**Summary**

Despite the recent dip in oil prices, the inertia of the shale gas boom will continue to move forward and as prices normalize and industry adjusts, the production of shale reservoirs will continue. One of the keys to understanding these reservoirs and maximizing their output is a better measurement of the natural fractures in the rock and how these fractures affect porosity. Porosity is the single most important petrophysical property. For shale reservoirs it is also critical to understand the natural fractures in the rock. A key indication of the quantity of the natural fractures can be obtained by measuring the amount of porosity contained in the fracture network. Shales have very small pores and therefore very short NMR \( T_2 \) relaxation times. The fractures are typically larger than the pores and therefore have longer \( T_2 \) relaxation times. In this work, we describe and demonstrate techniques using NMR that can obtain not only the total and effective porosity of shale samples but can also quantify the fracture porosity. Simple NMR measurements of the \( T_2 \) relaxation time were performed at different confining pressures to quantify the porosity loss as confining stress increases. This loss in porosity is interpreted as closing of the natural fractures in the rock as shale is not typically compressible at these pressures. The type and quantity of the fluid present in the shale is also of great importance. Bitumen (if present) can impact fracturing plans and production models. The relaxation times \( T_1 \) and \( T_2 \) are known to be affected by the viscosity of the fluid [1]. We can therefore quantify bitumen present by measuring \( T_1-T_2 \) maps at different temperatures, as the bitumen viscosity will change with temperature. \( T_1-T_2 \) maps can also be used to quantify the amount of water present, as \( T_1 \) and \( T_2 \) are similar for water in shale. The results of this \( T_1-T_2 \) mapping from different shale samples at different temperatures will also be presented and discussed.

**Introduction**

NMR has proven to be a valuable tool in conventional and unconventional oil and gas reservoir characterization. It is used in both logging and core analysis applications. Unconventional reservoir development in recent years has instigated an increase in NMR applications as conventional methods often fall short for proper characterization of unconventional reservoirs. The most commonly used NMR measurement in the oil and gas industry is the \( T_2 \) relaxation measurement of the fluids that are in the rock sample. If the sample is fully saturated, this can be a way to measure porosity since the measurement can quantify the amount of fluid detected. Additionally, \( T_2 \) relaxation of a fluid in the pore is related to pore size as follows:

\[
\frac{1}{T_2} = \frac{1}{T_2\text{-Bulk}} + \rho S V
\]

(1)

Where \( S/V \) is the surface to volume ratio of the pore, \( \rho \) is the relaxivity parameter and \( T_2\text{-bulk} \) is the \( T_2 \) relaxation time of the fluid.
In this study, $T_2$ measurements were used to measure total porosity of fully saturated shale samples at different confining pressures. Since most shale formations host more than one pore size network, $T_2$ relaxometry was also used to distinguish the pore networks and monitor effects of confining pressure to particular pore sizes.

New NMR techniques, such as $T_1$-$T_2$ maps, have been developed for fluid typing in shales [2]. $T_1$-$T_2$ ratio of a fluid is related to its viscosity, the higher the ratio the higher the viscosity [2], as shown in Figure 1. This idea was used to detect and quantify fluids (water and bitumen) in “as received” shale samples subject to different temperatures.

![Figure 1: Effect of viscosity on $T_1$ and $T_2$](image)

**Figure 1: Effect of viscosity on $T_1$ and $T_2$**

**Experiment**

Four shale samples from four different formations were used in the first part of this experiment (see Table 1 for sample details). Sample photographs are shown in Figure 2. Samples were saturated with 2% KCl brine by first applying a vacuum to samples for about one hour, back filling with brine for about an hour, and then subjecting them to 10000 psi of pressure in a Phoenix Instruments pressure vessel for about a week. NMR data was acquired using an Oxford Instruments GeoSpec 2-53 rock core analyzer [3] equipped with an Oxford Instruments P5 overburden NMR probe [4]. Confining pressure was provided by pressurizing the confining space of the P5 probe with Flourinert. NMR measurements were conducted at the following confining pressures: 0 psi, 1000 psi, 2000 psi, 3000 psi, 4000 psi and 5000 psi. NMR data acquisition and processing was attained with use of Green Imaging Technologies software [5]. Using $T_2$ NMR data, porosity and pore size distribution was determined at the different confining pressures. $T_1$-$T_2$ data was used for bitumen quantification. Data was acquired on two shale samples (see Table 1 samples DF-1 and DF-2 for details). A 40 mm NMR probe with no confining pressure was employed, while temperature was controlled by circulating hot air around the samples.
Table 1: Characteristic Information On Shale Samples Tested

<table>
<thead>
<tr>
<th>Shale</th>
<th>Formation</th>
<th>Sample Depth (ft)</th>
<th>Core Diameter (cm)</th>
<th>Core Length (cm)</th>
<th>Bulk Volume (cm$^3$)</th>
<th>Dry Core Mass (g)</th>
<th>Grain Density (g/cc)</th>
<th>Gas Permeability (mD)</th>
<th>He Porosity (p.u.)</th>
<th>Well Location</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Gates</td>
<td>1392</td>
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<td>3.8</td>
<td>18.7</td>
<td>51.68</td>
<td>2.74</td>
<td>4.28</td>
<td>5.9</td>
<td>British Columbia</td>
</tr>
<tr>
<td></td>
<td>Doig</td>
<td>5256</td>
<td>2.5</td>
<td>3.9</td>
<td>19.1</td>
<td>51.2</td>
<td>2.68</td>
<td>1.3</td>
<td>5</td>
<td>British Columbia</td>
</tr>
<tr>
<td></td>
<td>Muskwa</td>
<td>5422</td>
<td>2.5</td>
<td>4.4</td>
<td>21.6</td>
<td>55.0</td>
<td>2.61</td>
<td>0.301</td>
<td>7.4</td>
<td>British Columbia</td>
</tr>
<tr>
<td></td>
<td>Montney</td>
<td>6538</td>
<td>2.5</td>
<td>4.3</td>
<td>21.1</td>
<td>54.8</td>
<td>2.72</td>
<td>5.18</td>
<td>8.9</td>
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</tr>
<tr>
<td></td>
<td>DF-1</td>
<td>-</td>
<td>2.5</td>
<td>2.78</td>
<td>13.68</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>Middle East</td>
</tr>
<tr>
<td></td>
<td>DF-2</td>
<td>-</td>
<td>2.5</td>
<td>4.4</td>
<td>20.38</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>Middle East</td>
</tr>
</tbody>
</table>

Figure 2: Contrast-enhanced photographs of the shale samples tested

Results

T$_2$ distributions at different confining pressures for all samples are shown in Figure 3. Dashed lines represent cumulative porosity; solid lines represent incremental porosity. Cumulative porosity is calculated by summing the area under the solid lines.
From Figure 3 it is evident that total porosity in all four samples decreases as confining pressure is increased. This is to be expected as samples compress under pressure. The amount of compression is about 5% as the confining pressure is increased from 0 psi to 5000 psi. It is also evident, that shales 1 and 3 have a unimodal pore size distribution consisting of only micro pores. Shales 2 and 4 have a trimodal pore size distribution consisting of micro pores, meso pores and macro pores. For shale samples 2 and 4, the cumulative porosity of each network was estimated by summing the incremental porosity in certain $T_2$ ranges corresponding to the pore networks. The $T_2$ ranges for each pore network were determined by finding the minimum or inflection points between adjacent $T_2$ peaks [Figure 4]. For example, in shale 2 the three pore networks were micro ($T_2 < 1.7$ ms), meso ($T_2 1.7-55$ ms) and macro ($T_2 > 55$ms). Macro porosity likely includes some fractures.
Using T₂ cutoff analysis it is possible to examine the effects of overburden pressure on each pore network type individually. Figures 5 and 6 show this type of analysis for shales 2 and 4.

Figure 4: Pore distribution for shale samples 2 and 4 tested at 5000 PSI confining pressure
As pressure is increased, the total porosity of both samples decreases. Left panel of Figure 5 shows that most of shale 2 porosity comes from micro pores (~2.5 pu) with the least porosity contribution coming from macro pores (~0.5 pu). The center panel of Figure 5 shows porosity normalized to the 0 psi value. It is clear that different pore networks behave differently when subject to overburden pressure. Macro pores compress rapidly within the first pressure step and then stay constant up to 5000 psi. Meso pores show an approximate linear compression rate from 0 psi to 5000 psi. Micro pores do not compress at all. The right panel shows total porosity, being a sum of the three pore network
contributions, as a function of pressure. It is important to note that looking only at total porosity as a function of pressure masks the behavior of individual pore networks.

The same analysis was done on shale 4. It can be seen in left panel of Figure 6, that in shale 4 meso pores dominate the total porosity with ~5 pu. Macro pores contribute the least with ~0.5 pu. The behavior of the three different pore networks under pressure is the same as in shale 2 (Figure 6 center panel). Macro pores compress quickly within the first pressure step, meso pores compress approximately linearly with pressure increase while micro pores do not compress.

$T_1$-$T_2$ data was acquired on two shale samples. Data was acquired on “as received” samples at three temperatures, 35 C, 65 C and 110 C. Plots of this data are shown in Figure 7. Diagonal lines represent $T_1$-$T_2$ ratios on a logarithmic scale.

![Figure 7: $T_1$ – $T_2$ maps at different temperatures](image)

The graphs show the $T_2$ distribution at each $T_1$ value. Red intensity is high while blue is low. In shale, it is often useful to examine these types of maps because they can show two or more distinct peaks which correspond to different $T_1$/T2 ratios. The $T_1$/T2 ratio can be used to determine the origins of the signal in $T_1$-$T_2$ maps. Higher ratios indicate higher viscosity fluids such as bitumen, shorter ratio contribution are likely due to water (likely capillary and clay bound water). In sample DF-1 Only one fluid type is detectable and is a fluid that has its viscosity affected by temperature (i.e. bitumen/heavy oil). The effect is manifested by the signal shift from left to right, i.e. from higher $T_1$/T2 ratio to lower $T_1$/T2 ratio. Also as the sample gets heated, viscosity of bitumen decreases and more bitumen is detected, causing an increase in total NMR porosity. In sample DF-2 at least two distinct fluids are present, water and bitumen. The signal in lower box is disappearing, indicating a water loss due to drying. At 110 C the water loss is greater than bitumen signal increase (upper box) thus resulting in a net loss of total NMR porosity. Total NMR porosity from $T_1$-$T_2$ maps are shown in Table 2.
Table 2: Total NMR Porosity

<table>
<thead>
<tr>
<th>Shale</th>
<th>NMR Porosity, 35°C (pu)</th>
<th>NMR Porosity, 65°C (pu)</th>
<th>NMR Porosity, 110°C (pu)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DF-1</td>
<td>3.0</td>
<td>4.8</td>
<td>4.7</td>
</tr>
<tr>
<td>DF-2</td>
<td>3.0</td>
<td>2.8</td>
<td>2.5</td>
</tr>
</tbody>
</table>

Conclusions

NMR methodology was successfully used to identify and study the effects of overburden pressure on shale pore networks. It was shown that total porosity decreases as confining pressure increases. Some less obvious results were also demonstrated, such as the effect of confining pressure on micro, meso and macro pores. Overburden pressure did not affect micro pores. It affected meso pores and macro pores in such a way the meso porosity was decreasing linearly as pressure was increased while macro porosity decreased rapidly with initial pressure increase and then stabilized at higher pressure. Some of the macro porosity can likely be attributed to the existence of fractures in shale samples. This means that fracture and macro porosity would not have a large contribution to total porosity at overburden pressures of greater than 1000 psi.

$T_1$-$T_2$ maps proved to be a useful tool in detecting fluid types in shales. Doing the measurements at different temperatures helped to distinguish fluids since most water would evaporate and leave the sample at temperatures over 100°C. Bitumen viscosity decrease at higher temperatures was observed. This viscosity change allows the NMR techniques to detect more bitumen at higher temperatures because the more viscous (solid) bitumen becomes NMR visible.

References

5. GIT Systems and LithoMetrix User Manual, Revision 1.9, Green Imaging Technologies.