Project Title: **CHANGES IN COAL PROPERTIES WITH EXPOSURE TO CO\textsubscript{2}**

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**ABSTRACT**

The work reported is an integral part of a multi-year study, funded jointly by the Illinois Clean Coal Institute, the US Department of Energy, and a consortium of organizations, aimed at improving the modeling and simulation capability of operations where CO\textsubscript{2} is injected in gas bearing coals/shales. The objective of the study was to evaluate changes in coal/shale properties as a result of gas recovery and CO\textsubscript{2} injection, specifically those parameters required for modeling of coal “swelling”, its impact on CO\textsubscript{2} injectivity, and the strength of coal/shale with exposure to increasing concentrations of CO\textsubscript{2}.

The critical parameters when modeling flow are pore volume or cleat compressibility ($C_p$) and matrix shrinkage/swelling compressibility ($C_m$). The values of $C_p$ and $C_m$ were calculated using measured grain/solid compressibility ($C_g$) and bulk compressibility ($C_b$) under best replicated in situ conditions of stress and strain. The estimated values of $C_p$ and $C_m$ clearly showed that these two parameters are not constant. The two increase with depletion, the increase being more significant for CO\textsubscript{2}. Relatively speaking, coal is more compressible in a CO\textsubscript{2} environment although, at high pressures, there is no difference between values for methane and CO\textsubscript{2}. Hence, when injecting CO\textsubscript{2} in a depleted reservoir, compressibility would first increase at low pressures. This will be followed by a large decrease, with its value attaining the original value, when saturated with methane, at high pressure. At no time would it be lower with CO\textsubscript{2} injection than with methane.

In order to evaluate changes in the strength of coal as a result of CO\textsubscript{2} injection and determine if “weakening” of coal is a real phenomenon, ultrasonic velocities through the core were monitored continuously for increasing concentrations of CO\textsubscript{2}. First, based purely on increasing Poisson’s ratio and decreasing Young’s modulus as a signal of weakening, there is little loss of strength when methane is depleted and coal is exposed to CO\textsubscript{2}. Second, when injecting CO\textsubscript{2}, the injection should be carried out in a quantity-controlled manner, with small injection amounts at a time and allowing adequate time for attainment of equilibrium prior to subsequent injection, in order to avoid failure. Finally, a permeability reduction with continued CO\textsubscript{2} injection should be expected.

Preliminary results obtained for CO\textsubscript{2} injection in fractured shale suggest that, like in the case of coal, the change in strength is not significant. Also, the permeability of fractured shale increases with depletion, regardless of the gas used, suggesting that there would be no loss of permeability/injectivity with CO\textsubscript{2} injection. This may, however, be different for unfractured shale.
EXECUTIVE SUMMARY

This study was an integral part of a larger effort entitled “Coal-Seq III Consortium: Advancing the Science of CO\textsubscript{2} Sequestration in Coal Seam and Gas Shale Reservoirs.” It was a continuation of the study, funded jointly by the US Department of Energy (DOE), Illinois Department of Commerce and Economic Opportunity through the Illinois Clean Coal Institute (ICCI), and an industrial consortium, to evaluate the technical feasibility of CO\textsubscript{2} sequestration in deep coals and gas bearing shales while enhancing the production of methane. An important DOE program goal for CO\textsubscript{2} sequestration in unmineable coals and geologic media is to perform enhanced gas recovery field operations ensuring that injectivity is maintained at 90% (or greater) of its initial value. Since this objective is strongly dependent on how permeability varies with CO\textsubscript{2} injection, it is an obvious issue to address. Equally important is the change in permeability with continued methane production, followed by CO\textsubscript{2} injection, from these reservoirs. Efforts to model this effectively have not been very successful, necessitating changes in various input parameters without an adequate understanding of their behavior in multi-gas environments.

The first objective of this research study was to develop a means to improve the capability to model flow of gas in coal, particularly the variation in permeability with continued methane production, followed by CO\textsubscript{2} injection. The most disputed parameters required as an integral part of the modeling exercise are pore volume or cleat compressibility (C\textsubscript{p}) and matrix shrinkage/swelling compressibility (C\textsubscript{m}), since the two impact changes in permeability significantly. Models developed to date are divided as to the variation in values of these parameters with depletion/injection. Some models assume these values to be constant while others treat them as variables. Hence, the first part of the study was aimed at answering the basic question of whether values of these two critical parameters change with reservoir pressure, which decreases with methane production and increases with CO\textsubscript{2} injection.

Work completed to estimate values of C\textsubscript{p} and C\textsubscript{m} started with measuring the grain/solid compressibility of coal (C\textsubscript{g}), estimated using the measured volumetric strain induced purely due to mechanical compression of solid coal. This was followed by flooding the coal with methane and bleeding it to estimate the induced “shrinkage” associated with desorption and measuring the bulk compressibility (C\textsubscript{b}). Finally, the exercise was repeated using CO\textsubscript{2}. Results of this effort successfully demonstrated that values of C\textsubscript{p} and C\textsubscript{m} are not constant. Both increased with depletion of methane. The increase for a pressure reduction from 1000 psi was estimated to be more than three times. For CO\textsubscript{2} flooding, the permeability changed significantly with pressure, the change being dramatic at lower pressures. The change in the value of C\textsubscript{p} in the case of CO\textsubscript{2} was also more significant. These results showed that coal is more compressible when exposed to CO\textsubscript{2}. With continued CO\textsubscript{2} injection, the compressibility of the reservoir would decrease and approach that of methane but never fall below the corresponding value for methane.

The second objective of this study was to estimate changes in the strength of coal as a result of continued methane production and CO\textsubscript{2} injection. In the past, these tests have
not been successful due to pre-mature failure of test samples, giving rise to the suspicion that coal “weakens” when exposed to CO₂. Typically, testing to determine the strength of coal/rock is destructive in nature and does not permit establishing the dynamics with depletion or injection. Hence, a non-destructive technique using the through transmission pulse echo method was applied, where ultrasonic velocities were monitored continuously with depletion and injection and, using those measured velocities, elastic moduli and Poisson’s ratio were calculated. It was decided that a decrease in the value of Young’s modulus and an increase in Poisson’s ratio would be considered a signal of “weakening”.

To start with, the core was flooded with helium and longitudinal and shear velocities were measured. The pressure was then brought down in a step-wise manner. Results clearly suggested that depletion of helium did not affect the mechanical properties of coal in any way. Hence, the mechanical impact of methane depletion, CO₂ injection, or displacement of one by the other, cannot be significant in situations where CO₂ is injected in order to enhance gas production or for sequestration. For depletion of methane also, there was no change in mechanical properties of coal. When CO₂ was injected to displace coal, the value of Young’s modulus decreased and Poisson’s ratio increased although changes were really not significant. There probably was some softening of coal but it did not appear to become “weaker”. After displacing all of the methane, CO₂ pressure was increased to evaluate the incremental CO₂ storage capacity of the reservoir. Values of the two mechanical parameters remained fairly constant, indicating that there was no “weakening” effect resulting from additional injection. One issue that became clear during the experimental work is that CO₂ must be injected in a quantity-controlled manner, that is, injection of small amounts at a time with an adequate “soaking” period for the coal to stabilize and attain near-equilibrium conditions. Any pressure-controlled injection, where CO₂ is injected at high pressure, fails the coal, perhaps due to differential swelling at the point of injection resulting in large stresses, thus resulting in stress inequilibrium and failure.

The last phase of the experimental work involved repeating the above test for a core of New Albany shale. First, the initial permeability of shale was extremely low and the only way to induce any measurable flow through the core was to create a fracture in the core. Preliminary results of CO₂ injection in fractured shale suggested that, like in the case of coal, the change in the strength of shale is not significant. Also, the permeability of fractured shale increases with depletion, regardless of the gas used, suggesting that there would be no unexpected loss of permeability/injectivity with CO₂ injection. The change in permeability would be purely due to mechanical changes induced in shale due to changing pore pressure. At this time, it is not very clear if unfractured shale would exhibit the same behavior. However, since gas production from shale-gas reservoirs is possible only after hydrofracturing it, this is really not a concern at the present time.
OBJECTIVES

**Overall Project Objective(s):** The primary objective of this research was to evaluate changes in properties of coal, specifically the variation in its strength and compressibility as a result of methane depletion and injection of CO₂, and how these parameters affect CO₂ injectivity. The secondary objective was to initiate a preliminary study on evaluating the strength of shale and its variation with depletion of gas.

**Specific Objectives of This Study:** The overall study was aimed at determining if coal “weaks” with injection of CO₂, and if so, the degree of weakening. It included an evaluation of the impact of this effect on the permeability of coal, and hence, CO₂ injectivity. In order to model this effect correctly, the study included establishing the variation in pore volume (cleat) compressibility and matrix shrinkage/swelling compressibility of coal under field replicated conditions.

In order to achieve the stated objectives, work was divided into the following tasks:

**Task 1 – Measurement of Compressibility:** This task involved measuring grain/solid compressibility ($C_g$), followed by measurement of bulk compressibility ($C_b$), in an environment where methane is gradually replaced with CO₂. Using basic definitions of porosity ($\phi$), bulk, grain, and pore volume compressibilities ($C_b$, $C_g$, and $C_p$), and a mathematical relationship between these parameters, pore volume or cleat compressibility ($C_p$) and matrix shrinkage/swelling compressibility ($C_m$) were estimated to establish variation trends in their values with depletion and injection.

**Task 2 – Coal/Shale “Weakening” with Exposure to CO₂:** This task involved using a non-destructive experimental technique, specifically the “through transmission pulse echo method,” to estimate the dynamic strength properties of coal in an environment where methane is gradually replaced with CO₂ and with continued depletion of gas in shale.

INTRODUCTION AND BACKGROUND

Modeling of changes in permeability with continued production of gas from coalbed methane (CBM) reservoirs has been an ongoing effort. Several models have been developed to date [1-5] although only three are used extensively. Recently, these models have been modified/extended to include CBM operations where CO₂ is injected in reservoirs in order to enhance the production of methane and/or sequester carbon dioxide. These models are based on basic principles of rock mechanics and the theory of coal volumetric strain associated with sorption of gas, the so-called matrix “shrinkage/swelling” effect. However, there is a major fundamental difference between these three models. One treats pore volume or cleat compressibility ($C_p$) and matrix shrinkage compressibility ($C_m$) as constants throughout the production/injection period; the second treats $C_p$ as a constant, but $C_m$ as a variable; the third treats both as variables. At this time, there is a lack of fundamental understanding that would support any one of these assumptions.
Although $C_p$ and $C_m$ are critical parameters in determining changes in permeability of CBM reservoirs, they become even more critical when a higher sorbing gas, like CO$_2$, is injected into coal, since it has two distinct effects. First, the increase in gas pressure opens up cleats resulting in increased permeability. Second, the sorption associated swelling of the coal matrix tends to close cleats, reversing the increased permeability effect and resulting in an overall reduction in injectivity. The “swelling” of coal with sorption of CO$_2$ is a definite phenomenon and measurements have determined it to be significant [6]. Finally, a few research studies have reported that increased gas pressure reduces the strength of coal significantly [7, 8], while others claim that adsorption does not affect coal strength [9, 10].

Field pilots completed to date, and summarized in a previous ICCI report [6], suggest that there is an initial loss of injectivity with injection of CO$_2$, but that there is a reverse effect with continued injection, resulting in improved injectivity. The reason for this is not known with certainty although it is suspected that properties of coal change with exposure to CO$_2$. Two theories have been proposed to explain the increased injectivity. The first one states that coal “weakens” when exposed to CO$_2$ [11-14]. The second uses the theory of coal failure due to “excess” stresses, resulting from coal swelling in situ, and its inability to swell due to lateral confinement. Due to the destructive nature of rock strength testing procedures in the laboratory, work completed to date has been conducted on different coal samples and the extremely heterogeneous nature of coal has led to results of these studies being non-conclusive.

Given these controversial findings and concern about the overall permeability reduction associated with injection of CO$_2$, DOE has taken the stand that CO$_2$ sequestration in unmineable coals will be allowed only if it can be shown that injectivity is maintained at 90% (or greater) of its initial value. The overall objective of this research study was, therefore, two-fold. The first part involved answering the question as to whether values of $C_p$ and $C_m$ vary in CBM/ECBM/CO$_2$ sequestration operations by estimating their values for depletion and injection alternatives under best replicated field conditions. The second part involved estimating the strength of coal continuously as methane is depleted, or is gradually replaced with CO$_2$.

The US DOE is also considering sequestering CO$_2$ in non-coal formations, like shale gas reservoirs, where there is some value-added benefit, as opposed to saline aquifers, where the geologic formation simply serves as a CO$_2$ repository. However, studies on shale have been limited to numerical and simulation analysis. Hence, an extension of the primary objectives was to initiate similar experimental testing on shale.

**EXPERIMENTAL PROCEDURE**

**Task 1 – Measurement of Compressibility**

The experimental procedure for these tests and the theoretical background was presented in detail in a previous ICCI report [15].
Task 2 – Coal/Shale “Weakening” with Exposure to CO₂

A description of the experimental setup and procedure for these tests is presented in detail in a previous ICCI report [15].

Coal Sample Procurement and Preparation: Blocks of coal were obtained from Illinois and San Juan basins. Cylindrical cores, three inches in diameter and four to five inches in length, were prepared in the laboratory. The two end surfaces of the specimen were trimmed to enable proper placement in the triaxial cell as well as transmission of ultrasonic waves from the upper platen to the sample at the upstream end and from sample to the platen at the downstream end. The core was preserved in its native state to prevent any damage due to weathering by storing it in an environmental chamber with no source of light and under controlled conditions of temperature and humidity.

Shale Sample Procurement and Preparation: Cores of Albany shale, four inches in diameter, were provided by the Illinois State Geological Survey (ISGS). These cores were retrieved from a depth of ~5,050 feet. To enable proper placement of the sample in the triaxial cell, core diameter was reduced to three inches and sample length was reduced to four inches. Initially, intact sample was used for the experiment. Unfortunately, even after several days of injection, no gas flow through the sample was measured. Individuals with significant shale gas experience were contacted for advice. They were not surprised since it is typically not possible to produce shale gas in the field without fracturing the gas-bearing formation. They suggested replicating this by using a fractured core and repeating the experiment. This was done by creating artificial fractures in the sample, as shown in Figure 1.

Figure 1: Artificially fractured shale sample.
RESULTS AND DISCUSSION

**Task 1 – Measurement of Compressibility**

**Methane Displacement with CO\(_2\) Injection:** As the last step of the experiment for the unconstrained condition, after reporting results in the last report [15], methane displacement by step-wise CO\(_2\) injection was carried out. As expected, the volume of the coal matrix increased with CO\(_2\) injection under constant gas pressure at 850 psi. Results are shown in Figure 2. The volumetric strain increased from 0.3% to 1.15% for a decrease in partial pressure of methane from 850 psi to nearly zero. Hence, at 850 psi, the volumetric swelling strain due to CO\(_2\) adsorption is almost three times that with methane.

![Figure 2: Volumetric strain for methane displacement by CO\(_2\) injection.](image)

**Helium Injection under Constrained Condition:** The sample was flushed with helium at 100 psi to get rid of any residual air within the sample. Next, helium injection was carried out gradually to a final pressure of 1100 psi. After achieving equilibrium at 1100 psi, flowrate was measured. Helium pressure was then decreased from 1100 to ~100 psi in a step-wise manner. Under uniaxial strain conditions, horizontal stress decreased from 1400 to 550 psi for a decline in pressure from 1100 to 100 psi, as shown in Figure 3. Permeability was calculated using measured flowrates and results are shown in Figure 4. Helium permeability decreased from 1.07 to ~0.2 md during depletion, a five-fold reduction. The volumetric strain was continuously monitored during helium depletion and results are shown in Figure 5. The vertical stress over the duration of the experiment is also included in the plot. It is fairly constant throughout the experimental duration, satisfying the first condition of uniaxial strain. Using the measured vertical strain, coal bulk compressibility (C\(_b\)) for helium was calculated to be 9E-07 psi\(^{-1}\).
Figure 3: Changes in horizontal stress with helium depletion.

Figure 4: Changes in permeability with helium depletion.
Figure 5: Variation of vertical stress and volumetric strain for helium depletion.

**Methane Injection under Constrained Condition:** After completing the cycle, helium was flushed out and the sample was saturated with methane at 1100 psi. Flowrate measurements were then taken for different pressure steps and permeability was estimated in a step-wise manner. Permeability results are shown in Figure 6. There is an overall and continuous increase in permeability for decreasing pore pressure from 1100 to ~50 psi. It is apparent that the rate of increase is not uniform, with very little increase between 1100 and 500 psi, which becomes truly significant only below 500 psi. In addition to the desorption effect at low pressures, the second parameter that varies during depletion and influences permeability is the horizontal stress under uniaxial strain condition. Figure 7 shows corresponding changes in the applied horizontal stress for methane depletion. It is evident that the applied horizontal stress decreased linearly with pore pressure.

The volumetric strain with reduction in methane pressure is shown in Figure 8, along with the vertical stress to demonstrate the uniaxial stress condition. The polynomial best fit for these experimental results is given as:

\[ \varepsilon = -3E-09P^2 + 8E-06P - 0.0047 \]  

(1)
Figure 6: Changes in permeability with reduction in pore pressure for methane.

Figure 7: Variation in horizontal stress with decreasing pressure for methane.
Figure 8: Variation of vertical stress and volumetric strain for methane depletion.

The volumetric strain of methane was also modeled by a Langmuir-type equation and this is shown in Figure 9. The Langmuir-type best fit equation is modeled as:

\[ \varepsilon = \frac{0.01061P}{P + 1175} \]  

(2)

In order to compare the difference between results obtained for unconstrained [Figure 2] and constrained conditions, the two are plotted together, as shown in Figure 10. It is surprising that the difference in results between these two conditions is that small. Generally, it is believed that stress makes a significant difference due to the added resistance to swelling and shrinkage; however, results do not support this belief and logic. Nevertheless, it also suggests that unconstrained results can provide reasonable estimates for in situ conditions. This is a positive finding given the significant difference between the effort required to carry out experiments under unconstrained and constrained conditions.
Figure 9: Langmuir-type fit of volumetric strain for methane depletion.

\[ \varepsilon = \frac{0.01961 \phi}{P + 1175} \]

Figure 10: Comparison of Langmuir-type fits for constrained/unconstrained conditions.
Using measured volumetric strain under the constrained condition, bulk compressibility ($C_b$) for methane was calculated and plotted as a function of pore pressure, as shown in Figure 11. Mathematically, the slope of the volumetric strain plotted in Figures 8 or 9 is bulk compressibility ($C_b$). It increases from 2.1E-06 to 7.7E-06 psi$^{-1}$ for a pressure reduction from 1100 to ~100 psi. Also, bulk volume decreases faster at low pressures than at high pressures. Another interesting observation is that, even under constrained conditions, these values are two to eight times higher than the value for helium. Hence, the sorptive environment of methane results in a significant increase in the value of bulk compressibility.

**Figure 11: Variation in bulk compressibility under constrained condition for methane.**

**CO$_2$ Injection under Constrained Condition:** After completing the methane cycle, the sample was saturated with CO$_2$ at 850 psi and allowed to attain equilibrium. Flowrate measurements were then taken and permeability was estimated as shown in Figure 12. Permeability increases more than twenty times with CO$_2$ pressure depleting from 850 to ~100 psi, which is significantly higher than the increase measured for methane. This is as expected since the coal matrix shrinkage effect for desorption of CO$_2$ is stronger than for methane. The applied horizontal stress and vertical strain were monitored continuously. Results are shown in Figures 13 and 14, respectively.
Figure 12: Changes in permeability for reduction in pore pressure for CO$_2$.

Figure 13: Variation in applied horizontal stress with decreasing pressure for CO$_2$.
Using measured volumetric strain under the constrained condition, as shown in Figure 14, bulk compressibility ($C_b$) for CO$_2$ was calculated and plotted as a function of pore pressure. This is shown in Figure 15. It increases from 2.8E-06 to 2.5E-05 psi$^{-1}$ for a pressure reduction from 850 psi to ~100 psi, significantly higher than the range for methane. In order to compare the coal matrix volumetric behavior of methane and CO$_2$, a comparison of the two bulk compressibility values is presented in Figure 16. It is evident that the coal bulk is more compressible for CO$_2$ than for methane. With CO$_2$ depletion, the coal matrix volume decreases significantly below 400 psi and approaches the corresponding value for methane. The high volumetric strain above 400 psi is what appears to be responsible for the significant increase in permeability, explaining the sharp rise in its value in Figure 12.
Figure 15: Variation of bulk compressibility under constrained condition for CO$_2$.

Figure 16: Comparison of bulk compressibility for methane and CO$_2$. 
Task 2 – Coal/Shale “Weakening” with Exposure to CO₂

During the experiment, p- and s-ultrasonic velocities through the sample were measured at all step intervals. Measured velocities were used to calculate dynamic properties using the following equations [16]:

\[
\text{Poisson’s Ratio (v)} = \frac{1-\frac{v_s^2}{v_l^2}}{2\left(1-\frac{v_s^2}{v_l^2}\right)^2}
\]

(3)

\[
\text{Young’s Modulus of Elasticity (E)} = \frac{\rho v_s^2(3v_s^2 - 4v_l^2)}{(v_s^2 - v_l^2)}
\]

(4)

\[
\text{Shear Modulus (G)} = \rho v_s^2
\]

(5)

\[
\text{Bulk Modulus (K)} = \rho \left[v_l^2 - \frac{4}{3} v_s^2\right]
\]

(6)

where, \(v_s\) is ultrasonic s-wave velocity (m/s), \(v_l\) is p-wave velocity (m/s), and \(\rho\) is the density (kg/m³).

It was recommended that a decrease in the value of E and an increase in the value of \(v\) be taken as the signal that coal is “weakening”. The intent of this phase of the experimental work was not to actually measure any of the strength parameters but rather establish the trend of variation with depletion and injection.

Illinois Coal

The results of the first part of the experiment for the helium cycle were reported in detail in the previous ICCI report at the end of the first year of the study [15].

After completion of the helium cycle, step-wise injection of methane was carried out to a final pressure of \(-1000\) psi. Methane pressure was then decreased to \(800\) psi and, in the subsequent step, to \(600\) psi, maintaining uniaxial strain conditions. At this point, some methane was bled out and CO₂ was injected, maintaining the total pressure at \(600\) psi. Unfortunately, the sample failed prior to attaining methane/CO₂ equilibrium at \(600\) psi. The variation in velocities is shown in Figure 17. Calculated values of Young’s, Shear, and Bulk moduli are shown in Figure 18. For pressure decline from 1000 to \(600\) psi, measured velocities, and therefore, calculated moduli are fairly constant. There can be two explanations for this. First, there is no change in mechanical properties for methane depletion from 1000 to \(600\) psi as well as with initial injection of CO₂ although this lasted only a few minutes. It is unlikely that significant methane was displaced by CO₂ in the short duration. Second, values are constant since substantial desorption of methane and adsorption of CO₂ did not occur since the sample failed prematurely.
Figure 17: Variation in ultrasonic velocities of coal with depletion of methane and CO$_2$ injection at 600 psi – Illinois coal.

Figure 18: Variation in elastic moduli of coal with methane depletion and CO$_2$ injection at 600 psi – Illinois coal.
San Juan Coal

The core was placed in the triaxial cell and stressed gradually to 2100/1500 psi of vertical/confining stresses. A step-wise injection of helium was then carried out to a final pressure of 1000 psi. Helium pressure was decreased first to 800 psi, and in subsequent steps, to 600, 400 and 200 psi, maintaining uniaxial strain conditions. The ultrasonic p- and s-wave velocities were measured at each pressure step. Under uniaxial strain conditions, decreasing helium pressure increases the density of the sample slightly, and hence, there was a very small increase in the two velocities. As expected, calculated values of Young’s, Shear, and Bulk moduli also increased, but only slightly, with decreasing helium pressure. This is shown in Figure 19. The value of Poisson’s ratio remained constant at 0.36 throughout the helium cycle. Since Young’s modulus and Poisson’s ratio remained nearly constant during helium depletion, the strength of the sample is not affected by helium depletion from 1000 to 200 psi.

![Figure 19: Variation in elastic moduli with helium depletion for San Juan coal.](image)

After completing the helium cycle, step-wise injection of methane was carried out to a final pressure of 1000 psi. Methane pressure was then decreased to 800 psi, and in the subsequent step, to 600 psi, maintaining uniaxial strain conditions. After acquiring ultrasonic velocities at 600 psi, methane was gradually replaced with CO₂, keeping overall pressure constant at 600 psi. Small quantities of CO₂ were injected in a step-wise manner. This was a distinct deviation from the past practice of injecting CO₂ at constant pressure, that is, pressure controlled injection, which always resulted in premature failure of coal. After attaining equilibrium at each step, fractional CO₂ in the sample was determined by analyzing gas at the outlet using a gas chromatograph (GC). Partial pressures of methane at equilibrium for the various steps were 516, 354, and 186 psi. In the last step, all of the methane was displaced with CO₂ and the sample was completely
saturated with CO₂ at 600 psi. This is the first time that the test sample did not fail with CO₂ injection. Hence, it appears that the way to carry out injection is in a step-wise manner, controlling the amount of CO₂ injected rather than the injection pressure. At this time, the experiment was complete; however, the DOE Project Manager suggested one additional injection to evaluate the incremental sequestration capacity of coal. The CO₂ pressure in the sample was, therefore, subsequently increased to 800 psi and ultrasonic velocities were measured and used to calculate strength moduli and Poisson’s ratio.

Results for methane depletion, methane/CO₂ exchange, and CO₂ injection are shown in Figure 20. The value of Young’s modulus decreased slightly with increasing CO₂ concentration. It is not clear that micro-fracturing occurred during CO₂ injection, which would cause a significant decrease in the value of E. However, since there was some change in its value, Figure 21 shows the same results, exaggerated to exhibit the variation. Similarly, the value of Poisson’s ratio increased from ~0.36 to 0.39 during depletion, as shown in Figure 22 (also exaggerated). Decreasing values for Young’s modulus and increasing Poisson’s ratio indicate that the sample does get softer when methane is displaced with CO₂ although changes are not significant. It would be for geomechanics experts to provide the final verdict on whether these results support the “coal weakening” theory.

![Figure 20: Variation in Young’s modulus with methane/CO₂ injection – San Juan coal.](image-url)
Figure 21: Exaggerated view of changes in the value of Young’s modulus for methane/CO$_2$ injection – San Juan coal.

Figure 22: Variation in Poisson’s ratio for San Juan basin sample.
Shale Sample

A new experiment was initiated using a core from the Albany shale, taken from a depth of ~5050 feet. Shortly after starting the experiment, it was realized that the permeability of the core was very low and it would take extremely long to attain equilibrium. Also, measured permeability would be extremely low, in the order of a microdarcy. Two individuals with industrial experience working with shale gas were contacted. Based on their recommendation, artificial fractures were created in the core by cutting the sample orthogonally. The pieces were put together, placed in the triaxial cell and stressed gradually to 2100/1500 psi axial/confining stresses. A step-wise injection of helium was then carried out to a final pressure of 1000 psi. Helium pressure was decreased first to 800 psi, and in subsequent steps, to 600, 400 and 200 psi, maintaining uniaxial strain conditions. The p- and s- wave velocities were measured at each pressure step. There was a slight increase in values of p- and s-velocities with decreasing pore pressure, perhaps due to the increase in sample density resulting from helium depletion.

Using measured p- and s-wave velocities, Young’s, Shear, and Bulk moduli were calculated. These three values increased slightly with decreasing helium pressure. This is shown in Figure 23. The value of Poisson’s ratio did not change, maintaining a constant value of 0.28. Constant Young’s modulus and Poisson’s ratio indicate that helium injection did not affect the dynamic mechanical properties of shale. Based on experience with coal, this was expected.

Figure 23: Variation in elastic moduli with helium depletion for shale sample.
After completion of the helium cycle, methane injection was initiated where injected methane gradually replaced any residual helium and the sample was completely saturated with methane at 1000 psi. Methane pressure was then decreased first to 800 psi, and in subsequent steps, to 600, 400 and 200 psi, maintaining uniaxial strain conditions. Ultrasonic p- and s- wave velocities were measured at each pressure step. There was a slight increase and decrease in ultrasonic p- and s- wave velocities, respectively, an indication for material softening. This is further seen by decreased Young’s modulus from 3.7 x 10⁶ to 3.6 x 10⁶ psi (Figure 24) and increased Poisson’s ratio from 0.28 to 0.29 with reduction in methane pressure from 1000 to 200 psi. Results indicate that injection of methane changes the mechanical behavior of shale although these changes are not significant enough to support “weakening”.

After completing the methane cycle, CO₂ injection was initiated. Injected CO₂ gradually replaced methane until the sample was completely saturated with CO₂ at 800 psi. Gas pressure was then decreased to 200 psi, in steps of 200 psi, maintaining uniaxial strain conditions. Using measured p- and s- wave velocities for each pressure step, elastic moduli were calculated. Results are shown in Figure 25. The value of Young’s modulus decreased from 3.7 x 10⁶ to 3.6 x 10⁶ psi and Poisson’s ratio increased from 0.29 to 0.31 for the CO₂ depletion cycle. The decrease in the value of Young’s modulus and increasing Poisson’s ratio indicate that the sample did get softer with CO₂ injection although these changes are not significant.

Figure 24: Variation in elastic moduli with methane depletion for shale sample.
Overall changes in Poisson’s ratio for shale with injection of helium, methane, and CO$_2$ are shown in Figure 26. Poisson’s ratio remained almost constant for helium and increased with methane and CO$_2$ injection. These results show that helium injection does not change the mechanical behavior of shale. Injection of methane/CO$_2$ makes the sample softer although illustrations of this effect have been exaggerated in the figures provided. However, results suggest that, on a relative basis, CO$_2$ weakens/softens shale since the variation is larger than that for methane.

The permeability of the shale sample to the three gases was also measured to evaluate the effect of fracturing on flow in shale. The measured variation in permeability with depletion for three gases is shown in Figure 27. The permeability to helium is much higher than that to methane and CO$_2$. One of the reasons for this is probably the difference in the three viscosities, helium being higher than that of methane and CO$_2$. Hence, the relative increase in permeability for the three gases was calculated, where the base permeability was that measured at the highest pressure, that is, 1000 psi for helium and methane and 800 psi for CO$_2$. This is shown in Figure 28. The increase in the relative permeability is similar for all three gases and it increased with depletion. These results suggest that the permeability measured was purely the result of the fracture rather than the rock type.
Figure 26: Variation in Poisson’s ratio with gas depletion for shale sample.

Figure 27: Variation in permeability with gas depletion for shale sample.
CONCLUSIONS AND RECOMMENDATIONS

Based on the work completed, the following conclusions are made:

1. For methane displacement with CO\textsubscript{2} injection, the incremental volumetric swelling strain is almost three-fold. This is expected since coal has a much higher sorptive affinity for CO\textsubscript{2}. This is also in agreement with results from a few previous studies.

2. The permeability of coal with CO\textsubscript{2} injection should be expected to decrease significantly. It is unlikely that the 90% injectivity would be sustainable over the long-term.

3. The basic bulk compressibility of coal, that is, compressibility to helium, is estimated to be $9 \times 10^{-7}$ psi\textsuperscript{-1}.

4. The bulk compressibility increases for both methane and CO\textsubscript{2} depletion under uniaxial strain conditions, at a faster pace in the low pressure range than at high pressures. Bulk coal is more compressible for CO\textsubscript{2} than for methane. Hence, with CO\textsubscript{2} injection in depleted reservoirs, compressibility would decrease. However, it would never go below the corresponding value for methane. With increasing pressure, it would only approach the value for methane.

5. Applied horizontal stresses decrease linearly for helium, methane and CO\textsubscript{2} depletion. The magnitude of stress loss is in the following order: CO\textsubscript{2} > methane > helium.

6. Changes in dynamic mechanical properties, Poisson’s ratio, and elastic moduli, are related to changes in the strength of the rock/coal tested. Since injection of helium did
not change the dynamic mechanical behavior of either rock type, there appears to be no impact on the strength of coal as a function of depletion/injection alone. Any deviation from this behavior is attributed to ad-/de- sorption alone.

7. An important finding, based on the fact that several samples failed with constant pressure injection, is that injection should be carried out in a quantity-controlled manner rather than pressure-controlled. Pressure-controlled injection bombards coal with CO\textsubscript{2}, resulting in large swelling and strain in the area close to injection. This results in very large stresses and the coal fails in tension. Quantity-controlled injection, with adequate time between consecutive injections, allows movement of CO\textsubscript{2}, attainment of equilibrium before coal is exposed to additional CO\textsubscript{2}, and prevents premature failure. On the other hand, if it is desirable to fracture the coal in the area immediately surrounding the injection zone, injection should be carried out in a pressure-controlled manner.

8. Injection of methane/CO\textsubscript{2} resulted in a change in Poisson’s ratio and Young’s modulus of coal and shale. However, the measured changes were so small that it is difficult to support the theory of “weakening” associated with depletion or injection.

9. Finally, the permeability of fractured shale increases with gas depletion. However, the relative increase is similar for helium, methane and CO\textsubscript{2}. This behavior is distinctly different than that typically observed for coal. This appears to be due to the fact that the core was fractured rather than the rock characteristics.

Based on the findings of this study, it is recommended that the following topics be pursued for further research:

1. Since depletion of methane results in a significant increase in permeability, stability of the injected/stored CO\textsubscript{2} should be evaluated. In the event of CO\textsubscript{2} leakage for any reason, like an earthquake, the permeability of coal would increase at an accelerating pace and could result in catastrophic re-emission of stored CO\textsubscript{2}. The stability of coal under such conditions should also be evaluated.

2. The permeability of shale with depletion should be studied further. Typically, it is believed that permeability decreases with continued production; however, preliminary results obtained suggest that this may not be the case. It is equally important to explain this behavior. For example, similar behavior in the case of coal is now well understood and explained as a consequence of matrix shrinkage.

3. The recommended signal of weakening, i.e., a decrease in the value of Young’s modulus and an increase in Poisson’s ratio, need to be studied in detail. Specifically, the magnitude of the change prior to using it as a predictive tool must be defined properly.
REFERENCES


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