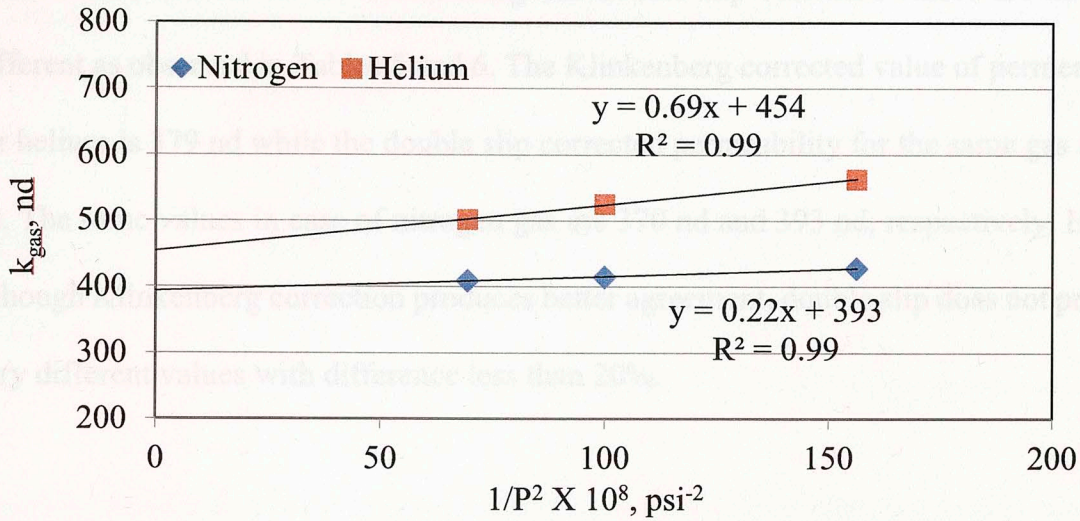


Figure 27: Double slip correction for Wolfcamp sample XX88 (pore pressure range: 800 to 1200 psi).

Table 6: Data for double slip corrected permeability for Wolfcamp sample XX88 (pore pressure range: 800 to 1200 psi).

Gas	Corrected permeability (Intercept)	Slope	R ²	Corrected permeability ratio (k _N / k _{He})
Nitrogen	393 ± 1	.22 ± .008	0.99	0.87
Helium	454 ± 2	.7 ± .02	0.99	

Based on observations from figure 27 and Table 6, the double slip correction results in slightly different values of permeability over the pore pressure range of 800 to 1200 psi. The ratio of double slip corrected permeability with nitrogen to that of helium is 0.87. Though the difference in double slip permeabilities is not very large (13%), but the difference for Klinkenberg corrected permeabilities is smaller (2%). Based on the larger

difference of permeability in double slip correction, Klinkenberg is suggested to be the better correction. However, Klinkenberg and double slip corrected values are not very different as observed in Tables 5 and 6. The Klinkenberg corrected value of permeability for helium is 379 nd while the double slip corrected permeability for the same gas is 454 nd. The same values in case of nitrogen gas are 370 nd and 393 nd, respectively. Hence, although Klinkenberg correction produces better agreement, double slip does not provide very different values with difference less than 20%.

Pore pressure range: 1800 - 2200 psi

The third pore pressure range in the flow regime test is from 1800 to 2200 psi. Three steady state permeability measurements are made at varying pore pressures (1800, 2000 and 2200 psi) using nitrogen and helium. The confining pressure is again changed in such a way that the effective pressure remains a constant at 3000 psi.

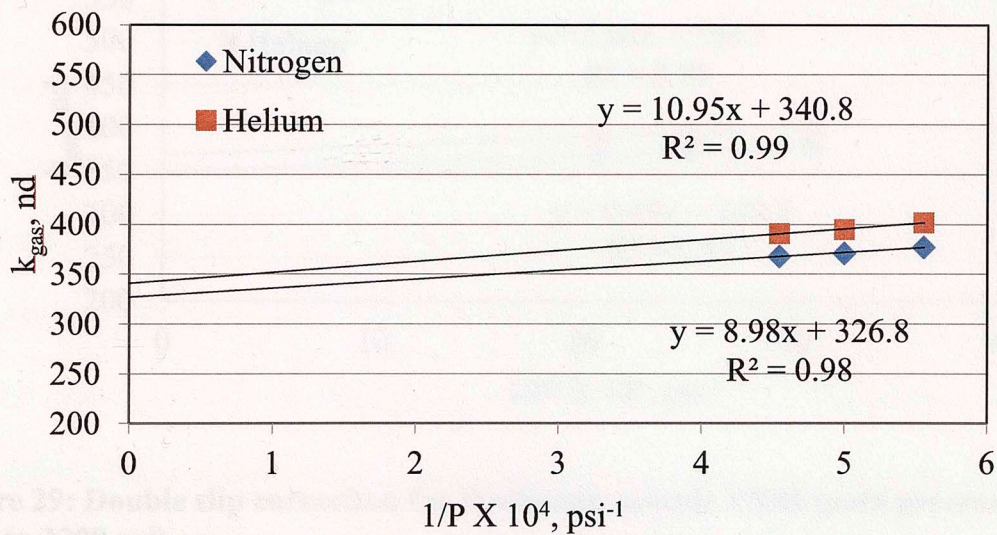


Figure 28: Klinkenberg correction for Wolfcamp sample XX88. (Pore pressure range: 1800 to 2200 psi).

Table 7: Data for Klinkenberg corrected permeability for Wolfcamp sample XX88 (pore pressure range: 1800 to 2200 psi).

Gas	Corrected permeability (Intercept)	Slope	R ²	Corrected permeability ratio (k _N / k _{He})
Nitrogen	327 ± 6	9 ± 1.2	0.98	0.96
Helium	341 ± 6	11 ± 1.1	0.99	

Based on observations from figure 28 and Table 7, the Klinkenberg correction results in similar values of permeability for the pore pressure range of 1800 to 2200 psi. The ratio of Klinkenberg corrected permeability of nitrogen to that of helium is 0.96. This agreement of the permeability values suggest the Klinkenberg correction is appropriate for the pore pressure range of 1800 to 2200 psi.

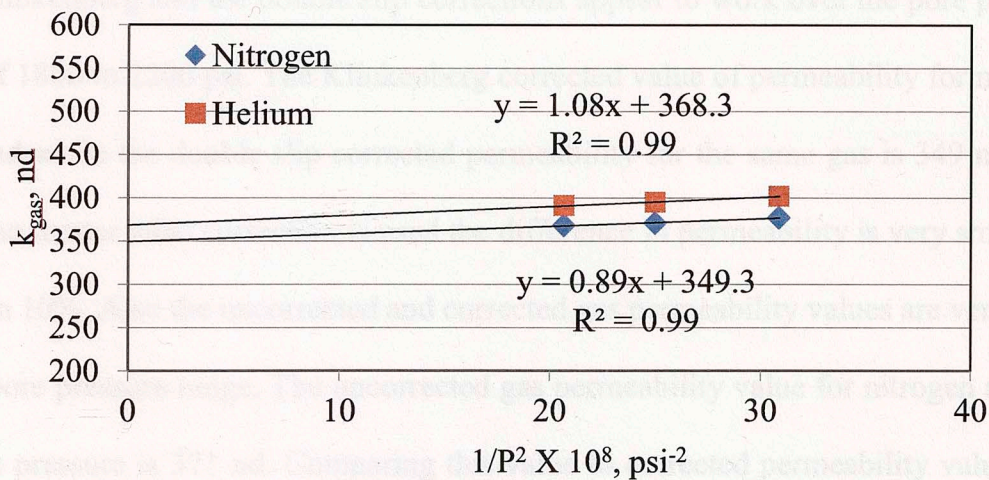


Figure 29: Double slip correction for Wolfcamp sample XX88 (pore pressure range: 1800 to 2200 psi).

Table 8: Data for double slip corrected permeability for Wolfcamp sample XX88 (pore pressure range: 1800 to 2200 psi).

Gas	Corrected permeability (Intercept)	Slope	R ²	Corrected permeability ratio (k _N / k _{He})
Nitrogen	349 ± 2.4	.9 ± .1	0.99	0.95
Helium	368 ± 2	1.1 ± .1	0.99	

Based on observations from figure 29 and Table 8, the double slip correction results in similar values of permeability over the pore pressure range of 1800 to 2200 psi. The ratio of double slip corrected permeability with nitrogen to that of helium is found to be 0.95. This similarity in permeability values means that even double slip correction can be used for the pore pressure range of 1800 to 2200 psi.

Both Klinkenberg and the double slip corrections appear to work over the pore pressure range of 1800 to 2200 psi. The Klinkenberg corrected value of permeability for nitrogen is 327 nd while the double slip corrected permeability for the same gas is 349 nd. This means no matter what correction is used the difference in permeability is very small, i.e. less than 10%. Also the uncorrected and corrected gas permeability values are very close at this pore pressure range. The uncorrected gas permeability value for nitrogen at 2000 psi pore pressure is 371 nd. Comparing this value to corrected permeability values, i.e. 327 nd for Klinkenberg correction and 349 nd for double slip indicates that a correction of less than 20 % is required to give corrected permeability values at pore pressures close to 2000 psi. This can save significant measurement time. Rather than conducting

measurements for at least 3 different pore pressures and then correcting them, a single permeability value measured at a pore pressure of 2000 psi can be regarded as a corrected permeability value.

Pore pressure range: 2800 - 3200 psi

The results for the last pore pressure range stretching from 2800 to 3200 psi are similar to those obtained from the previous range of 1800 to 2200 psi. Figures 30 and 31 provide the plots for Klinkenberg and double slip corrections, respectively, for the pore pressure range of 2800 to 3200 psi. Tables 9 and 10 provide the corresponding statistical data for figures 30 and 31, respectively. Both corrections seem to work over this pressure range. The permeability values obtained from both corrections are very similar with differences less than 20 %. Also the difference between uncorrected permeability values and the corrected permeability values are within 15%.

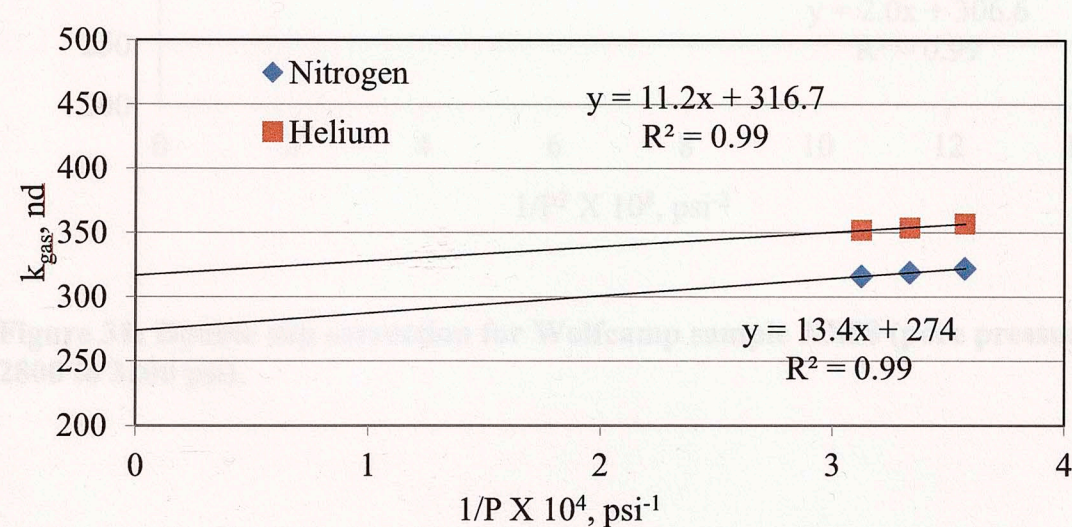


Figure 30: Klinkenberg correction for Wolfcamp sample XX88 (pore pressure range: 2800-3200 psi).

Table 9: Data for Klinkenberg corrected permeability for Wolfcamp sample XX88 (pore pressure range: 2800 to 3200 psi).

Gas	Corrected permeability (Intercept)	Slope	R ²	Corrected permeability ratio (k _N / k _{He})
Nitrogen	274 ± 1	13.4 ± 0.5	0.99	0.87
Helium	317 ± 1	11.2 ± 0.9	0.99	

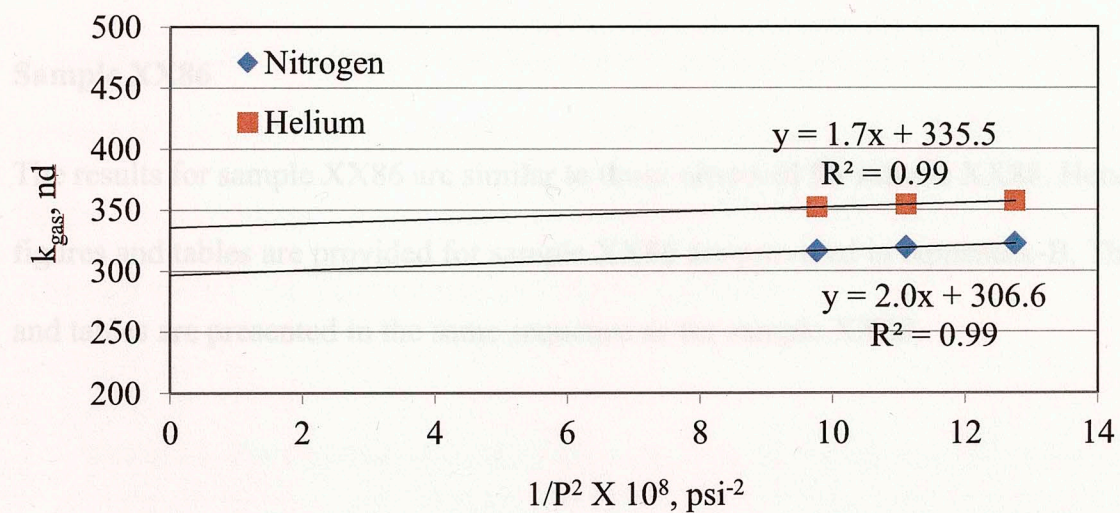


Figure 31: Double slip correction for Wolfcamp sample XX88 (pore pressure range 2800 to 3200 psi).

4.2 Round Robin test

Table 10: Data for double slip corrected permeability for Wolfcamp sample XX88 (pore pressure range: 2800 to 3200 psi).

Gas	Corrected permeability (Intercept)	Slope	R ²	Corrected permeability ratio (k _N / k _{He})
Nitrogen	307 ± 2.4	2.0 ± 0.1	0.99	0.92
Helium	336 ± 2	1.7 ± 0.01	0.99	

Sample XX86

The results for sample XX86 are similar to those observed for sample XX88. Hence only figures and tables are provided for sample XX86 are provided in Appendix-B. The plots and tables are presented in the same sequence as for sample XX88.

Table 11: Pore pressure range and methods used by the four laboratories

S. No.	Laboratory	Pore pressure range	Method of permeability measurement
1	Core Laboratory	20 – 30 psi	Steady state
2	Weatherford	25 – 30 psi	Steady state
3	Total	125 – 130 psi	Step decay (Lasson et al., 2012)
4	CO-RP	25 – 100 psi	Pressure build up

The permeability and orientation of the nine samples used in this study are given below in

Table 12: The porosity of the samples ranged from 1 to 7 %

4.2 Round Robin test

Four laboratories participated in a round robin permeability measurement test. Permeability of nine pyrophyllite samples was measured in this test. Different measurement methods were used by different laboratories. This test thus could potentially help in finding variances or similarities in permeability measured with different methods. Pyrophyllite has no organics and is a stable non-reacting clay which is in the illite family. The confining pressure was kept a constant at 1000 psi pressure by all the four laboratories. The pore fluid used in the measurement was 99.999% pure nitrogen. Each laboratory performed the measurement for at least three different pore pressures in order to apply the gas correction. The details regarding the mean pore pressure range and the method of permeability measurement are summarized in Table 11. The pore pressure ranges are well below 250 psi and hence transition flow is thought to be the dominant. All plugs were 1'' in diameter and the length varied from $\frac{3}{4}$ '' to 1''.

4.2.1 Results and discussions

Table 11: Pore pressure range and methods used by the four laboratories.

S. No.	Laboratory	Pore pressure range	Method of permeability measurement
1	Core Laboratory	20 - 35 psi	Steady state
2	Weatherford	25 - 90 psi	Steady state
3	Total	125 – 175 psi	Step decay (Lasseux et al., 2012)
4	OU- IC ³	25 – 100 psi	Pressure build up

The porosities and orientations of the nine samples used in this study are given below in Table 12. The porosity of the samples ranges from 1 to 7 %.

There is a large discrepancy in permeability values. The permeability values differ by as much as a factor of 40.

Table 12: Porosity and orientation of the samples used in the test

Sample code	Porosity	Orientation
1-H	1.5	Horizontal
2-H	7.0	Horizontal
3-H	1.7	Horizontal
4-H	2.7	Horizontal
1-V	7.2	Vertical
2-V	2.6	Vertical
3-V	4.3	Vertical
4-V	1.2	Vertical
5-V	3.2	Vertical

4.2.1 Results and discussions

The permeability data from Weatherford, Core laboratories, and OU reported permeability values at different pore pressures. Hence, different gas corrections should be applied. Data from Total comprised of corrected permeability values using the Klinkenberg correction.

To compare the permeability values from different laboratories, Klinkenberg correction was applied initially. Permeability values are plotted against reciprocal of mean pore pressure and the intercept provides the corrected permeability values. Figure 32 is a comparison of Klinkenberg corrected permeability values from the different laboratories.

There is a large discrepancy in permeability values. The permeability values differ by as much as a factor of 20.

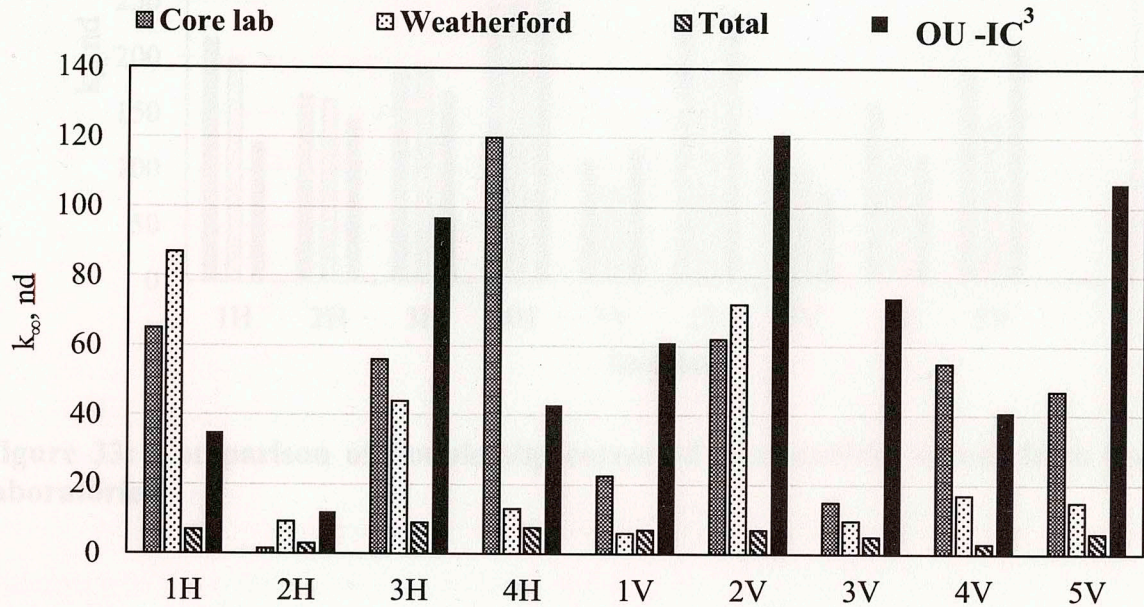


Figure 32: Comparison of Klinkenberg corrected permeability values from 4 laboratories

We applied the double slip correction and the result is shown in Figure 33. As Total's data consisted of only Klinkenberg corrected permeability values, we did not use them for this comparison.

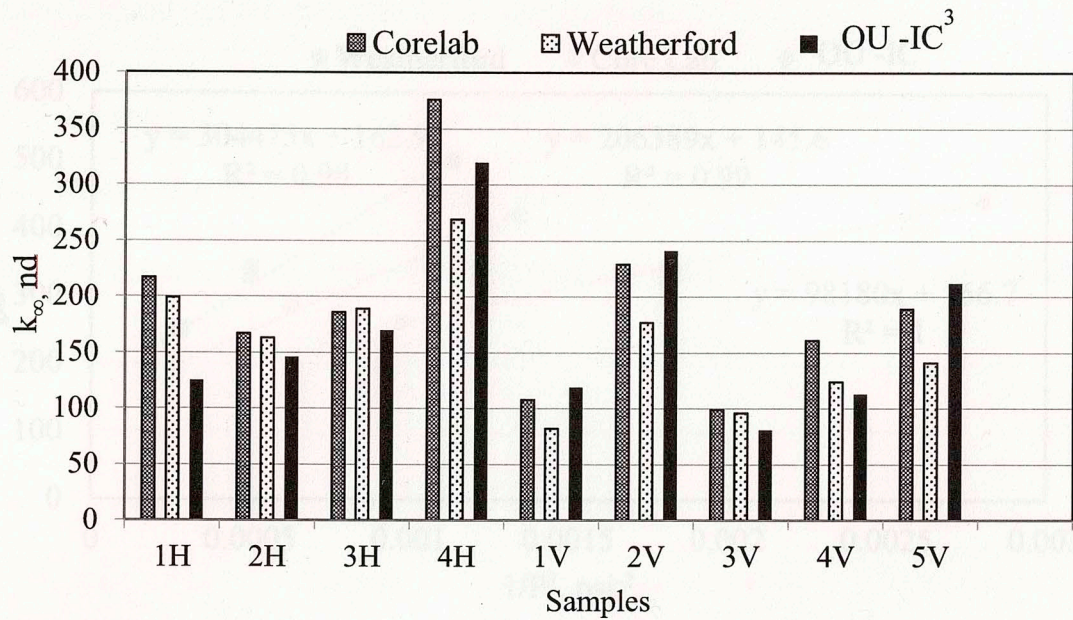


Figure 33: Comparison of double slip corrected permeability values from the 3 laboratories

The permeability values show improved agreement using the double slip correction (see figure 33). The average difference in permeability is less than 30%.

Figure 34 shows the use of double slip correction for one of the samples (2H) in the dataset. The intercepts for the curves in figure 34 from the three laboratories are very close to each other. The corrected double slip permeability values vary less than 15 % for this sample. The slopes though are different for each of the samples.

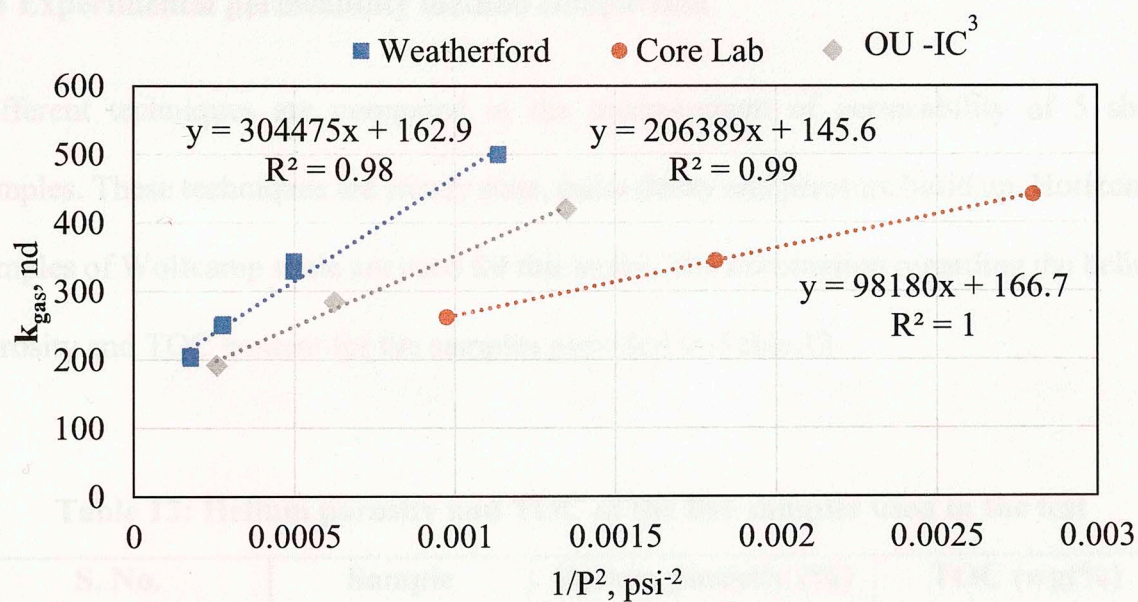


Figure 34: Double slip correction for the permeability values obtained from the three laboratories (sample 2H)

Summary of the round robin permeability tests:

- Double slip correction should be applied for pyrophyllite samples for pore pressures less than 175 psi; this implies that the transition flow regime is dominant at these low pore pressures.
- Transient techniques such as pressure build up can provide permeability values consistent with steady state value with differences less than 30%.

4.3 Experimental permeability method comparison

Different techniques are compared in the measurement of permeability of 5 shale samples. These techniques are steady state, pulse decay and pressure build up. Horizontal samples of Wolfcamp shale are used for this study. The information regarding the helium porosity and TOC content for the samples provided in Table 13.

Table 13: Helium porosity and TOC of the five samples used in the test

S. No.	Sample	Helium porosity (%)	TOC (wgt%)
1	XX83	5.5	1.4
2	XX66	2.4	3.1
3	XX33	1.8	2.3
4	XX65	5.3	0.4
5	XX07	7.1	1.0

The porosity of the samples range from 1.8 to 7.1 % while the TOC values range from 0.4 to 3.1 wgt%. Figure 35 provides FTIR mineralogy for the samples; the clay content varies from 7 to 34 %. Illite is the dominant clay mineral.

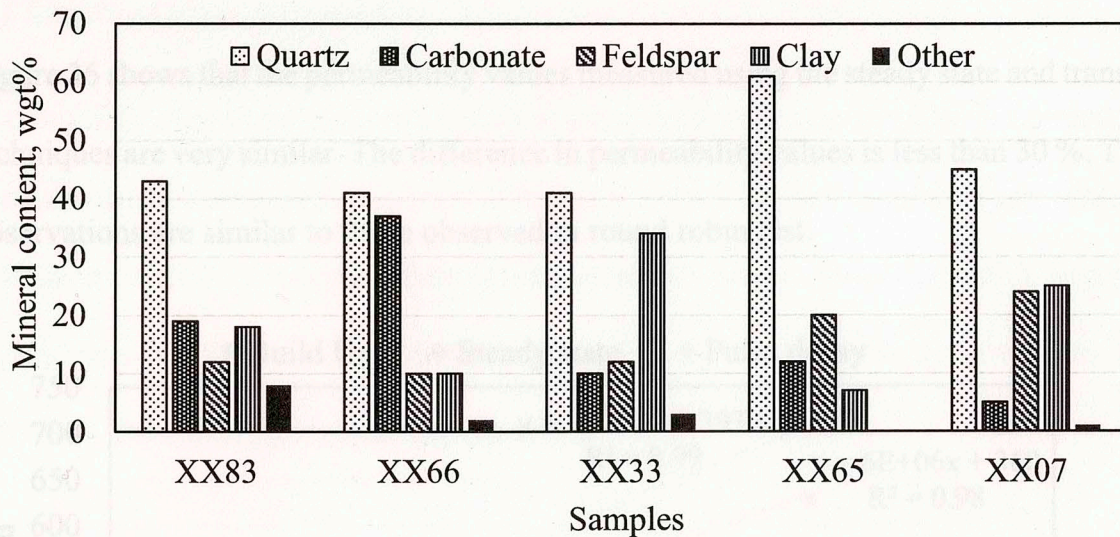


Figure 35: FTIR mineralogy for the five horizontal Wolfcamp samples used in this study

The effective pressure was 3000 psi. Nitrogen was the flowing fluid. Three different pore pressures were used for the measurement for each sample within the range of 100 to 250 psi. Double slip correction was applied. The double slip corrected permeability values are plotted for the five samples in figure 36. The range of measured permeabilities is 4 to 550 nd.

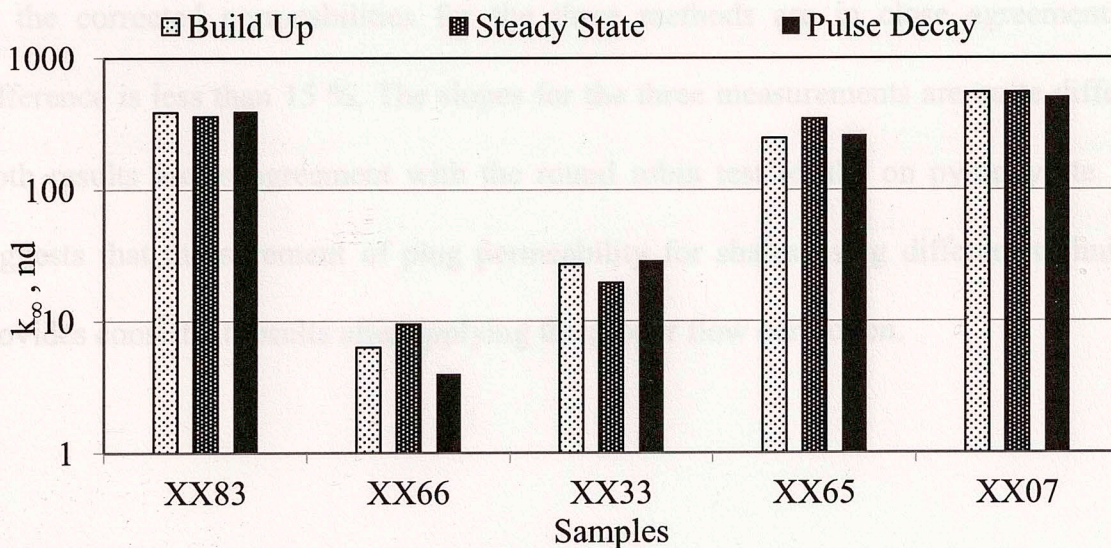


Figure 36: Comparison of double slip corrected permeabilities of build-up, steady state and pulse decay measurements on Wolfcamp shale.

Figure 36 shows that the permeability values measured using the steady state and transient techniques are very similar. The difference in permeability values is less than 30 %. These observations are similar to those observed in round robin test.

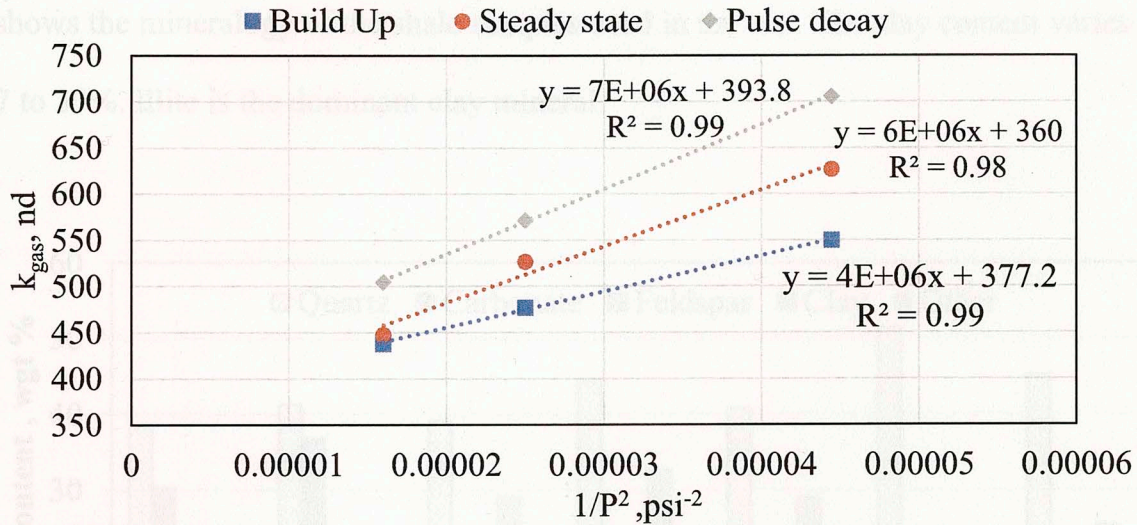


Figure 37: Double slip corrected permeabilities for sample XX83 for the three measurement techniques

Figure 37 shows the double slip corrected permeabilities for sample XX83. The intercepts or the corrected permeabilities for the three methods are in close agreement; the difference is less than 15 %. The slopes for the three measurements are quite different. Both results are in agreement with the round robin test results on pyrophyllite. This suggests that measurement of plug permeability for shales using different techniques provides consistent results after applying the proper flow correction.

4.4 Liquid and gas permeability comparison

Permeability variation due to difference in pore fluids was also examined. Permeability measurements were performed on 7 Wolfcamp shale samples using nitrogen (gas) and dodecane (liquid). The orientations of all the tested samples were horizontal. Figure 38 shows the mineralogy of the shale samples used in the test. The clay content varies from 7 to 33%. Illite is the dominant clay mineral.

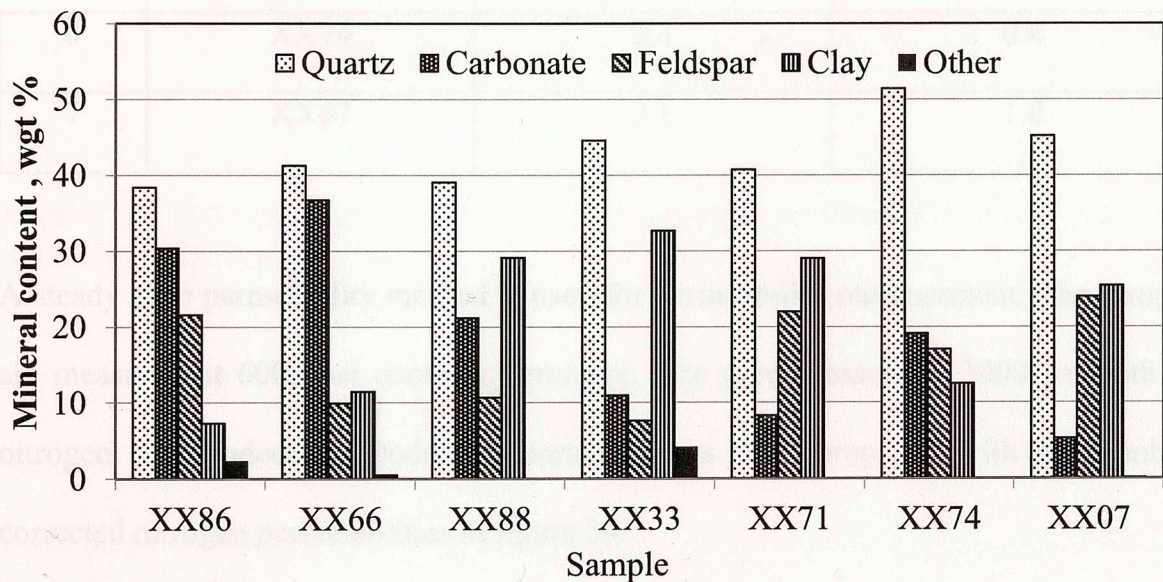


Figure 38: FTIR mineralogy of the Wolfcamp shale samples used in the liquid versus gas permeability comparison test.

Table 14 shows the range of TOC and porosity values of the Wolfcamp shale samples used in this test. The TOC varies from 0.2 to 4 wt% while the porosity ranges from 1.8 % to 10.3%.

Table 14: TOC and porosity values for the Wolfcamp shale samples used in the liquid versus gas comparison test.

S. No.	Shale sample	Helium porosity (%)	TOC (wgt%)
1	XX86	7.2	1.2
2	XX66	2.4	3.1
3	XX88	2.7	4.0
4	XX33	1.8	2.3
5	XX71	10.3	0.2
6	XX74	5.3	0.4
7	XX07	7.1	1.0

Figure 39: Nitrogen (gas) and dodecane (liquid) permeability comparison for Wolfcamp shale samples.

A steady state permeability method is used for permeability measurement. The samples are measured at 6000 psi confining pressure. The pore pressure is 3000 psi both for nitrogen and dodecane. Dodecane permeabilities are compared with Klinkenberg corrected nitrogen permeabilities in figure 39.

The difference in liquid and gas permeability values is compared with findings reported by Cui et al. (2013) and Chou et al. (2015).

NMR measurements have been conducted on the sample before and after permeability measurements using nitrogen and dodecane. The acquisition of NMR data was made at 2MHz ($\gamma = 17.6$ rad) using Oxford NMR console instrument. The signal to noise ratio was 100 for the measurements.

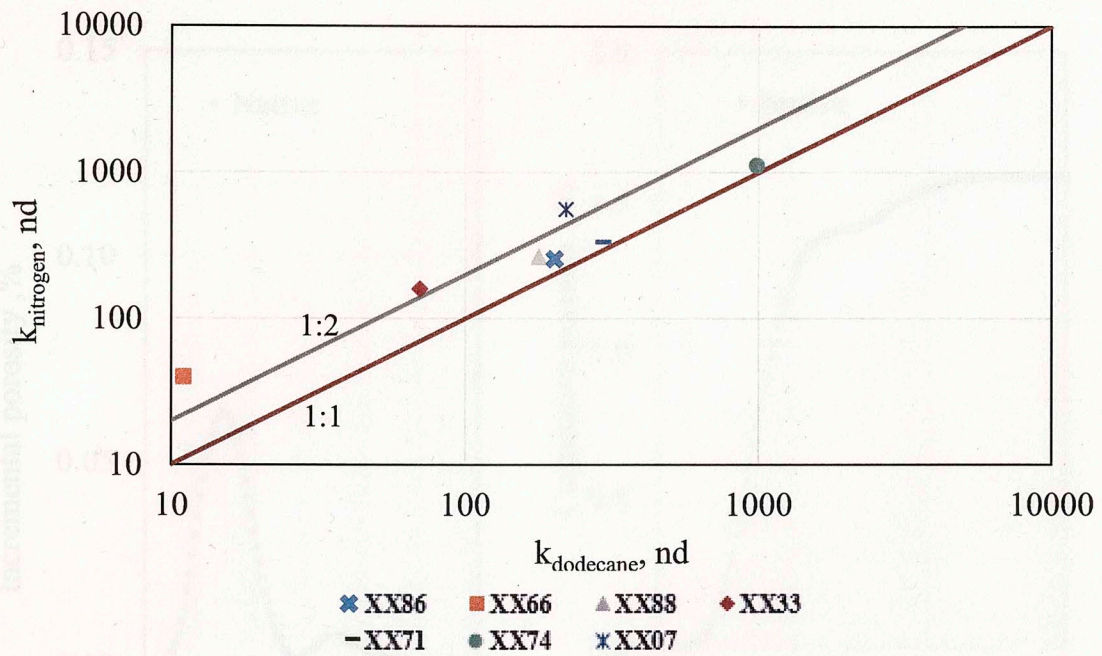


Figure 39: Nitrogen (gas) and Dodecane (liquid) permeability comparison for Wolfcamp shale samples.

The gas permeability in figure 39 is generally greater than the liquid permeability by a factor of 2. Note that some samples fall on the 1:1 line. Hence, we cannot see a common trend in the differences between measured liquid and gas permeabilities. The difference in liquid and gas permeability values is consistent with findings reported by Cui et al. (2013) and Chhatre et al. (2014).

NMR measurements have been conducted on the sample before and after permeability measurements using nitrogen and dodecane. The acquisition of NMR data was made at 2MHz ($\tau = 57\mu\text{sec}$) using Oxford Maran GeoSpec instrument. The signal to noise ratio was 100 for the measurements.

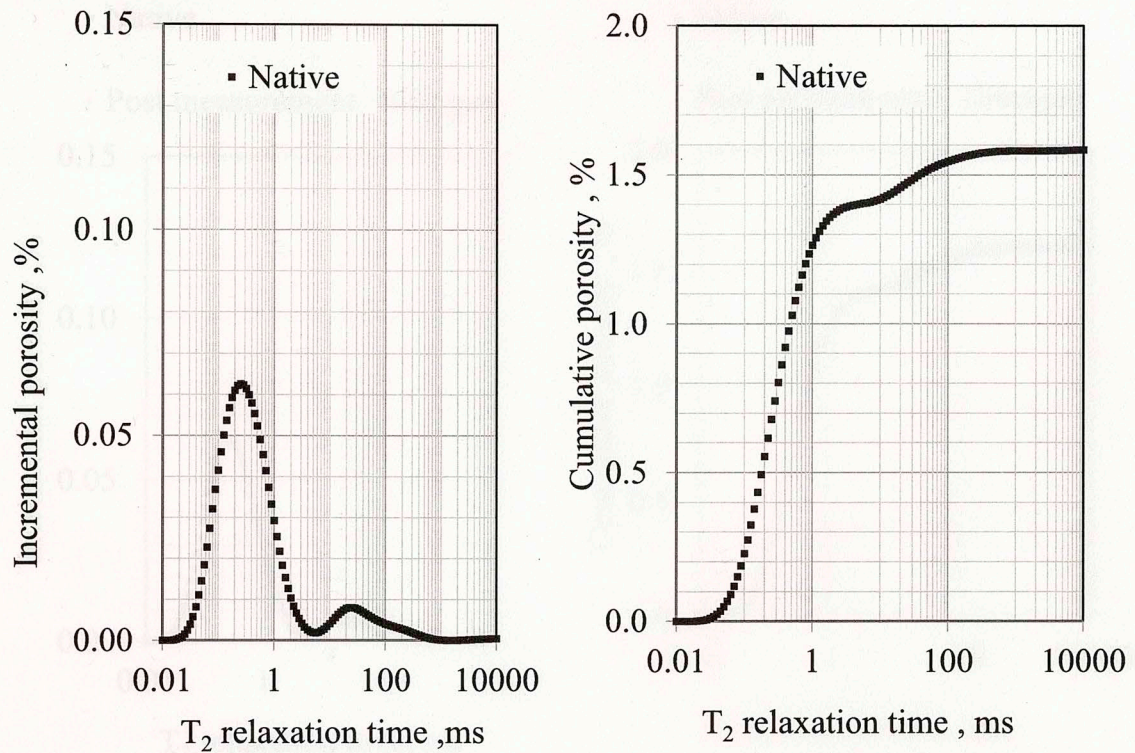


Figure 40: Native NMR incremental and cumulative porosity curves for sample XX86 Wolfcamp sample before permeability measurement.

Figure 40 shows the incremental and cumulative NMR porosity plots for the Wolfcamp sample XX66 before the permeability measurement, i.e. initial native state. The sample shows a T2 peak at 0.25ms. Tinni et al. (2014) show that the NMR peak in shales less than 1ms correspond to water wet clay pores. A cumulative NMR porosity for the sample at this stage is found to be 1.6 %, this is an equivalent water saturation of 22 %.

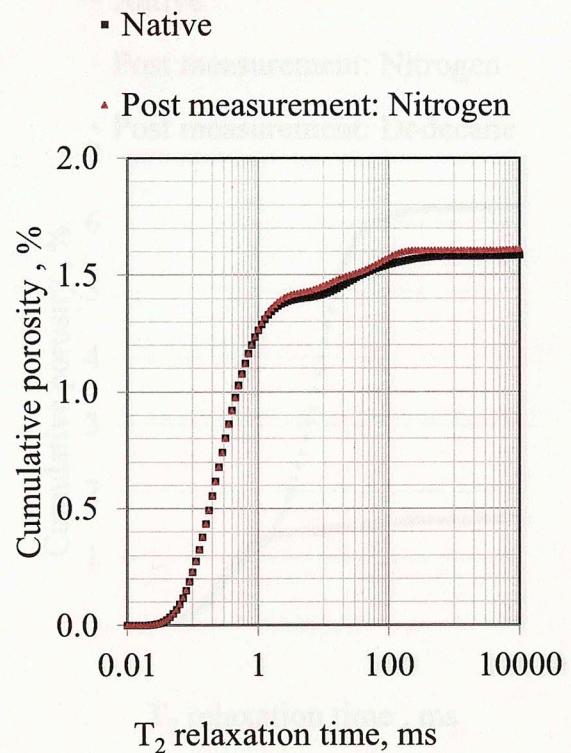
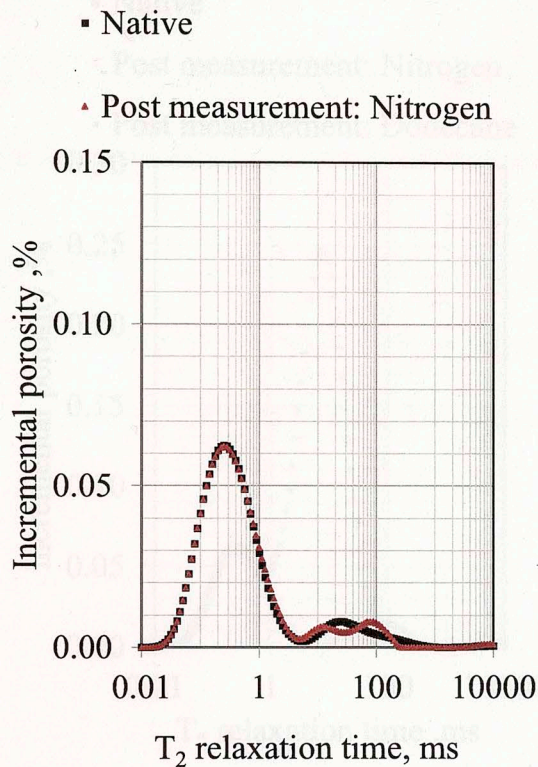


Figure 41: NMR incremental and cumulative porosity curves for sample XX86 after permeability with nitrogen.

Figure 41 shows the incremental and NMR porosity curves for the initial sample and post permeability measurement with nitrogen. Note the vertical scales are different for incremental and cumulative plots but are consistent between the before and after plots. The NMR curves remain unaltered. This indicates that the water saturation remained unchanged at 22% after permeability measurement with nitrogen.

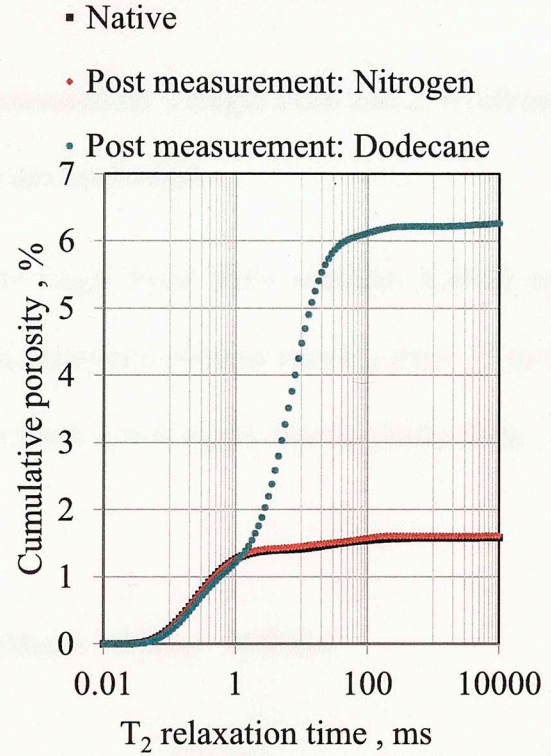
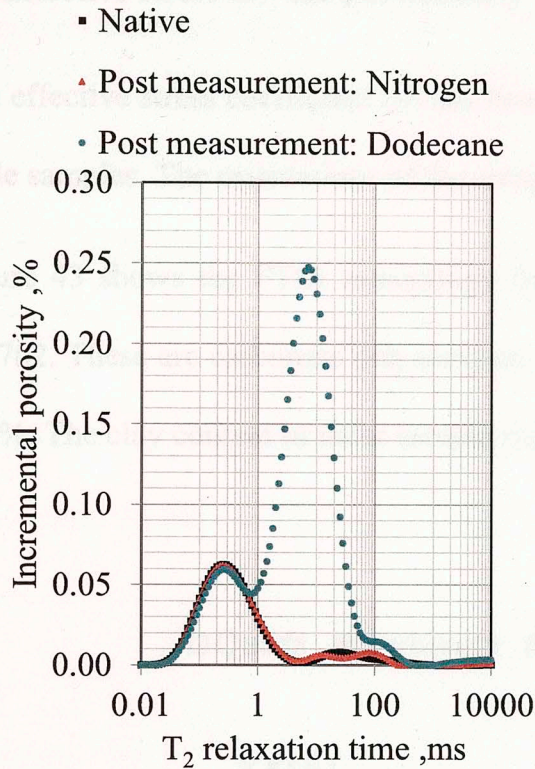


Figure 42: NMR incremental and cumulative porosity curves for sample XX86 after permeability with dodecane.

Figure 42 shows the incremental and NMR porosity curves for the initial sample, post nitrogen permeability measurement and post dodecane permeability measurement. After measurement with dodecane the incremental graph shows two peaks one at 0.25 ms as seen before and a new one at 7 ms. Tinni et al. (2014) show that the peak around 6-20 ms generally corresponds to oil (dodecane) in shales. The amplitude of the peak associated with water wet clay pores at 0.25 ms remains same. This indicates that the water saturation after permeability measurement with dodecane still remains at 22%.

4.5 Effective stress law for permeability

The effective stress coefficient (n) has been measured on 2 Eagle Ford and 2 Wolfcamp shale samples. The orientations of the samples are horizontal.

Figure 43 shows the FTIR mineralogy for the Eagle Ford shale samples XX692 and XX702. These are carbonate rich samples with carbonate content varying from 71 to 80 wgt%. The clay content in these sample ranges from 4 to 6 wgt% and is mostly illite.

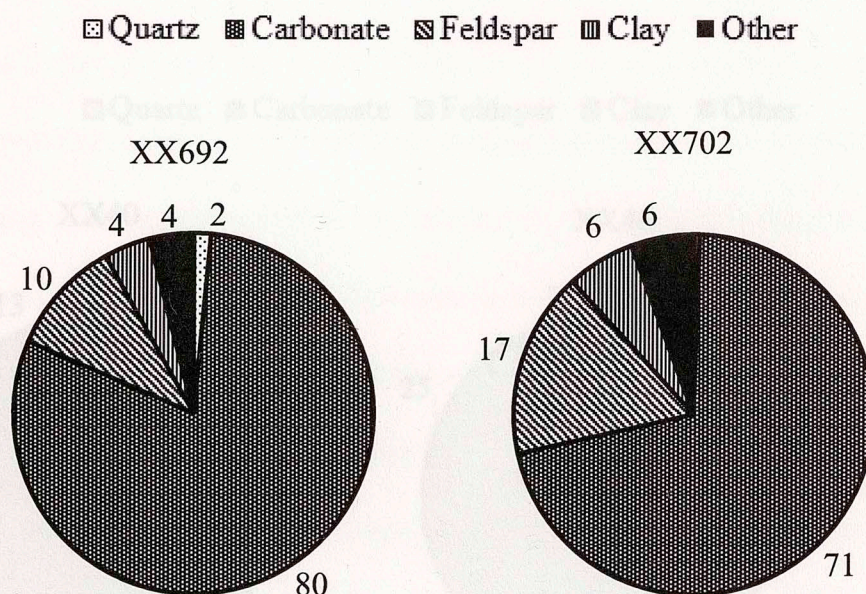


Figure 43: FTIR mineralogy of the two Eagle Ford shale samples used in the effective stress law test. Eagle Ford is actually a carbonate. Percentages are wgt%.

The helium porosity and TOC content of the two samples is given in Table 15. The porosity of the samples range from 8 to 9 % while the TOC content is ~ 6 wgt% for both the samples.

Table 15: Porosity and TOC data the two Eagle Ford shale samples used in the effective stress law test.

Sample	Helium porosity (%)	TOC (wgt%)
XX692	9.2	5.5
XX702	8.3	5.8

The FTIR mineralogy of the 2 Wolfcamp shale samples is given below in figure 44. The clay content of the 2 samples is ~ 25%. The dominant clay is illite.

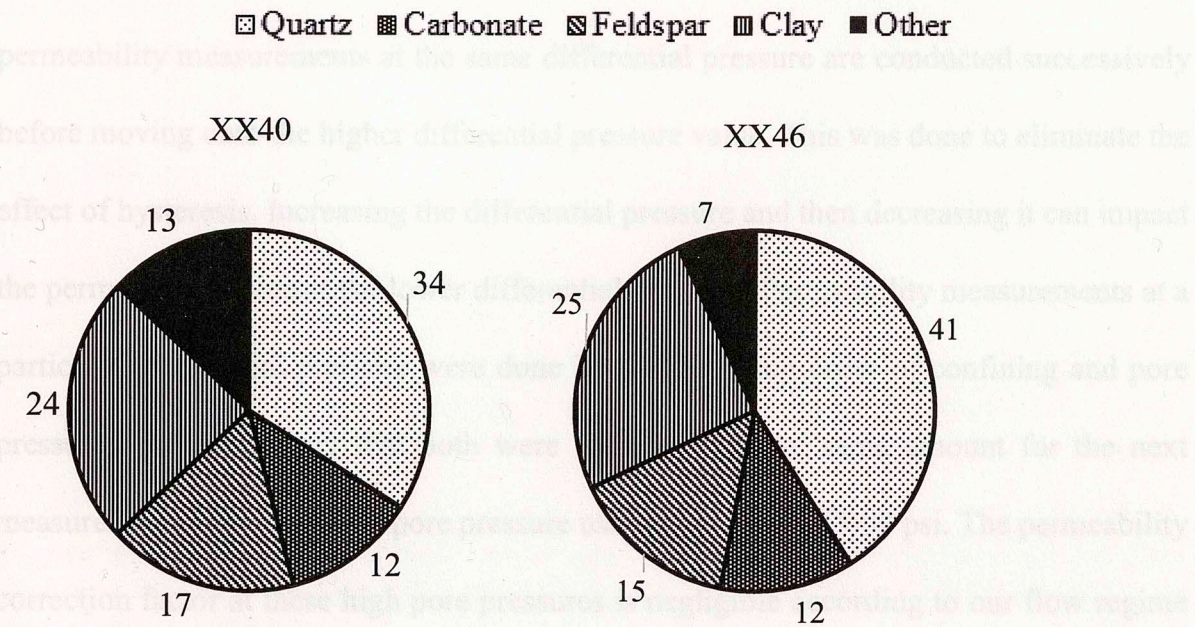


Figure 44: FTIR Mineralogy of the two Wolfcamp shale samples used in the effective stress law test. Percentages are wgt%.

The helium porosity and TOC content of the two Wolfcamp shale samples is given in Table 16.

Table 16: Porosity and TOC data the two Wolfcamp shale samples used in the effective stress law test.

Sample	Helium porosity (%)	TOC (wgt%)
XX40	4.3	1.1
XX46	5.6	2.0

The Todd and Simmons (1972) method has been used to calculate the effective pressure coefficient. To calculate effective pressure coefficient, permeability measurements have to be performed at different pore and confining pressures. The sequence of permeability measurements can impact the value of effective pressure coefficient. In this study permeability measurements at the same differential pressure are conducted successively before moving onto the higher differential pressure value. This was done to eliminate the effect of hysteresis. Increasing the differential pressure and then decreasing it can impact the permeability value at the lower differential pressure. Permeability measurements at a particular differential pressure were done in the ascending order of confining and pore pressures in such a way that both were increased by the same amount for the next measurement. The minimum pore pressure used in this test is 2000 psi. The permeability correction factor at these high pore pressures is negligible according to our flow regime tests. Hence effect of gas slippage would be negligible in this study. The discussion of permeability measurements for the two shale formations is presented separately. Pressure build-up technique was used for permeability measurement of the Eagle Ford samples. The pore pressures used in the study ranged from 2000 to 3000 psi in 500 psi increments. Nitrogen was used as the pore fluid. The different differential pressures (P_d) used in the study range from 1000 to 4000 psi in 1000 psi increments. P_d is the simple difference

between the confining and pore pressures. Figure 45 and 46 show the permeability values measured for Eagle Ford samples XX692 and XX702. For both samples permeability reduces by more than 1 order of magnitude as differential pressure increases from 1000 to 4000 psi. Permeability measurements at 2000 psi pore pressure and the four differential pressures were made thrice to get the error in measurement which was found to be 5%. For all other pressure points permeability was just measured just once.

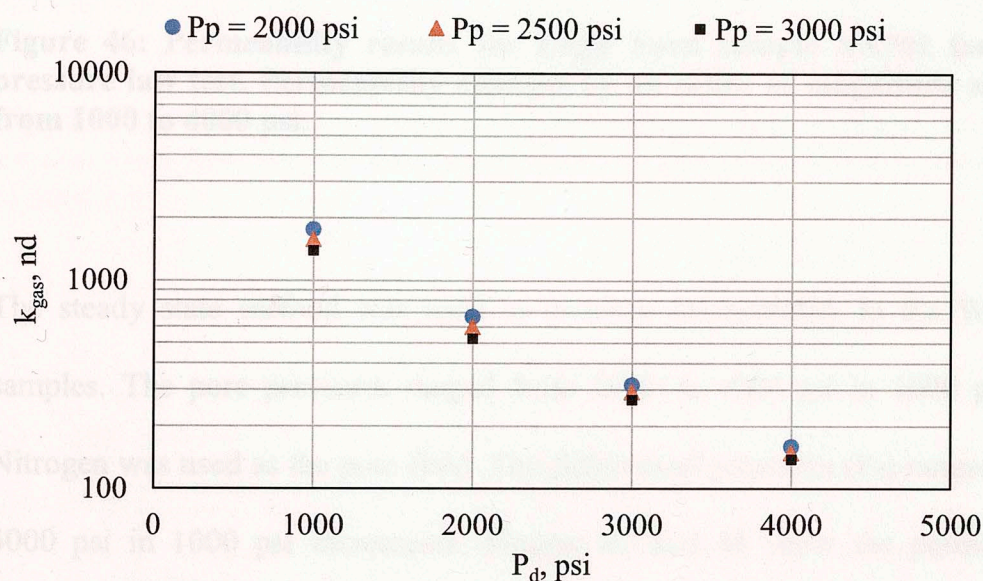


Figure 45: Permeability results for Eagle Ford sample XX692 for the effective pressure law test. Permeability changes by an order of magnitude as P_d increases from 1000 to 4000 psi.

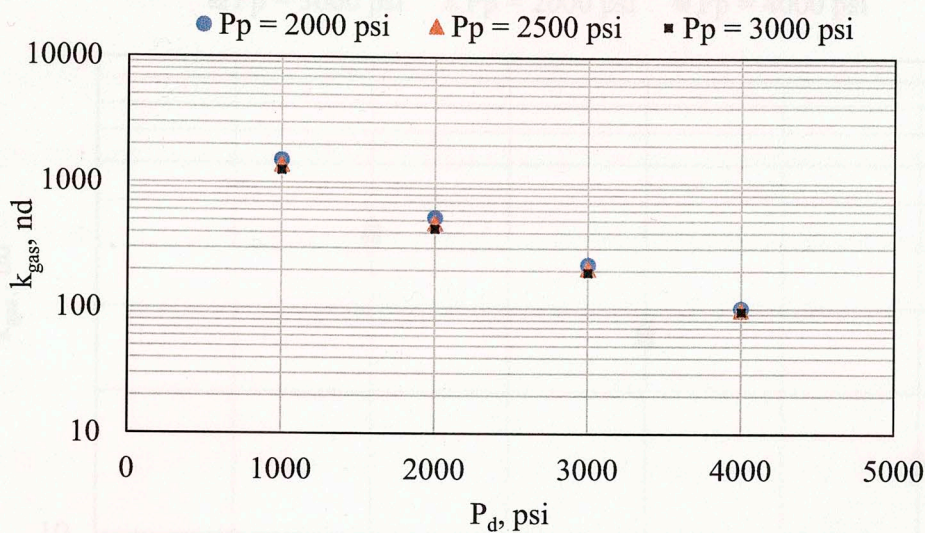


Figure 46: Permeability results for Eagle Ford sample XX702 for the effective pressure law test. Permeability changes by an order of magnitude as P_d increases from 1000 to 4000 psi.

The steady state method was used to measure permeability in the Wolfcamp shale samples. The pore pressures ranged from 2000 to 4000 psi in 1000 psi increments. Nitrogen was used as the pore fluid. The differential pressures (P_d) ranged from 1000 to 3000 psi in 1000 psi increments. Figures 47 and 48 show the permeability values measured for Wolfcamp samples XX40 and XX46. For both samples permeability reduces by a factor of 3 as differential pressure increases from 1000 to 3000 psi.

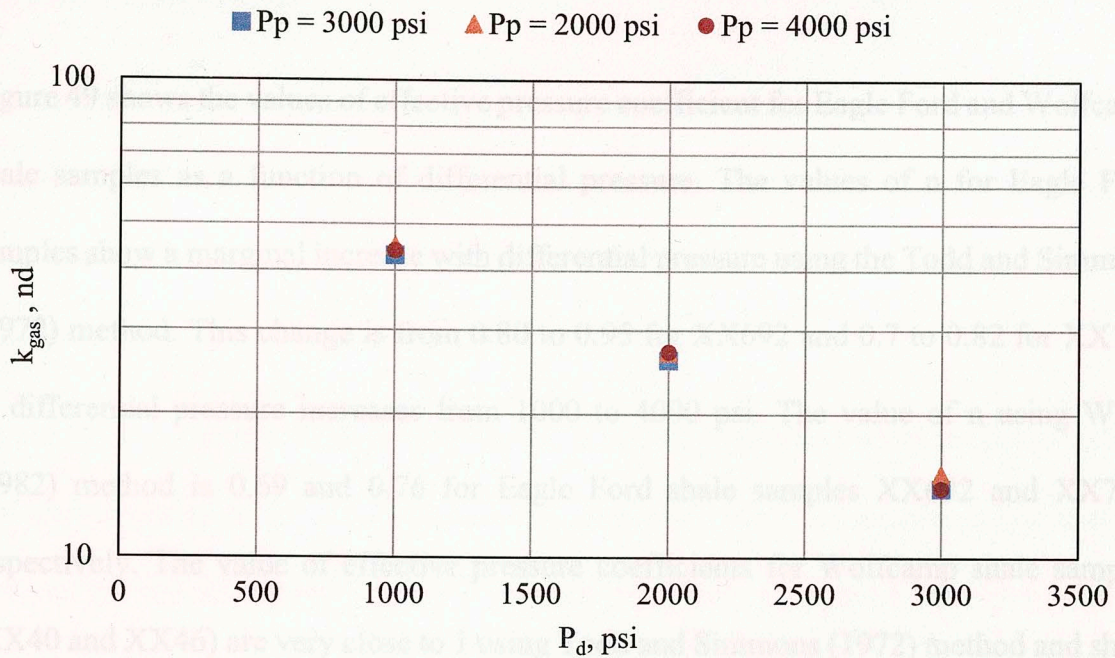


Figure 47: Permeability results for Wolfcamp sample XX40 for the effective pressure law test. Permeability decreases by a factor of 3 as P_d changes from 1000 to 3000 psi.

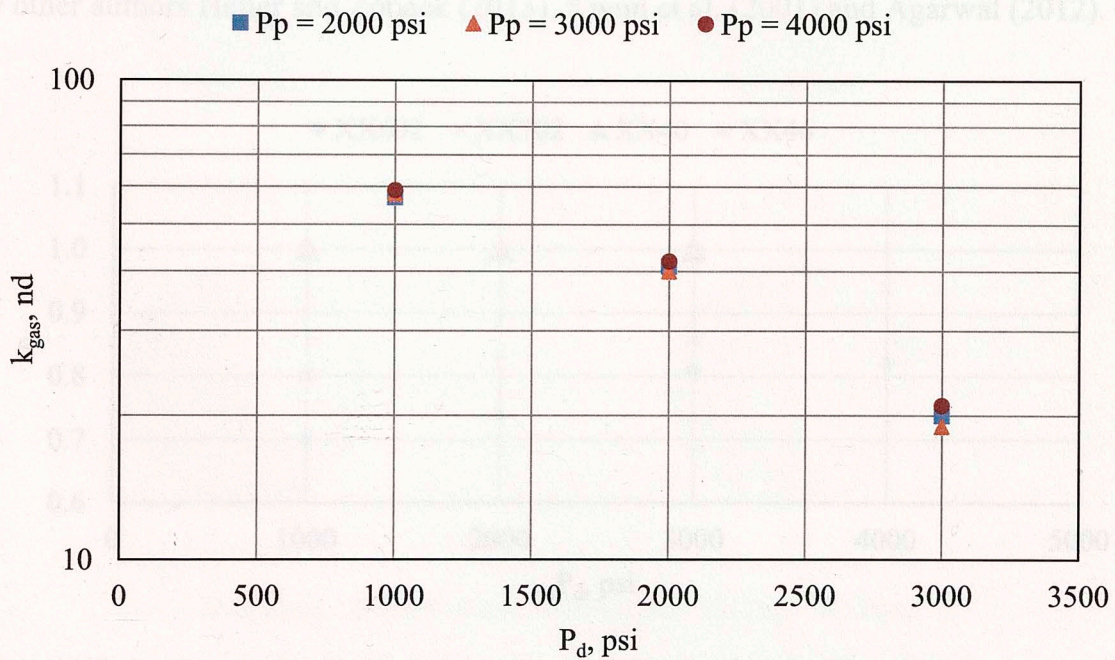


Figure 48: Permeability results for Wolfcamp sample XX40 for the effective pressure law test. Permeability decreases by a factor of 3 as P_d changes from 1000 to 3000 psi.

Figure 49 shows the values of effective pressure coefficient for Eagle Ford and Wolfcamp shale samples as a function of differential pressure. The values of n for Eagle Ford shale samples show a marginal increase with differential pressure using the Todd and Simmons (1972) method. This change is from 0.80 to 0.95 for XX692 and 0.7 to 0.82 for XX702 as differential pressure increases from 1000 to 4000 psi. The value of n using Walls (1982) method is 0.69 and 0.76 for Eagle Ford shale samples XX692 and XX702, respectively. The value of effective pressure coefficients for Wolfcamp shale samples (XX40 and XX46) are very close to 1 using Todd and Simmons (1972) method and show no pressure dependence. The value of n was 1 using Bernabe (1987) method as well as Walls (1982) method for the Wolfcamp shale samples. The values of n are thus smaller than or equal to 1 for both the shales. This data is in agreement with what has been found by other authors Heller and Zoback (2013), Kwon et al. (2001) and Agarwal (2012).

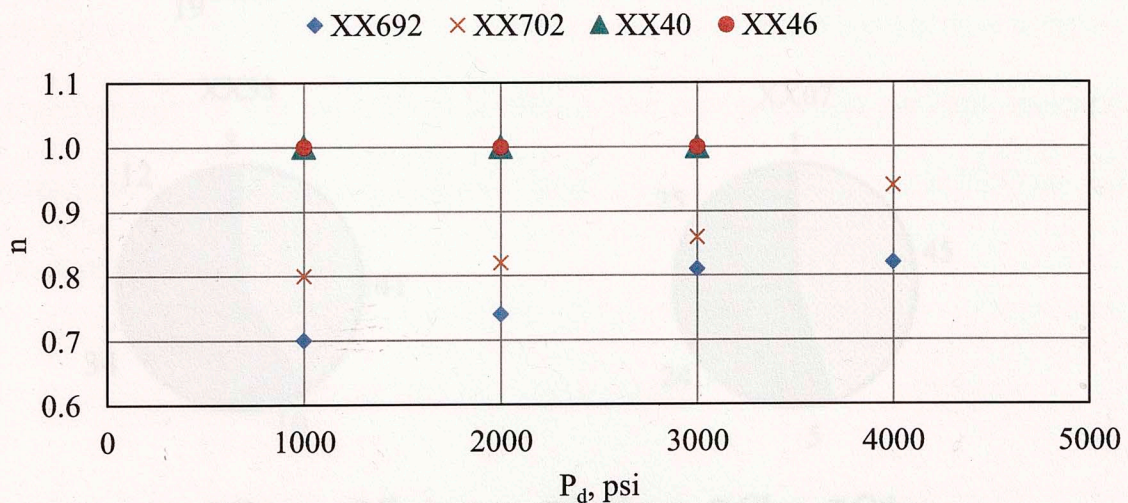


Figure 49: Values of the effective pressure coefficient (n) using Todd and Simmons (1972) for Eagle Ford (XX692, XX702) and Wolfcamp (XX40, XX46) shale samples. The value of n varies little with P_d and is generally close to 1.

4.6 Permeability creep

Permeability creep measurements require application of constant confining and pore pressures while observing permeability changes over time. Creep measurements were performed on Wolfcamp, Eagle Ford and Vaca Muerta shale samples. Sample description and creep results are discussed separately for each shale.

Figure 50 provides the FTIR mineralogy for the four Wolfcamp shale studies on which permeability creep studies was studied. The orientation of all the samples is horizontal.

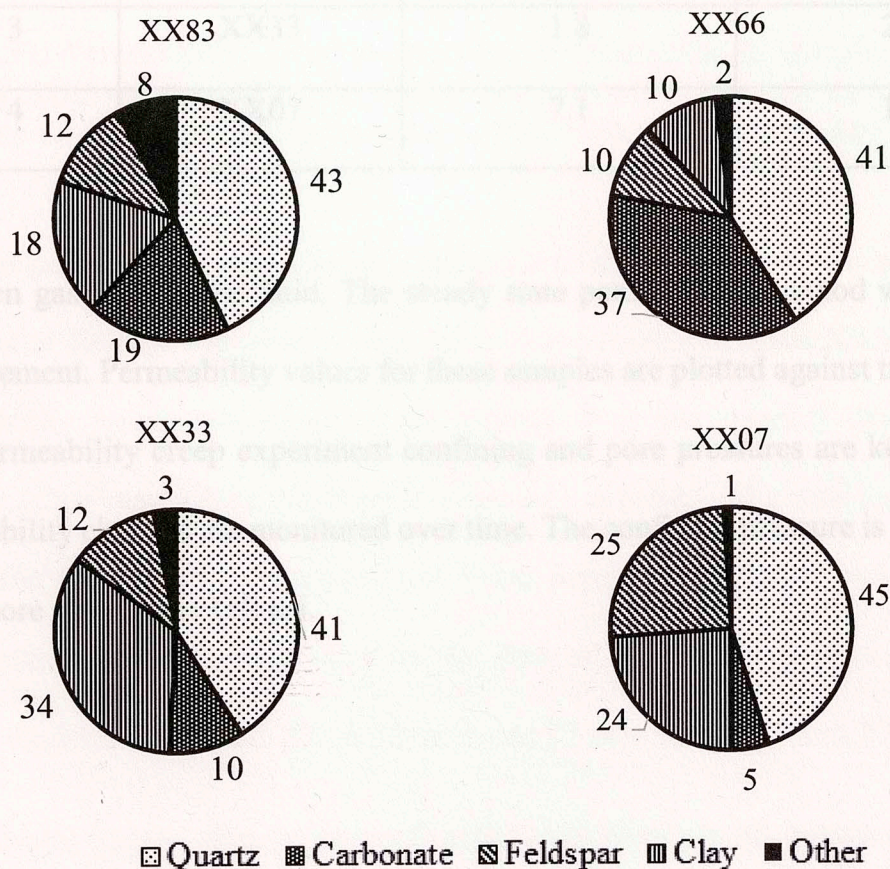


Figure 50: FTIR mineralogy for 4 Wolfcamp shale samples used in creep study.

Table 17 provides the porosity and TOC values of the Wolfcamp shale studies. The range of porosity values in these samples is from 1.8 to 7.1 %. TOC content varies from 1 to 3.1 wgt%. Illite is the dominant clay mineral.

Table 17: Porosity and TOC values of the Wolfcamp shale samples used in the creep study

S. No.	Sample	Helium porosity (%)	TOC (%)
1	XX83	5.5	1.4
2	XX66	2.4	3.1
3	XX33	1.8	2.3
4	XX07	7.1	1.0

Nitrogen gas is the pore fluid. The steady state permeability method was used for the measurement. Permeability values for these samples are plotted against time in figure 51. In a permeability creep experiment confining and pore pressures are kept constant and permeability changes are monitored over time. The confining pressure is 3000 psi and the mean pore pressure is 150 psi.

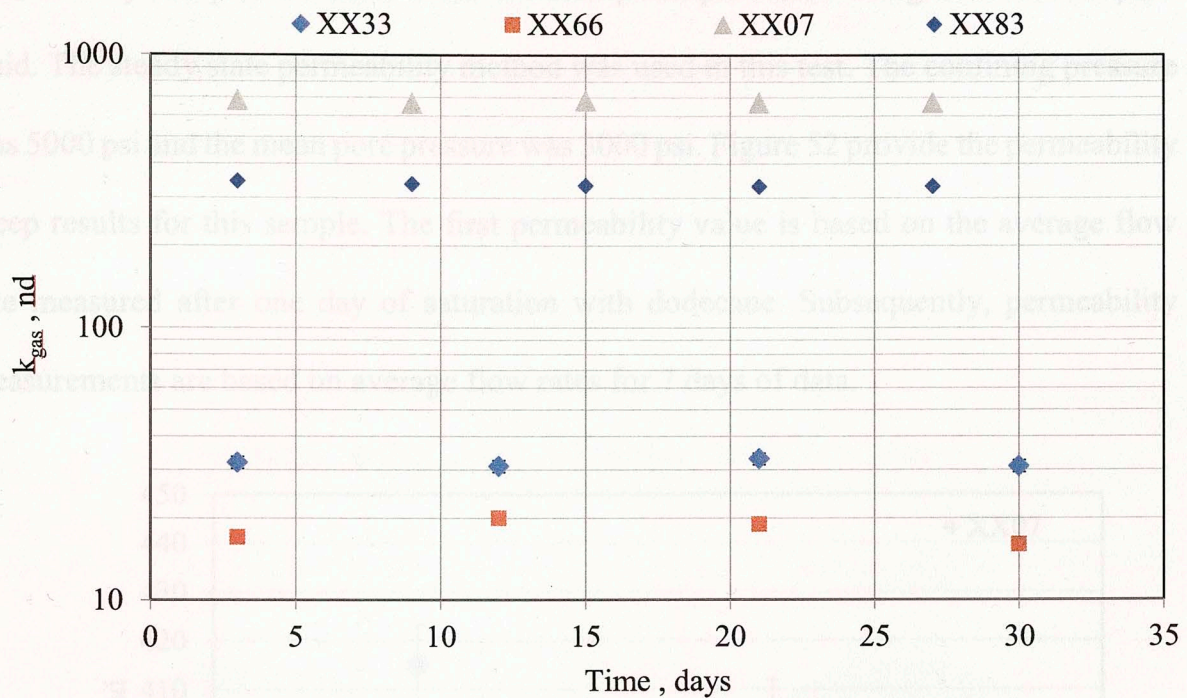


Figure 51: Permeability creep for the four Wolfcamp shale samples using nitrogen as pore fluid. Permeability was measured with a steady state technique. There is basically no observed change in permeability with time over 30 days.

The first permeability value for each sample was calculated using the average flow rate obtained in the first 3 days after saturation; subsequently samples XX33 and XX66 the permeability was measured using the average flow rate every 9 days. However for samples XX07 and XX83 this time was reduced to 6 days. The time for samples XX33 and XX66 was greater because of their lower permeability. More time was required to get a better correlation coefficient for the flow rate data. The duration of measurement was 30 days for XX33 and XX66 while it was 27 days for XX07 and XX83.

Permeability values change insignificantly with time for all samples. The range of measured permeability values range from around 20 nd to 700 nd. No reduction in permeability is observed for any sample.

Permeability creep test was done on Wolfcamp sample XX07 using dodecane as pore fluid. The steady state permeability method was used in this test. The confining pressure was 5000 psi and the mean pore pressure was 3000 psi. Figure 52 provide the permeability creep results for this sample. The first permeability value is based on the average flow rate measured after one day of saturation with dodecane. Subsequently, permeability measurements are based on average flow rates for 7 days of data.

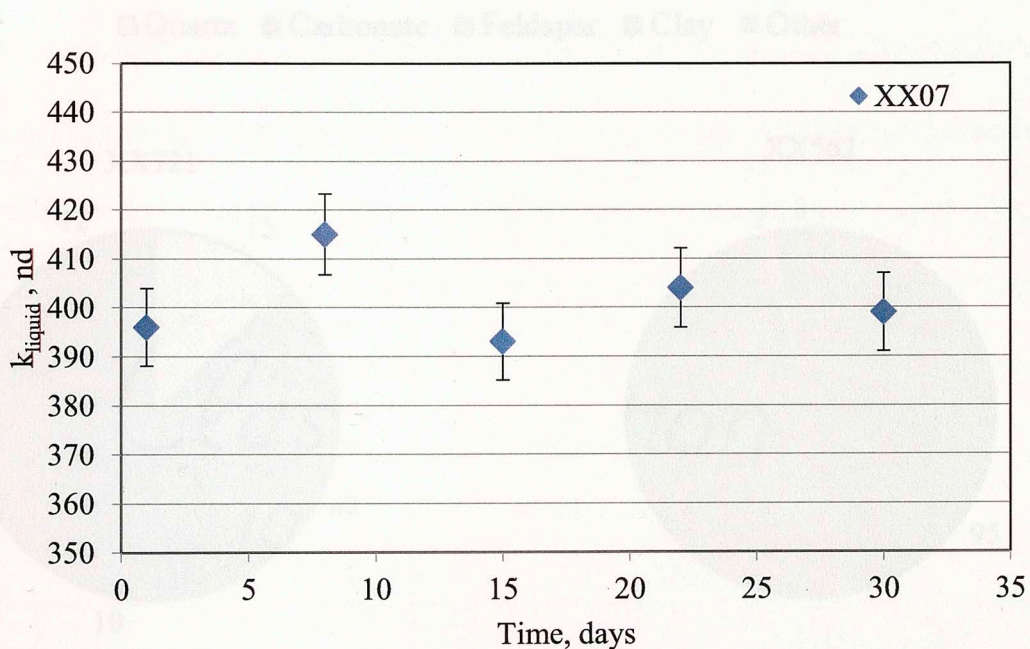


Figure 52: Permeability creep for Wolfcamp shale sample XX07 saturated with dodecane. Permeability was measured with a steady state technique.

The complete test is conducted for a month. It is observed that the change in permeability is within the range of experimental error. The mean value of permeability is close to 400 nd and the permeability oscillates between 390 and 420 nd during the period of measurement. No significant reduction in permeability is observed.

The second shale on which permeability creep measurements were made was the Eagle Ford shale. Mineralogy of the two samples used in the test is given in figure 53. Illite is the dominant clay mineral.

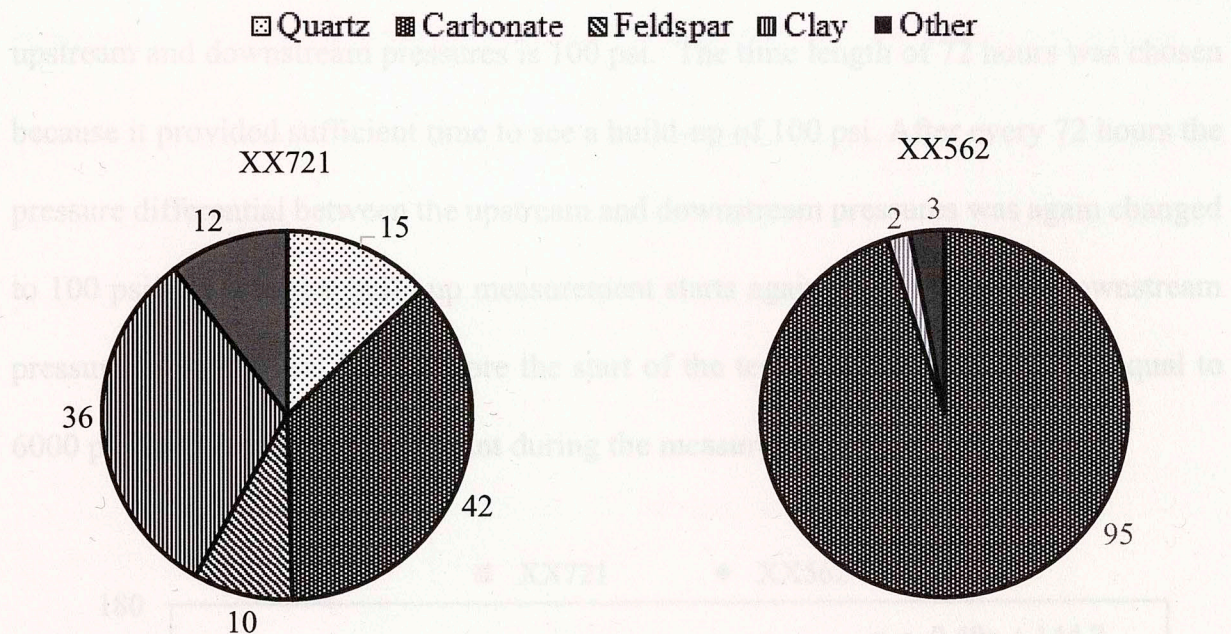


Figure 53: FTIR mineralogical composition for the two Eagle Ford shale samples used in the creep study. Numbers are in wgt%. Note XX721 has 36% clay

Table 18 provides the porosity and TOC values of the Eagle Ford shale samples.

Table 18: Porosity and TOC values of the Eagle Ford shale samples used in the creep study

Sample	Helium porosity (%)	TOC (wgt%)
XX721	5.7	3.5
XX562	8.5	4.4

The Eagle Ford samples were tested for permeability creep using nitrogen gas as pore fluid. Permeability values for these samples are plotted against time in figure 54. The confining pressure is 6000 psi and the mean pore pressure is 3000 psi. Permeability changes are monitored with time using the pressure build up data. Permeability measurements are made every 72 hours. The initial pressure differential between upstream and downstream pressures is 100 psi. The time length of 72 hours was chosen because it provided sufficient time to see a build-up of 100 psi. After every 72 hours the pressure differential between the upstream and downstream pressures was again changed to 100 psi and pressure build up measurement starts again by bringing the downstream pressure to its original value before the start of the test. Confining pressure is equal to 6000 psi and is maintained constant during the measurement.

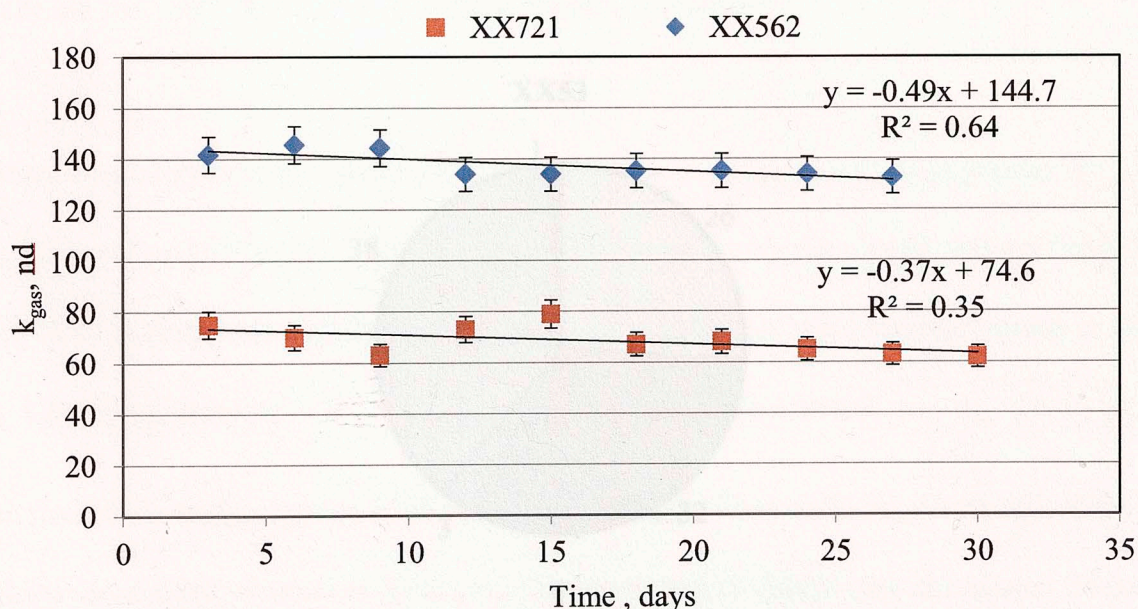


Figure 54: Permeability creep test for Eagle Ford shale samples XX721 and XX562 while using nitrogen as pore fluid. The confining pressure for the measurement is 6000 psi and the mean pore pressure is 3000 psi.

No significant reduction in permeability for the 2 samples is observed (see figure 54). The permeability of sample XX721 decreases from 79 to 62 nd in 30 days while the permeability of the sample XX562 decreases from 142 nd to 133 nd in the same period. The changes in permeabilities for the 2 samples are within 25%.

The third shale on which permeability creep measurement was performed is the Vaca Muerta shale. This creep measurement was performed at different effective pressures.

The FTIR mineralogy of the Vaca Muerta shale XX41 used in this test is provided by figure 55. The TOC of the Vaca Muerta sample XX53 is 5.8 wgt% and the helium porosity is 2.5 %. The sample consists of 28% mixed clays in wgt%.

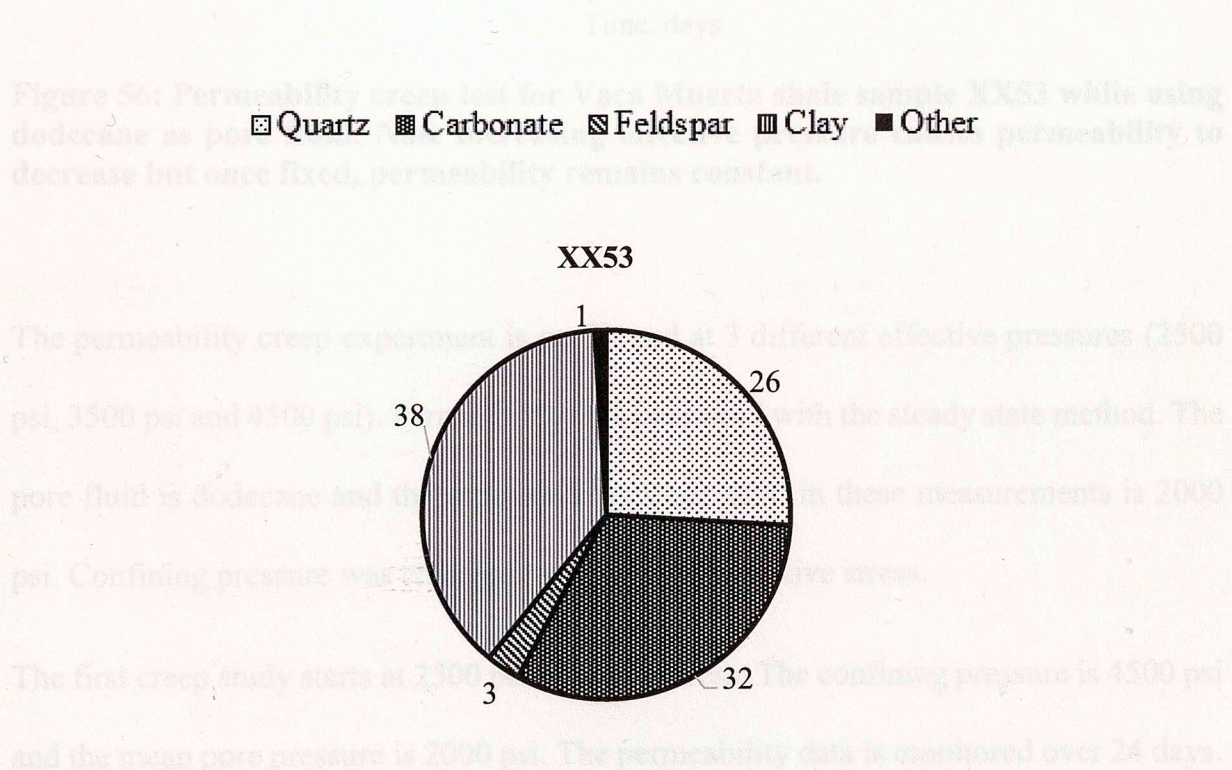


Figure 55: Mineralogical composition for the Vaca Muerta shale sample XX53 used in the creep study. Note values are in wgt%.

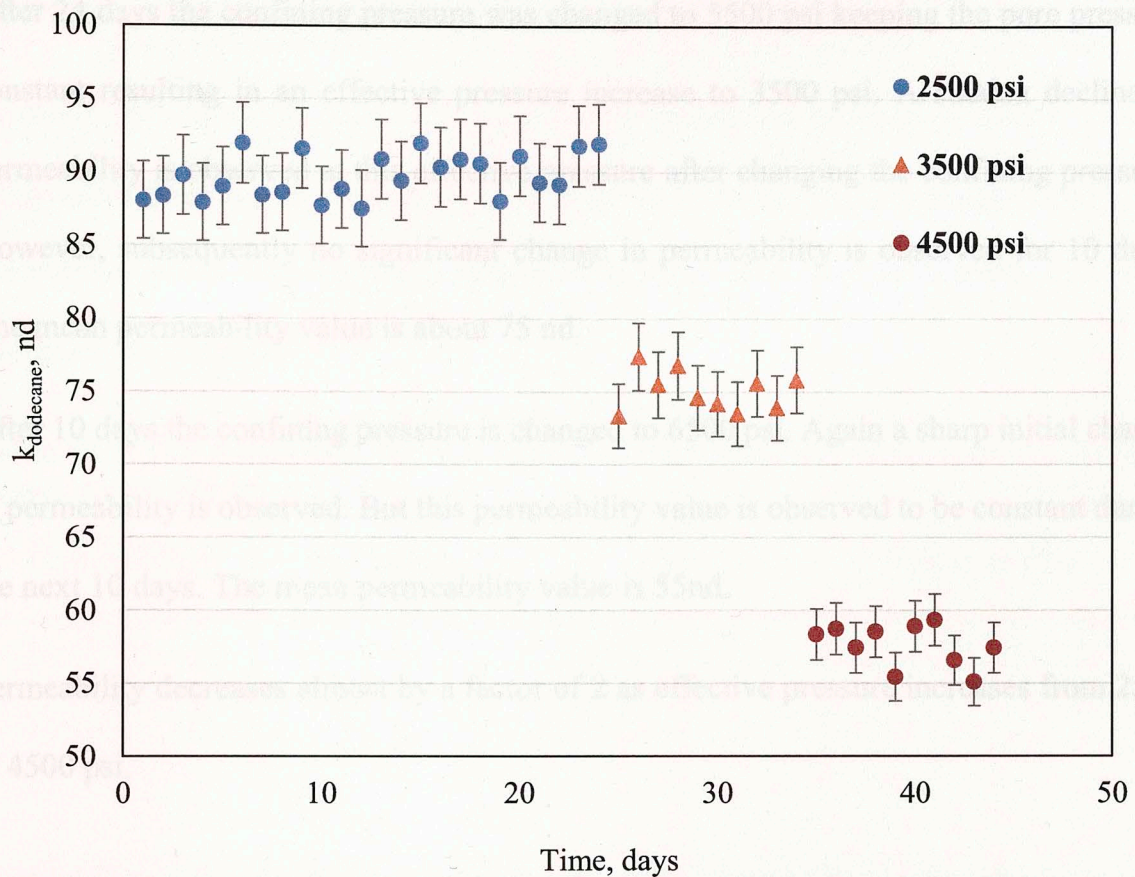


Figure 56: Permeability creep test for Vaca Muerta shale sample XX53 while using dodecane as pore fluid. Note increasing effective pressure causes permeability to decrease but once fixed, permeability remains constant.

The permeability creep experiment is performed at 3 different effective pressures (2500 psi, 3500 psi and 4500 psi). Permeability was measured with the steady state method. The pore fluid is dodecane and the mean pore pressure used in these measurements is 2000 psi. Confining pressure was changed to change the effective stress.

The first creep study starts at 2500 psi effective stress. The confining pressure is 4500 psi and the mean pore pressure is 2000 psi. The permeability data is monitored over 24 days. Permeability is calculated based on the average flow rate for each day. The permeability values oscillate around 90 nd. No significant reduction in permeability is observed.

After 24 days the confining pressure was changed to 5500 psi keeping the pore pressure constant resulting in an effective pressure increase to 3500 psi. A sudden decline in permeability is observed at this effective pressure after changing the confining pressure. However, subsequently no significant change in permeability is observed for 10 days. The mean permeability value is about 75 nd.

After 10 days the confining pressure is changed to 6500 psi. Again a sharp initial change in permeability is observed. But this permeability value is observed to be constant during the next 10 days. The mean permeability value is 55nd.

Permeability decreases almost by a factor of 2 as effective pressure increases from 2500 to 4500 psi.

4.7 Permeability versus effective pressure: Eagle Ford shale

Permeability was measured for 22 Eagle Ford samples as a function of effective stress.

FTIR mineralogy for these shale samples is given in Table 19. TOC ranges from 1.9 to 7.3 wgt% and the helium porosity ranges from 1.9 to 10 % for the samples Illite is the dominant clay mineral

Table 19: FTIR mineralogy for the 22 measured Eagle Ford shale samples. Note all values are in wgt %.

Quartz (%)	Carbonate (%)	Clay (%)	Feldspar (%)
0 - 15	42 - 95	2 - 36	0 - 10

Permeability of the samples was measured using the pressure build up method. The pore fluid was nitrogen. The mean pore pressure was 2000 psi; this eliminated the effect of gas slippage. So although the permeabilities are not corrected, they are very close to the corrected permeability value, with difference being within 20 % as found from the flow regime test. Permeability measurements were made as a function of effective pressure by changing the confining pressure. Initially, the range of effective pressure for the measurements was from 1000 to 6000 psi. Figure 57 shows the results of permeability results for some samples.

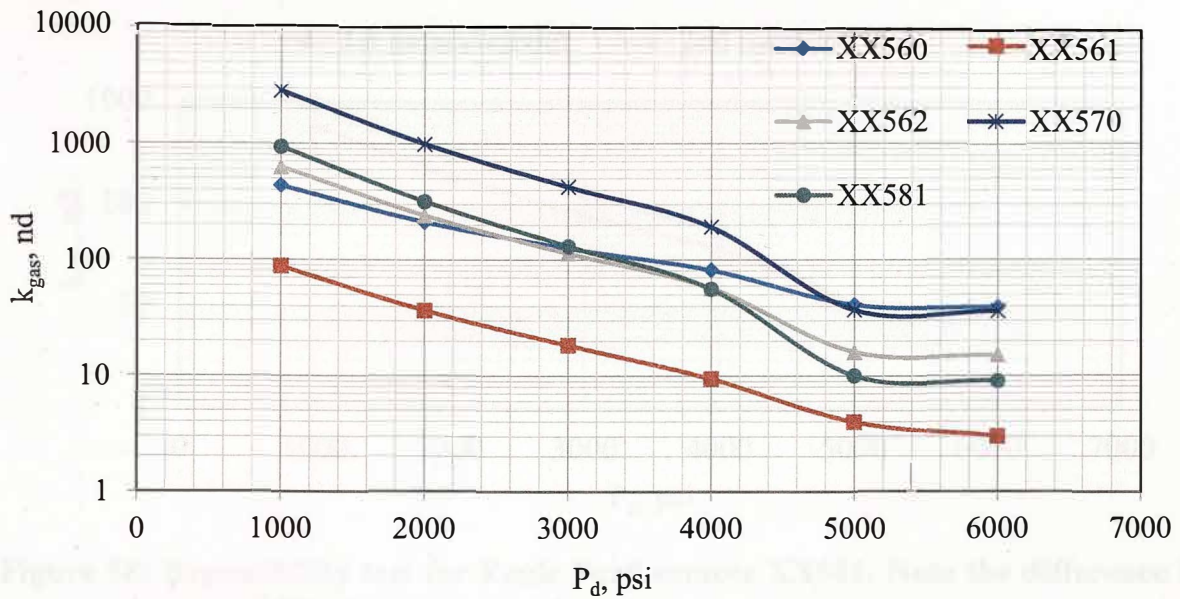


Figure 57: Permeability versus effective pressure for Eagle Ford shale samples for effective pressure range 1000 to 6000 psi. Permeability was measured with pressure build-up method with nitrogen as pore fluid.

After reaching the effective pressure value of 4000 psi a sharp drop in permeability is observed for the samples (see figure 57). The effective reservoir stress on these samples is close to 4000 psi. The sharp decrease in permeability after the effective pressure value of 4000 psi might be an indication of pore collapse and hence permanent damage to the sample.

In order to test this, permeability measurement of a sample XX581 was repeated (see figure 58). Permeability measurements were made as a function of increasing effective pressure ranging from 1000 to 6000 psi. The same test was then repeated.

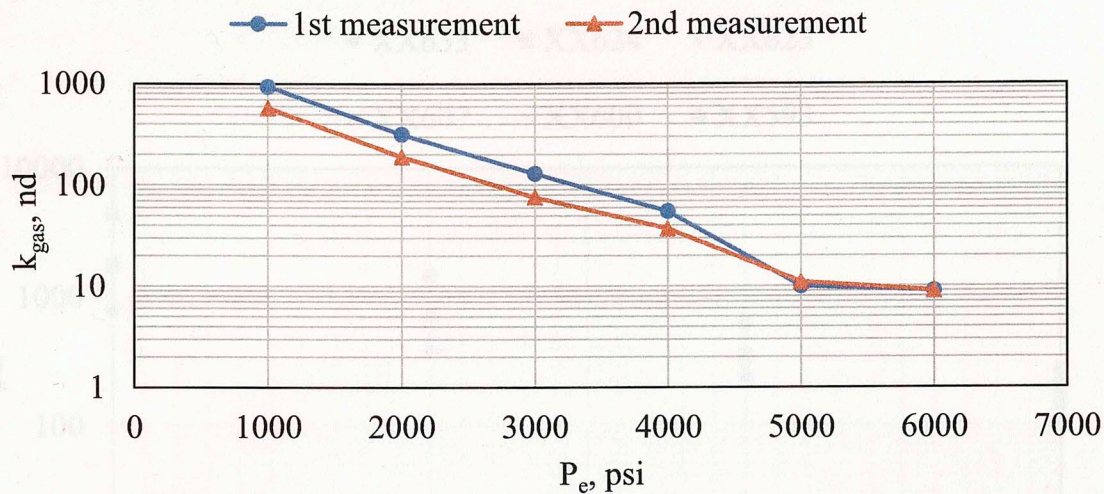


Figure 58: Repeatability test for Eagle Ford sample XX581. Note the difference in measured permeability between experiments.

On comparing the permeability values of the two measurement runs, permeability decreases for the second run within a factor of 2 of the first run at effective pressures less than 4000 psi. If there was no permanent damage to the sample, both the measurements should have produced similar results. Change in permeability values in the two tests suggest sample was damaged after the first run.

Permeabilities of 22 Eagle Ford shale samples have been measured up to 4000 psi effective pressures (see figures 59, 60, 61 and 62). Permeability is reduced by more than order of magnitude over this range.

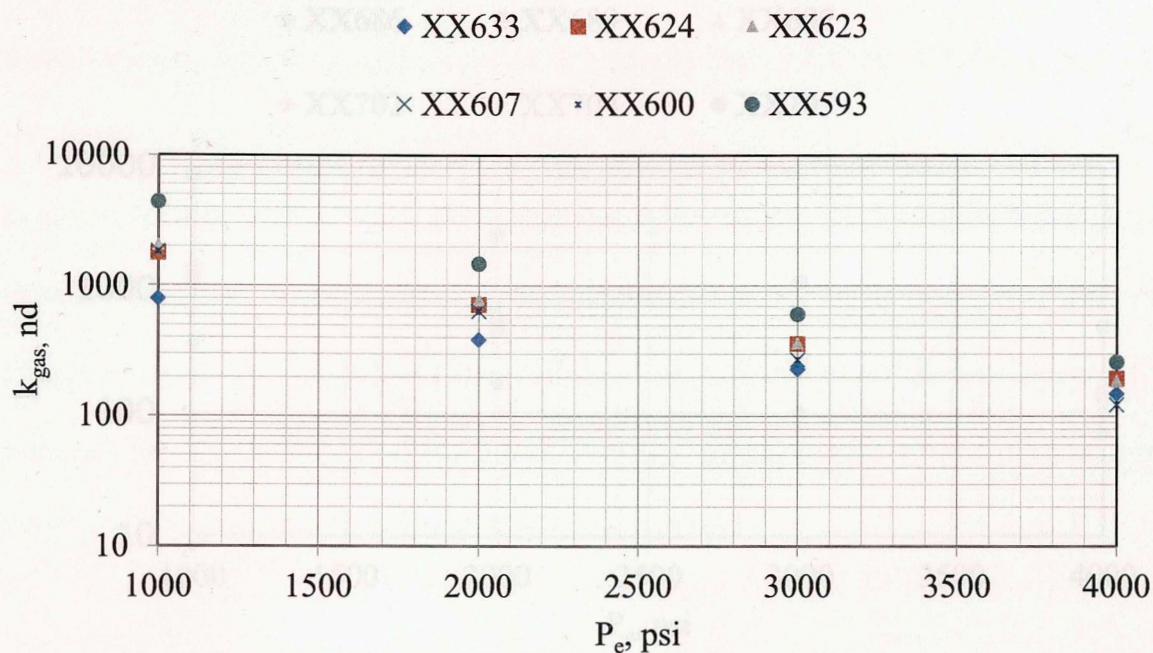


Figure 59: Permeability versus effective pressure for Eagle Ford shale samples over the effective pressure range 1000 to 4000 psi (1).

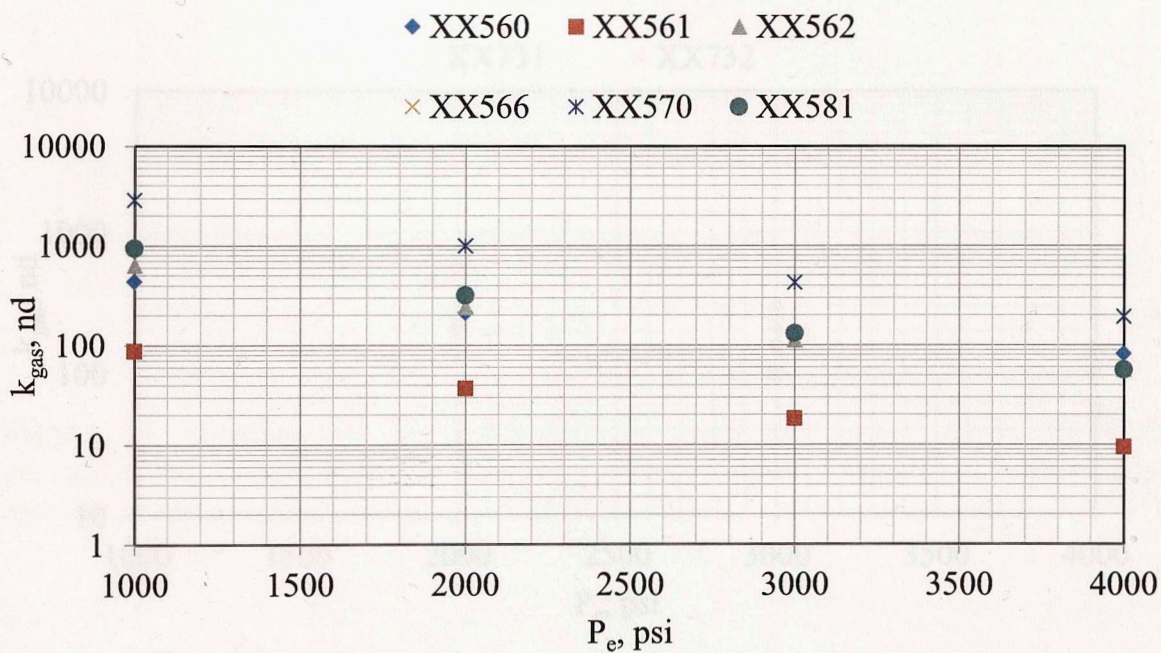


Figure 60: Permeability versus effective pressure for Eagle Ford shale samples over the effective pressure range 1000 to 4000 psi (2).

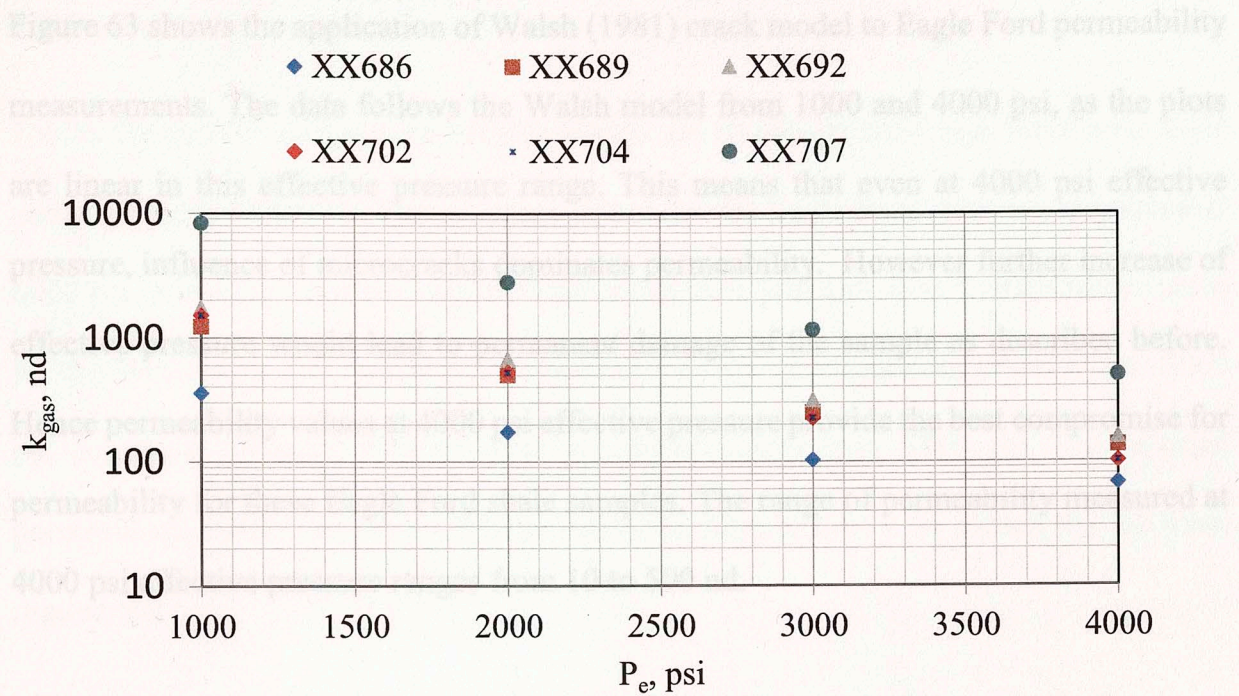


Figure 61: Permeability versus effective pressure for Eagle Ford shale samples over the effective pressure range 1000 to 4000 psi (3).

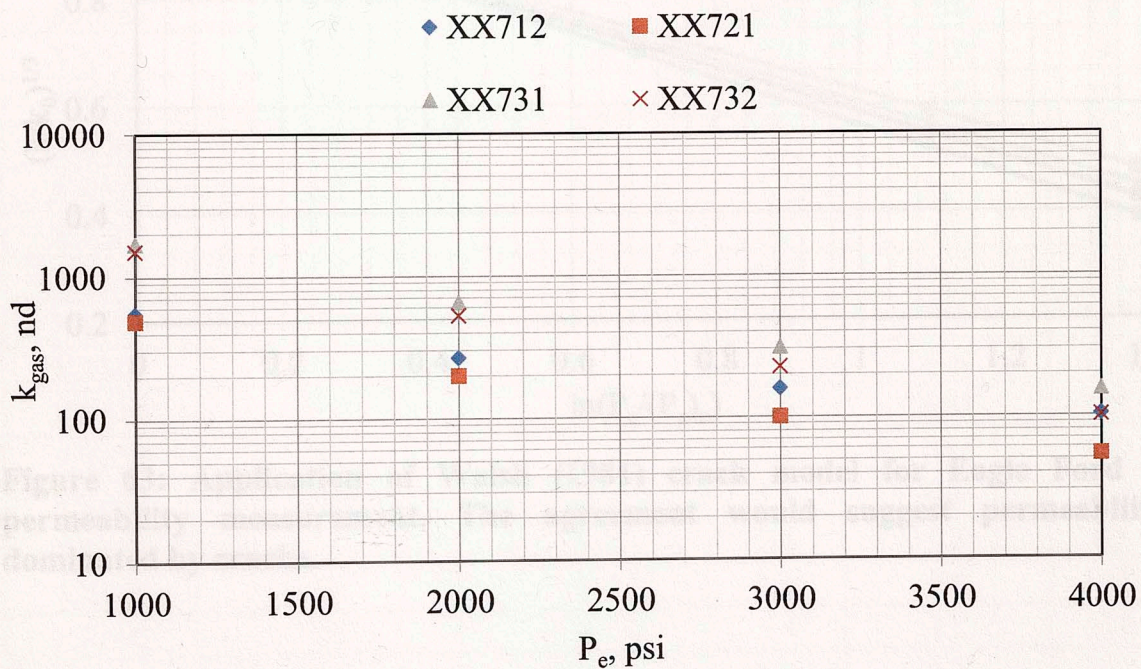


Figure 62: Permeability versus effective pressure for Eagle Ford shale samples over the effective pressure range 1000 to 4000 psi (4)

Figure 63 shows the application of Walsh (1981) crack model to Eagle Ford permeability measurements. The data follows the Walsh model from 1000 and 4000 psi, as the plots are linear in this effective pressure range. This means that even at 4000 psi effective pressure, influence of microcracks dominates permeability. However further increase of effective pressure would lead to permanent damage of the sample as described before. Hence permeability values at 4000 psi effective pressure provide the best compromise for permeability for these Eagle Ford shale samples. The range of permeability measured at 4000 psi effective pressure ranges from 10 to 500 nd.

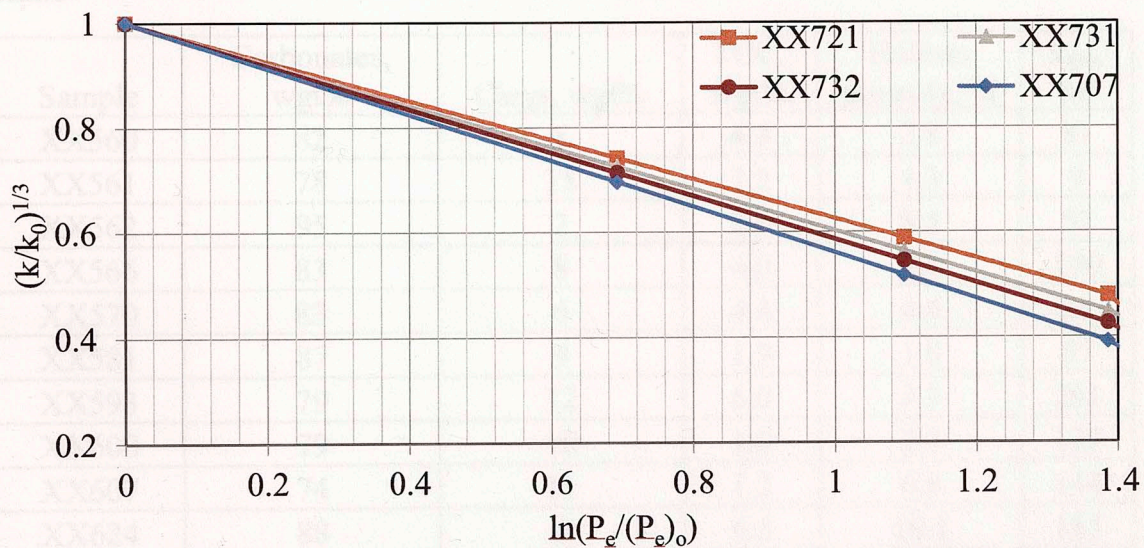


Figure 63: Application of Walsh (1981) crack model for Eagle Ford shale permeability measurement. The agreement would suggest permeability is dominated by cracks.

4.8 Permeability results

Tables 20 and 21 list the permeability results for the Eagle Ford and Wolfcamp shale samples respectively. The permeability values for the Eagle Ford shale are measured at 6000 psi confining pressure and 2000 psi mean pore pressure. The permeability values for the Wolfcamp shale samples are measured at 6000 psi confining pressure and 3000 psi confining pressure. Due to pore pressures greater than 2000 psi, the reported permeability values are very close to corrected permeabilities as shown for the flow regime test.

Table 20: Permeability and important petrophysical properties for Eagle Ford shale

Sample	Carbonates, wgt%	Clays, wgt%	TOC, wgt%	Helium porosity, %	k _{gas} , nd
XX560	82	8	4.3	2.8	81
XX561	78	15	3.7	4.3	9
XX562	95	2	3.5	8.5	57
XX566	83	8	4.0	2.8	190
XX570	83	6	4.4	4.4	38
XX581	87	9	1.9	1.8	57
XX593	79	12	6.0	8.3	261
XX600	79	10	1.0	2.4	122
XX607	74	11	1.2	6.8	144
XX624	88	5	6.5	10.1	185
XX625	85	6	5.9	10.3	190
XX633	80	8	5.8	9.6	149
XX686	71	8	2.6	5.5	62
XX689	83	8	2.6	7.0	135
XX692	80	9	5.4	9.2	159
XX702	71	16	5.8	8.3	162
XX704	81	13	2.5	7.3	101
XX707	64	27	7.3	7.8	494
XX712	76	5	1.9	3.0	102
XX721	42	36	4.4	5.7	54
XX731	58	36	4.6	5.8	155
XX732	89	8	2.6	3.3	101

Table 21: Permeability and important petrophysical properties for Wolfcamp shale

Sample	Quartz, wgt%	Carbonates, wgt%	Clays, wgt%	TOC, wgt%	Helium porosity, %	k _{gas} , nd
XX86	38	30	7	1.2	7.2	228
XX88	39	21	29	4.0	2.7	274
XX66	41	37	11	3.1	2.4	40
XX33	44	10	34	2.3	1.8	161
XX07	45	5	25	0.0	7.1	561
XX71	41	8	29	0.2	10.3	337
XX74	51	19	13	0.4	5.3	1122

The permeability for Eagle Ford shale varies from 9 to 494 nd. It does not correlate to any petrophysical property mentioned in the table. It must be noted here is that these permeabilities are affected by the influence of microcracks.

The permeability for Wolfcamp shale varies from 40 to 1122 nd. It increases with increasing quartz content but decreases with increasing TOC (see figures 64 and 65).

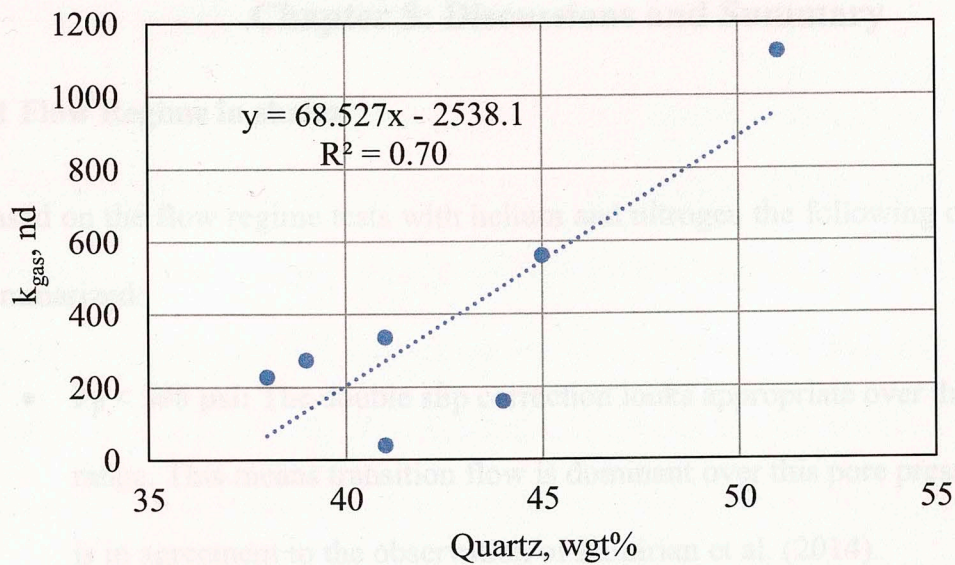


Figure 64: Permeability of Wolfcamp shale versus quartz content. Note increasing quartz content has a strong correlation with permeability.

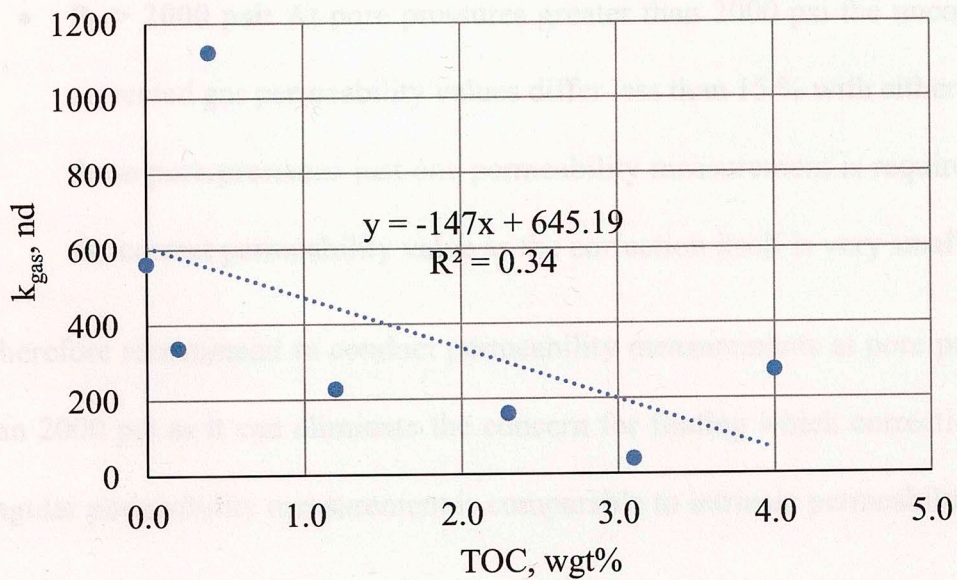


Figure 65: Permeability variation of Wolfcamp shale with TOC. Permeability decreases with increasing TOC.

Chapter 5: Discussions and Summary

5.1 Flow Regime in shales

Based on the flow regime tests with helium and nitrogen the following observations are summarized:

- **$P_p < 500$ psi:** The double slip correction looks appropriate over this pore pressure range. This means transition flow is dominant over this pore pressure range. This is in agreement to the observation of Zamirian et al. (2014).
- **$P_p = 800 - 1200$ psi:** The Klinkenberg correction appears superior to double slip correction. This indicates slip flow over this pore pressure range.
- **$P_p > 2000$ psi:** At pore pressures greater than 2000 psi the uncorrected and the corrected gas permeability values differ less than 15 % with either corrections. At these pore pressures just one permeability measurement is required to determine the correct permeability value as the correction itself is very small.

I therefore recommend to conduct permeability measurements at pore pressures greater than 2000 psi as it can eliminate the concern for finding which correction to use and a singular permeability measurement is comparable to intrinsic permeability value.

Also if the reservoir pore pressure is itself less than 500 psi and laboratory measurement of permeability are being conducted at these low pressures to reproduce reservoir conditions, then double slip correction should be applied to correct the permeability data.

5.2 Liquid vs. gas permeability

In the steady state gas and liquid permeability measurements, it was found that gas permeability can be greater than the liquid permeability by as much as a factor of 2. Wettability seems to be a potential reason for this discrepancy. NMR measurements conducted before and after permeability measurements show that dodecane does not flow through the water wet clay pores (T_2 peak at 0.25 ms) but instead travels through oil wet pores (T_2 peak at 7 ms), probably the pores in organic matter. As nitrogen does not have any NMR response, it is difficult to gauge its exact flow path; however it is shown to not change the initial water saturation in clay pores. Flow of dodecane will be hindered in oil wet pores and might be the reason for reduced permeability.

These NMR measurements also tell us about the flow and connectivity in shales. The water saturation in the sample remain unchanged at 22% after permeability measurement with nitrogen and dodecane. The NMR response of dodecane suggest that it follows a selective flow path that does not entail the entire pore spectrum but rather just the oil wet pores. Dodecane that not compete with the native water present in the sample while flowing. For this reason, this measurement cannot be termed as a relative permeability measurement. It is better to say that the permeability measurements have been conducted at an irreducible water saturation of 22 %.

The difference in liquid and gas permeability values can have a major impact on calculating the optimum number of fracture stages in oil shales. As observed above, liquid permeability values differ from gas permeability values by as much as a factor of 2; hence, using gas permeability values in the calculation for stage spacing for oil shales would

overestimate stage spacing by as much as a factor of $\sqrt{2}$ or 1.4. This will therefore result in more fracture stages. This will help in increased production from parts of the reservoir volume which could not have been drained with an initial larger fracture spacing.

5.3 Effective pressure coefficient

The value of effective pressure coefficient is found to be less than or equal to 1 for Wolfcamp and Eagle Ford Shales. A smaller value of effective pressure coefficient means less sensitivity of effective pressure to pore pressure. Hence as pore pressure reduces during production the reduction in permeability will be small and linearly related to pore pressure.

5.4 Permeability creep

In all the shales measured i.e. Wolfcamp, Eagle Ford and Vaca Muerta shale, little to no change in permeabilities are observed over time. This has a positive implication on the permeability measurement time for shales. If creep was observed, sufficient time should be allowed for the permeability values to stabilize after any pressure changes.

When laboratory measurements on a new shale formation are being conducted, I suggest to make creep measurements on at least some samples for a period of 30 days as a function of effective pressure. This is to check for permeability creep in the new shale and how much effect it will have on time for the measurements.

5.5 Permeability versus effective pressure and Recommendations

6.1 Conclusions

While making permeability measurements as a function of effective stress, repeatability of the measurement should be checked to ensure no damage has been done to the sample. It is better to know the effective reservoir stress and not to go beyond it during measurements. Permeability measurements for some samples can be conducted as a function of effective stress going up to the effective reservoir stress and then repeated to find out whether the sample has undergone any deformation.

b. $P_p = 1000$ psi: Slip flow is prevalent and Klinkenberg correction should be used to correct gas permeabilities.

c. $P_p \geq 2000$ psi: The correction to permeability values are negligible. A single permeability measurement can substitute for corrected gas permeability.

2. Permeability measurements made with transient techniques such as pressure pulse decay and pressure build up provide permeability values consistent with the steady state technique within 30% with no particular trend regarding which one is greater.
3. Comparison of gas and liquid permeability measurements on Wolfcamp shale shows corrected gas permeability values are greater than liquid permeability values by a factor of 2.
4. Shale permeability experiments generally measure relative permeability but the saturations are unknown. NMR measurements on Wolfcamp shale show that (1) the water saturation is 22% and (2) it did not change after permeability measurements with gas or liquid.

Chapter 6: Conclusions and Recommendations

6.1 Conclusions

The main conclusions of this study are:

1. Flow regime in shales was characterized for gas permeability measurements over different pore pressure ranges
 - a. $P_p \leq 250$ psi: Transition flow is prevalent and second order corrections like double slip are required.
 - b. $P_p \approx 1000$ psi: Slip flow is prevalent and Klinkenberg correction should be used to correct gas permeabilities.
 - c. $P_p \geq 2000$ psi: The correction in permeability values are negligible. A single permeability measurement can substitute for corrected gas permeability.
2. Permeability measurements made with transient techniques such as pressure pulse decay and pressure build up provide permeability values consistent with the steady state technique to within 30% with no particular trend regarding which one is greater.
3. Comparison of gas and liquid permeability measurements on Wolfcamp shale shows corrected gas permeability values are greater than liquid permeability values by a factor of 2.
4. Shale permeability experiments generally measure relative permeability but the saturations are unknown. NMR measurements on Wolfcamp shale show that (1) the water saturation is 22% and (2) it did not change after permeability measurements with gas or liquid.

5. Permeability creep studies conducted on Wolfcamp, Eagle Ford and Vaca Muerta shales for over a month show negligible change in permeability. This is in contrast to creep measurement reported by Chhatre et al. (2014).
6. The values of effective pressure coefficients were found to be less than or equal to one for Eagle Ford and Wolfcamp shales. This has a positive impact on production as permeability is less sensitive to changes in decreasing pore pressure.
7. Permeability of Eagle Ford shale changes by over an order of magnitude as effective pressure varies from 1000 to 4000 psi. Permeability values follow the Walsh (1981) fracture model in this range of effective pressure which indicates the influence of microcracks. The range of permeability measured for Eagle Ford shale at 4000 psi effective pressure is 10 to 500 nd.

6.2 Recommendations

These are some of the recommendations for further study on this topic.

1. More experiments must be conducted to compare liquid and gas permeability values. For some samples the difference is found to be a factor of 2 while for others there is a smaller difference in gas and liquid permeability values. Hence more measurements are needed to understand the reason for the difference in liquid and gas permeability measurements.

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Appendix-A

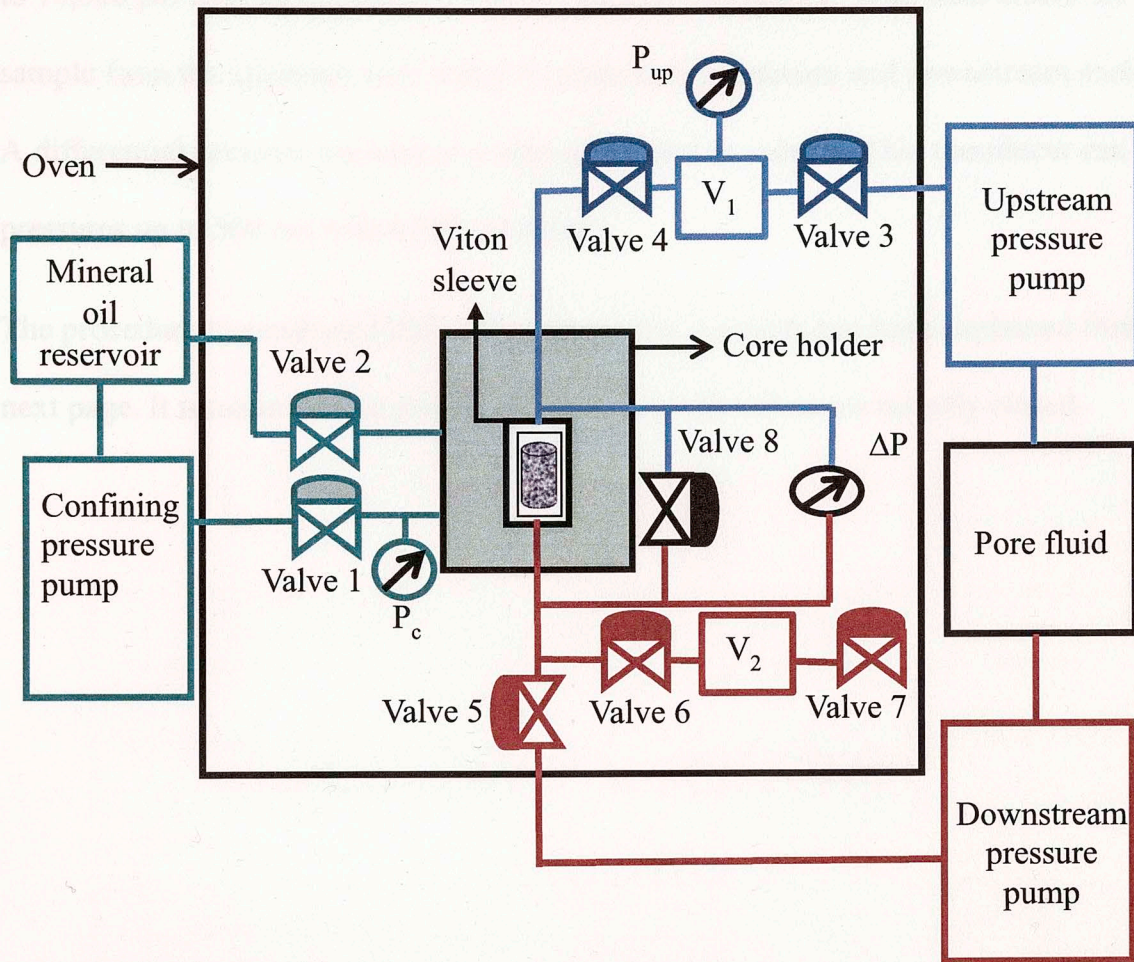


Figure 66: Experimental apparatus for permeability measurement. The core holder is placed in an oven for thermal insulation. The confining pressure pump develops overburden stress and the upstream and downstream pumps develop pore pressures on the sample. Pressures are monitored using pressure gauge in the upstream and a differential pressure gauge.

The sequence of developing pore pressure in the system starts from the downstream pressure pump followed by the upstream pressure pump. In the downstream section the pore fluid can flow from the downstream pressure pump to the core sample if valve 5 is opened. If valve 6 is opened pore fluid enters into a system volume V_2 . This is also connected to a discharge valve (valve 7) which is open to atmosphere. In the upstream section, the pore fluid flows from the upstream pressure pump to a system volume, V_1 ,

when valve 3 is opened. A pressure transducer is connected to V_1 which can read pressures to 10,000 psi with an accuracy of 0.02%. On opening valve 4, pore fluid enters the core sample from the upstream end. Valve 8 connects the upstream and downstream sections. A differential pressure transducer is also connected to valve 8. This transducer can read pressures up to 500 psi with 0.25% accuracy.

The procedure to conducted different permeability methods has been explained from the next page. It is assumed that prior to measurement all valves are initially closed.

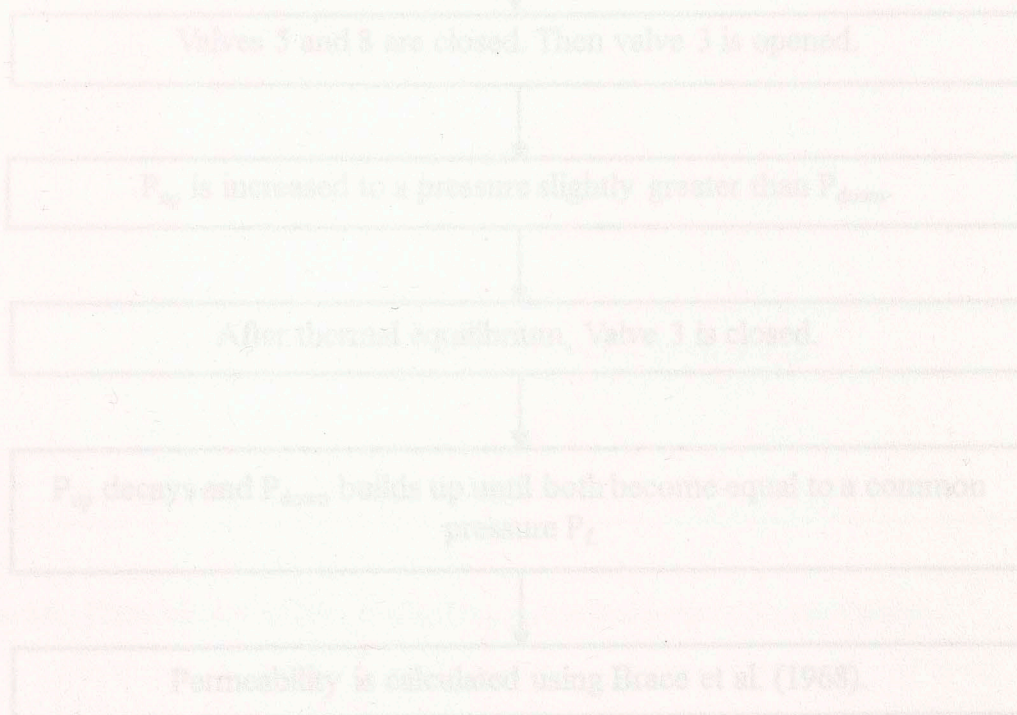


Figure 67: Flow diagram for conducting pressure pulse decay experiment.

- **Pressure pulse decay operational sequence**

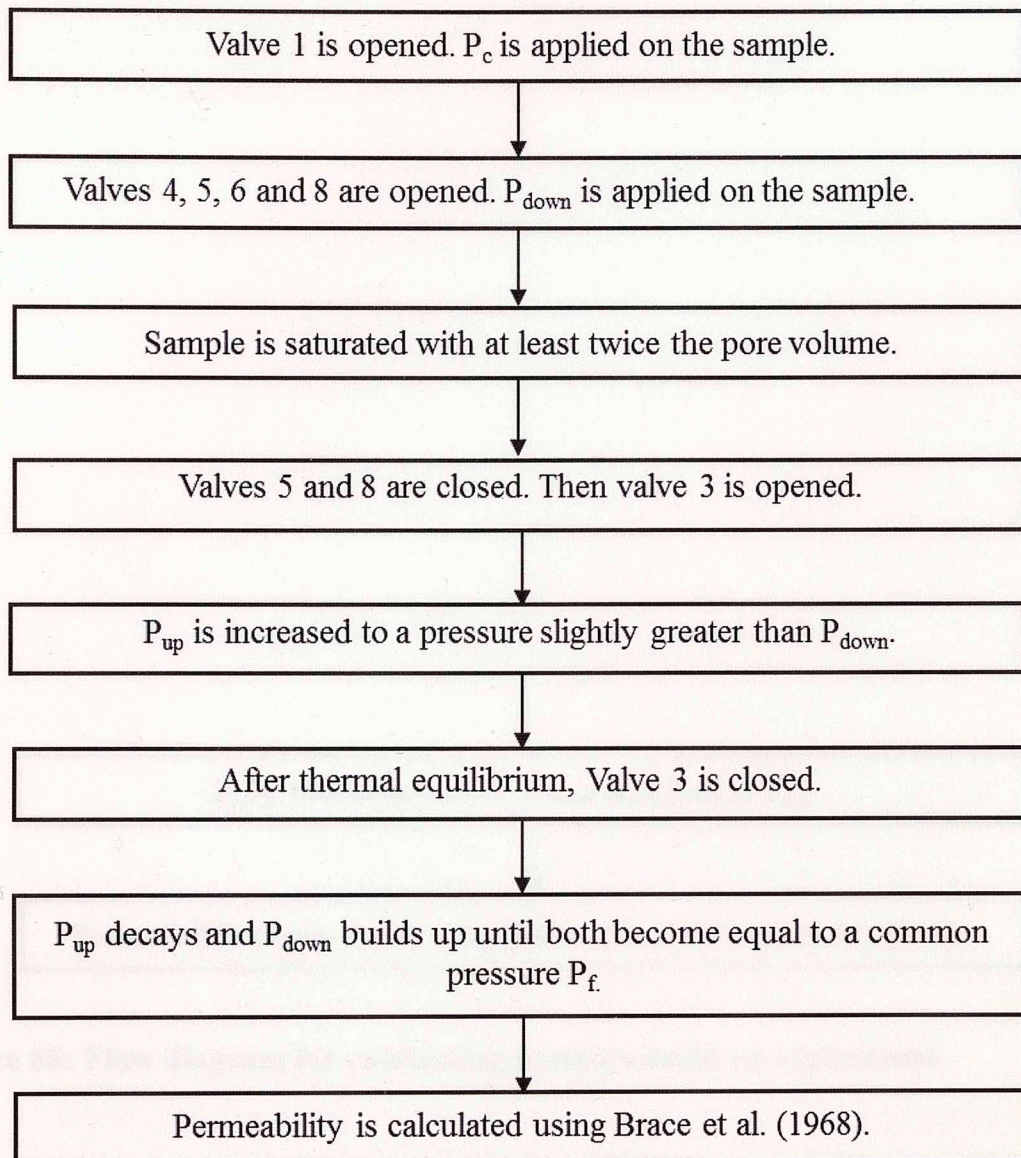


Figure 67: Flow diagram for conducting pressure pulse decay experiment.

Sample is saturated with at least twice the P_{down}

Valves 5 and 8 are closed. Then valve 3 is opened.

P_{up} is increased to a pressure slightly greater than P_{down} .

P_{down} builds up until it becomes equal to P_{up} .

Permeability is calculated using Metwally and Sondergeld (2011)

- **Steady state permeability operational sequence**

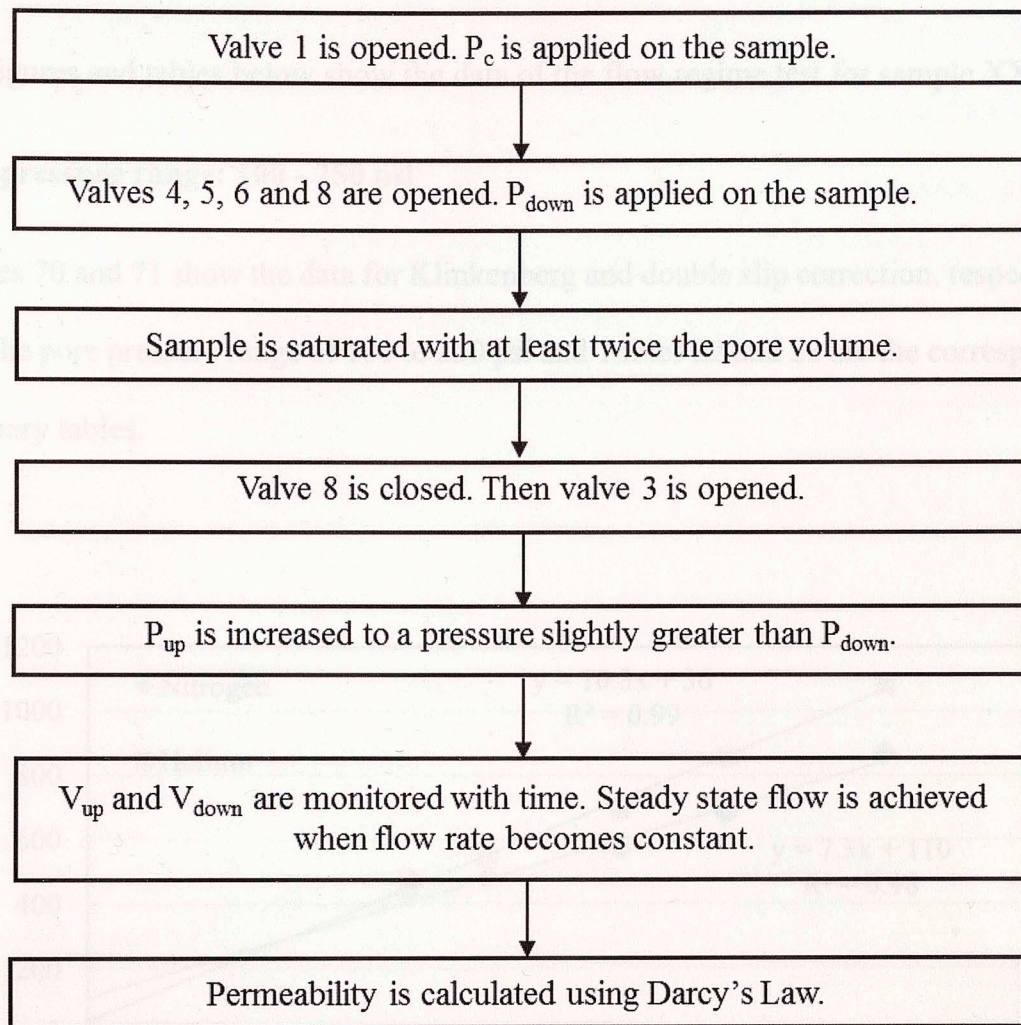


Figure 69: Flow diagram for conducting steady state experiment.

Appendix- B

The figures and tables below show the data of the flow regime test for sample XX86.

Pore pressure range: 100 - 250 psi

Figures 70 and 71 show the data for Klinkenberg and double slip correction, respectively, over the pore pressure range of 100 to 250 psi and Tables 22 and 23 are the corresponding summary tables.

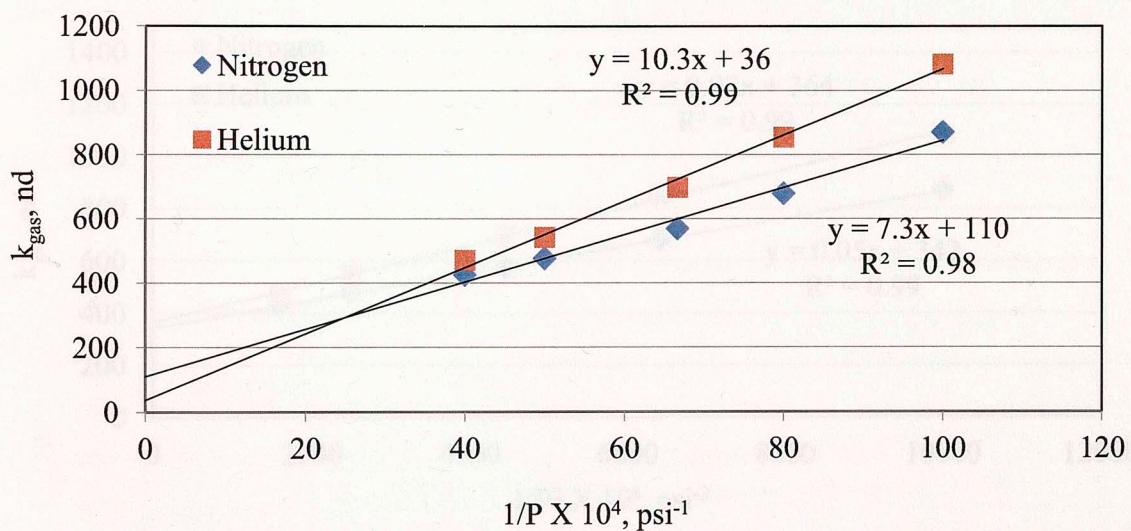


Figure 70: Klinkenberg correction for Wolfcamp sample XX86 (pore pressure range: 100 to 250 psi).

Table 22: Data for Klinkenberg corrected permeability for Wolfcamp sample XX80 (pore pressure range: 100 to 250 psi).

Gas	Corrected permeability (Intercept)	Slope	R ²	Corrected permeability ratio (k _N / k _{He})
Nitrogen	110 ± 22	7.3 ± 0.6	0.99	3.05
Helium	36 ± 12	10.3 ± 0.5	0.99	

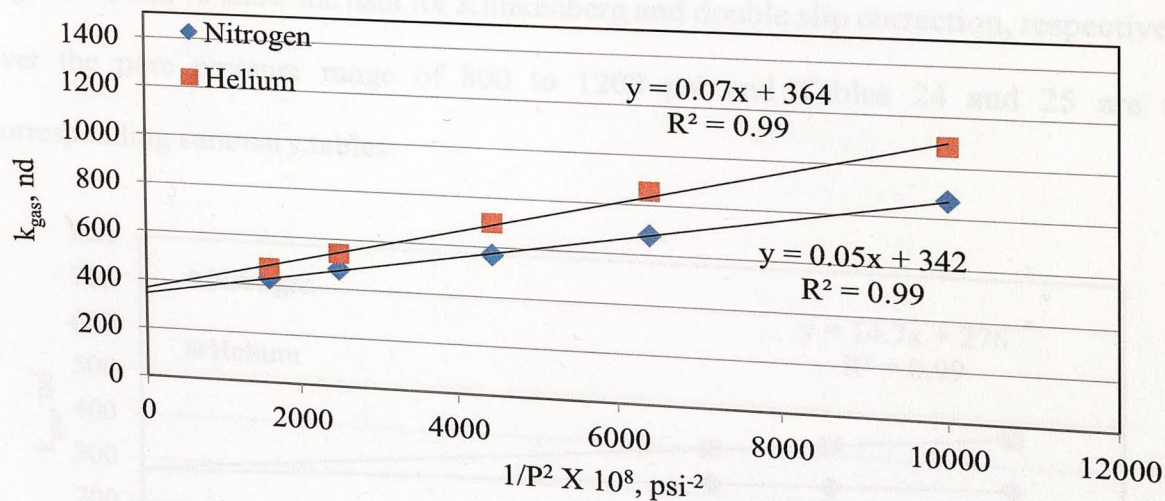


Figure 71: Double slip correction for Wolfcamp sample XX80 (pore pressure range: 100 to 250 psi).

Table 23: Data for double slip corrected permeability for XX86 (pore pressure range: 100 to 250 psi).

Gas	Corrected permeability (Intercept)	Slope	R ²	Corrected permeability ratio (k _N / k _{He})
Nitrogen	342 ± 4	0.05 ± 0.0006	0.99	0.94
Helium	364 ± 15	0.07 ± 0.0025	0.99	

Pore pressure range: 800 - 1200 psi

Figures 72 and 73 show the data for Klinkenberg and double slip correction, respectively, over the pore pressure range of 800 to 1200 psi and Tables 24 and 25 are the corresponding summary tables.

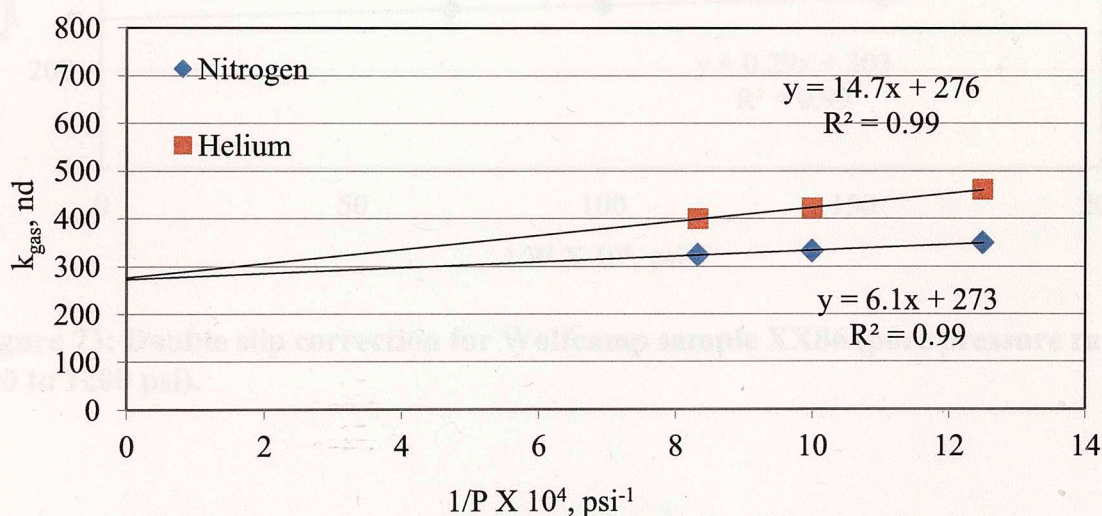


Figure 72: Klinkenberg correction for Wolfcamp sample XX86 (pore pressure range: 800 to 1200 psi).

Table 24: Data for Klinkenberg corrected permeability for Wolfcamp sample XX86 (pore pressure range: 800 to 1200 psi).

Gas	Corrected permeability (Intercept)	Slope	R ²	Corrected permeability ratio (k _N / k _{He})
Nitrogen	273 ± 6	6.1 ± 0.55	0.99	0.99
Helium	276 ± 7	14.7 ± 0.65	0.99	

Pore pressure range: 1800 - 2200 psi

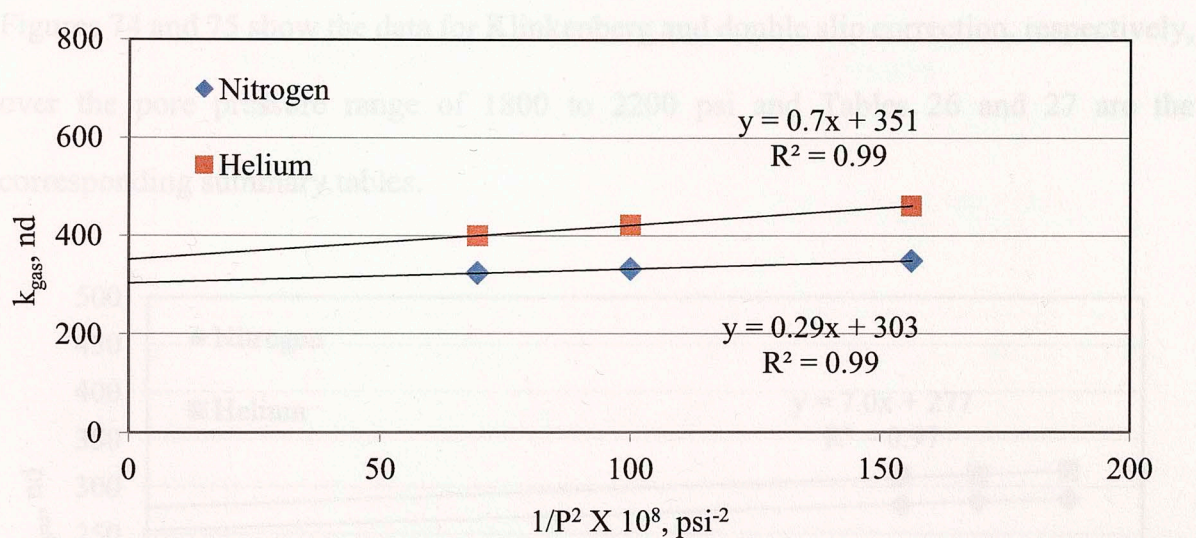


Figure 73: Double slip correction for Wolfcamp sample XX86 (pore pressure range: 800 to 1200 psi).

Table 11: Data for double slip corrected permeability for Wolfcamp sample XX86. (pore pressure range: 800 to 1200 psi).

Gas	Corrected permeability (Intercept)	Slope	R ²	Corrected permeability ratio (k _N / k _{He})
Nitrogen	303 ± 1	.29 ± .01	0.99	0.86
Helium	351 ± 1	0.7 ± .007	0.99	

Pore pressure range: 1800 - 2200 psi

Figures 74 and 75 show the data for Klinkenberg and double slip correction, respectively, over the pore pressure range of 1800 to 2200 psi and Tables 26 and 27 are the corresponding summary tables.

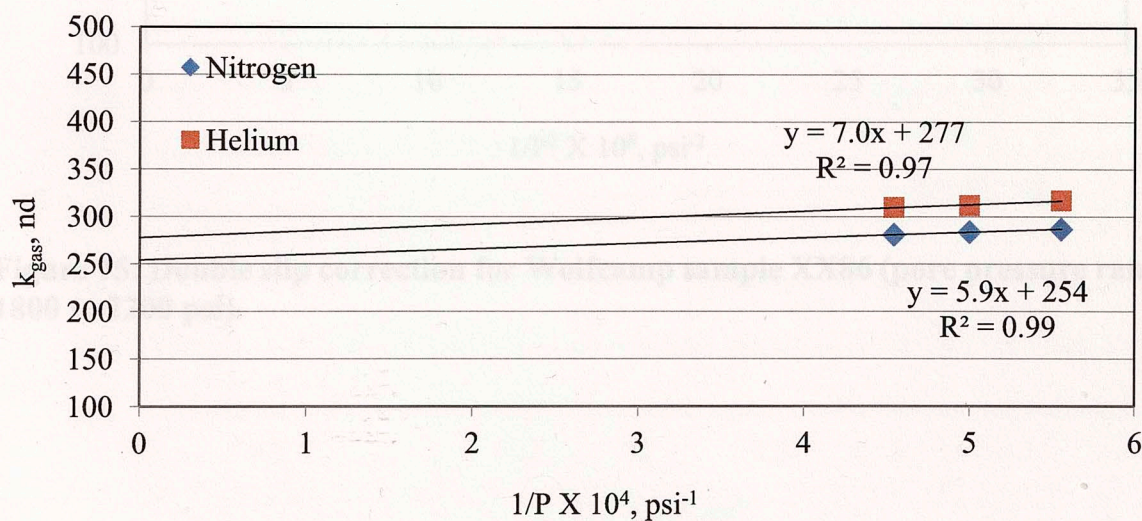


Figure 74: Klinkenberg correction for Wolfcamp sample XX86 (pore pressure range: 1800 to 2200 psi).

Table 26: Data for Klinkenberg corrected permeability for Wolfcamp sample XX86 (pore pressure range: 1800 to 2200 psi).

Gas	Corrected permeability (Intercept)	Slope	R ²	Corrected permeability ratio (k _N / k _{He})
Nitrogen	254 ± 2	5.9 ± 0.3	0.99	0.92
Helium	277 ± 7	7 ± 1.3	0.97	

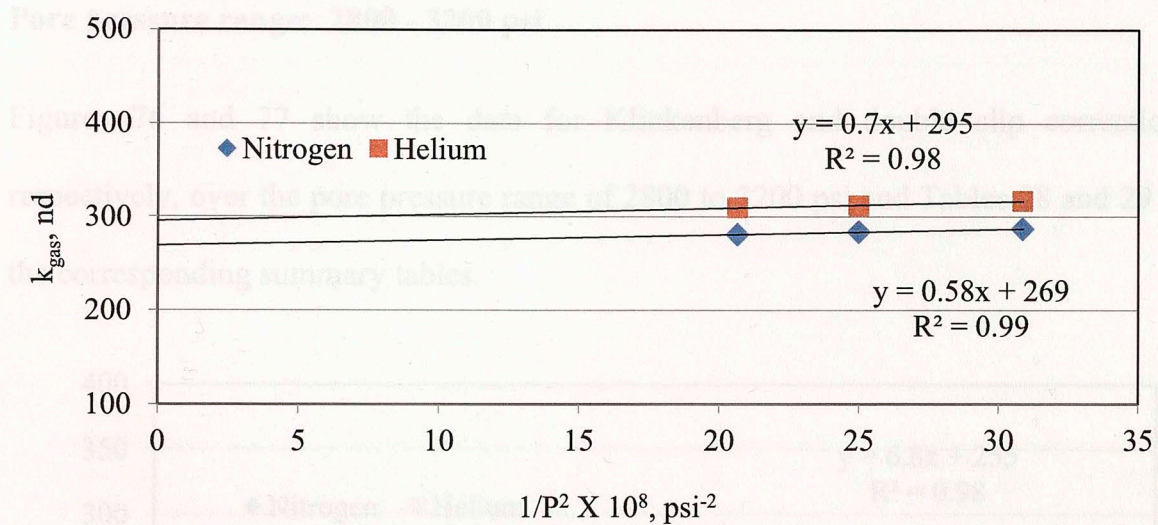


Figure 75: Double slip correction for Wolfcamp sample XX86 (pore pressure range: 1800 to 2200 psi).

Table 27: Data for double slip corrected permeability for Wolfcamp sample XX86 (pore pressure range: 1800 to 2200 psi).

Gas	Corrected permeability (Intercept)	Slope	R ²	Corrected permeability ratio (k _N / k _{He})
Nitrogen	269 ± 1	0.58 ± 0.05	0.99	0.91
Helium	295 ± 3	0.7 ± 0.1	0.98	

Pore pressure range: 2800 - 3200 psi

Figures 76 and 77 show the data for Klinkenberg and double slip corrections, respectively, over the pore pressure range of 2800 to 3200 psi and Tables 28 and 29 are the corresponding summary tables.

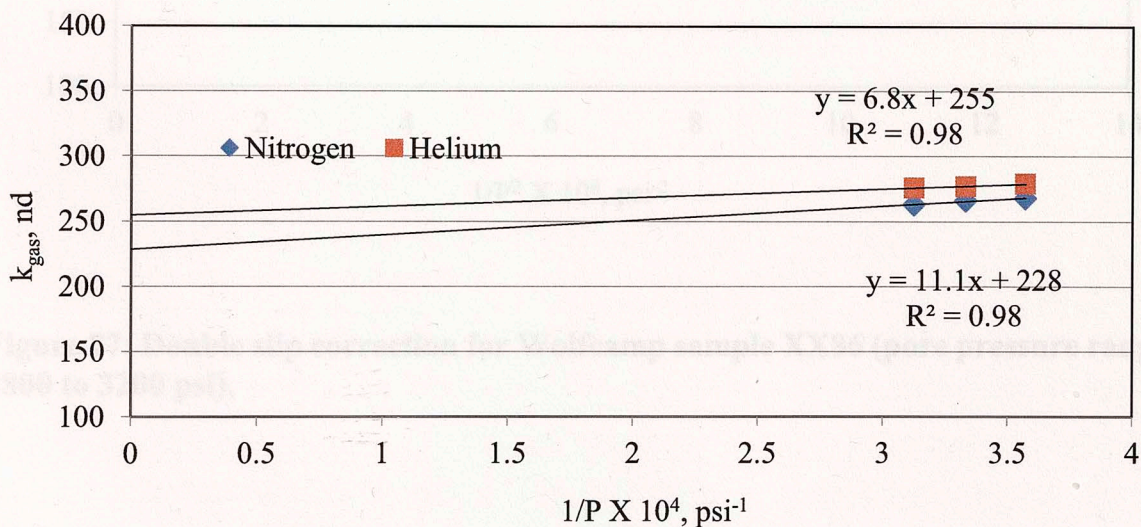


Figure 76: Klinkenberg correction for Wolfcamp sample XX86. (Pore pressure range: 2800 to 3200 psi).

Table 28: Data for Klinkenberg corrected permeability for Wolfcamp sample XX86 (pore pressure range: 2800 to 3200 psi).

Gas	Corrected permeability (Intercept)	Slope	R ²	Corrected permeability ratio (k _N / k _{He})
Nitrogen	228 ± 6	11.1 ± 1.7	0.98	0.9
Helium	255 ± 4	6.8 ± 1	0.98	

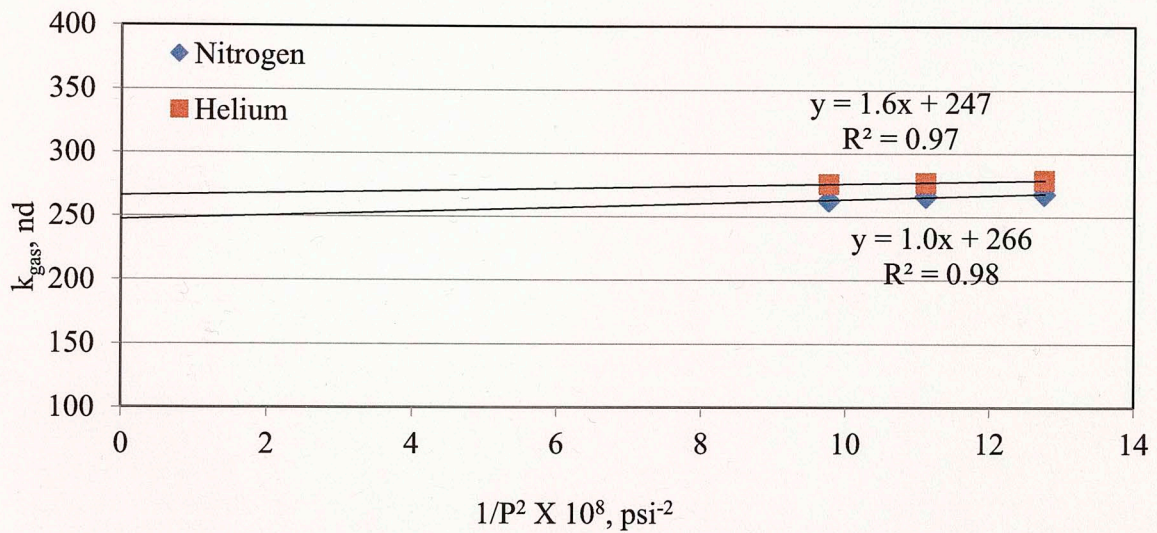


Figure 77: Double slip correction for Wolfcamp sample XX86 (pore pressure range: 2800 to 3200 psi).

Table 29: Data for double slip corrected permeability for Wolfcamp sample XX86 (pore pressure range: 2800 to 3200 psi).

Gas	Corrected permeability (Intercept)	Slope	R ²	Corrected permeability ratio (k _N / k _{He})
Nitrogen	247 ± 3	1.6 ± 0.3	0.98	0.93
Helium	266 ± 2	1 ± .13	0.97	

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