Carbon Dioxide Seal Capacity, Carbine ESSCI, Browse Basin, Western Australia

Dr R. F. Daniel

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Executive Summary

This report details the results of MICP (Mercury Injection Capillary Pressure) analyses of samples of the Prudhoe Deltaics Member (Turnstone Formation) and the Borde Marl from Carbine 1 in the Carbine Ponded Turbidite ESSCI, Browse Basin.

Mercury injection capillary pressure data are used to determine threshold or breakthrough pressure used in the interpretation of the carbon dioxide retention height of sealing rocks. Pore throat size distributions are also calculated for the analysed samples and the laboratory mercury/air values are converted to subsurface supercritical carbon dioxide (scCO₂) values to determine saturation versus height relationships.

The supercritical carbon dioxide seal retention column for the Prudhoe Deltaics Member is 20m and for the Borde Marl Formation ranges from 43 to 53m. These analyses were carried out on dried unwashed bulk cuttings collected at the time of drilling and hence the shale cuttings may have experienced some degradation of shale structure resulting in lower than expected column heights.

XRD analyses showed that the Prudhoe Deltaics sample consisted of mostly dominant quartz, with kaolinite and smectite/illite, whereas the Borde Marl samples were dominantly calcite and quartz with kaolinite and smectite/illite.
Introduction

The Carbine Ponded Turbidite (ESSCI) in the Browse Basin is the subject of a study to determine the viability of storing super critical carbon dioxide within the Puffin Sandstone and cap sealed by the Borde Marl and Prudhoe Deltaics Member. Three samples were analysed at the Australian School of Petroleum, two being from the Borde Marl and one from the Prudhoe Deltaics Member.

Subsurface pressure, temperature and salinity data were obtained from the Carbine 1 well completion report. Carbon dioxide density, formation water density and interfacial tension were determined from this data, which enabled the calculation of maximum column height and plotting of super critical carbon dioxide saturation versus height and seal retention height.

An outline of mercury injection capillary pressure analysis and the determination of pore throat size and seal capacity is presented under Methodology. Table 1 provides primary details of the rock samples analysed.

Table 1: Depth and type of seal samples analysed for the Carbine ESSCI, Browse Basin, Northwest Shelf, WA.

<table>
<thead>
<tr>
<th>Well</th>
<th>Depth (m)</th>
<th>Fm; Core/ Cuttings/ SWC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbine 1</td>
<td>1070 - 1073</td>
<td>Prudhoe Deltaic Mbr; Cuttings</td>
</tr>
<tr>
<td>Carbine 1</td>
<td>1157 – 1160</td>
<td>Borde Marl Formation; Cuttings</td>
</tr>
<tr>
<td>Carbine 1</td>
<td>1250 - 1253</td>
<td>Borde Marl Formation; Cuttings</td>
</tr>
</tbody>
</table>
Methodology

Mercury Injection Capillary Pressure (MICP)

The theory of the mercury porosimeter is based on the physical principle that a non-reactive, non-wetting liquid will not penetrate pores until sufficient pressure is applied to force its entrance. The relationship between the applied pressure and the pore size into which mercury will intrude is given by the Washburn equation (1921):

\[ P_c D = 4 \gamma \cos \theta \]  

(1a)

More recently, however, this equation has developed into a more useful style for determining pore throat radius as suggested by Purcell (1949) and Schowalter (1979):

\[ P_c r = 2 \gamma \cos \theta \]  

(1b)

Where \( P_c \) is the applied capillary pressure, \( D \) is the pore throat diameter, \( r \) is the pore throat radius, \( \gamma \) is the surface tension of mercury (480 dyne cm\(^{-1}\)) and \( \theta \) is the contact angle between mercury and the pore wall, usually near 140°. This equation assumes that all pores are right circular cylinders. As pressure increases, the instrument senses the intrusion volume of mercury by the change in capacitance between the mercury column and a metal sheath surrounding the stem of the penetrometer. As the mercury column shortens, the pressure and volume data are continuously acquired by an attached computer.

Mercury porosimetry is a technique, which is rate limited, as predicted by the Darcy equation. This describes the general function of pressure drop vs. flowrate:

\[ \frac{\rho_1 - \rho_2}{L} = \frac{\alpha \mu N}{g_c} \]  

(2)

Where \( \rho_1 \) is the upstream pressure, \( \rho_2 \) is the downstream pressure, \( L \) is the pore length, \( 1/\alpha \) is the permeability coefficient, \( \mu \) is the fluid viscosity, \( V \) is the superficial velocity of fluid and \( g_c \) is a dimensional constant. The velocity of flow in a viscous liquid such as mercury is proportional to the pressure drop and inversely proportional to the length and surface area of the pore. Hence, given a specific limited flow velocity, the complete filling of a porous network will be a function of time. The
larger the volume of pores the more time is required to fill the total pore volume completely. Therefore mercury porosimetry is most accurate when mercury is allowed to fill all the available pores, at equilibration.

The mercury injection porosimetry analyses for this study were carried out using a Micromeritics Autopore 9410 instrument. This is composed of two separate systems, one for low pressure runs and the second for the high pressure runs. The low pressure run must always be done first, followed quickly by the high pressure run, to preclude the possibility of extra mercury intrusion into the sample by capillary action while the sample is held at atmospheric pressure at the conclusion of the low pressure run.

The system operates using the equilibration by time method - after the required pressure for a reading is attained it is held for twenty seconds to allow the amount of mercury entering the pores to stabilise. This is done because the process of mercury filling the pores is not an instantaneous one. Mercury begins entering the pores as soon as the pressure exceeds the value required for the pore throats' diameter, but the time required to fill the pores depends on the volume and shape of the pores. The equilibration by time process allows the pores to fill. If equilibration is not allowed, then the filling may not be complete when the reading is taken, which leads to estimation of lower pore volumes and smaller pore sizes than is actually the case. Readings of mercury intrusion are taken by measuring the electrical capacitance of the penetrometer. This varies as the mercury is intruded from the precision bore stem into the pore space of the sample by the increasing pressure (see Graph (A) of each sample analysis).

Each sample is usually dried at 60°C for at least twenty four hours, weighed and placed into a penetrometer (a glass chamber attached to a precision bore glass tube, which has been nickel-plated) and the entire assembly is weighed. This is placed in the low pressure port and evacuated to 0.05 torr. This vacuum is held for thirty minutes to ensure that no vapour remains in the sample. After this time the penetrometer is filled with mercury and the low pressure run is carried out. The pressure is increased incrementally from 13.8 kPa (2 psia) to 199.5 kPa (28.94 psia), with a reading taken after 20 seconds of equilibration at each pressure. At the end of the low pressure run the penetrometer returns to atmospheric pressure. It is removed from the instrument and weighed to obtain its weight plus that of the mercury.

The penetrometer is then placed in the high pressure chamber, which uses hydraulic oil to take the pressure incrementally from 199.5 kPa (28.94 psia) to 413.7 MPa (60,000 psia). Again readings are taken after a twenty second equilibration period (see Graph A of the MICP analyses). The pressure is then decreased incrementally from 413.7 MPa (60,000 psia) to 139kPa (20 psia), with readings taken after the equilibration period. The sample is removed from the penetrometer and weighed. The specific gravity and porosity of the sample can be calculated when it is removed from the penetrometer and weighed.
Pore Throat Size Distribution

Using the following data for the air mercury system from (Vavra et al, 1992), capillary pressure data were converted to effective pore throat size:

Air/mercury contact angle \((\theta_{a/m}) = 140^\circ\),

Interfacial tension \((\sigma_{a/m}) = 481\) dyne/cm,

e.g. \(\sigma_{a/m} \cos \theta_{a/m} = 367.7\)

Solving for \(r\) in equation (1b) results in the following relationship of capillary pressure to pore throat size:

\[
1\ \text{psi} \approx 100\ \mu\text{m};\ 10\ \text{psi} \approx 10\ \mu\text{m};\ 100\ \text{psi} \approx 1\ \mu\text{m};\ 1000\ \text{psi} \approx 0.1\ \mu\text{m}\ (100\ \text{nm})
\]

Graph (B) of each analysis represents the distribution of pore throat size and from this the critical pore throat size can be determined.

Determination of Seal Capacity

MICP studies done solely for the purpose of determining pore throat radius and pore throat size distribution are valuable. However, there is great benefit in using the MICP analytical data to relate the mercury saturation of the sedimentary rock to the intruding pressure as a function of height above the free water level (FWL). This function indicates the height of a CO\(_2\) column that a sealing rock will support. The process of determining column height is described below.

Before the mercury injection data can be applied to seal capacity they must be converted to a subsurface CO\(_2\)/brine system using the following equation (after Schowalter, 1979):

\[
P_{b\text{CO}_2} = P_{a/m} \left( \frac{\sigma_{b\text{CO}_2} \cos \theta_{k/b\text{CO}_2}}{\sigma_{a/m} \cos \theta_{a/m}} \right)
\]  

(3)

Where \(P_{b\text{CO}_2}\) is the capillary pressure in the brine/CO\(_2\) system, \(P_{a/m}\) is the capillary pressure in the air/mercury system, \(\sigma_{b\text{CO}_2}\) and \(\sigma_{a/m}\) are the interfacial tensions of the brine/CO\(_2\) and the air mercury systems respectively, \(\theta_{k/b\text{CO}_2}\) and \(\theta_{a/m}\) are the contact angles of the brine/CO\(_2\)/solid and air/mercury/solid systems respectively.
The role of wettability (contact angle) and interfacial tension (IFT) in determining column height is significant and in the petroleum industry these parameters are well known both experimentally and practically. The role of wettability, in the geological storage of carbon dioxide, is not well known with little published research available.

Recent experimental data by Chiquet and Broseta, (2005) show that quartz and mica (as test minerals for fine grained rocks) under low pressures, become less water wet in the presence of scCO₂ i.e. contact angles vary from 0° to 20° for mica (a clay proxy, having a similar structure to clay) and 20° to 30° for quartz. Under higher pressures the contact angle increases to 60° - 80° for mica and 40° to 55° for quartz. These experiments were carried out above the substrate and when subsequently carried out below the substrate the angles were 10-15° less at low pressure with negligible differences at high pressures (10MPa).

The experiments were carried out on single mineral plates rather than a shaly surface. Experimental studies on shaly surfaces have not been reported. As a result of this study it appears that contact angle sensitivities (0° to ~60°) need to be used to determine the range of carbon dioxide column heights that can be held back by a given seal.

The initial pressure at which the mercury first displaces the air at the surface pores is referred to as the "entry pressure" (Pe). In the subsurface, buoyancy pressure drives CO₂ (the non-wetting phase) movement and forces it into the pore throats of a rock, displacing water (the wetting phase). Buoyancy is simply the density difference between CO₂ and brine multiplied by the column height and a constant (k) gravitational factor, which is 0.433. The greater the column thickness, the greater the buoyancy pressure driving the CO₂.

Pe in the reservoir system is the pressure at which CO₂ first entered the pore system by displacing the water. Different rocks with different pore throat sizes will have different displacement pressures and different saturations as a function of height (h) above the free water level (FWL). Thus, in any given reservoir section, the lowest indication of live (vs. residual) CO₂ in a particular rock type approximates the entry pressure (Pe) for that rock. The Pe can thus be considered as the CO₂ – water contact for that particular rock type. It should be remembered that a reservoir may have several CO₂ water contacts (as a function of pore properties controlled by rock type), but will have only one FWL. It is therefore of significance to determine the FWL.

In order to do this, capillary pressure data must first be converted to height above free water level information by using the equation:

$$P_{cb/co2} = h (\rho_b-\rho_{co2}) \times 0.433$$  \hspace{1cm} (4)

Where: $P_{cb/co2}$ = Capillary Pressure (psi) reservoir brine/CO₂ system

$h$ = height (in ft.)
\[ \rho_b = \text{brine density (gm/cc)} \]

\[ \rho_{co2} = \text{CO}_2 \text{ density (gm/cc)} \]

**Browse Basin Subsurface fluid properties:**

Super critical \(\text{CO}_2\) Density - 0.65gm/cc (variable)

Brine Density- variable in the range of 1.03 to 1.07 gm/cc.

Interfacial Tensions were determined from the CO2CRC web-site carbon dioxide calculator (see spreadsheet of parameters for each well). Generally the interfacial tension for brine/\(\text{CO}_2\) is approximately 25 dynes/cm and the contact angle is 0° (with included sensitivities).

Once the capillary pressure values have been converted to \(h\) (height in metres), a plot of height versus mercury (non-wetting phase) saturations can be constructed. However, conversion of mercury (non-wetting phase) to \(\text{CO}_2\) (non-wetting phase) will now result in a height versus \(\text{CO}_2\) saturation plot. Since water (wetting phase) saturation is more commonly used in the oil and gas industry, the non-wetting phase saturation needs to be converted to water (wetting-phase) saturation (Schowalter, 1979). This is done using the simple conversion:

\[ Sw = 1 - Snw \]  \hspace{1cm} (5)

Where: \(Sw = \text{wetting phase (water) saturation} \)

\(Snw = \text{non-wetting phase (CO}_2\) saturation}\)

A plot of height (above FWL) versus water saturation results from combining equations (4) and (5) as highlighted in the third graph (C) shown of each sample in Appendix 2. This graph indicates the water saturation at varying column heights, which aids in determining relative permeability.
Results

The results of MICP analysis of Carbine-1 samples from the Prudhoe Deltaics Member (Turnstone Formation) and the Borde Marl indicate the carbon dioxide retention columns of 20 m (Prudhoe Deltaics Mbr) and an average of 48 m (Borde Marl). The two samples from the Borde Marl were 43 m and 53 m (Table 2). The XRD analyses showed that the Prudhoe Deltaics sample consisted of mostly dominant quartz, with kaolinite and smectite/illite, whereas the Borde Marl samples were dominantly calcite and quartz with kaolinite and smectite/illite. Trace amounts of mud chemicals were also present in the samples.

CO2 column height sensitivities have been calculated from 0º to 60º in 20º increments indicating increasing wettability with a commensurate decrease in column height. This sensitivity is based on the experimental evidence of Chiquet and Broseta (2005) (see above) who showed that scCO2 is partially wetting with respect to quartz and mica rocks under subsurface conditions. The maximum sensitivity contact angle of 60º shows column heights varying from 10 m to 26 m (Table 2).

Table 2: Primary and derived data used to determine the carbon dioxide column height from cuttings samples, Browse Basin, WA.

<table>
<thead>
<tr>
<th>Well Name</th>
<th>Depth of sample (m TVDSS)</th>
<th>Pressure at sample depth (MPa)</th>
<th>Temp at sample depth (ºC)</th>
<th>Salinity at sample depth (ppm)</th>
<th>CO2 density (g/cm³)</th>
<th>Interfacial tension (dyn/cm)</th>
<th>Contact Angle (º)</th>
<th>Seal Threshold Pressure (bar N system) (psi)</th>
<th>Reservoir Threshold Pressure (bar N system) (psi)</th>
<th>Seal Threshold Pressure (bar N system) (psi)</th>
<th>Reservoir Threshold Pressure (bar N system) (psi)</th>
<th>Height of CO2 Column (ft)</th>
<th>Height of CO2 Column (m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbine 1 1076-1073</td>
<td>10.98</td>
<td>49.50</td>
<td>6000</td>
<td>0.5277</td>
<td>1.0078</td>
<td>26.44</td>
<td>0</td>
<td>199.00</td>
<td>6.00</td>
<td>14.31</td>
<td>0.36</td>
<td>84</td>
<td>26</td>
</tr>
<tr>
<td>Carbine 1 1157-1160</td>
<td>11.84</td>
<td>53.40</td>
<td>5000</td>
<td>0.5174</td>
<td>1.0063</td>
<td>25.67</td>
<td>0</td>
<td>413.00</td>
<td>5.00</td>
<td>29.96</td>
<td>0.36</td>
<td>140</td>
<td>43</td>
</tr>
<tr>
<td>Carbine 1 1250-1253</td>
<td>12.79</td>
<td>57.20</td>
<td>6000</td>
<td>0.5209</td>
<td>1.0045</td>
<td>26.90</td>
<td>0</td>
<td>600.00</td>
<td>6.00</td>
<td>36.68</td>
<td>0.37</td>
<td>173</td>
<td>53</td>
</tr>
</tbody>
</table>

CO2 column height sensitivities have been calculated from 0º to 60º in 20º increments indicating increasing wettability with a commensurate decrease in column height. This sensitivity is based on the experimental evidence of Chiquet and Broseta (2005) (see above) who showed that scCO2 is partially wetting with respect to quartz and mica rocks under subsurface conditions. The maximum sensitivity contact angle of 60º shows column heights varying from 10 m to 26 m (Table 2).
Figure 1a: Carbon dioxide retention heights for samples from Carbine 1 (linear). Contact angle sensitivities from 0° to 60° are included, based on experimental work by Chiquet and 2005

Figure 1b: Carbon dioxide retention heights for samples from Carbine 1 (lognormal). Contact angle sensitivities from 0° to 60° are included, based on experimental work by Chiquet and 2005
Discussion and Conclusions

The results of MICP analysis of samples from the Carbine Ponded Turbidite ESSCI show that the calculated carbon dioxide column heights are fairly low (column heights from 20 m to 53 m) when compared with what could be expected from the reported lithologies in the Carbine No.1 well report. Overall, the condition of the cuttings showed a significant degree of softness and friability indicating a relaxation of integrity or ‘weathering’ time. Unfortunately, this characteristic is very difficult to determine prior to MICP analysis.

This process appears to be a result of the shale cuttings absorbing atmospheric water into the clay structure with a commensurate expansion and degradation of the cuttings from the time the samples are collected to when they are analysed. This process is probably exacerbated by the smectite content of the samples, which tends to absorb water over time resulting in an expansion of the rock.

Cuttings that are washed and dried at the time of drilling invariably retain a higher degree of integrity than unwashed cuttings slowly drying in the presence of drilling fluid at the drill site. Unfortunately the Carbine-1 cuttings were unwashed bulk samples, which mean the determined column heights probably represent minimum heights. It could be that analysis on fresh core/ cuttings would be significantly higher than presently indicated.

To determine whether the column heights have been affected by aging, it would be useful to perform MICP analyses on either sidewall cores (SWC) or washed and dried cuttings from more recent wells within the Carbine ESSCI.


Appendix

Mineralogy (X-ray Diffraction)

Table 3: Semi quantitative mineralogy of the Carbine ESSCI cap seal samples

<table>
<thead>
<tr>
<th>Mineral</th>
<th>Carbine-1 1073</th>
<th>Carbine-1 1160</th>
<th>Carbine-1 1253</th>
</tr>
</thead>
<tbody>
<tr>
<td>Clays; Smectite, Illite</td>
<td>M</td>
<td>M</td>
<td>M</td>
</tr>
<tr>
<td>Quartz</td>
<td>D</td>
<td>CD</td>
<td>CD</td>
</tr>
<tr>
<td>Kaolinite</td>
<td>M</td>
<td>M</td>
<td>M</td>
</tr>
<tr>
<td>Muscovite</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Plagioclase</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Calcite</td>
<td>Tr-M</td>
<td>CD</td>
<td>CD</td>
</tr>
<tr>
<td>Barite – mud chem</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Halite – mud chem</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sylvite – mud chem</td>
<td>Tr</td>
<td>Tr</td>
<td>Tr</td>
</tr>
<tr>
<td>Gypsum – mud chem</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pyrite</td>
<td></td>
<td>Tr</td>
<td></td>
</tr>
<tr>
<td>Siderite</td>
<td></td>
<td>Tr-M</td>
<td></td>
</tr>
</tbody>
</table>

Table 4: Approximate mineral percentages

<table>
<thead>
<tr>
<th>Notes:</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>D - Dominant</td>
<td>&gt;60%</td>
</tr>
<tr>
<td>CD - Co-dominant</td>
<td>sum of components &gt;60%</td>
</tr>
<tr>
<td>SD - Sub-dominant</td>
<td>20-60%</td>
</tr>
<tr>
<td>M - Minor</td>
<td>5-20%</td>
</tr>
<tr>
<td>T - Trace</td>
<td>&lt;5%</td>
</tr>
</tbody>
</table>
Figure 2: XRD Carbine-1 1073m

Figure 3: XRD Carbine 1 1160m
Figure 4: XRD Carbine-1 1253m

<table>
<thead>
<tr>
<th>k</th>
<th>kaolinite</th>
</tr>
</thead>
<tbody>
<tr>
<td>cl</td>
<td>chlorite</td>
</tr>
<tr>
<td>m</td>
<td>montmorillonite/smectite</td>
</tr>
<tr>
<td>i</td>
<td>illite</td>
</tr>
<tr>
<td>kf</td>
<td>K-feldspar</td>
</tr>
<tr>
<td>q</td>
<td>quartz</td>
</tr>
<tr>
<td>ca</td>
<td>calcite</td>
</tr>
<tr>
<td>s</td>
<td>sylvite/mud chem</td>
</tr>
</tbody>
</table>

Counts

Degrees 2 Theta

Carbine-1 1253m

m, cm, k, cl, m, i, kf, cl, k, s, ca, q, ca, s
Graphs of (A) Injection Pressure vs. Hg Saturation/Incremental Pore Volume, (B) Pore Throat Size vs. Frequency and (C) CO$_2$ Seal Capacity above FWL vs. Water Saturation

Figures 5a - 5c: MICP Data for Carbine-1 1073m
Figures 6a - 6c: MICP Data for Carbine-1 1160m

A

Carbine-1 1160m

Mercury Saturation, %

Injection Pressure, psia

B

Carbine-1 1160

Incremental Pore Volume, %

Distribution of Pore Throat Size, microns
Carbine-1 1160m

Maximum CO₂ Column Height - 43m
Minimum CO₂ Column Height - 21m

Water Saturation, %

Height above FWL, m

CA - 0  CA - 20  CA - 40  CA - 60
Figures 7a – 7c: MICP Data for Carbine-1 1253m

A

Carbine-1 1253m

注射压力, psia

汞饱和度, %

B

Carbine-1 1253m

分布于喉径大小, microns

增量孔体积, %
Maximum CO₂ Column Height - 53m
Minimum CO₂ Column Height - 26m
an emission free vision for the future

Contact CO2CRC

Canberra

Dr Peter Cook, Chief Executive
GPO Box 463, Canberra, ACT 2601
Ph: + 61 2 6120 1600  Fax: +61 2 6273 7181
Email: pcook@co2crc.com.au

Ms Carole Peacock, Business Manager
GPO Box 463, Canberra, ACT 2601
Ph: + 61 2 6120 1605  Fax: +61 2 6273 7181
Email: cpeacock@co2crc.com.au

Ms Carmel Anderson, Communication Manager
GPO Box 463, Canberra, ACT 2601
Ph: + 61 2 6120 1607  Fax: +61 2 6273 7181
Email: canderson@co2crc.com.au

Adelaide

Dr John Kaldi, Chief Scientist
Australian School of Petroleum,
The University of Adelaide, SA 5005
Ph: + 61 8 8303 4291  Fax: +61 8 8303 4345
Email: jkaldi@co2crc.com.au

Perth

Mr Sandeep Sharma, Pilot Project Manager
PO Box 1130, Bentley Western Australia 6102
Ph: +61 8 6436 8736  Fax: +61 8 6436 8555
Email: ssharma@co2crc.com.au

Mr David Hilditch, Commercial Manager (CO2TECH)
PO Box 1130, Bentley, Western Australia, 6102
Ph: + 8 6436 8655  Fax: +61 8 6436 8555
Email: dhilditch@itcpl.com.au

Melbourne

Mr Barry Hooper, Chief Technologist
University of Melbourne, Dept. of Chemical & Biomolecular Engineering, Melbourne, 3010
Ph: +61 3 8344 6622  Fax: +61 3 8344 4153
Email: bhooper@co2crc.com.au
reducing carbon dioxide emissions to the atmosphere