Reservoir Rock Characterization Using Centrifuge and Nuclear Magnetic Resonance: A Laboratory Study of Middle Bakken Cores

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Abstract

In tight liquid-rich shale formations, fast and accurate techniques to determine reservoir key parameters, such as oil and water end-point saturations, capillary pressures, relative permeabilities, and pore-size distribution (PSD) are of great interest. In this paper, we use centrifuge and nuclear magnetic resonance (NMR) to determine these parameters for several Bakken cores. First, we measured helium porosity and Klinkenberg permeability of clean and dry Middle Bakken core plugs. After saturating these core plugs with brine, we performed drainage measurements using a high-speed centrifuge. Before and after centrifuge experiment, we measured the transverse relaxation time (T2) with a 2-MHz NMR instrument for use for pore-size distribution. Our results indicate that, using both centrifuge and NMR, we can obtain important petrophysical information for use in reservoir engineering and numerical simulation applications.

Introduction

Unconventional liquid-rich shale reservoirs have become important contributors to the hydrocarbon production in the United States. However, production from these reservoirs is challenging. The first step is to better understand shale reservoir production mechanisms by characterizing reservoir cores. One of the important reservoir rock characterization parameters is pore size distribution which has a significant effect on capillary pressure. Small pore throat size in tight formations makes the effect of capillary pressure on fluid distribution more significant compared to conventional formations (Alamdari et al. 2012). Capillary pressure is used both in petrophysical and reservoir engineering applications such as saturation height modeling (Altunbay et al. 2001), pore size distribution (Washburn 1921), and wettability (Amott 1959).

Capillary pressure is measured using various techniques e.g. mercury intrusion (Purcell 1949; Comisky et al. 2011), fluid displacement using centrifuge instrument (Rajan 1986; O’Meara et al. 1992) and porous plate (Christoffersen and Whitson 1995). Capillary pressure measurement on tight formations requires a significant force to displace fluids. Thus, centrifuge is the best instrument to measure capillary pressure in tight formations. Mercury intrusion experiment (MICP) is known for fast capillary pressure measurements, but we believe centrifuge measurements provide the correct capillary pressure curves because we use formation or synthetic fluids which preserve the rock-fluid interactions in measurements. The main
The disadvantage of all capillary pressure measurement techniques is limited accessibility to small pores, especially in tight formations. NMR transverse relaxation time (T2) experiments are known as representation of pore size distribution. Thus, with proper calibration, T2 distributions can be converted to pore size distribution. This calibration is achieved by correlating the T2 distributions with the pore size distributions calculated from either mercury intrusion (Marschall et al. 1995; Kleinberg 1996; Kenyon 1997; Rivera et al. 2014) or centrifuge capillary pressure curves (Coates et al. 1999; Chen et al. 2006). Since NMR is the only downhole logging tool that provides a representation of pore size distribution, building the correlation between centrifuge and NMR experiments can be used for logging interpretation applications. However, this correlation, especially in tight rocks, is scarce in the literature.

In this study, we conducted centrifuge experiments on low-permeability shale cores to measure flow properties such as capillary pressure, residual saturation and recovery factor. Before and after each centrifuge experiment cycle we performed NMR T2 and saturation profile experiments to not only measure the fluid saturation but also the distribution of oil and water in the cores. We also monitored the saturation changes at each stage using both techniques. Combination of NMR and centrifuge experiments extended our capability to produce capillary pressure curves beyond the limitation of the centrifuge experiments.

In addition, it is important to determine wettability of cores. Wettability is one of the key parameters in improved oil recovery design. Wettability also affects capillary pressure and relative permeability. We used NMR data to determine where different fluids reside in pores to better understand the wettability of rocks (Hsu et al. 1992; Looyestijn 2008; Rios et al. 2014).

**Materials**

**Core:** We used three clean Middle Bakken cores.

**Brine:** We used synthetic brine in our experiments: 20,000 ppm KCl in deionized water; 50,000 ppm KCl in deionized water; 100,000 ppm KCl in deionized water.

It is important to note that Middle Bakken formation is composed of a mixture of carbonate and sandstone minerals. Introduction of fresh brine to cores may dissolve some carbonate minerals, and create secondary porosity; therefore, we used synthetic brine after it reached equilibrium with the sample end pieces (personal communication with Dr. Andre Revil, Colorado School of Mines). More details are presented in the *Appendix A*.

**Oil:** We used decane as an alternative for Bakken formation live oil (personal communication with Mr. Jerry Warne, Core Lab). Because, decane has lower viscosity (0.92 cp at ambient conditions) compared to our Bakken crude oil sample (2.4 cp at ambient conditions) which has already lost its light components at ambient pressure and temperature (Karimi and Kazemi 2015).

**Reservoir Characterization Parameters Measured/Calculated**

We measured various rock and fluid properties such as porosity, permeability, capillary pressure and NMR response. In this section we discuss reservoir characterization parameters and measurement techniques.

**Porosity and Permeability**

CMS-300™ from Core Lab was used to measure porosity, air permeability and klinkenberg permeability of clean Middle Bakken cores. Helium gas is used in measurements with CMS-300™. We used 1,000 and 1,500 psi as confining pressures (minimum confining pressure required is 1,000 psi).

**Capillary Pressure**

We measured drainage capillary pressure and corresponding pore size accessed in drainage cycle, residual saturation and production from Middle Bakken cores using an ACES-200™ centrifuge from Core Lab. In
centrifuge experiments, we place fully fluid-saturated cores in drainage or imbibition centrifuge cups and spin the cores with several different rotational speeds. Spinning the cores creates centrifugal force along the axis of the cores and displaces fluid inside the pores with another type of fluid which is surrounding the cores in the centrifuge cups. Production is recorded with a high resolution camera in the form of change in fluid interface position with respect to time and rpm.

We conducted first drainage cycle using five different rotational speeds (Table 1) to capture the curvature of capillary pressure versus fluid saturation curve. Required run time to reach stabilization (when production from cores ceases at each rotational speed) for Middle Bakken cores was 60-70 hrs. We used Equation 1 to calculate capillary pressure (Ayappa et al. 1989).

<table>
<thead>
<tr>
<th>#</th>
<th>Rotational Speeds Chosen for Drainage Cycle (rpm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5,000</td>
</tr>
<tr>
<td>2</td>
<td>7,000</td>
</tr>
<tr>
<td>3</td>
<td>9,000</td>
</tr>
<tr>
<td>4</td>
<td>11,000</td>
</tr>
<tr>
<td>5</td>
<td>13,000</td>
</tr>
</tbody>
</table>

$$P_c(r) = \frac{1}{2} \Delta \rho \omega^2 \left( r_2^2 - r_1^2 \right)$$ \hspace{1cm} \text{(1)}

Where,

$$\Delta \rho = \rho_w - \rho_o$$ \hspace{1cm} \text{(2)}

$$\omega = 2\pi \left( \frac{N}{60} \right)$$ \hspace{1cm} \text{(3)}

$P_c(r)$ = capillary pressure at distance $r$ from the center of rotation

$\rho_w$ = brine density

$\rho_o$ = oil density

$\omega$ = angular rotation

$r_2$ = distance between the center of rotation to the outlet end of core

$N$ = number of rotations per minute (rpm)

Low rpm does not provide enough force to displace fluid in tight samples. For Middle Bakken cores, Karimi and Kazemi (2015) showed that the minimum rotational speed of 5,500 rpm provides a reasonable starting point on the capillary pressure curves. Although, various drainage and imbibition experiments can
be conducted using the centrifuge instrument, in this study we only report on first drainage results. All centrifuge experiments were conducted at laboratory pressure and temperature.

In addition to drainage experiments, we conducted spontaneous imbibition measurements on cores for two weeks using Amott cell. Amott cell is a glass instrument used to measure production from core plugs at zero capillary pressure condition.

**Nuclear Magnetic Resonance (NMR)**

We measured transverse ($T_2$) and longitudinal ($T_1$) relaxation time distributions using Carr, Purcell, Meiboom and Gill (CPMG) (Carr and Purcell 1954; Meiboom and Gill 1958) and inversion recovery free induction decay (IRFID) (Dunn et al. 2002) pulse sequences, respectively. We also acquired two dimensional (2D) $T_1$-$T_2$ maps, and one dimensional (1D) profile using magnetic resonance imaging techniques. The measurements were conducted on bulk fluids and Bakken cores at various fluid saturations at laboratory pressure and temperature. We used a 2 MHz Magritek Rock Core Analyzer™ equipped with z-direction gradient coil. $T_2$ distributions were measured using 100 $\mu$s echo spacing (TE), 2000-15000 ms polarization time (depending on the fluid relaxation rate), 2000-40000 number of echoes (depending on the fluid relaxation rate) and minimum 100 signal to noise ratio (SNR). The $T_1$ distributions were measured using 20 logarithmically spaced wait times ranging from 0.07 to 3000 ms. The IRFID and CPMG raw data were inverted using inverse Laplace non-negative least square fitting (Lawson and Hanson 1974; Buttler et al. 1981) to generate $T_1$ and $T_2$ distributions, respectively. We calculated the smoothing parameter using the methodology described by Dunn et al. (1994).

**Experimental Procedure**

A series of drainage, imbibition and NMR experiments were conducted on each core. A step by step procedure for these measurements is presented in Figure 2. The cores were received in cleaned and dried condition and no further cleaning was performed to prevent additional damage to the cores.

First, we saturated the cores with KCl brine with different salinities to study the effect of salinity on the spontaneous imbibition (Table 2). The saturation procedure is presented in the Appendix A. NMR $T_2$ distribution was measured on fully brine-saturated cores.

<table>
<thead>
<tr>
<th>Core ID</th>
<th>Synthetic Brine (KCl in DI Water) Salinity (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C1</td>
<td>20,000</td>
</tr>
<tr>
<td>C2</td>
<td>50,000</td>
</tr>
<tr>
<td>C3</td>
<td>100,000</td>
</tr>
</tbody>
</table>

![Figure 2](image_url)
Next, we conducted centrifuge first drainage cycle to displace brine by decane. Then, NMR $T_2$ distribution was measured to study fluid distribution and saturation changes. The same NMR experiments were performed after keeping the cores submerged in 50,000 ppm KCl brine in Amott cell for two weeks. Remaining steps of the experimental procedure in Figure 2 are being conducted currently.

Results

Porosity and Permeability
Porosity, air permeability and Klinkenberg permeability of three Middle Bakken cores used in this experiment are presented in Table 3. As indicated from this table, porosity varies from around 2.4 to 7.0 porosity units — a rather broad porosity variation.

<table>
<thead>
<tr>
<th>Core ID</th>
<th>Net Confining Pressure (Psi)</th>
<th>Porosity (%)</th>
<th>Air Permeability (μD)</th>
<th>Klinkenberg Permeability (μD)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1500</td>
<td>6.99</td>
<td>—</td>
<td>95.06</td>
</tr>
<tr>
<td>2</td>
<td>1000</td>
<td>2.43</td>
<td>2.14</td>
<td>0.807</td>
</tr>
<tr>
<td>3</td>
<td>1500</td>
<td>4.57</td>
<td>2.84</td>
<td>1.11</td>
</tr>
</tbody>
</table>

Capillary Pressure
We present the results of first drainage and spontaneous imbibition on three clean Middle Bakken cores. First drainage capillary pressure versus water saturation curves for the cores are plotted in Figure 3. Figure 4 presents results of spontaneous imbibition on the cores. Due to measurement difficulty in recording data for 5,000 rpm, we decided to estimate the displaced fluid volume based on NMR fluid saturation calculations for this specific rpm.
In spontaneous imbibition measurement using Amott cell, we surrounded all the cores with 5,000 ppm KCl brine. Core C1 saturated with 20,000 ppm KCl brine produced negligible amount of oil, Core C2 saturated with 50,000 ppm KCl brine produced decane uniformly from all faces of the core, and Core C3 saturated with 100,000 ppm KCl brine produced decane from top face of the core.

NMR
We conducted various NMR experiments, $T_1$, $T_2$, $T_1-T_2$ and saturation profile, on bulk fluids and cores at different fluid saturation stages. In the following, we present the $T_2$ and saturation profile results for all experiments. We also show examples of $T_1$ and $T_1-T_2$ maps for a subset of samples.

NMR $T_2$ Measurements on Bulk Fluids and Cores  Results of NMR $T_2$ measurements on bulk fluids used in our measurements are presented in Figure 5. KCl brine shows longer relaxation compared to decane. We also conducted $T_2$ measurements on cores based on the procedure shown in Figure 2. Figure 6 (C1-A, C2-A, and C3-A) presents cumulative and incremental porosity of fully brine-saturated cores. Figure 6 (C1-B, C2-B, and C3-B) shows the $T_2$ results after first drainage by decane, and Figure 6 (C1-C, C2-C, and C3-C) shows the results after spontaneous imbibition. We make the following observations:

- All cores show uni-modal or bi-modal distribution with a dominant peak at brine-saturated stage (A series in Figure 6).
- Drainage by decane reduced the signal amplitude of the dominant peak (10 ms) significantly, and shifted the peak to shorter relaxation times. After first drainage another peak was observed at 100 ms (B series in Figure 6).
- After spontaneous imbibition more discontinuity in NMR response is observed, and the signal amplitude at 1-10 ms decreased, while the amplitude at 100 ms increased.

We calculated brine and decane saturations by applying a threshold time to $T_2$ distributions before and after spontaneous imbibition to differentiate brine and decane responses. Table 4 presents the threshold times, NMR porosity and fluid saturation for different saturation stages.
Figure 5—NMR T₂ relaxation time measurement on bulk fluids. The logarithmic mean of the T₂ relaxation time for all fluids are shown for each distribution.

Figure 6—NMR T₂ relaxation time for Middle Bakken Cores C1, C2, and C3, where each row represents one core. First column (A) presents T₂ distributions for brine-saturated cores, second column (B) T₂ distribution for first drainage cycle, and third column (C) T₂ distribution after spontaneous imbibition in Amott cell.
We measured T₁ and two-dimensional (2D) T₁-T₂ maps for all samples at different fluid saturation stages. Figure 7 presents the results of T₁ and T₁-T₂ measurements on Core C3 at fully brine-saturated condition (Figure 7a and 6b) and after first drainage by decane (Figure 7c and 6d). Similar peaks in T₂ (Figure 6-1A and 6-1B) and T₁ (Figure 7a and 7c) are observed. Since brine and decane have approximately similar T₁/T₂ ratio, the T₁-T₂ maps and T₁ distributions result in similar pore size distribution and fluid saturation analysis. Thus, in this study we mainly use T₂ distribution which is also the most common acquired NMR response in downhole NMR logging.

**Table 4—Threshold time, calculated fluid saturation and porosity from NMR measurements.**

<table>
<thead>
<tr>
<th>Core ID</th>
<th>Water-Oil Saturation Threshold time (ms)</th>
<th>After Saturation by Brine</th>
<th>After First Drainage</th>
<th>After Spontaneous Imbibition</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Φ (%)</td>
<td>Sₘ (%)</td>
<td>Sₖ (%)</td>
<td>Φ (%)</td>
</tr>
<tr>
<td>C1</td>
<td>23.27</td>
<td>8.60</td>
<td>0</td>
<td>100</td>
</tr>
<tr>
<td>C2</td>
<td>12.75</td>
<td>6.83</td>
<td>0</td>
<td>100</td>
</tr>
<tr>
<td>C3</td>
<td>16.45</td>
<td>8.20</td>
<td>0</td>
<td>100</td>
</tr>
</tbody>
</table>

**NMR T₁ and T₁-T₂ Measurements on Cores** We measured T₁ and two-dimensional (2D) T₁-T₂ maps for all samples at different fluid saturation stages. Figure 7 presents the results of T₁ and T₁-T₂ measurements on Core C3 at fully brine-saturated condition (Figure 7a and 6b) and after first drainage by decane (Figure 7c and 6d). Similar peaks in T₂ (Figure 6-1A and 6-1B) and T₁ (Figure 7a and 7c) are observed. Since brine and decane have approximately similar T₁/T₂ ratio, the T₁-T₂ maps and T₁ distributions result in similar pore size distribution and fluid saturation analysis. Thus, in this study we mainly use T₂ distribution which is also the most common acquired NMR response in downhole NMR logging.
**NMR One-Dimensional Profile Measurements** Profile measurements are used to study fluid distribution in cores before and after saturation changes in drainage and imbibition experiments. Figure 8 presents the profile for all samples at fully brine-saturated stage, after first drainage and after spontaneous imbibition. This figure shows that fluid distribution is fairly uniform in the fully brine-saturated cores (orange curve in Figure 8). After first drainage, higher signal amplitude was observed at the inlet face of the cores, where decane was introduced. At the end of the spontaneous imbibition time period (two weeks), fluid distribution is uniform across the sample, although, the signal amplitude is higher than previous steps.

Figure 7—(a) One-dimensional $T_1$ and $T_2$ distributions for Core C3 at fully brine-saturated stage; (b) Two-dimensional $T_1$-$T_2$ for sample C3 at fully brine-saturated stage; (c) One-dimensional $T_1$ and $T_2$ for Core C3 after first drainage; and (d) Two-dimensional $T_1$-$T_2$ for Core C3 after first drainage. Brine and decane have approximately similar $T_1/T_2$ ratios, $T_1$-$T_2$ maps, and $T_1$ distributions in similar pore-size distributions and fluid saturations. The solid diagonal blue line in (b) and (d) represent the $T_1$-$T_2$ correlation.
In this section, we analyze and discuss experimental results presented in previous sections. We discuss porosity measurement results from different methods, drainage and spontaneous imbibition capillary pressure curves versus water saturation, saturation distributions and profile, and pore size distributions.

**Porosity Measurement**

In Table 1, we present helium porosity using CMS-300™ and porosity measured using a 2-MHz NMR apparatus - before and after each centrifuge desaturation stage. NMR porosities are significantly higher than CMS-300™ porosities. However, there is only a small difference between NMR porosities for all desaturation stages. We used water and decane hydrogen index (HI) of one in the calculations because both brine and decane indicated the same HI.

There are several reasons for CMS-300™ porosity being lower than NMR porosity. This includes (1) core exposure to air moisture and water condensation on pore minerals and (2) short equilibrium time for helium to access very small pores in CMS-300™ experiments.

**Capillary Pressure**

Figure 3 presents first drainage capillary pressure curves measured on Cores C1, C2, and C3. Core C1 and Core C3 have similar capillary pressure curves, but Core C2 has a slightly different capillary pressure curve. The irreducible water saturation after first drainage with decane for Cores C1, C2, and C3 are 43, 34, and 42 %, respectively. We cut Core C1 and Core C3 from a longer core plug; despite different brine
salinities, they have similar capillary pressure curves. This similarity is because we used clean cores, which are typically water wet, and we displaced brine with decane, which is not wettability altering.

Earlier, we measured capillary pressure of a Middle Bakken core plug with 3.4 % porosity (Karimi and Kazemi 2015). For this core, the irreducible water saturation at the end of drainage was 62 %. Comparing those results with the irreducible water saturations measured in this study for Cores C1, C2, and C3 shows the effect of porosity, pore size distribution and oil viscosity on irreducible water saturation. That is, rocks with lower porosity and smaller pores have higher irreducible water saturations (19-28 %).

Next, we conducted spontaneous imbibition measurement on the same cores (Figure 4). Spontaneous imbibition measurement shows negligible oil production from Core C1, saturated with 20,000 ppm KCl brine and surrounded by 50,000 ppm KCl brine. This happens because the salinity of brine around the core is higher than salinity of brine inside pores; thus, creating a negative osmotic pressure. Core C2 was saturated with 50,000 ppm KCl brine and surrounded with 50,000 ppm salinity brine. Oil was produced uniformly from all surfaces of the core (Figure 4a). This production is the result of spontaneous imbibition of brine in the water-wet core. Core C3 was saturated with 100,000 ppm KCl brine and surrounded by 50,000 ppm KCl brine. Core C3 produced more oil than other cores because of a combination of spontaneous imbibition and positive osmotic pressure.

Using NMR experiments on clean cores in this study, we could not measure oil production in the spontaneous imbibition stage; thus, we estimated produced oil volume from photographic images (Figure 4). On the other hand, for preserved cores (not shown here) NMR experiments, we were able to measure the spontaneous imbibition oil production.

Fluid Distribution in Pores

NMR T2 distributions for various fluid displacement experiments are plotted in Figure 9 for comparison. The NMR T2 of brine-saturated cores (black solid curves in Figure 9) shows a dominant single peak. The T2 distribution at brine-saturated state represents pore size distribution of the cores which are uni-modal. After displacing brine by decane (first drainage cycle-purple dotted curves in Figure 9), the amplitude of initial brine peak decreased and a longer relaxation decane peak was observed. Separation of peaks after decane displacement is due to the water wetness of the cores. Brine T2 relaxation (2.4 s) is longer than decane (1.4 s) (Figure 5), but as seen in Figure 9, decane relaxation in pores is longer. Since the samples are water wet, a thin layer of water on the minerals separates the mineral surface from decane and minimizes the surface relaxation in decane phase. This phenomenon is illustrated schematically in Figure 10.

![Figure 9—NMR T2 distributions at different stages on Middle Bakken cores. (Inc_Sat: Incremental porosity after cores were fully brine-saturated; Inc_F_D: Incremental porosity after first drainage; and Inc_SP_IMB: Incremental porosity after spontaneous imbibition measurement)](image)
A decrease in decane peak amplitude, as a result of oil production, was expected. However, NMR T2 distributions after spontaneous imbibition show an increase in decane peak (Figure 9). We speculate that the increase is due to residing the imbibed water in big pores with minimal surface relaxation. This speculation can be verified using profile measurements shown in Figure 8. In the following we take a closer look at the profile measurements.

Acquisition time for profile measurement is rather long and water is affected by surface forces (e.g. water in fully brine-saturated samples) (Figure 10a) relaxes before data acquisition starts. As a result, the profile amplitude for the fully brine-saturated cores is low (Figure 8-orange curves). After introducing decane, in the first drainage cycle, the profile amplitude increased. This increase is because of longer relaxation of decane which is not affected by surface forces (Figure 10b). The profile also showed non-uniform distribution of decane and brine after drainage due to centrifugal forces (Figure 8-green curves).

Uniform profiles after spontaneous imbibition (Figure 8-black curves) indicate that oil and brine were re-distributed during the spontaneous imbibition experiment time period (two weeks). In addition, the profile amplitude increased.

**Capillary Bound-Water Cut-off Determination**

It is important to determine capillary bound-water in tight rocks for log calibration and oil in place estimation purposes. We used the methodology presented in Coates et al. (1999). In their methodology the wetting phase was displaced by air which is the non-wetting phase. In this study, we displaced the wetting phase (water) by a non-wetting phase (decane) instead of air. As shown in previous section, different phases were identified (using the thresholds reported for saturation calculations) and the capillary bound-water cut-off time was calculated using cumulative porosity curves. For clean cores, we used the NMR T2 distributions before and after displacement of brine by decane in drainage cycle. The results are presented in Table 6.

![Figure 10](https://via.placeholder.com/150)

**Figure 10**—Schematic illustrating increase in decane saturation before and after drainage cycle. Before drainage, pores are filled with brine (a). After drainage, since the cores are water wet, a thin layer of water separated the mineral surfaces from decane and minimized the surface relaxation in decane phase (b).

<table>
<thead>
<tr>
<th>Sample</th>
<th>Threshold (ms)</th>
<th>Cut-off Time (ms)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C1</td>
<td>23.27</td>
<td>3.65</td>
</tr>
<tr>
<td>C2</td>
<td>12.75</td>
<td>9.66</td>
</tr>
<tr>
<td>C3</td>
<td>16.45</td>
<td>4.88</td>
</tr>
</tbody>
</table>

Table 6—Threshold time and capillary bound-water cut-off time for clean Middle Bakken cores.
Pore Size Distribution (PSD)

NMR T2 is a representation of the PSD as a result cumulative T2 distribution represents capillary pressure curves with appropriate conversion. However, the T2 distribution is in time domain and a conversion factor called “surface relaxivity” is required to convert T2 data to PSD. A combination of the pore size calculated using the centrifuge capillary pressure data and the cumulative NMR T2 distribution can be used to calculate the surface relaxivity.

To calculate pore size from capillary pressure curves, first, we measured contact angle of decane-brine-rock system using Drop Shape Analyzer DSA 100™ at ambient conditions (Figure 11). Then, we calculated the pore size using Equation (4) (ref).

\[
P_c = \frac{2 \times \sigma \times \cos \theta}{r}
\]

Where,
- \( P_c \) = capillary pressure
- \( \sigma \) = interfacial tension (IFT)
- \( \theta \) = contact angle
- \( r \) = pore throat radius
Next, we matched cumulative NMR $T_2$ distribution with centrifuge pore size and converted NMR $T_2$ distribution to a full spectrum of PSD for the cores. The surface relaxivity of Cores C1, C2, and C3 are 0.35, 0.22, and 0.28 respectively. The calculated surface relaxivities are in agreement with reported values by Saidian and Prasad (2015) for Middle Bakken cores.

**Conclusion**

In this study, we conducted measurements using centrifuge and NMR techniques to quantify reservoir rock characteristics. Based on the scope of this study we draw the following conclusions:

1. The irreducible water saturation after first drainage with decane for Cores C1, C2, and C3 are 43, 34, and 42 %, respectively.
2. Oil production as a result of spontaneous imbibition reduced the oil saturation in cores up to 11%. Salinity difference between pore brine and surrounding brine and water wetness of cores controls oil production via spontaneous imbibition. Surrounding the cores by low salinity brine increased oil production due to a combination of osmotic pressure and mineral water wetness effects.
3. Capillary bound-water cut-off time for the Middle Bakken cores ranges between 3 to 10 ms.
4. Middle Bakken cores show water-wet characteristics. We compared fluid distribution in pores at different stages of our experiments using NMR $T_2$ time measurements. Our observations show that water resides in smaller pores and oil resides in larger pores in both clean and preserved cores.
5. The effective surface relaxivity of the cores were calculated in the range of 0.35, 0.22 and 0.28 $\mu$m/s.

**Acknowledgement**

The authors are grateful to Whiting Petroleum, Marathon center of excellence for reservoir studies (MCERS) and Oil, Clay, Sand and Shale (OCLASSH) consortium for their support. We also thank Professor Andre Revil, CSM, and Mr. Jerry Warne, Core Lab, for their valuable advice.

**References**


Appendix A

Fluid and Rock Sample Preparation Procedure

**Synthetic Brine:** We used synthetic brine in measurements as well. We prepared synthetic brine using KCL and deionized (DI) water. To avoid creating secondary porosity in the cores due to dissolution of carbonate minerals, we put small pieces of rock (from the same rock type or core) in brine, and let brine and rock pieces stay for twenty-four hours to reach equilibrium. To find out if the equilibrium has been reached, one can measure conductivity of brine every hour after adding rock pieces. Equilibrium between brine and rock will be reached when brine conductivity is fairly stable not changing.

**Decane:** We used decane in some measurements as well. To make the interface between decane and brine visible (for centrifuge measurement purposes), we used red dye (SIGMA-ALDRICH Oil-Red O). The dye is only soluble in decane.

**Cleaned Core Samples:** For cleaned core samples, we used them as received.

To saturate cleaned cores by brine:

1. We put core samples under vacuum in vacuum chamber for twenty-four hours to get the air out of the pores as much as possible.
2. We poured brine (produced formation brine or synthetic brine) slowly on the cores. After brine level was high enough to cover the cores, we let the cores and brine stay under vacuum for another five hours. Figure A.1 is a schematic of vacuum chamber used to saturate the cores.

![Figure A.1—Schematic of vacuum chamber used to saturate cores.](image)

It is important to note that leaving brine under vacuum will make brine more saline due to evaporation and conductivity of brine will change. Thus, long vacuum time is not recommended. However, we are hoping that brine which has already saturated the pores won’t change under vacuum.

3. To make sure cores are fully saturated by brine, we put the cores in a high pressure vessel. The cores were surrounded by brine and 3,000 psi pressure was applied on the system for about three weeks. High pressure helps brine to penetrate to smaller pores and fully saturate the core samples. Figure A.2 presents a schematic of the high pressure vessel we used to saturate the cleaned cores.
Figure A.2—High pressure vessel used to saturate cleaned core samples.