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# RECOVERY OF STORED GAS IN COAL BY NITROGEN INJECTION – A LABORATORY STUDY

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**ABSTRACT:** With increasing worldwide concern on Green House Gas (GHG) emission and its reduction, significant interest is now directed toward finding a practical and economical ways of enhancing methane gas containment in coal deposits. Carbon dioxide sequestration has been tried successfully for the recovery of methane from coal measure rocks. A laboratory study was undertaken to examine the effect of displacing the adsorbed gases in coal with N<sub>2</sub> injection. To study the feasibility of removing the initially adsorbed gas from coal with another gas, tests were carried out using an in-house built multi-function outburst research rig (MFORR). Accordingly the following laboratory tests were carried out: sorption and desorption characteristics of CO<sub>2</sub>/CH<sub>4</sub> mixed gas in coal, displacement characteristics of adsorbed mixed gases with N<sub>2</sub> injection, displacement characteristics of adsorbed CO<sub>2</sub> with N<sub>2</sub> injection, and displacement characteristics of adsorbed CH<sub>4</sub> with N<sub>2</sub> injection. The study revealed that CO<sub>2</sub> desorption increased by almost 30% as a result of N<sub>2</sub> injection, which is about double that obtained without N<sub>2</sub> injection. This finding has significant bearing in solving the drainage difficulties experienced at West Cliff Colliery and Panel 500 area, which had difficulty in draining CO<sub>2</sub> gas concentration zones.

## INTRODUCTION

With increasing worldwide concern on Green House Gas (GHG) emission and its reduction, significant interest is now directed toward finding a practical and economical ways of enhancing methane gas release from coal deposits and the subsequent recovery of this gas from both mineable and unmineable coal deposits.

Carbon dioxide sequestration has been tried successfully, for some time now, for the recovery of methane from coal measure rocks. The increased attraction of the CO<sub>2</sub> to coal is commonly attributed to the coal's affinity to carbon dioxide. As a result of CO<sub>2</sub> injection, the methane gas is stripped from its monolayer adherence to the coal matrix surface and retained in coal fracture space, which would then be readily driven out of coal by the reduction in gas pressure. A major drawback of the application of CO<sub>2</sub> to methane recovery is the hazards associated with coal gas outburst in underground coal mining. Therefore, this technique is limited to unmineable coal deposits.

In recent years, however, the use of N<sub>2</sub> injection has been tried for methane recovery, in a number of locations in the USA and in Canada, with reported success, the case study of Tiffany Project, San Juan Basin, USA, Reeves and Oudinot, (2004) and Koperna *et al* (2009), has indicated that the recovery of CH<sub>4</sub> gas from coal have increased between 10-20 % with N<sub>2</sub> injection. There has also been some growing interest in the possible use of CO<sub>2</sub>/N<sub>2</sub> mixture as an alternative approach to enhancing methane recovery, particularly from mineable coal deposits. The injection of mixed gas is considered to possibly provide a synergy of production mechanisms which would result in lowering of CO<sub>2</sub> in mine air (Reeves and Oudinot, 2004).

The storage of gas in general is dependent on the coal rank especially in virgin seams. Higher rank coals such as higher rank bituminous and anthracite coals retain methane in preference to other gases, but in some other coal seams, such as the Bulli seam of the Sydney basin, NSW, there are areas where the dominant seam gas is CO<sub>2</sub> rather than CH<sub>4</sub>. In fact, CO<sub>2</sub> and mixed gas CH<sub>4</sub>/CO<sub>2</sub> have been found in a number of locations in Tahmoor, Metropolitan, Appin and West Cliff Mines. A typical difficult to drain site is at West Cliff Mine and panel 500, where some sections of the longwall panels, with CO<sub>2</sub> gas, are extremely difficult to drain, despite the extensive gas drainage drilling programme. Field studies on the use of sand-propped hydraulic fracturing failed to increase the gas drainage from such sites, although the technique was proven in other coal deposits as reported by Mills *et al* (2006). It is suggested that the highly stressed and low permeability coal is preventing carbon dioxide from being effectively drained.

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Little has been reported in literature on the application of nitrogen for carbon dioxide stripping from coal, particularly from underground coal mines, and accordingly a laboratory study was undertaken by the gas and outburst research group at the University of Wollongong, to examine the effect of displacing the adsorbed gases in coal with N<sub>2</sub> injection.

## EXPERIMENTAL PROCEDURE

### Equipment

To study the feasibility of removing or displacing the initially adsorbed gas from coal with another gas, tests were carried out using an in-house built multi-function outburst research rig (MFORR). This apparatus consists of a number of components which can be utilised in a variety of investigations, for example, it was initially built for the study of the influence of the gas environment on coal strength. The description and utilisation of the equipment have already been reported previously in various publications, Lama (1995), Aziz, Hutton and Indraratna (1996) and Aziz and Ming (1999). To reiterate, the integrated components of the MFORR include:

1. High pressure chamber, which has a load cell for measuring the load applied on the coal samples
2. Main apparatus support frame
3. Precision drill
4. Drill cutting collection system
5. Universal socket for vertical loading of coal sample in the gas pressure chamber
6. Flow meters
7. Gas chromatographer (GC)
8. Data acquisition System.

Figure 1 shows the schematic drawing of the apparatus. Figure 2 shows the general view of the apparatus. The gas pressure chamber is a rectangular prism of cast iron with removable front and back viewing plates. Its dimension is 110 mm x 110 mm x 140 mm. The viewing windows are made of 20 mm thick glass in a cast iron frame. Access to the chamber is possible by unbolting the front steel frame to the chamber. The chamber is made leak proof by inserting packers between the frame and the box as well as fitting O-rings around the loading shaft situated at the top of the chamber.

Housed in the chamber was a load cell with the capacity of 40 kN for monitoring the applied axial load. A pair of specimen loading plates with locating lips was used for holding a cylindrical specimen. Thus the main features of the MFORR with regard to coal gas sorption studies include:

- application of stress,
- application of gas suction,
- gas pressure confinement ( gas flooding),
- sample strain measurement, and
- gas flow rate measurement

Three flow meters connected in series were used to measure the flow characteristics of the escaping gas from the coal in the high pressure chamber. The flow rate range of different meters was 0-100 mL/min, 0-2 L/min, 0-15 L/min respectively. The composition of the discharged gases was measured by an on line GC.

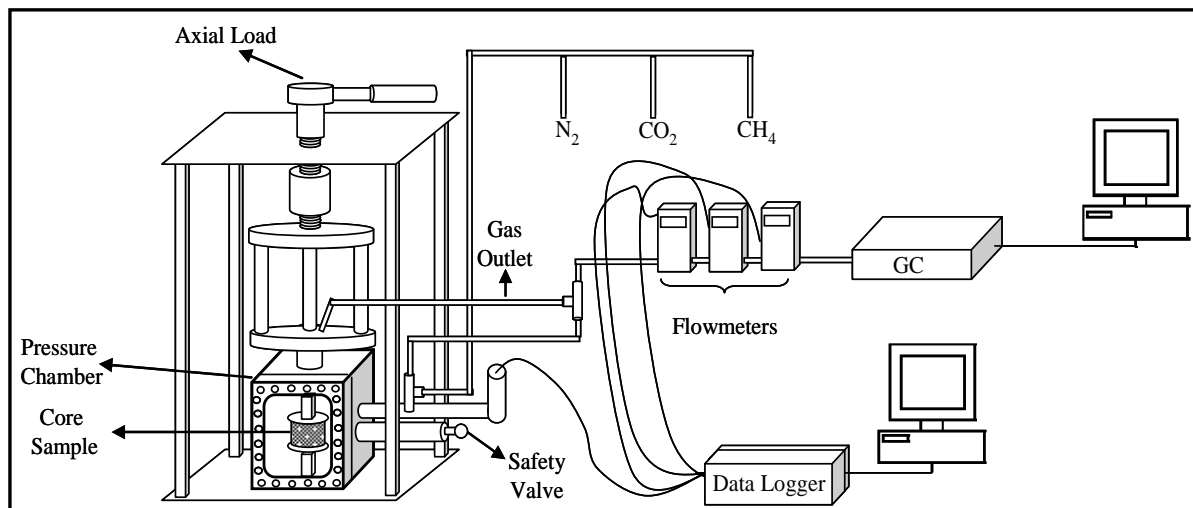


Figure 1 - Schematic view of experimental set-up.

### Sample Preparation

Coal samples used in this study were obtained from the Bulli Seam in Mine A. Bulk samples were taken from different locations along the longwall panel 519. Once collected the coal samples were sealed in plastic bags and transported to the University of Wollongong mine gas laboratory. In the laboratory the coal lumps were cut into regular and manageable sizes and immersed in water to minimise the effect of adverse climatic conditions.

Core samples (Figure 3) of 54 mm in diameter and height of 50 mm were then prepared for testing. The preparation of the coal core samples was carried out in accordance with the International standard for rock core sample preparation and testing (ISRM, 1981). A 2 mm diameter hole was then drilled through each sample for draining of the gas flowing through the coal core.

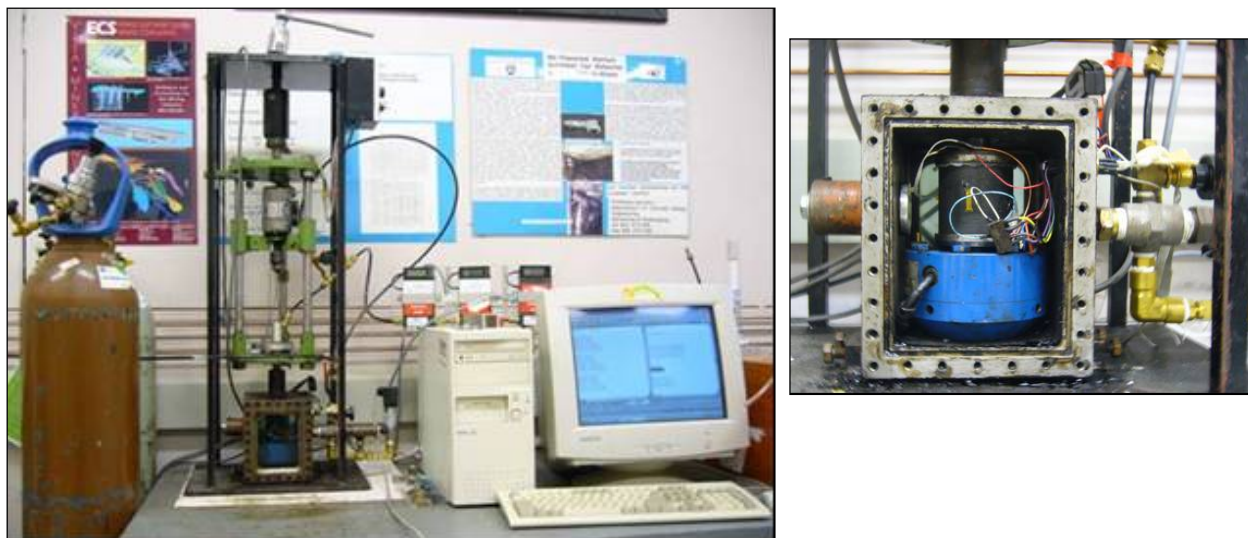
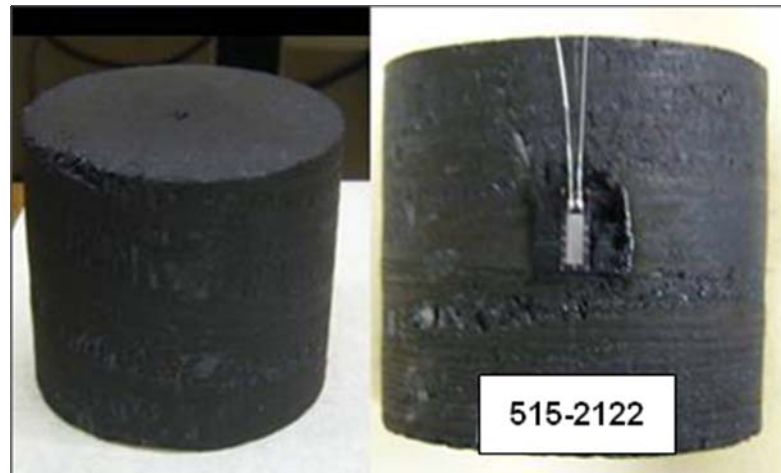


Figure 2 - A photograph of MFORR apparatus and closer view of the high-pressure gas chamber.



**Figure 3 - A representative sample used in tests.**

Prior to the sorption test, each coal core sample was fitted with both axial and circumferential strain gauges to monitor the volumetric changes in the sample during the gas sorption and desorption processes.

### TEST PROCEDURE

Each coal sample used for the injection study was placed in the high pressure gas chamber of the MFORR and sealed tight. In test 1, the sample was loaded axially to a predetermined initial axial loading of 200 kg which after saturation reached a 365 kg load. These loads were equivalent to a vertical stress of 0.87 MPa and 1.60 MPa respectively.

The general procedure used for testing was to initially saturate the coal sample with a specific gas (such as CH<sub>4</sub>) and then recharge the coal sample by injecting N<sub>2</sub>. The aim was to study the displacement characteristics of the initially saturated gas in coal. The applied confining gas pressure in the gas chamber was maintained constant at 3.2 MPa, thus creating a confining condition with the lateral to vertical pressures, acting on the coal sample, being in the order of 3.7:1 to 2:1 ratio. The maximum pressure ratio was marginally greater than the ground stress conditions in the Bulli Seam of the Southern coalfield of NSW.

All tests were carried under strict environmental and laboratory conditions. The room temperature was maintained constant at 22 °C throughout the experiment. This controlled experimental environment condition was considered useful with respect to coal bed methane production, carbon dioxide sequestration research, and for mine gas outburst control. Accordingly the following laboratory tests were carried out:

- Sorption and desorption characteristics of CO<sub>2</sub>/CH<sub>4</sub> mixed gas in coal (Test 1)
- Displacement characteristics of adsorbed mixed gases with N<sub>2</sub> injection (Test 3)
- Displacement characteristics of adsorbed CO<sub>2</sub> with N<sub>2</sub> injection (Test 4)
- Displacement characteristics of adsorbed CH<sub>4</sub> with N<sub>2</sub> injection (Test 5)

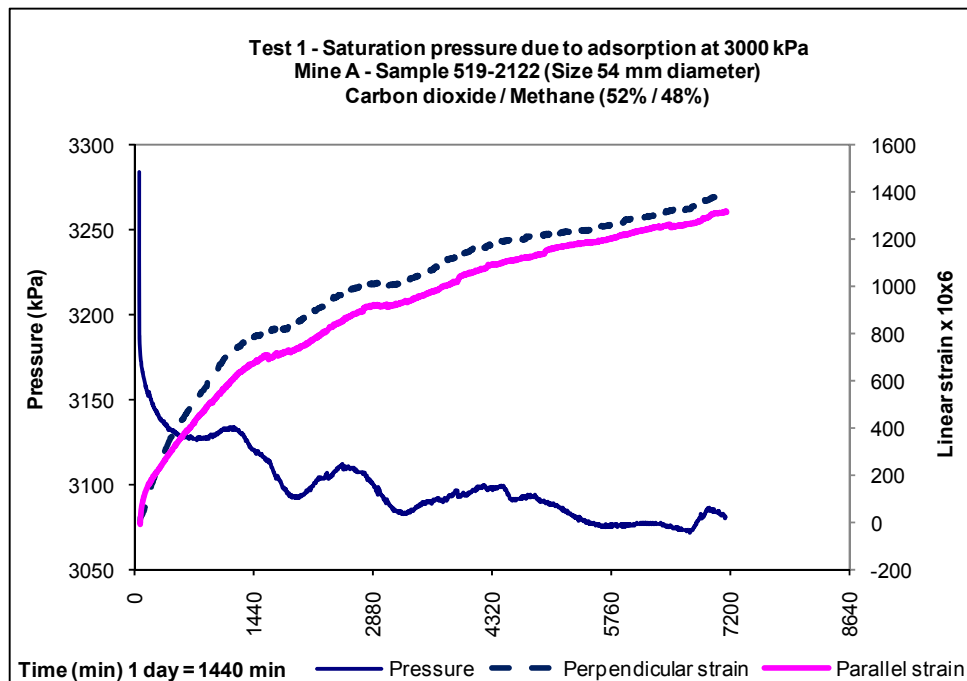
It is noted that sorption test 2 is not reported in this paper as the test was similar to test 1.

#### **Adsorption and desorption characteristics of CO<sub>2</sub>/CH<sub>4</sub> mixed gas in coal**

Prior to the N<sub>2</sub> injection test, a series of tests were carried out to examine the sorption (adsorption and desorption) behaviour of mixed gas (CO<sub>2</sub>/CH<sub>4</sub>) in coal when it is subjected to both axial and lateral confining pressures. During the first stage of the sorption test (Test 1) the coal sample was saturated with the mixed gas at a total pressure of 3.2 MPa. The gas saturation of coal was achieved by flooding the high gas pressure chamber initially with the mixed gas to the required pressure level. Figure 4 shows the fluctuation of gas pressure levels from the initial charged pressure level of about 3.2 MPa down to the final level of about 3 MPa over a period of around 5 days.

The pressure curve is a typical gas adsorption profile in coal with high micro porosity, which is translated to large volume of molecular structure (matrix). The initial CO<sub>2</sub>/CH<sub>4</sub> composition was in a 52/48 ratio. The coal saturation level was monitored according to the minimum duration time required for full saturation with this gas type, and as reported by Florentin *et al*, (2009). It is interesting to note from Figure 4 that the gas pressure fluctuates during the saturation period of the coal, the intermittent movement of the gas in and out of coal structure indicates that the coal sample is in a continuous state of gas adsorption and desorption. The amplitude of the gas sorption reduces as the coal nears its full saturation.

At the equilibrium point the percentage of molecules adsorbed is equal to the percentage of molecules desorbed. The amplitude of fluctuation levels in the adsorption profile show how far the gas molecules move from the macro to the micro pores in the coal structure. In the first sorption fluctuation step shown in the Figure 4, the coal sample is almost fully saturated mainly with carbon dioxide. The volumetric strain profile confirms clearly that the sample never stop from swelling. In the following fluctuation step, the amount of methane adsorbed is increased whereas the carbon dioxide component decreases until it is almost the same as the methane. However, in order to reach the equal proportions of the gases sorbed in coal, it is necessary to run the experiment much longer. This means that time is a deciding factor for the coal to adsorb methane in equal amount that carbon dioxide.



**Figure 4 - Variation in saturation pressure of adsorbed CO<sub>2</sub>/CH<sub>4</sub> (0.52:0.48) gas in coal with time.**

Figure 5 shows the effects of the room temperature on the confining gas pressure during the process of pressure equilibrium. Two thermocouples were located nearby the MFORR to monitor the room temperature periodically. The changes in temperature improve gas sorption (adsorption and desorption) in coal by varying the equilibrium pressure duration.

Figure 6 shows the changes in gas composition over time. The early part, of the released gas is due mostly to the confining chamber gas pressure and, the latter part is a combination of both the confining chamber gas and the desorbed gas from the coal sample. The initial gas composition of 52%/48% (CO<sub>2</sub>/CH<sub>4</sub>) ratio was changed to 51%/49% ratio during the first five days of coal sample saturation, and measured just prior to the release of the confining gas, which indicates a differential gas component adsorption in coal.

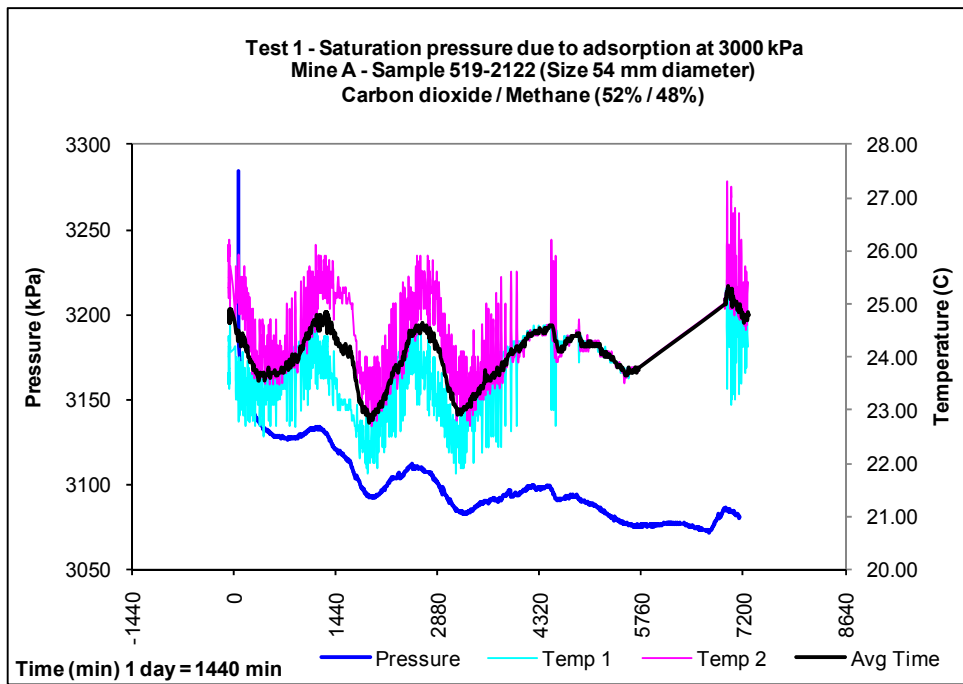


Figure 5 – Effect of the room temperature on confining gas pressure at saturation

The methane concentration is marginally higher than the carbon dioxide during the early stages of the adsorption process. Higher CH<sub>4</sub> concentration is attributed to the preferential adsorption of CO<sub>2</sub> in coal (CO<sub>2</sub> affinity to coal) and the fact that the marginal increase is due to relatively short sorption time. However, the mixed gas percentages returns to the original 52/48 level after some 30 min of gas confining pressure in the bomb being dropped down to around 500 kPa from the initial pressure level of about 3.2 MPa. The changes in mixed gas composition then reached a 56/44 ratio when the chamber pressure was almost at atmospheric level. Table 1 shows that at the initial desorption stage, CH<sub>4</sub> desorption was greater than that of CO<sub>2</sub>. However, at the end of the test, both CO<sub>2</sub> and CH<sub>4</sub> desorptions increase by a similar amount of almost 15%.

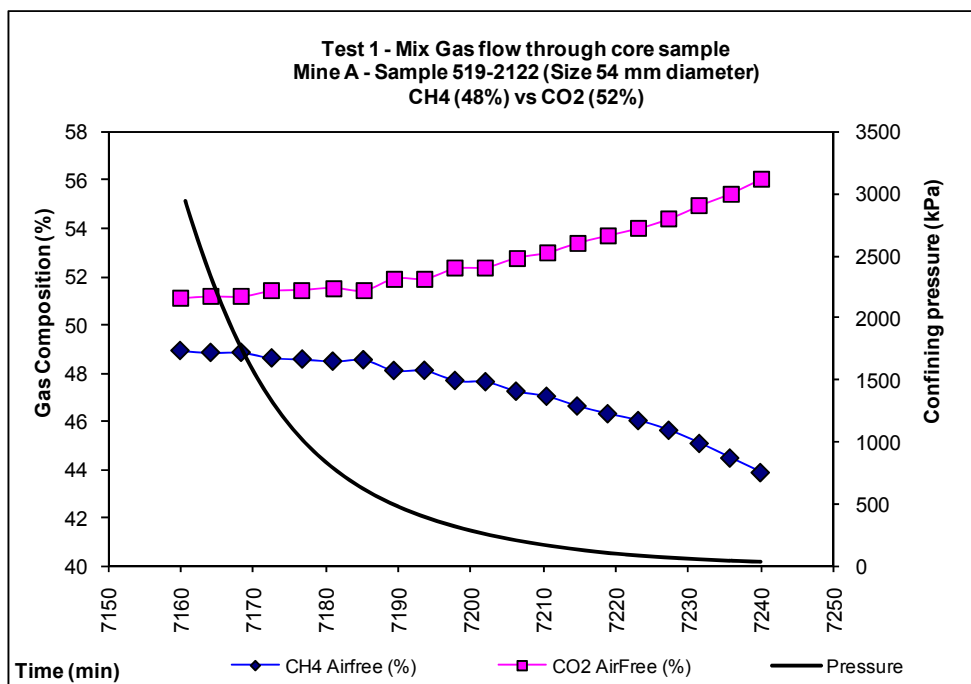


Figure 6 - Change in confining gas composition intermittently and pressure during Test 1

TABLE 1 - Gas composition in desorption (Test 1).

TEST 1 - Mixed gas					
% Desorbed			Period	Status	CO2/CH4 ratio
CO2	N2	CH4			
48.93%	-	51.09%	Start test		0.96
56.01%	-	43.88%	End test		1.28
14.47%	-	-14.11%	Improvement		

The gas composition measured at the GC is the sum of the confined and adsorbed gas. Confined gas refers to the combination of the free confining chamber gas, and the free gas in the cleat and fracture systems due to the flooded confined gas. At this short duration saturation level (about five days) it is difficult to predict accurately the composition of both adsorbed and free gases when released. Commonly, at high pressures the gas composition measured at the GC inlet is mostly from both free gases that pass through the coal sample. Most of the desorbed gas (from openings and matrices) is likely to be measured at low pressure as it takes a longer time to be released.

Figure 7 shows the decrease in both parallel and perpendicular strains to bedding planes, which is a clear indication that the coal sample volumetrically shrank as the coal sample starts to desorb its gas. The slope profile of the coal strain perpendicular to bedding decreases almost linearly due basically to the gas desorption. This was not the case of the strain parallel to bedding, which was increased while the confining pressure was reduced. Furthermore decreases in the axial load, confining pressure, and perpendicular strain gave a clear indication that gas was desorbing from coal. The changes in both perpendicular and parallel strains are due to the Poisson effect. The axial load falls because of the gas desorption which allows the coal sample to regain its initial structural shape. It is likely that coal shrinkage will occur mostly at the macro porosity level and very little at micro pores level. These volumetric changes occur particularly during the first 30 min of gas pressure drop, and until the confining pressure drops from around 3 000 kPa down to 500 kPa, which is the point where the parallel strain in desorption is maximum. After this point, the matrix desorption is significant with the confining pressure almost zero. Thus both perpendicular and parallel strains decrease due to the coal shrinkage mainly at molecular structure level

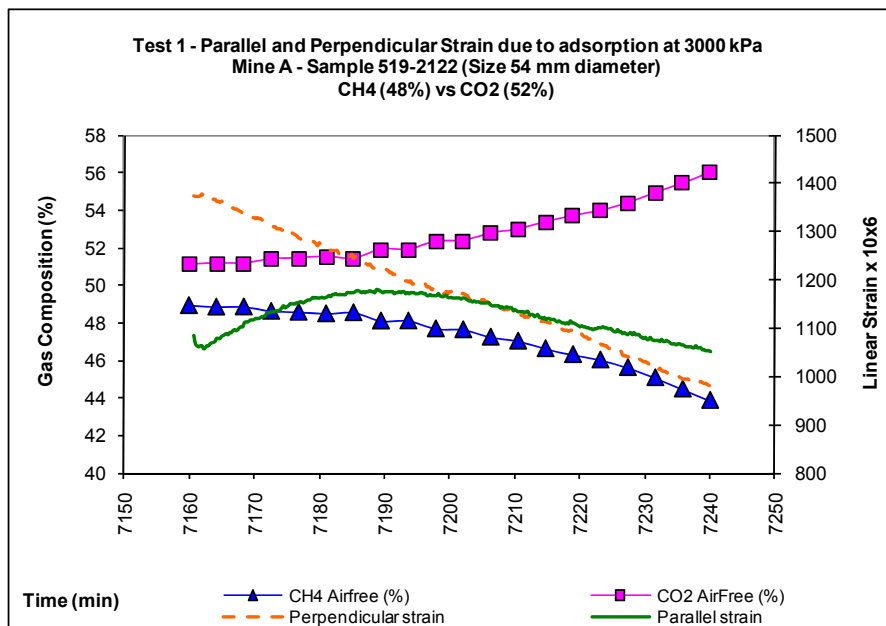


Figure 7 - Variation in gas compositions and coal sample deformation with time



Figure 8 shows the variations of the flow rate and confining gas pressures decay with sorption time. There is a strong gas pressure dependency on the process of gas movement. For the same reason similar gas behaviour will occur in the coal permeability profile.

Both the flow rate discharge and pressure decay occurred at higher rates particularly during the first 10 min of desorption time where the confining gas pressure dropped almost to a third of its value. This is because the tested coal sample was highly fractured at macro pore level. Similar results were obtained from other samples from the same location. At a pressure of 3.2 MPa, the maximum gas flow through the coal sample was about 2.3 L/min (3.3 m<sup>3</sup>/D) with maximum cleat permeability measured at around 11.7 mD.

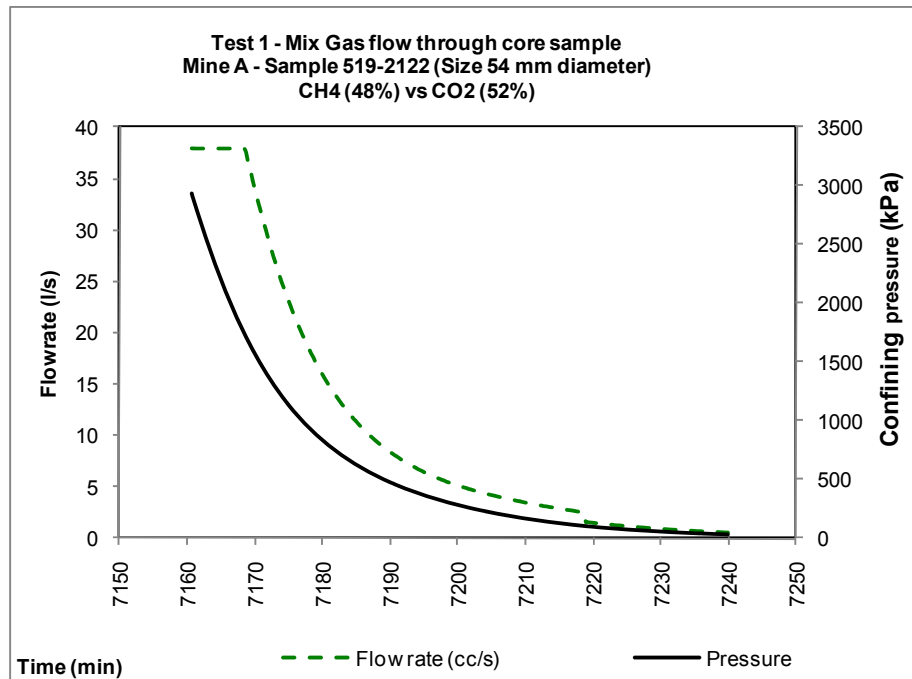


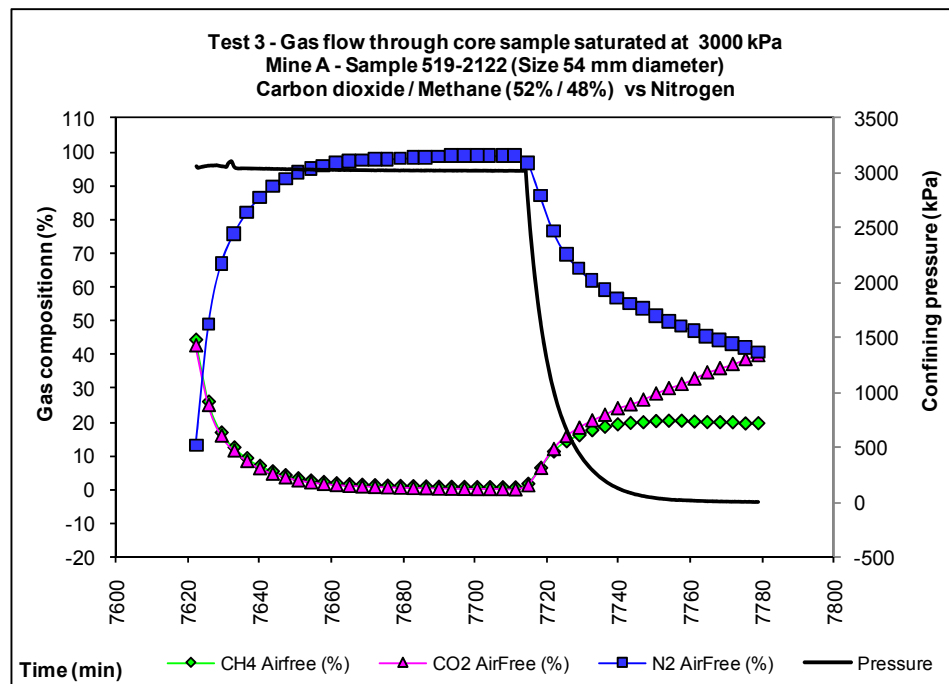
Figure 8 - Variation in flow rate and pressure during Test 1

## N<sub>2</sub> injection

In this part of the study, nitrogen gas was injected into the high pressure chamber housing the initially saturated coal sample used in the study for the purpose of improving the pressure gradient and the gas concentration gradient in the coal, and to examine the displacing influence of N<sub>2</sub> on the initially adsorbed gas, which is considered important for methane gas recovery from the coal and for carbon dioxide sequestration. Three gas types were used for the initial saturation phase; they were CO<sub>2</sub>/CH<sub>4</sub> (52/48) mixed gas, CO<sub>2</sub> and CH<sub>4</sub> respectively. The experimental procedure was carried out in two stages, referred as stage A and B. In Stage A, nitrogen gas was injected to a high pressure chamber housing the saturated coal sample with an initially adsorbed gas at a pressure of 3.2 MPa. The injected N<sub>2</sub> gas was maintained for a predetermined period until the confining nitrogen gas concentration as measured by the GC was almost 100%. As a result of N<sub>2</sub> the initial mixture gas composition was found to be reduced to almost zero.

Stage B commenced when N<sub>2</sub> gas ceases injection. The discharged gas (which is part free gas as a confining gas, and part as adsorbed gas) from the saturated coal samples was monitored for the rate of flow and gas composition by flow meters and gas chromatographer as described previously.

Figures 9, 10, 11 and 12 show the results of the N<sub>2</sub> injection tests carried out on coal samples initially saturated with different gases of CO<sub>2</sub>/CH<sub>4</sub> mixed, CO<sub>2</sub> and CH<sub>4</sub> respectively. The coal sample saturation periods in different initially charged gases were different. Thus, the duration period for each of mixed CO<sub>2</sub>/CH<sub>4</sub>, CO<sub>2</sub> and CH<sub>4</sub> gases were 5.3, 10.8 and 4.0 days respectively. These periods of initial charging were carried out just prior to the beginning of nitrogen injection.



**Figure 9 - Variation in gas composition and pressure in Test 3**

As seen in Figure 9 (Test 3), as soon as  $N_2$  was injected into the gas chamber in Stage A, its composition began to increase sharply. The rate of  $N_2$  increases occurred at the expense of the mixed gas  $CO_2/CH_4$ , and the near zero reduction in the composition of the  $CO_2/CH_4$  mixed gas occurred after 30 min of  $N_2$  injection. At the same time the latter gas concentration increased to almost 100%. The increased rate of mixed gas dilution was due to the large volume of  $N_2$  gas injection into the chamber at high gas pressure. The rate of the mixed gas decline was almost an opposite mirror image of the injected  $N_2$  gas increase, and as a result the combined confined gas pressure remained constant at 3.2 MPa. It is interesting to note that the mixed gas dilution ratio measured in every four min during the first 90 min of the  $N_2$  injection in Stage A was smaller than the initial 52/48 mixture ratio as shown in Figure 10. This is because  $CO_2$  gas was predominantly adsorbed in coal during the early stage of the saturation. Note that the released gas was mainly the free gas -confining gas or gas located on the macro pores. Once  $N_2$  gas injection was stopped, the mixed gas began to gradually reappear.

When  $N_2$  gas injection was stopped, it marked the beginning of stage B. The confining gas pressure dropped quickly, reaching almost zero after some 40 min of injection stoppage. During the same period the mixed gas composition level began to increase and some 20 min later the proportion of the discharged mixed gas component began to diverge with the rate of  $CO_2$  gas discharged reaching almost the double that of  $CH_4$ . The  $CH_4$  composition stabilised at 20 %, after some 30 min, while the  $CO_2$  increased to 40%. The  $CO_2$  desorption increases by almost 30% which was the double that those obtained in Test 1 (without  $N_2$  injection). Table 2 summarises the results of  $N_2$  injection.

Since the coal still retained some gas in its matrices, it is likely that these ratios could change, over longer periods of testing, especially for  $N_2$  and  $CO_2$ . It is worth remembering that in stage A the gases measured at the GC were mainly from free gases with a small amount being from desorbed gases, while in stage B it is supposed that the measured compositions were mainly from desorbed gases.

From Figures 9 it can be inferred that the  $CH_4$  composition is due to molecules desorbing at a steady rate from the coal matrices. However, the  $CO_2$  composition profile is more likely to be due to molecules desorbing from openings- cleats, cracks and fissures- which is the expected location for the adsorbed gas in a short period of adsorption time. The  $N_2$  desorption profile is mostly due to the free molecules passing through the core sample and is decreasing with pressure. In summary;  $N_2$  displaces  $CO_2$  basically due to the improvement of the concentration gradient, and the location of the adsorbed molecules. However,  $CH_4$  is apparently less affected by  $N_2$  injection for the same reasons. In a mixed gas adsorption, most of the  $CH_4$  is adsorbed in coal matrices, while the  $CO_2$  component is adsorbed mostly in the macro pores with a small amount being stored in coal matrices because of the so called  $CO_2$  affinity to coal. Thus  $CO_2$  prevails over methane in obtaining the sweetest spots available on the macro pores.

Table 2 - Gas composition in desorption (Test 3)

TEST 3 - Mixed gas with N2 injection					
% Desorbed			Period	Status	CO2/CH4 ratio
CO2	N2	CH4			
44.13%	13.23%	42.64%	Start test		
50.86%		49.14%	Start test	Free N2	1.03
39.74%	40.75%	19.50%	End test		
67.08%		32.92%		Free N2	2.04
31.90%		-33.02%	Improvement		

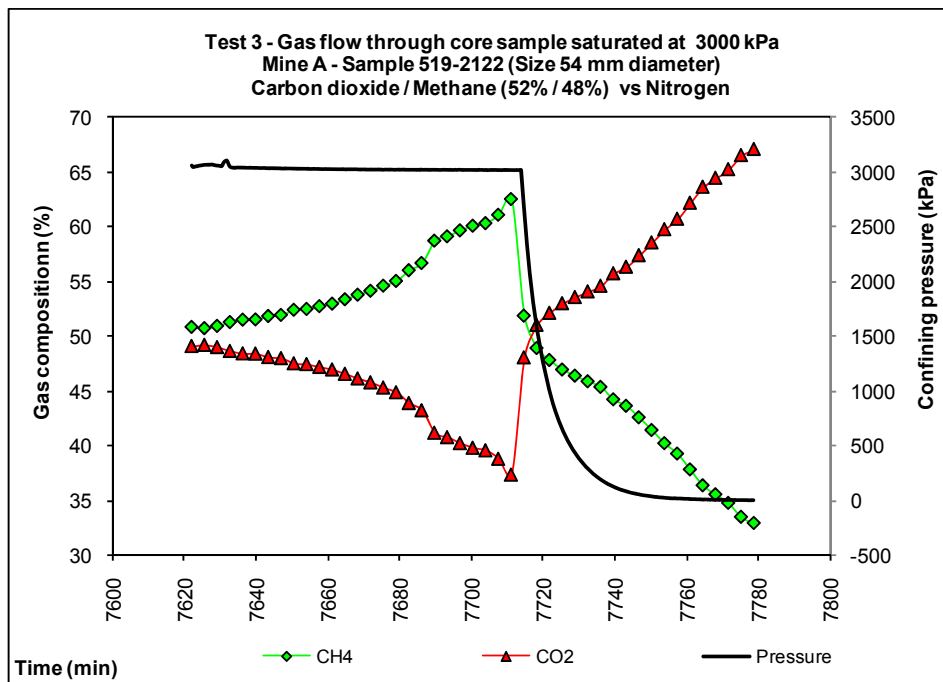


Figure 10 - Variation in gas concentration without the contribution of N<sub>2</sub> in Test 3

Figures 11 and 12 show the results of similar tests carried out separately with each of CO<sub>2</sub> (Test 4) and CH<sub>4</sub> gases (Test 5) respectively. As seen in Figure 11, N<sub>2</sub> was injected for about 110 min when full N<sub>2</sub> concentration was achieved. During the second phase (stage B), starting from 15 710 min, the CO<sub>2</sub> concentration began to increase sharply, from 0 to 50% in 20 min, and at the same rate that N<sub>2</sub> concentration dropped. Thus 50 % concentration point was achieved at the 15 730 min mark. The level of CO<sub>2</sub> concentration of the out-flowing gas passing through the GC approached 75% after 215 min of GC analysis and testing, which suggests that the injection of N<sub>2</sub> gas into coal appears to have a significant influence on CO<sub>2</sub> displacement and removal from coal. This level of N<sub>2</sub> influence on CO<sub>2</sub> is almost four fold greater than for CH<sub>4</sub>. During the early phase of stage B, the discharged N<sub>2</sub> gas was mostly free confining gas. Later, after the 15 800 min. the small amount of nitrogen released appears to be an adsorbed gas. Also any amount of the released carbon dioxide is more likely to be a desorbed gas because the initial pure CO<sub>2</sub> free confining gas was mostly diluted during the N<sub>2</sub> injection process (Stage A) and passed through the coal sample.

As can be seen from Figure 12 the removal of CH<sub>4</sub> was at best 20% which was achieved after 90 min of testing. Note that this methane composition was almost the same as that measured in Test 3. Hence the application of N<sub>2</sub> for methane recovery appears to be not a viable method in the current laboratory environment conditions. This finding has a significant bearing in solving the drainage difficulties experienced at West Cliff Colliery Panel 500 area, which has difficulty to drain CO<sub>2</sub> gas concentration zones.

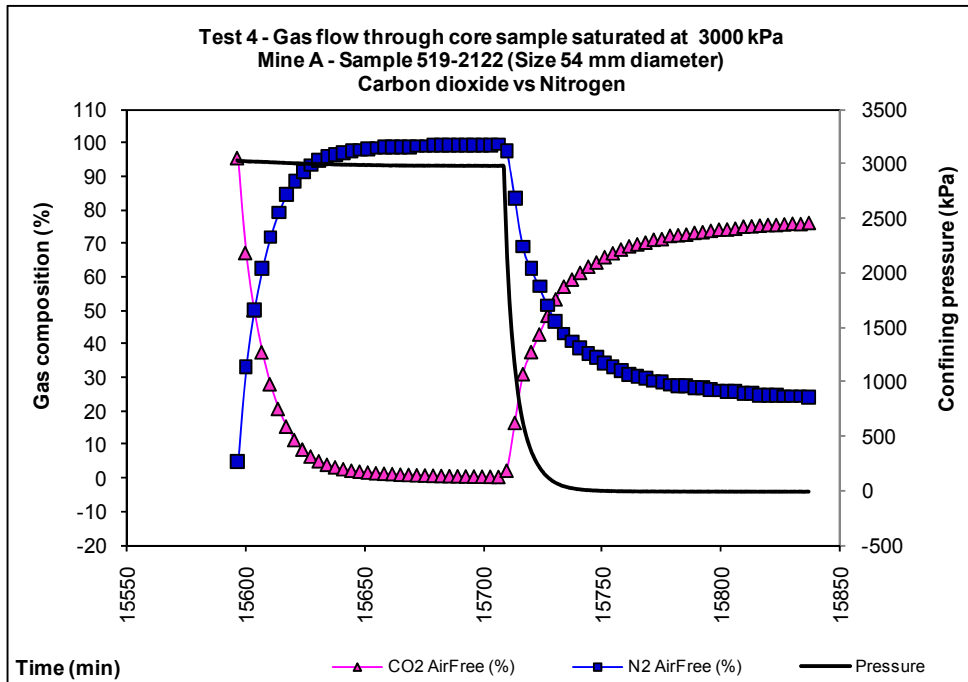


Figure 11 - Variation in gas concentration and pressure in Test 4

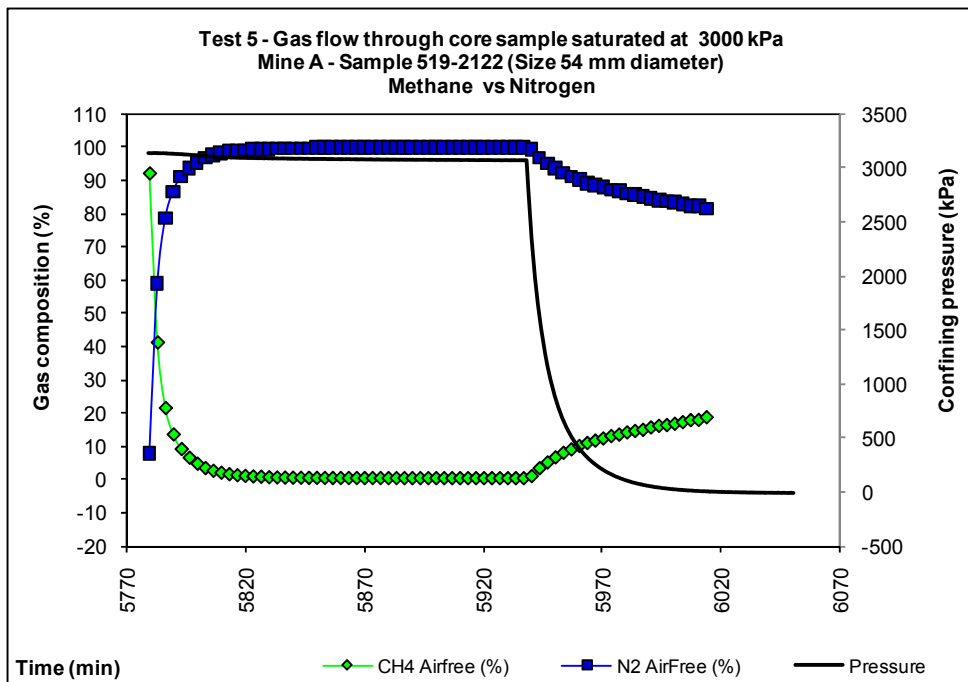


Figure 12 - Variation in gas concentration and pressure in Test 5

### CONCLUSIONS

The following were inferred from the experimental studies which were conducted under strict environmental and laboratory conditions;

1. Nitrogen appears to displace both carbon dioxide and methane; however the degree of displacement varies according the gas type. N<sub>2</sub> gas appears to displace CO<sub>2</sub> more than CH<sub>4</sub>.

2. Coal appears to adsorb greater CO<sub>2</sub> than CH<sub>4</sub> in a mixed gas adsorption. However, longer saturation duration improves CH<sub>4</sub> adsorption until it equals the CO<sub>2</sub> level.
3. Injection of N<sub>2</sub> gas resulted in changes in coal volume. These changes occur both perpendicular and parallel to coal layering/ bedding. However, and based on the results of this study, coal swelling occurred only axially when N<sub>2</sub> injection was stopped.
4. Coal shrinkage or swelling is attributed to the nature of gas sorption in coal joints, fissures and cracks as well as in the coal matrix.
5. On the basis of this study, N<sub>2</sub> injection has greater effect on CO<sub>2</sub> removal than on CH<sub>4</sub> removal. This finding has significant bearing in solving the drainage difficulties experienced in Panel 500 areas at West Cliff Colliery, which has with difficult to drain CO<sub>2</sub> gas concentration zones

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