Experimental Methods of Shale Permeability

by

Feng Guo
BEng

THE UNIVERSITY OF WESTERN AUSTRALIA

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Abstract

This thesis explores the permeability of shale caprock (Green River Shale). Dynamic coupling between fluid transport and mechanical response have been observed in this unconstrained sorbing and swelling shale. We report laboratory experiments that investigate the permeability evolution of shale sample as a function of applied stress and pore pressure at room temperature and gas transport between fracture and matrix. Experiments were conducted on 2.5 cm diameter by 1.2cm long cylindrical samples at confining and axial stresses of 7 MPa. Permeability and sorption characteristics were measured using a pulse transient method that was also used to measure volumetric strains for both helium (He) and carbon dioxide (CO₂). From these experiments it was found that gas adsorption is closely linked with the apparent permeability of the sample. As pore pressure increased, the permeability to He increased from 6.01×10⁻¹⁹ m² to 9.74×10⁻¹⁹ m² while the permeability to CO₂ which is a larger molecule with higher absorbability increased from 3.03×10⁻¹⁹ to 4.32×10⁻¹⁹ m². Gas transport between fracture and matrix also played a significant role in permeability evolution. During gas transport, gas diffused gradually from the fractures to the matrix and the area affected by gas transport expanded dynamically from the fractures towards to the matrix. This suggests permeability
evolution is dynamic during gas transport and can be divided into two stages. In the first stage, the gas-influenced area is limited to fracture surfaces, which causes the fracture apertures to decrease, thereby reducing permeability. In the second stage, the gas-influenced area penetrates further into the matrix which causes the fracture apertures to increase, resulting in permeability recovery. This hypothesis is supported by our experimental observations. Between two stages of the gas transport process, where local swelling dominated, the permeability of both He and CO₂ was lower than at the final equilibrium state. At the final equilibrium state, where uniform macro swelling occurred, permeability of both He and CO₂ increased as pore pressure increased. Our experiments reveal that not only adsorption-induced swelling and effective stress but also gas transport has an important impact on permeability of shale caprock.
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Chapter 1

Introduction

Global development has led to increased demand for affordable clean energy. Energy consumption in both the developed and the developing world is increasing, and is particularly high in developing countries. Electricity is produced primarily from non-renewable sources such as fossil fuels and nuclear power but also from renewable sources that include hydropower, wind, geothermal, solar and biomass. Globally, coal provides for 29.6% of primary energy needs and generates 42% of the world’s electricity. The low cost of coal relative to other fuel sources means it is a critical resource for developing economies. As a result, massive amounts of carbon dioxide are being discharged into the atmosphere as coal is consumed in these countries. To reduce emission of carbon dioxide, technologies need to be developed to efficiently capture and store carbon dioxide from the atmosphere.

Carbon sequestration is a carbon capture and storage technology that reduces anthropogenic carbon dioxide emission by storing carbon dioxide deep under the earth’s surface or deep in the ocean. The main source of carbon dioxide for sequestration is from power plants. The common places
for sequestration are saline aquifers, coal seams and depleted oil and gas reservoirs.

For the effective and long-term storage of carbon dioxide in saline aquifers, it is necessary that the storage basin is large, isolated and the reservoir caprock has good sealing capacity. Carbon dioxide can be sequestrated in these aquifers in four ways: (1) solubility trapping where the carbon dioxide dissolves in the formation water, (2) mineral trapping where geochemical reactions trap the carbon dioxide within the aquifer fluids and rocks, (3) structural trapping which occurs when carbon dioxide rises to the top of geological structures with an impermeable top seal and is stored there due to capillary pressure, (4) hydrodynamic trapping which occurs when the aquifer does not allow the carbon dioxide plume to seep out of the targeted reservoir zone, and (5) retention of CO2 in pore space (Bachu S, 2003).

After carbon dioxide is injected into sequestration formations, there is a possibility it could flow back throughout the caprocks and migrate back to atmosphere. This migration through caprocks back to the atmosphere can occur as a result of four different pathways: (1) molecular diffusion of dissolved carbon dioxide in the pore water from the reservoir zone into the caprock formation, (2) CO2 diphasic flow after capillary breakthrough, (3)
CO₂ flow through the caprock after hydraulic fracturing and (4) CO₂ flow through existing open fractures, faults or wells (Chadwick et al., 2008).

Leakage due to CO₂ diffusion in saline occurs over geological time scales, and therefore diffusion-induced leakage through caprock is unlikely to significantly impact the safety of the CO₂ reservoir (Lu et al., 2009). Flow of CO₂ through a newly created pathway or existing open fractures or faults is most dangerous, but only happens when accumulated pressure beneath the caprock is high enough to overcome the minimum compressive stress surrounding the caprock. Usually the caprocks of sequestration sites are deep enough that this leakage is easy to avoid.

In contrast, leakage due to capillary breakthrough is a major threat to the efficiency of caprock sealing. This leakage occurs when the pressure difference of CO₂ and the water phase in the pores adjacent to the caprock is higher than the capillary entry pressure of the caprock. This leakage occurs more frequently than leakage due to a newly created pathway or existing open fracture because the capillary entry pressure is often much smaller than the minimum compressive stress. In addition, capillary entry pressure is controlled by the interfacial tension of the fluids, the wettability of the solid surface (wetting angle) with respect to the fluid, and the structure of the pore system. The migration of CO₂ may also be accelerated
by other mechanisms such as chemical alteration of the mineral composition of the caprock formation under the influence of acid water, re-opening of pre-existing fracture or micro-cracks induced by overpressure of the reservoir below, or a combination of the two (Fleury et al, 2010).

Once carbon dioxide penetrates into a water-saturated caprock, the CO₂-saline displacement process in the caprock layer can be described by diphasic flow. Permeability is an important property of caprocks as it influences the CO₂-saline displacement process and has a direct impact on the sealing capacity of caprock. Although the initial permeability of shale caprock is usually lower than 1 μD, it changes when CO₂ penetrates into the caprocks (Gherardi et al., 2007; Wang and Peng, 2014). To accurately predict the permeability evolution of shale caprocks, it is important to assess the sealing effectiveness of the caprock. As previous studies of shale caprock permeability have shown that shale and coal have a similar dual pore (Smith et al., 2013; Deng et al., 2013) and organic matter composition (Weniger et al., 2010; Kang et al., 2011), the investigation of shale-gas interactions can be based on extensive coal-gas studies. The permeability of coal is mainly determined by pore size affected by shrinkage and swelling of caprock, the effective stress of caprock (which involves pore pressure, axial pressure and confining pressure) and gas transport.
Experimental and modelling studies have shown that rock shrinkage and swelling with gas desorption/adsorption has an important influence on the evolution of permeability (Bustin et al., 2008; Cui et al., 2007; Harpalani and Chen, 1995; Kelemen et al., 2006; Seidle and Huitt, 1995). Laboratory measured coal permeabilities to non-adsorbing or lightly adsorbing gases such as He and N₂ have been found to be higher than those permeabilities to adsorbing gases such as CH₄ and CO₂ (Chen et al., 2011; Sirwardane et al., 2009; Somerton et al., 1975). With constant axial and confining pressure, the permeability of these adsorptive gases drops when pore pressures increase due to coal swelling (Mazumder and Wolf, 2008; Pan et al., 2010; Robertson and Christiansen, 2005; Wang et al., 2010), and rise when pore pressures decrease due to matrix shrinkage (Cui and Bustin, 2005; Harpalani and Chen, 1997; Harpalani and Schraufnagel, 1990; Seidle and Huitt, 1995). Swelling of the coal matrix has also been found to be caused by the preferential sorption of CO₂ over CH₄ by reducing or closing the aperture of existing cleats to reduce net permeability during CO₂ injection in constrained coals (Chikatamarla et al., 2004b; Gu and Chalaturnyk, 2005; Levine, 1996).
A variety of coal permeability models have also been used to access the influence of rock shrinkage and swelling on gas desorption/adsorption (Cui and Bustin, 2005; Pan and Connell, 2007; Robertson and Christiansen, 2006; Seidle and Huit, 1995). The model P&M model derived by Palmer and Mansoori (1996) has been widely used to study permeability in coal as a function of effective stress and matrix shrinkage. This model was improved by Palmer et al. in 2007. Liu et al. (2011) have also used the cubic coal model to examine how permeability is affected by swelling/shrinkage in two extreme cases: (1) where the matrix blocks and fracture in a coal sample are assumed to be completely separated from each other, and permeability is not affected by matrix swelling/shrinkage, and (2) where the coal sample is assumed to be completely constrained and permeability is not affected by the swelling/shrinkage strain of the sample. A number of other models have also shown that gas adsorption and related swelling is largely impacted by coal rank, sorption capacity and the composition of the permeating gas and water saturation (Chikatamarla et al., 2004a, b; Han et al., 2010; Levine, 1996; Pone et al., 2010).

Many experiments have also been undertaken to evaluate the influence of stresses on permeability evolution. These experiments have shown that gas permeability in coal is sensitive to geo-stress because the fissures and pores in the coal body tend to close under external compressive forces (Somerton et al., 1975). As applied stress increases along a gentle gradient,
permeability decreases along a steep gradient (Mckee et al., 1987; Seidle et al., 1992). In the stress range of 250-2000 psi, permeability decreases by more than two orders of magnitude in response to hydrostatic or triaxial stress, although this decrease can be controlled by the mean effective stress (Somerton et al., 1975; Liu et al, 2011). A number of studies using non-adsorbing gas have also shown that permeability increases with decreasing effective stress on the sample (Pini et al., 2009; Pan et al., 2010). Zeng et al. (2011) reported that the permeability of coal samples under triaxial stress tend to decrease as effective stress in each loading direction increases and is controlled by the evolution of cracks in the samples.

Another factor impacting on permeability evolution is gas transport between the matrix and fractures. Coal is a typical dual porous medium. If a state of equilibrium does not exist between the fracture and the matrix, a local mechanical response determines permeability alternation rather than a macro mechanical response. Liu et al. (2011) believes that these permeability changes are related to the process of gas transport and proposed a permeability switching model that describes how permeability can switch instantaneously from reduction to enhancement. This model also described how coal matrix swelling can transit from local swelling to macro-swelling under the influence of unconstrained swelling.
Migration of CO₂ in sedimentary formations can be also be affected by mineral composition (e.g. Kaszuba et al., 2003, 2005; Kharaka et al., 2006; Hangx and Spiers, 2008; Wollenweber et al., 2010; Alemu et al., 2011). Aqueous chemistry and dissolution of minerals and mineralogical changes may occur between shale and CO₂-saline, largely as a result of the formation of bicarbonate and a decrease in pH. Experimental studies have shown that carbonate-rich shale is more reactive than clay-rich shale at experimental temperatures of 250°C (Alemu et al., 2011), suggesting that the reactivity of caprock is highly governed by its mineral composition. Changes in permeability and porosity are limited as a result of interactions between CO₂, saline and caprock (e.g. Wollenweber et al., 2010; Alemu et al., 2011).

While shale has a similar microstructure and mineral composition to coal, this may not result in the same response to stress, adsorption and gas transport. Therefore, further investigation of the permeability of shale is required. In this thesis, laboratory measurements were performed on a shale sample from Green River Formation in USA, using He and CO₂ at room temperature. The effects of gas adsorption, gas permeability under tri-axial conditions (with respect to the pore and confining pressures) and the
geomechanical properties of the shale were measured. Gas transport at room temperature was also analysed.
Chapter 2

Experimental Investigation

2.1 Apparatus

Experiments were completed using a simple tri-axial apparatus capable of applying defined effective stress paths and concurrently measuring permeability and adsorbing capacity as shown in Figure 1-1. A tri-axial core holder (Temco) was used to accept membrane-sheathed cylindrical samples (2.5cm diameter and 5 cm long) and apply independent loading in the radial and axial directions. This holder can apply a maximum to $35 \pm 0.007$ MPa of confining and axial stresses by dual cylinder syringe pumps (ISCO 500D). Constant upstream pressure can be applied by a third syringe pump (ISCO 500D) with the downstream reservoir open to the atmosphere to measure both water and gas permeabilities to $10^{-23}$ m$^2$. Pressure can be controlled by the syringe pumps consisting of a piston-cylinder assembly with a maximum internal volume of 507.38 ml, which can be operated either in constant pressure mode (up to 35 MPa), constant volume mode (up to 507.38 ml), or constant flow rate mode (up to 204 ml min$^{-1}$). The pump displacement calculated from stepper-motor drive increments (resolution 31.71 nl) can be used to reflect volume changes in the pore fluid.
system. Constant temperature to 0.1 °C can be maintained by a temperature control jacket.

The Temco core holder was used to sandwich the cylindrical shale sample within the between two cylindrical stainless steel loading platens with through-going flow connections and flow distributors. A polyvinyl chloride (PVC) rubber jacket was used to isolate the sample and axial platens from the confining fluid. When measuring permeability by the pressure transient method, the end-platens were connected to two low-volume stainless steel reservoirs by tubing and isolating valves. The volumes of the upstream and downstream were 16 cm$^3$ and 2.94 cm$^3$ respectively and the volumes of reservoirs were significantly higher than the total adsorption capacity of the sample used in the experiments. Upstream and downstream gas pressures were recorded by pressure transducers (PDCR 610 and Omega PX302-5KGV) to a resolution of 0.03 MPa. The gas-pressurized upstream reservoir was discharged through the sample to the downstream reservoir with the equilibration time defining the permeability of the sample (Brace et al., 1968; Hsieh et al., 1980). Volume change effects due to the high compressibility of gas in the reservoirs were minimized by covering the gas reservoirs with thermal insulation material to maintain constant temperature. The mass balance method was used to measure the mass of gas sorbed into the shale samples.
The flow rate, pressure and fluid volume signals obtained from the ISCO pumps were recorded by a National Instruments Labview program using a serial connection (RS-232) between the pumps and the logging computer. Output signals from the pressure transducers were converted at 16-bit resolution using a 16-channel data acquisition system (National Instruments - USB 6211). This Labview software can record signals digitally at a sampling rate of 1 Hz to 1 kHz.

Before starting the series of experiments, the calibration of the stiffness of the Temco tri-axial cell was calibrated to allow the true sample deformation to be measured when samples were loaded. The apparatus was calibrated with a stainless steel dummy sample with a known Young's modulus, loaded axially in the core holder at conditions identical to those used in shale sample experiments. Also, a He leakage check was conducted for each gas permeability test to make sure there were no leaks in the system prior to loading.
Fig. 1. Schematic of experimental set-up
2.2 Sample preparation

The experiments were performed on shale from the Green River formation of Pennsylvania recovered as a large block from a depth of 1100m, drilled perpendicular to the bedding plane. The sample was built by first placing shrink-wrap tubing over the end of first platen with the empty tubing pointing upwards. A frit was inserted on the platen face and this open tube was then filled with sample to a height of two inches. A frit and second platen were then inserted on top of the sample, and an additional wrap was placed around the sample to ensure no leakage. Spacers were placed at either end of the platen assembly to make the desired length for the tri-axial vessel holder. Everything not covered by the shrink-wrap was then covered by rubber jackets, and tied with metal wires to prevent the confining fluid from leaking into the sample or out of the vessel. The sample was then loaded into the vessel and the open end of the tube was closed. The flow lines exiting the pressure vessel were then connected to upstream and downstream reservoirs. Details of the sample preparation procedure are shown in Appendix A.
2.3 Method and Analysis

2.3.1 Permeability

The pressure transient method was used to conduct the gas flow experiments in the low permeability samples (Brace et al., 1968; Hsieh et al., 1980). The governing equation for the pressure pulse through sample can be written as follows.

\[ P_{up}(t) - P_{dn}(t) = (P_{up}(to) - P_{dn}(to))e^{-\alpha t} \]  \hspace{1cm} (1)

\[ \alpha = \frac{kA/\mu\beta L}{1/V_{up} + 1/V_{dn}} \]  \hspace{1cm} (2)

Where \( P_{up}(t) - P_{dn}(t) \) is the pressure difference between the upstream and downstream reservoirs at time \( t \); \( (P_{up}(to) - P_{dn}(to)) \) is the initial pressure difference between the upstream and downstream reservoir at time \( to \); \( \alpha \) is the slope of the line when plotting the pressure decay \( P_{up}(t) - P_{dn}(t) \) on semi-log paper against time(Fig.2); \( A \) and \( L \) are the cross-sectional area and length of the sample, respectively, which define the dimensions of the sample; \( \mu, \beta, V_{up}, \) and \( V_{dn} \) are the dynamic viscosity and compressibility of the gas, and the volume of the upstream reservoir and downstream reservoir, respectively; and permeability \( k \) is calculated from Equation (2) where it is the only unknown.

2.3.2 Sorption

The pressure transient method was used to measure the void volume of the...
sample as a direct consequence of measuring the permeability to He. Firstly, we evacuated air from the system, injected a known amount of He in the upstream reservoir and opened the shut-off valve. As He is inert, the void volume can be calculated from the pressure of the upstream and downstream reservoirs and cell, and the volumes from upstream and downstream reservoirs, as shown in Equation (3):

$$\frac{(P_{up}V_{up}/ZRT)_{\text{pulse,10}}}{P_{up}V_{up}/ZRT} = \frac{(V_{up}+V_{dn}+V_{vd})/ZRT)_{\text{pulse,1}}}{P(V_{up}+V_{dn}+V_{vd}/ZRT)_{\text{pulse,1}}}$$  \hspace{1cm} (3)

Where \( P_{\text{pulse,10}} \) denotes the initial condition for the pressure pulse; \( P_{\text{pulse,1}} \) represents the final condition for the pressure pulse 1; \( Z \) is the compressibility factor of the gas at corresponding temperature and pressure; \( R \) is the universal gas constant; \( T \) is the absolute temperature; \( P \) is the final pressure of the system; and \( V_{vd} \) is the unknown void volume of the sample corresponding to pressure pulse 1.

This method was repeated for multiple cycles and to obtain the void volume of the sample corresponding to different pressures:

$$\frac{(P_{up}V_{up}/ZRT)_{\text{pulse,no}}}{P_{up}V_{up}/ZRT} = \frac{(P_{up}V_{up}+V_{dn}+V_{vd})/ZRT)_{\text{pulse,n}}}{P(V_{up}+V_{dn}+V_{vd}/ZRT)_{\text{pulse,n}}}$$  \hspace{1cm} (4)

Where \( P_{\text{pulse,no}} \) denotes the initial condition for the pressure pulse; and \( P_{\text{pulse,n}} \) represents the final condition for the \( n_{th} \) pressure pulse.
After the void volume of the sample was obtained at different pressures, tests were repeated using CO₂. Since CO₂ is a sorbing gas, the mass balance equation can be expressed as follows:

\[
(P_{up}V_{up}/ZRT)_{pulse, no} + (P(V_{dn}+V_{vd})/ZRT)_{pulse, n-1}
= (P(V_{up}+V_{dn}+V_{vd})/ZRT)_{pulse, n} + n_{pulse, n} \tag{5}
\]

Where \( n_{pulse, n} \) is the amount of gas adsorbed in the sample in moles for the \( n_{th} \) pressure pulse.

These steps were repeated until the desired final pressure level was reached. The cumulative amount of gas adsorbed in the sample was then calculated by summing up the quantities measured in each pressure pulse step.

2.3.3 Morphology

The morphology of samples and the thickness of coatings were examined using a scanning electron microscope (Zeiss 1555 VP FE-SEM, Oberkochen, Germany) equipped with measurement software and an EDS system.
Chapter 3

Experimental Observations and Discussion

3.1 Experimental observations

A series of experiments were run on one sample to determine permeability at same total stress and different pore pressure. The experiments are conducted at room temperature. We define effective stress as the difference between confining stress and pore pressure inside the sample. For each sample permeability was measured at confining stress and axial stress 7 MPa, and the pore pressure was increasing from 1MPa to 5MPa. The influence of effective-stress-driven changes in volumes were examined with non-sorbing He. The superposed influences of swelling were examined with sorbing gas CO\textsubscript{2}. The apparatus had upstream and downstream reservoir characteristics as identified in Table 3-1.

Table 3-1 Experimental constants

<table>
<thead>
<tr>
<th>Experiment constants</th>
<th>Value</th>
<th>Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>Volume upstream</td>
<td>1.6E-5</td>
<td>m\textsuperscript{3}</td>
</tr>
<tr>
<td>Volume downstream</td>
<td>2.94E-6</td>
<td>m\textsuperscript{3}</td>
</tr>
<tr>
<td>Area of cross section of sample</td>
<td>5.06E-7</td>
<td>m\textsuperscript{3}</td>
</tr>
<tr>
<td>Length of sample</td>
<td>1.2E-2</td>
<td>m\textsuperscript{3}</td>
</tr>
</tbody>
</table>
Permeabilities of the samples to He and CO$_2$ as the permeating fluid were measured as defined previously by recording the equilibration of pressures with time. The change in pressure with time was plotted for both upstream and downstream reservoirs. In ideal conditions the two should meet at equilibrium pressure. The equilibrium pressure was then subtracted from the pressure in the upstream and then plotted vs time on a semi-log plot. The slope of the resulting line gave the value of $\alpha$, which was used to calculate permeability using Equation 2.

3.1.1 Results of permeability to He

Five experiments were conducted to measure the permeability to He under different pore pressure. We increased the initial pore pressure from 1.34MPa to 5.26MPa while the downstream initial pressure increased from 0.03MPa to 4.09MPa. The pressure variations of upstream and downstream vs time were recorded by Fig 2, Fig 4, Fig6, Fig 8 and Fig 10. Data was stopped to record when pressures mostly kept constant and the equilibrium pressure was the average value of upstream and downstream pressure. It is obvious that the figures have a same trend: at beginning, pressure sharply decreased and increased for upstream and downstream, respectively; then the pressure had a trend to flatten and finally to reach constantly or meet together. Pressure decay $P_{up}(t) - P_{dn}(t)$ on semi-log paper against time were
reflected by Fig 3, Fig 5, Fig 7, Fig 9 and Fig11. Based on the figures, we can measure 5 different \( \alpha \) (slopes of the lines). The basic experimental data is showed in the table 3-2.

Table 3-2 Basic data from experimental results of He

<table>
<thead>
<tr>
<th></th>
<th>Upstream initial pressure(MPa)</th>
<th>Downstream initial pressure(MPa)</th>
<th>Equilibrium pressure(MPa)</th>
<th>( \alpha )(E-3)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.34</td>
<td>0.03</td>
<td>1.20</td>
<td>0.60</td>
</tr>
<tr>
<td>2</td>
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<td>3.07</td>
<td>4.10</td>
<td>2.35</td>
</tr>
<tr>
<td>5</td>
<td>5.26</td>
<td>4.09</td>
<td>5.10</td>
<td>4.30</td>
</tr>
</tbody>
</table>

Fig.2. Pressure-pulse decay with non-adsorbing gas (Helium) during a transient pulse decay permeability test with initial upstream reservoir pressure 1.3 MPa under constant axial and confining pressure 7 MPa
Fig. 3. Pressure decay $P_{up}(t) - P_{dn}(t)$ on semi-log paper against time for Helium with initial upstream reservoir pressure 1.3 MPa

$$y = -6.04 \times 10^{-4}x + 3.45 \times 10^{-1}$$

$R^2 = 0.99\times 10^{-1}$

Fig. 4. Pressure-pulse decay with non-adsorbing gas (Helium) during a transient pulse decay permeability test with initial upstream reservoir pressure 2.2 MPa under constant axial and confining pressure 7 MPa

$0$ $0.5$ $1$ $1.5$ $2$ $2.5$ $3$ $3.5$ $4$

$0$ $1000$ $2000$ $3000$ $4000$ $5000$ $6000$ $7000$

time(S)

$0$ $500$ $1000$ $1500$ $2000$ $2500$ $3000$

time(s)

$1$ $1.2$ $1.4$ $1.6$ $1.8$ $2$ $2.2$ $2.4$

Pressure(MPa)

downstream pressure

upstream pressure
Fig. 5. Pressure decay $P_{up}(t) - P_{dn}(t)$ on semi-log paper against time for Helium with initial upstream reservoir pressure 1.3MPa

Fig. 6. Pressure-pulse decay with non-adsorbing gas (Helium) during a transient pulse decay permeability test with initial upstream reservoir pressure 3.3MPa under constant axial and confining pressure 7 MPa
Fig. 7. Pressure decay $P_{up}(t) - P_{dn}(t)$ on semi-log paper against time for Helium with initial upstream reservoir pressure 3.3MPa

$y = -2.15E-03x + 2.33E-01$
$R^2 = 9.99E-01$

Fig. 8. Pressure-pulse decay with non-adsorbing gas (Helium) during a transient pulse decay permeability test with initial upstream reservoir pressure 4.3MPa under constant axial and confining pressure 7 MPa
Fig. 9. Pressure decay $P_{up}(t) - P_{dn}(t)$ on semi-log paper against time for Helium with initial upstream reservoir pressure 4.3 MPa.

$$y = -2.35E-03x + 2.57E-01$$

$R^2 = 9.99E-01$

Fig. 10. Pressure-pulse decay with non-adsorbing gas (Helium) during a transient pulse decay permeability test with initial upstream reservoir pressure 5.3 MPa under constant axial and confining pressure 7 MPa.
As the viscosity of gas varies with pressure, viscosity of He can be checked from NIST Chemistry WebBook. Permeability can be measured by Equation 6 and all the relative data is summarized in Table 3-3.

\[ k = \alpha \mu \beta L V_{up} V_{dn} / (A (V_{up} + V_{dn})) \] (6)

Table 3-3 Data for permeability calculation for He

<table>
<thead>
<tr>
<th></th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \alpha ) (E-3)</td>
<td>0.60</td>
<td>1.45</td>
<td>2.15</td>
<td>2.35</td>
<td>4.30</td>
</tr>
<tr>
<td>( \mu ) (E-5 Pa s)</td>
<td>1.98</td>
<td>1.99</td>
<td>1.99</td>
<td>1.99</td>
<td>2.00</td>
</tr>
<tr>
<td>L(E-2 m)</td>
<td>1.20</td>
<td>1.20</td>
<td>1.20</td>
<td>1.20</td>
<td>1.20</td>
</tr>
<tr>
<td>( V_{up} ) (E-5 m(^3))</td>
<td>1.60</td>
<td>1.60</td>
<td>1.60</td>
<td>1.60</td>
<td>1.60</td>
</tr>
<tr>
<td>( V_{dn} ) (E-6 m(^3))</td>
<td>2.94</td>
<td>2.94</td>
<td>2.94</td>
<td>2.94</td>
<td>2.94</td>
</tr>
<tr>
<td>A(E-4 m(^2))</td>
<td>5.06</td>
<td>5.06</td>
<td>5.06</td>
<td>5.06</td>
<td>5.06</td>
</tr>
<tr>
<td>Peq(MPa)</td>
<td>1.20</td>
<td>2.00</td>
<td>3.00</td>
<td>4.10</td>
<td>5.10</td>
</tr>
<tr>
<td>( V_{up} + V_{dn} ) (E-5 m(^3))</td>
<td>1.89</td>
<td>1.89</td>
<td>1.89</td>
<td>1.89</td>
<td>1.89</td>
</tr>
<tr>
<td>( k ) (E-19 m(^2))</td>
<td>6.01</td>
<td>8.50</td>
<td>8.57</td>
<td>6.73</td>
<td>9.74</td>
</tr>
</tbody>
</table>
The permeability vs equilibrium pressure (defined as pore pressure) is plotted in Fig.12. It can be seen that the permeability has an increasing trend with pore pressure rising. Firstly, the permeability had a sharp rise from pore pressure 1.2MPa to 2MPa, and then continued to increase a little to $0.857 \times 10^{-19}$ m². However, the permeability dropped to 0.8 times ($0.673 \times 10^{-19}$ m²) of that with pore pressure 2 MPa. Finally, the permeability increased sharply again to $0.974 \times 10^{-19}$ m².

3.2.2 Results of permeability to CO₂

Another five experiments was conducted to measure the permeability to CO₂ under different pore pressure. We increased the initial pore pressure
from 1.32MPa to 5.41MPa while the downstream initial pressure increased from 0.02MPa to 4.17MPa. The pressure variations of upstream and downstream vs time were recorded by Fig 13, Fig 15, Fig17, Fig 19 and Fig 21. We stopped recording data when pressures mostly maintained constant and the equilibrium pressure was the average value of upstream. It is obvious that the figures of CO₂ has a similar trend to Helium; at beginning, pressure sharply decreased and increased for upstream and downstream, respectively; then the pressure tended to flatten and finally to reach constantly or meet together. Pressure decay $P_{up}(t) - P_{dn}(t)$ on semi-log paper against time are reflected by Fig 14, Fig 16, Fig 18, Fig 20 and Fig22. Based on the figures, 5 different $\alpha$ (slopes of the lines) can be achieved to calculate the permeability to CO₂. The basic experimental data is summarized in the table 3-4.

<table>
<thead>
<tr>
<th></th>
<th>Upstream initial pressure(MPa)</th>
<th>Downstream initial pressure(MPa)</th>
<th>Equilibrium pressure(MPa)</th>
<th>$\alpha$ (E-3)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.32</td>
<td>0.02</td>
<td>1.10</td>
<td>0.38</td>
</tr>
<tr>
<td>2</td>
<td>2.28</td>
<td>1.08</td>
<td>1.99</td>
<td>0.85</td>
</tr>
<tr>
<td>3</td>
<td>3.49</td>
<td>2.07</td>
<td>3.00</td>
<td>1.30</td>
</tr>
<tr>
<td>4</td>
<td>4.36</td>
<td>3.18</td>
<td>4.17</td>
<td>1.47</td>
</tr>
<tr>
<td>5</td>
<td>5.41</td>
<td>4.17</td>
<td>5.05</td>
<td>2.18</td>
</tr>
</tbody>
</table>
Fig.13. Pressure-pulse decay with adsorbing gas (CO$_2$) during a transient pulse decay permeability test with initial upstream reservoir pressure 1.3MPa under constant axial and confining pressure 7 MPa

\[ y = -3.78 \times 10^{-4}x + 4.28 \times 10^{-1} \]
\[ R^2 = 9.96 \times 10^{-1} \]

Fig.14. Pressure decay $P_{up}(t) - P_{dn}(t)$ on semi-log paper against time for CO$_2$ with initial upstream reservoir pressure 1.3MPa
Fig. 15. Pressure-pulse decay with adsorbing gas (CO₂) during a transient pulse decay permeability test with initial upstream reservoir pressure 2.2 MPa under constant axial and confining pressure 7 MPa.

Fig. 16. Pressure decay $P_{up}(t) - P_{dn}(t)$ on semi-log paper against time for CO₂ with initial upstream reservoir pressure 2.2 MPa.
Fig. 17. Pressure-pulse decay with adsorbing gas (CO₂) during a transient pulse decay permeability test with initial upstream reservoir pressure 3.35MPa under constant axial and confining pressure 7 MPa

\[ y = -1.30E-03x + 4.44E-01 \]
\[ R^2 = 9.95E-01 \]

Fig. 18. Pressure decay \( P_{up}(t) - P_{dn}(t) \) on semi-log paper against time for CO₂ with initial upstream reservoir pressure 3.35MPa

\[ y = -1.30E-03x + 4.44E-01 \]
\[ R^2 = 9.95E-01 \]
Fig. 19. Pressure-pulse decay with adsorbing gas (CO₂) during a transient pulse decay permeability test with initial upstream reservoir pressure 4.35 MPa under constant axial and confining pressure 7 MPa.

Fig. 20. Pressure decay $P_{\text{up}}(t) - P_{\text{dn}}(t)$ on semi-log paper against time for CO₂ with initial upstream reservoir pressure 4.35 MPa.

The equation for the line of best fit is $y = -1.47 \times 10^{-3}x + 2.13 \times 10^{-1}$ with $R^2 = 9.99 \times 10^{-1}$. 

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Fig. 21. Pressure-pulse decay with adsorbing gas (CO₂) during a transient pulse decay permeability test with initial upstream reservoir pressure 5.3 MPa under constant axial and confining pressure 7 MPa.

![Graph showing pressure-pulse decay](image)

Fig. 22. Pressure decay $P_{\text{up}}(t) - P_{\text{dn}}(t)$ on semi-log paper against time for CO₂ with initial upstream reservoir pressure 3.3 MPa.

![Graph showing pressure decay](image)

The linear regression equation is $y = -2.18 \times 10^{-3}x + 2.49 \times 10^{-1}$ with $R^2 = 0.97 \times 10^{-1}$.
As the viscosity of gas varies with different pressure, viscosity of CO₂ can be achieved from NIST Chemistry WebBook. The permeability can be measured by Equation 6 and all the relative data is summarized in Table 3-5.

Table 3-5 Data for permeability calculation for CO₂

<table>
<thead>
<tr>
<th></th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>α (E-3)</td>
<td>0.38</td>
<td>0.85</td>
<td>1.30</td>
<td>1.47</td>
<td>2.18</td>
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<tr>
<td>µ (E-5 Pa·s)</td>
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<td>1.52</td>
<td>1.55</td>
<td>1.60</td>
<td>1.70</td>
</tr>
<tr>
<td>L(E-2 m)</td>
<td>1.20</td>
<td>1.20</td>
<td>1.20</td>
<td>1.20</td>
<td>1.20</td>
</tr>
<tr>
<td>V_{up}(E-5 m^3)</td>
<td>1.60</td>
<td>1.60</td>
<td>1.60</td>
<td>1.60</td>
<td>1.60</td>
</tr>
<tr>
<td>V_{dn}(E-6 m^3)</td>
<td>2.94</td>
<td>2.94</td>
<td>2.94</td>
<td>2.94</td>
<td>2.94</td>
</tr>
<tr>
<td>A(E-4 m^2)</td>
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<td>5.06</td>
<td>5.06</td>
<td>5.06</td>
<td>5.06</td>
</tr>
<tr>
<td>Peq(MPa)</td>
<td>1.10</td>
<td>1.99</td>
<td>3.00</td>
<td>4.17</td>
<td>5.05</td>
</tr>
<tr>
<td>V_{up}+V_{dn}(E-5 m^3)</td>
<td>1.89</td>
<td>1.89</td>
<td>1.89</td>
<td>1.89</td>
<td>1.89</td>
</tr>
<tr>
<td>k(E-19 m^2)</td>
<td>3.03</td>
<td>3.84</td>
<td>3.95</td>
<td>3.32</td>
<td>4.32</td>
</tr>
</tbody>
</table>

The permeability to CO₂ vs equilibrium pressure has similar trend to Helium, plotted in Fig.23. It can be found the permeability has an increasing trend as pore pressure rises. Firstly, the permeability kept increasing from $3.03 \times 10^{-19}$ m² to $3.95 \times 10^{-19}$ m² under pore pressure 1.1MPa and 3MPa respectively. After a sudden drop to $3.32 \times 10^{-19}$ m², permeability finally increased sharply to $0.974 \times 10^{-19}$ m².
Fig. 23. Permeability evolution to CO$_2$ as a function of applied pore pressure with constant axial and confining pressure 7 MPa
3.2 Discussion

From Fig.24, it can be seen that as pore pressure increased, the permeability to He ranges from $6.01 \times 10^{-19}$ m$^2$ to $9.74 \times 10^{-19}$ m$^2$ while the permeability to CO$_2$ was almost halved in the range $3.03 \times 10^{-19}$ m$^2$ to $4.32 \times 10^{-19}$ m$^2$. It can be explained by two reasons: (1) the molecule of He is much smaller than CO$_2$, so the molecule of He can permeate into more small fractures in the sample; (2) as CO$_2$ can be adsorbed by the organic matters in the shale sample, more CO$_2$ can be stored in the matrix during gas transport. It can be also found although the permeabilities to He and CO$_2$ fluctuate as pore pressure increasing, there is still an increasing trend due to effective stress decreasing. Fig.25 showed that with effective stress increasing, permeabilities to both He and CO$_2$ tended to decrease. The permeabilities to He and CO$_2$ increased by 43% and by 30% respectively, as effective stress decreased from 6MPa to 4MPa, and finally increased to 1.62 times and 1.43 times of the initial permeability at effective stress 2MPa.
Fig. 24. Evolution of permeability as a function of applied pore pressure with non-sorbing gas (Helium) and sorbing gas (CO2) under constant axial and confining pressure 7MPa.

Fig. 25. Evolution of permeability as a function of applied pore pressure with constant axial and confining pressure 7MPa.
Experimental observations illustrate that permeability evolution is closely linked with gas transport between fracture and matrix. Based on the model of Liu et al. (2011), during gas transport, gas diffused gradually from the fractures to the matrix and the area affected by gas transport expanded dynamically from the fractures towards to the matrix. This suggests permeability evolution is dynamic during gas transport and can be divided into two stages (Fig.26). In the first stage, the gas-influenced area is limited to fracture surfaces, which causes the fracture apertures to decrease, thereby reducing permeability. In the second stage, the gas-influenced area penetrates further into the matrix which causes the fracture apertures to increase, resulting in permeability recovery. This hypothesis is supported by our experimental observations.

<table>
<thead>
<tr>
<th>Initial Equilibrium State</th>
<th>Intermediate State</th>
<th>Final Equilibrium State</th>
</tr>
</thead>
<tbody>
<tr>
<td>Matrix</td>
<td>$p_m = 0$</td>
<td>$p_m = 0$</td>
</tr>
<tr>
<td>Fracture Compartment $p_f$</td>
<td>$0 &lt; p_m &lt; p_f$</td>
<td>$p_m = p_f$</td>
</tr>
</tbody>
</table>

(a) No Swelling Everywhere ($t = 0$)  
(b) Local Swelling ($t$)  
(c) Uniform Swelling ($t \rightarrow \infty$)

Fig.26. Illustration of the difference between local swelling and marco swelling
Between two stages of the gas transport process, where local swelling dominated, the permeability of both He and CO$_2$ was basically lower than at the final equilibrium state. The permeability with 4MPa did not reach final equilibrium, so this is the reason why the permeabilities to He and CO$_2$ decreased from pore pressure 3 MPa to 4MPa and then increased from pore pressure 4 MPa to 5MPa. At final equilibrium state when the uniform macro swelling occurred, permeability for both He and CO$_2$ increased as pore pressure increased, only determined by effective stress decreasing.
Chapter 4

Conclusion

A pulse transient method was applied to investigate the evolution of shale sample permeability, affected by stress, sorption and gas transport between the fracture and the matrix. Based on the experimental results, it can be concluded that:

- Green River Shale has a low permeability to CO$_2$ in the order of magnitude of $10^{19}$ m$^2$, which indicates its good seal efficiency as a caprock for CO$_2$ sequestration.

- Gas transport in the shale sample matches the gas transport predicted by Liu et al. (2011):
  - At the initial stage of CO$_2$ injection, gradual diffusion of gas from the fractures to the matrix resulted in matrix swelling within the vicinity of the fracture compartment. This confirms that shale permeability is controlled by the internal fracture boundary condition.
• Furthermore, as the matrix swelling continued and expanded to macro-swelling, the external boundary condition became the dominant factor affecting the permeability of the shale.

Further research

This thesis has investigated the permeability of shale with He and CO₂. The further research will focus on the effect of water on the evolution of permeability and breakthrough pressure of this shale. Some modelling could also be built to combine the experimental data and simulations, aiming to provide a certain scientific and practical value for CO₂ sequestration.
References


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Weniger, P., Kalkreuth,W., Busch, A., Krooss, B.M., 2010. High-pressuremethane and carbon dioxide sorption on coal and shale samples from the Parana basin, Brazil. Int. J. Coal Geol. 84, 190-205.


Appendix A

Photo documentation of Sample preparation

Step 1: Two spacers and an end platen are placed in that order onto the end with piston of the cell. They are covered up to half the end platen with a rubber sleeve.
Step 2: The sample is covered by foil to prevent the CO2 from leaking and reacting with rubber.
Step 3: A black tape is used to cover end platens and sample.
Step 4: Another heat shrink rubber tube is shrunk around the first one.
Step 5: A white tape is used to cover end platens and rubber tubes to prevent leakage.
Step 6: Wires are tied on the heat shrink tubes and tapes, which is used to fasten them.
Step 7: Another black tapes are used to cover the wires.
Step 8: Another rubber sleeve is connected to the platen with two spacers inside. The spacer is placed in such a way as there is in excess of 20 mm of free space at the top. A similar black tape is used on the rubber sleeve and rubber tube.
Step 9: A long heat shrink rubber tube is shrunk around the last one with two black tapes covered.
Step 10: It is then placed in the cell and closed using the other end cap.
Step 11: Axial and confining pressure is applied to the sample in the cell using pumps filled with water. Pore pressure lines carrying gas are connected to reservoirs. Transducers help measure upstream and downstream pressure.
A block of shale rock and the drilled sample
Appendix B

Micrographs of sample

Fig.1. Some phanerozoic microorganisms can be observed in the sample.

Fig.2. Fractures with porous media can be observed.