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Application of SEM Imaging and MLA Mapping Method as a Tool for Wettability Restoration in Reservoir Core Samples for SCAL Experiments

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Abstract: In reservoir engineering, special core analysis experiments (SCAL) are performed in the lab to evaluate the production capabilities of an oil reservoir. A critical component of SCAL experiments is core wettability restoration to its original wettability, i.e., oil wet condition. Typically, aging is performed by saturating the core with oil and aging at reservoir temperature where time is the variable in question dictating whether the resulting restored core is strongly or weakly oil-wet. In the lab, core wettability is often experimentally validated using contact angle measurements or USBM (United States Bureau of Mines) wettability tests, which are often time consuming, expensive and prone to error. In this study we developed a novel method by using Scanning Electron Microscope (SEM) and mineral liberation analysis (MLA) imaging (at low vacuum conditions) to determine the wettability of rocks saturated with reservoir fluids such as oil and brine. For this work a systematic approach was applied with comparing the SEM-MLA method against conventional methods to quantify the degree of uncertainty linked to a) wettability estimation and b) the aging time. We have used a comprehensive suite of core samples such as Berea, Silurian Dolomite and Chalk to represent the bulk of oil reservoirs in the world.

Keywords: SEM-MLA; special core analysis (SCAL); wettability; USBM

1. Introduction

Special core analysis (SCAL) data are important for reservoir characterization and secondary and tertiary production optimization or enhanced oil recovery (EOR). An important consideration for ensuring quality and reliability in SCAL experiments is the restoration of core wettability. Incomplete core sample restoration may lead to unrealistic estimates of residual oil saturation, and inaccurate capillary pressure and relative permeability measurements—key parameters in simulating the reservoir productivity. Reservoir rock is generally considered to be oil-wet and can be achieved either from native or from restored states [1]. Obtaining native state core samples is challenging both economically and operationally. It requires suspending production, protecting the sample from drilling fluids, and preventing any evaporation or contamination [2]. Hence, laboratory experiments are routinely performed on core samples that have been cleaned of drilling and remaining reservoir fluids and then restored. Restoration typically involves cleaning the core sample with various solvents, rendering it water wet. The cleaned samples are then saturated with brine at reservoir conditions (to establish connate water saturation) and crude oil to capillary pressure. The oil/brine saturated cores are aged at reservoir conditions. Literature is replete with various aging strategies, but the conundrum is the lack of a commonly accepted wettability restoration period for either sandstones or carbonates.

Wettability is the tendency of one fluid to spread on the solid surface in the presence of another immiscible fluid. Wettability is of paramount importance in oil recovery from
low permeability chalk to high permeability sandstones as they control the flow and distribution of fluids [1]. In the past, numerous studies have indicated multiple factors influencing wettability including oil composition, rock mineralogy, fluid saturation, brine composition, temperature and aging time [2]. Although carbonate reservoirs tend to be intermediate to strongly oil wet, laboratory investigations on core restoration has many uncertainties [3,4].

Aging time and temperature are generally accepted to be the two most important factors contributing to the aging process. Anderson (1986) [2] indicated that 1000 h (40 days) of aging at reservoir temperature is sufficient for wettability equilibrium. Additionally, during the aging process it is important to saturate the core with brine prior to oil to ensure the wettability effects due to brine chemistry are not ignored [5]. Numerous studies have demonstrated the effect of increased brine salinity on rendering the sample water wet and ultimately increasing the oil recovery on carbonate rocks [4,6]. Alternately, it has also been widely demonstrated that brine composition containing mainly sulfate ions to have a positive impact in recovering oil from carbonate reservoirs by altering the surface charge on the rocks and making it water wet [6–8].

Wettability is generally quantified by contact angle measurements or United States Bureau of Mines (USBM) method or both. For a reservoir rock to be deemed oil-wet, the contact angle in an oil–brine–rock system that should be >105° [2] or wettability index to be –1. USBM wettability index is calculated from the drainage and imbibition capillary pressure curves and Robin (2001) [8] demonstrated a qualitative differentiation between oil-wet to water-wet capillary pressure curves. Lately, USBM wettability methods are increasingly applied to understand shale formation wettability in unconventional oil reservoirs [9–11]. In addition to the above two methods, digital imaging methods like SEM analysis are increasingly applied for wettability characterization. CRYO SEM (cryogenic scanning electron microscopy) and ESEM (environmental scanning electron microscopy) methods [9–12] were initially used to analyze wettability in rocks and packed glass beads that were saturated with reservoir fluids. However, these analyses were not accurate as it often compromised the sample integrity due to extreme changes in the physical state because of cooling and polishing. In a seminal method, we applied the SEM-MLA (scanning electron microscopy-mineral liberation analysis) method by testing the sample without any changes to its physical state.

In this paper we try to determine the threshold duration beyond which the aging does not change the wetting state of Berea sandstone, Silurian dolomite and outcrop chalk. After aging the cores at high temperature for varying periods of time, wettability determination was performed using contact angle measurements, the USBM method, and compared against SEM-MLA analysis. Specifically, the study was carried out on outcrop chalk and Berea samples as laboratory core flooding and SCAL experiments are routinely performed on core samples from restored state [1]. A common SCAL program can last for months if not for years. A significant portion of this time is spent on aging the reservoir cores and validating the wettability. Any significant reduction time in wettability validation will contribute immensely to reducing cost and concentrated effort on characterizing the reservoir. We aim to achieve this by applying this low vacuum SEM-MLA method to validate the state of wettability.

2. Materials and Methods

Porous media: The porous media used for this work was outcrop chalk from Kansas, which is a low permeable (2 mD) high porosity rock. The Berea samples came from Cleveland Quarries (Ohio, USA) and had a porosity in the range of 18–21% and gas permeability of 350 mD. The Silurian dolomite was obtained from Kocurec Industries, Texas, USA. Porosity of the dolomites varied from 13 to 14% and the gas permeability was measured to be 120 mD. The petrophysical characteristics of the core samples are listed in Table 1. Core samples were cut to 1.5" diameter by about 2" length. The core samples were initially sonicated to remove any fines and dried at 90 °C for two days before being subject
to saturation. Porosity was determined by the saturation test. Air and brine permeability were measured by conventional methods (permeameter).

Table 1. Experimental measurement of wettability by contact angle, SEM-(MLA) and United States Bureau of Mines (USBM) method for Berea, chalk and dolomite samples at different aging times.

| Core Sample | Sample ID | Porosity (%) | Aging Time (Weeks) | Contact Angle (°) | SEM-MLA Oil % | Wettability Index (USBM) | Wettability State |
|-------------|-----------|--------------|--------------------|-------------------|--------------|-------------------------|-----------------|
| Berea 1     | B1        | 18.45        | 0.5                | 70                | 0            | 0.282                   | Water wet       |
| Berea 2     | B2        | 18.19        | 2                  | 82                | 7.42         | 0.424                   | Water wet       |
| Berea 3     | B3        | 19.71        | 4                  | 95                | 22.43        | −0.013                  | Intermediate    |
| Berea 4     | B4        | 20.69        | 6                  | 103               | 37.53        | 0.034                   | Oil wet         |
| Berea 5     | B5        | 18.24        | 8                  | 112               | 73.47        | 0.278                   | Oil wet         |
| Chalk 1     | C1        | 38.57        | 1                  | 65                | 6            | 0.368                   | Water wet       |
| Chalk 2     | C2        | 38.49        | 2                  | 66                | 10           | 0.510                   | Water wet       |
| Chalk 3     | C3        | 37.76        | 3                  | 78                | 14           | 0.005                   | Intermediate    |
| Chalk 4     | C4        | 36.93        | 4                  | 85                | 24           | −0.018                  | Oil wet         |
| Chalk 5     | C5        | 38.04        | 5                  | 102               | 36           | −0.165                  | Oil wet         |
| Dolomite 1  | D1        | 14.06        | 0.5                | 65                | 0            | 0.263                   | Water wet       |
| Dolomite 2  | D2        | 13.63        | 4                  | 95                | 36.78        | −0.364                  | Intermediate    |

Fluids: The first part of the experiment involved saturation test to determine porosity. Only one type of brine was used for all experiments. The physical properties of brine used in this work are presented in Table 2. After saturation all the core samples were aged at constant temperature of 90 °C and the aging period was varied. Conventional dead crude oil with 6 cP viscosity and 858 kg/m³ density was used as the non-wetting phase. The composition of the crude oil used for this work is presented in Table 3. The oil was filtered and degassed by vacuuming it for 48 h to prevent any gas production during the drainage. During the aging process the core samples were circulated with few pore volumes of oil at constant intervals.

Table 2. Composition and properties of brine.

| Brine Salinity (NaCl) (ppm) | Density (kg/m³) | Viscosity (cP) | IFT with Oil (mN/m) |
|-----------------------------|-----------------|---------------|---------------------|
| 64,000                      | 1060            | 1.05          | 70.8                |

Table 3. Composition of crude oil.

| Components | Mass Fraction | Mole Fraction | Volume Fraction |
|------------|---------------|---------------|-----------------|
| CO₂        | 0.0000        | 0.0000        | 0.0000          |
| N₂         | 0.0000        | 0.0000        | 0.0000          |
| C₁         | 0.0000        | 0.0000        | 0.0000          |
| C₂         | 0.0000        | 0.0000        | 0.0000          |
| C₃         | 0.0002        | 0.0009        | 0.0003          |
| i-C₄       | 0.0003        | 0.0012        | 0.0005          |
| n-C₄       | 0.0018        | 0.0070        | 0.0026          |
| i-C₅       | 0.0028        | 0.0086        | 0.0040          |
| n-C₅       | 0.0054        | 0.0165        | 0.0075          |
| C₆         | 0.0163        | 0.0427        | 0.0206          |
| C₇⁺        | 0.9732        | 0.9231        | 0.9646          |

The core samples for testing were initially cut to 2” length with two 5 mm sections cut from top and bottom of the core. The idea was to use the core sample for capillary pressure measurement (USBM wettability measurement) and the thin section being used for the contact angle measurement and SEM-MLA analysis. The samples were then sonicated for 20 min and dried in an oven for 24 h before being saturated with the representative brine as outlined in the experimental plan.

In the first stage of experiment, the core samples were brought to connate water condition and oil saturation. The brine saturated samples (core + thin section) were loaded in to a coreholder with an overburden pressure of 6900 kPa (1000 psi) and centrifuged...
in drainage mode. A Rotosilenta 630RS refrigerated centrifuge from Vinci technologies (Nanterre, France) was used for this purpose. The drainage test with oil displacing brine was carried out in 7 centrifugation steps starting from 500 rpm to a maximum of 3500 rpm with 3 h of equilibration time per rpm step.

After centrifuging, the coreholders were disassembled to inspect the oil saturation in the core samples. The samples were again loaded in the core holder and the overburden pressure was adjusted prior to placing them in the oven for aging. Once aging was completed, the top (thin) section of the core sample was loaded in a Vinci IFT 700 instrument (Vinci Technologies, Nanterre, France) to measure the contact angle by the sessile drop method using brine as the drop fluid. The measured contact angles for the aged core samples are listed in Table 1. Figure 1 shows representative contact angles for the brine sessile drop in the presence of air for all the core samples. Figure 1a–e are the contact angle measurements for Chalk 1 through Chalk 5 that were aged at increasing time of 1 to 5 weeks. Figure 1f–j are the contact angle measurements of Berea 1 through Berea 5 that were aged at increasing time of 0.5 to 8 weeks. Figure 1k,l are the contact angle measurement for clean and 4 weeks aged Silurian dolomite samples.

In the second stage, the bottom (thin) section of the aged core sample was utilized for SEM-MLA analysis. FEI Quanta 650 FEG (Brno, Czech Republic) scanning electron microscope, equipped with Bruker high throughput energy dispersive X-ray (EDX) system (Billerica, MA, USA) and backscattered electron detectors was used for this purpose. Imaging on the flat sample surfaces was carried out at very low vacuum conditions (79 Pa) to prevent evaporation of fluids. Additionally, the samples were not subject to any metallic or carbon coating on the surface, except for the liquid graphite coating on the sample holder. Instrument conditions and parameters include a high voltage of 25 kV, spot size of 5.75, working distance of 13.5 mm, 10 nA beam current, 16 µs BSE dwell time, 10 pixel minimum size (400 pixel frame resolution for 1 mm HFW) and 12 ms spectrum dwell for EDX. Each of these MLA acquisitions was completed using version 3.1.4.683 MLA™ software and took between 3 and 4 h per sample. Minerals and fluids in the core sample were calculated through a custom classification script that accounted for porosity and minerals. The mineralogy was determined using GXMAP measurement mode within FEI Mineral Liberation Analyzer™ software, (version 3.1.4.683) equipped on a FEI Quanta 650 Field Emission Gun (FEG) SEM (Brno, Czech Republic). Each mineral identified must be within an 80% match to a known standard x-ray. For the determination of porosity, the “Pores” were determined using a custom classification, in which material that had a greyscale of certain range was scripted to be “Pores” instead of background material. This darker material of “Pores” was not matched to any x-ray standard. The results for individual samples were acquired as digital map of the minerals and a data table listing their mineral composition. Figure 2 is an example of mineral map and BSEM image of a core samples aged for varying time periods at 90 °C. Figure 2a–c are the SEM image of Chalk 1, Chalk 3 and Chalk 5 respectively and Figure 2g–i are the corresponding MLA images. Figure 2d,e are the SEM images of Berea 1 and Berea 5 with Figure 2i,j representing the corresponding MLA images. Figure 2f is the SEM image of Silurian dolomite 2 and Figure 2l is the corresponding MLA image. The minerals identified from the mineral map for chalk samples are listed in Table 4. Wettability assessment was based on the organic (oil) content of the sample in direct comparison to brine and mineral composition prior to saturation. The organic content for each sample is listed in Table 1 as SEM-MLA oil%.
Figure 1. Brine droplet of size $5 \times 10^{-4}$ mL placed onto the aged top end piece. (a) Chalk 1 ($\theta = 65^\circ$); (b) Chalk 2 ($\theta = 66^\circ$); (c) Chalk 3 ($\theta = 78^\circ$); (d) Chalk 4 ($\theta = 85^\circ$); (e) Chalk 5 ($\theta = 102^\circ$); (f) Berea 1 ($\theta = 70^\circ$); (g) Berea 2 ($\theta = 82^\circ$); (h) Berea 3 ($\theta = 93^\circ$); (i) Berea 4 ($\theta = 102^\circ$); (j) Berea 5 ($\theta = 112^\circ$); (k) Silurian dolomite 1 ($\theta = 65^\circ$); (l) Silurian dolomite 2 ($\theta = 95^\circ$).
Figure 2. Cont.
After the primary drainage test was completed, the imbibition step was started to force brine into the aged core sample to displace oil. The core samples were loaded in the core holder (in imbibition mode) with overburden pressure of 6900 kPa (1000 psi). The receiving tubes were filled with the representative brine for each sample and the samples were centrifuged from 500 to 3500 rpm in seven steps of 3 h duration in each step. At the end of the imbibition test, the secondary drainage step was carried out by forcing oil through the brine saturated samples. The secondary drainage process was also carried out in seven steps. The secondary drainage data and the imbibition data were analyzed and the area under each curve was calculated. Figures 3 and 4 are the capillary pressure curves generated for samples saturated with oil at different brine concentration (drainage (D)) and displacement of oil under different brine concentrations (imbibition (I)). The USBM wettability index was calculated based on the area under the curve for both secondary drainage \(A_1\) and primary imbibition \(A_2\) using the formula \(W = \log(A_1/A_2)\). The trapezoidal method was used to estimate the area under the curves using the capillary pressure points as the basis for each sample. A sample calculation for area under the capillary pressure curves for dolomite sample is presented in Figure A1 and Table A1 (Appendix A). Typically, the wettability index ranges from >0 for water wet, to <0 for oil wet and 0 for neutrally wet. In comparison with the contact angle measurement, the USBM

### Table 4. Mineral list from the SEM-MLA analysis for chalk samples.

| Mineral     | No. 1 (Area %) | No. 2 (Area %) | No. 3 (Area %) | No. 4 (Area %) | No. 5 (Area %) |
|-------------|----------------|----------------|----------------|----------------|----------------|
| Carbonate   | 83             | 81             | 80             | 35             | 38             |
| Halite      | 7              | 9              | 1              | 27             | 24             |
| Oil         | 6              | 10             | 14             | 24             | 36             |
| Others      | 4              | 0              | 5              | 14             | 2              |

Figure 2. (a–f) SEM images of Chalk 1, Chalk 3, Chalk 5, Berea 1, Berea 5 and Silurian dolomite 2 respectively. (g–l) are the MLA images of Chalk 1, Chalk 3, Chalk 5, Berea 1, Berea 5 and Silurian dolomite 2 respectively.
method provides a macroscopic average of the core plugs used in this study [13,14]. The wettability index calculated via the USBM method for all the core samples are listed in Table 1.

![Capillary pressure curves for Berea and Silurian dolomite samples.](image1)

**Figure 3.** Capillary pressure curves for Berea and Silurian dolomite samples.

![Capillary pressure curves for chalk samples.](image2)

**Figure 4.** Capillary pressure curves for chalk samples.

### 3. Results

The premise of this work is to establish an alternative method to determine wettability in sandstone and carbonates. Wettability was altered by aging the samples with brine and crude oil at constant temperature. The tests were initiated by bringing the core samples to residual water and initial oil saturation using the centrifugal method. The oil saturated core samples with crude oil and connate water were aged for the different times and further analysis was carried out.

Contact angle measurements were first measured to determine wettability and were carried out on thin slices from aged core samples. The sessile drop method was applied with brine as the drop fluid on the aged core sample. For samples that were aged for few days less than a week, a stable drop could not be attained as it started spreading. Hence, the contact angle was considerably less and it was below $70^\circ$. Figure 1 is a display of all the contact angle images obtained from the samples used for this work. For samples that were aged more than a week, a more stable drop was formed and the contact angle increased
to almost 80. Further, the difference in contact angle for samples of increasing aging time is indicated in Figure 1. As the aging time was increased to more than 4 weeks, more oil wet characteristics were observed with contact angle reaching more than 100. However, typical oil wet characteristics was displayed for samples that were aged above 6 weeks as the contact angle was more than 105. Increasing contact angle was also evident from the Figures 5 and 6 where a direct comparison of contact angle against SEM-MLA data was plotted for Berea and chalk.

![Figure 5. Comparison of contact angle data and SEM-MLA data for Berea.](image)

![Figure 6. Comparison of contact angle data and SEM-MLA data for chalk.](image)

The wettability index from the USBM method was analyzed using the area under the secondary drainage curve and the imbibition curve using the formula $I = \log (A_1/A_2)$. After the samples were aged, primary imbibition test was carried out followed by secondary drainage. Beside the uncertainty with confining pressure, the sample length impacted the extent of saturation in the chalk samples. The sample sizes were reduced to almost 1” to produce reasonable oil saturation, production and secondary drainage. As it can be seen from Table 1 and Figure 7, the wettability index did not follow a trend that was observed in contact angle measurement and SEM-MLA analysis. This is largely due to the inconsistency with data acquisition through laborious experimental process of bringing the
core sample to ambient condition before changing from drainage to imbibition coreholders and vice versa.

Figure 7. Comparison of USBM wettability index data between Berea and chalk.

4. Discussion

It was evident that the breakthrough pressure for chalk was significantly higher than Berea and Silurian dolomite because of its low permeability. Additionally, the pore size distribution is quite narrow in the range of 50 µm. In spite of the variations in the USBM results, we are able to demonstrate oil-wet characteristics for the chalk sample that were aged more than 3 weeks. Contrarily, Berea samples showed oil-wet characteristics when aged more than 4 weeks. The wettability index measured from USBM method was in close agreement with other published results [14,15] in predicting optimal time of 4 weeks for chalk, 6 weeks for Berea and more than 4 weeks for dolomite to render reservoir rocks oil-wet [1].

The last method to validate wettability was using SEM-MLA analysis where the samples were analyzed to provide an estimate of oil present. Initial tests on thin slices aged core samples provided inconclusive results as the saturation at the lower part of the core sample was not uniform. Thin slices from top of the core were later imaged and the corresponding mineral composition for a sample is shown in Table 3. As the chalk mineral composition was mainly carbonate and the brine composition was made of only NaCl, the MLA analysis straight forward compared to Berea and Silurian dolomite, which has more quartz and calcite respectively. A clean sample of each type of rock with crude oil and one with brine saturation were initially analyzed to detect the corresponding oil/brine signature and saved in the SEM database to match aged core samples. This was more effective in mineral liberation analysis and resulted in each sample yielding the corresponding oil residue values. The individual estimates for oil present in the aged core samples are tabulated in Table 1. Figures 5 and 6 were a direct comparison of the SEM-MLA data against the contact angle measurement. It is evident SEM-MLA method showed increasing oil residue in core sample with aging time corroborating the results from contact angle measurement.

5. Conclusions

1. A suite of Berea, dolomite and outcrop chalk cores were prepared and aged at increasing duration at constant temperature. Wettability was validated using three diverse methods, i.e., contact angle, USBM wettability index and oil content using SEM-MLA analysis.
2. With increasing aging time, the core samples indicated increasing oil-wet characteristics. Contact angle measurements indicated agreed strongly oil-wet characteristics for increased aging time. Contact angle values varied from 60° (water-wet) to 120° (oil-wet). Above aging time of 4 weeks the contact angle measurements were stable with a contact angle value around 110°.

3. A new method to estimate wettability was tested using SEM-MLA analysis, which provided more direct and convincing results. Oil presence in core samples is quantified via MLA analysis and the strategy to change wettability with increasing aging time was validated. Interestingly MLA analysis on chalk and Berea samples were straightforward with a simplified mineral list with increasing oil residue for increased aging time.

4. A comprehensive estimate of more than 5 weeks for chalk and 6 weeks for Berea was commonly agreed between three different wettability measurements to ensure core samples are oil wet.

5. The test on Silurian dolomite can further be strengthened by increasing the number of test samples with aging time to provide a similar estimate as Berea and chalk.

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Data Availability Statement: The data presented in this study are available in Tables 1–4 in this document. Data presented in this study relevant to Figures 2–4 are available upon request from the corresponding author.

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Appendix A

Table A1. Area under the curve calculation using trapezoidal rule for dolomite 2.

| $P_c$ (psi) | $S_w$ (%) | Area     |
|------------|-----------|----------|
| 26.20      | 78.03     | 1157.11  |
| 28.40      | 35.64     | 454.83   |
| 30.20      | 20.12     | 190.91   |
| 35.60      | 14.32     | 215.47   |
| 46.50      | 9.07      | 152.80   |
| 56.50      | 6.10      | 63.04    |
| 60.50      | 5.02      | 182.53   |
| 70.20      | 2.23      | 125.26   |
| 79.30      | 0.55      | 182.53   |
| **Area under drainage curve ($A_1$)** |          | 2541.96  |
| **Area under imbibition curve ($A_2$)** |          | 5862.43  |
| **Wettability Index (USBM)** | Log($A_1/A_2$) | −0.363   |
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