

APPLICATION OF AN OPTIMISATION METHOD FOR THE RESTORATION OF CORE SAMPLES FOR SCAL EXPERIMENTS – Part II

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This paper was prepared for presentation at the International Symposium of the Society of Core Analysts held in Vienna, Austria, 27 August – 1 September 2017

ABSTRACT

The restoration of wettability in reservoir rock core samples to its original state is highly critical in preliminary core preparation for SCAL experiments. Conventional core restoration methods to either strongly or weakly oil-wet are largely influenced by increasing or decreasing the aging time at reservoir temperature. There is a lack of consistent or reliable methods to restore core to its original state which is normally obtained from preserved core samples. This paper is a continuation of the work that was presented in SCA2016-002 where we examined the optimum level of three important parameters, brine salinity, restoration temperature, and restoration time (age in number of days) for restoring wettability. In this paper, we extend the Box Behnken model of surface response methodology to analyze wettability determined via three methods: contact angle, Amott-USBM, and via a new method using SEM-MLA over extended ranges of brine salinity (10,000, 100,000 and 200,000 ppm total dissolved salts), temperature (60, 90 and 120° C), and age of conditioning (2, 4 and 8 weeks). The samples for this study included 15 Berea sandstone and Silurian dolomite samples aged in crude oil and brine of varying salinity. The samples for this work underwent extended aging time to overcome the shortcoming in the previous work which again was carried out using coreholders under confining pressure. The wettability was experimentally validated using contact angle measurements, Amott-USBM tests, and a novel SEM-MLA imaging (at low vacuum conditions). A seminal effort in applying SEM-MLA image analysis for wettability determination was also explored. Linear regression models were developed and the adequacy of predicting the output variables (wettability) to nearly all conditions were verified. The study showed a comprehensive influence of brine salinity, aging time, and temperature towards wettability restoration. Further 2-D and 3-D surface plots were generated to show the interaction between the three independent variables in establishing a wettability value.

INTRODUCTION

Specialised Core Analysis (SCAL) data, specifically capillary pressure (P_c) and relative permeability (K_r) are important for reservoir characterization, production optimization and simulation. In this study, we developed an optimization methodology to restore wettability in core samples for SCAL experiments as laboratory experiments are routinely performed

on core samples from restored state [4]. In practice, aging strategies vary between organizations and there is no one commonly accepted restoration process. In this paper, we try to determine the optimal value of the three parameters that are generally agreed upon as contributing to the aging process [2, 15]: brine salinity, aging time, and aging temperature for a given rock mineralogy and oil composition. While it is understood that mineralogic composition of the reservoir rock and oil composition (polar components, asphaltenes) play a significant role in wettability alteration and aging, the interest of this study is to establish optimal aging time for a given reservoir rock and oil. Our interest is to determine the optimal aging conditions using Hibernia light crude oil (35°API) with an asphaltene content of < 1% for aging the core samples. The crude oil composition is presented in Table 6. A Berea core sample with 80% quartz content and less than 2% clay content was used for this study. Although wettability is largely influenced by rock mineralogy and crude oil composition, the restoration process is impacted by the aging time, temperature and brine salinity.

Aging time and temperature are generally accepted to be the two most important factors contributing to the aging process. Anderson (1986) indicated that 1,000 hours (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. Numerous studies [5, 13] have demonstrated the effect of increased brine salinity on oil wet characteristics exhibited in Berea sandstone.

Wettability is generally quantified by contact angle measurements or by USBM method or both. For a reservoir rock to be deemed oil-wet, the contact angle in an oil-brine-rock system should be $> 105^\circ$ (Anderson 1986) or wettability index to be -1. USBM wettability index is calculated from the drainage and imbibition capillary pressure curves and Robin (2001) demonstrated a qualitative differentiation between oil-wet to water-wet capillary pressure curves. Lately, USBM wettability methods are increasingly applied on understanding the wettability nature of shale formations in unconventional oil productions [3, 6, 8]. In addition to the above two methods, digital imaging methods like SEM analysis are increasingly applied for wettability characterization. CRYO SEM and ESEM methods [7, 9, 10, 11] were initially used to analyse wettability in rocks and packed glass beads that were saturated with reservoir fluids. But 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 have applied SEM-MLA method by testing the sample without any changes in its physical state.

This paper is a continuation of the work that was presented in SCA 2016 [12]. We have extended the range of three input parameters that influence wettability to address the gaps that were identified in the past work [12]. Additionally, we have extended the model to include three response factors instead of one for determining wettability, they include contact angle measurement, USBM wettability index and organic content from SEM-MLA analysis.

EXPERIMENTAL METHODOLOGY

For this study we have chosen statistical design of experiments (DOE) as it provides an understanding of the parametric effects controlling a process with the benefit of a decrease in the number of experiments required. Box Behnken response surface methodology was applied to optimise the input parameters i.e., aging time, temperature and brine salinity for the desired output parameter that is wettability. Box Behnken requires only 15 trials for a three factor experiment and providing maximum efficiency for a surface model. In the previous work, the model was tested using one response factor i.e. wettability using contact angle. In this study, we have extended the model to include three response factors: contact angle measurement, USBM wettability index method and organic content (%) from SEM-MLA analysis. Table 2 summarises the experimental plan and the real values for input parameters. Trials at optimal levels were duplicated twice to ensure repeatability. Design Expert ® software was used for the DOE, response surface model analysis, and input parameters optimisation.

A wider range of brine salinities from 10,000 to 200,000 ppm were used for this work which was markedly different from 60,000 to 120,000 ppm that was used in the previous study [12]. The composition of brine includes some divalent calcium and magnesium ions and the complete composition is presented in Table 1. The aging period was increased from 6 weeks to 8 weeks with the maximum aging temperature changed from 90 to 120°C. Hibernia (offshore Newfoundland) dead crude oil with 5.9 cP viscosity and 878 kg/m³ density was used for saturation. The oil was filtered and degassed by vacuuming it for 48 hours to prevent any gas production during the drainage. During the aging process the core samples were circulated with few pore volumes of oil on a weekly basis. Berea Sandstone used for this work came from Cleveland Quarries with porosity in the range of 18 - 20% and gas permeability ~ 150 mD.

The core samples for testing were initially cut to 6.3 cm length with two 5 mm sections cut from the 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 contact angle measurement and SEM-MLA analysis. The samples were then sonicated twice for 20 minutes each for a total of 40 minutes and dried in an oven for 24 hours 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 core holder at overburden pressure of 3000 psi and centrifuged in drainage mode. A Rotosilenta 630RS refrigerated centrifuge from Vinci Technologies 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 3,500 rpm with 3 hours of equilibration time per rpm step.

After centrifuging, the core holders 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 the aging was completed, the top (thin) section of the core sample was loaded in a Vinci IFT 700 instrument to measure the contact angle by sessile drop method using brine as the drop fluid. The measured contact angles for the aged Berea Sandstone are listed in Table 2. Figure 1 shows representative contact angles for the brine sessile drop in the presence of air for some of the rock samples.

In the second stage, the bottom (thin) section of the aged core sample was utilised for SEM-MLA analysis. FEI Quanta 650 FEG scanning electron microscope, equipped with Bruker high throughput energy dispersive x-ray (EDX) system and backscattered electron detectors was used for this purpose. Imaging on the flat sample surfaces was carried out at very low vacuum conditions (0.6 Torr) to prevent evaporation of fluids [7]. Additionally, the samples were not subject to any metallic or carbon coating on the surface, except for 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 μ s BSE dwell time, 10 pixel minimum size (400 pixel frame resolution for 1mm 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 3-4 hours per sample. Minerals and fluids in the core sample were calculated through a custom classification script that accounted for porosity and minerals. The results for individual samples were acquired as digital map of the minerals and a data table listing their mineral composition. Figure 3 is an example of mineral map and BSEM image of a sample aged for 4 weeks at 90°C. Wettability assessment was based on the organic (oil) content of the sample in direct comparison to brine and mineral composition prior to saturation. Values are in Table 2.

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 3000 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 3-hour steps. 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 analysed and the area under each curve was calculated. Figure 2 is the capillary pressure curves generated for samples saturated with oil at different brine concentration (secondary drainage) and displacement of oil under different brine concentrations (imbibition). The USBM wettability index was calculated based on the area under the curve for both secondary drainage (A_1) and imbibition (A_2) using the formula $W = \log (A_1/A_2)$. 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 method provides a macroscopic average of the core plugs used in this study [8]. The wettability index calculated for the 15 samples are listed in Table 2.

RESULTS AND DISCUSSION

Box Behnken response surface methodology, with three factors and three responses, was chosen to investigate and optimise the core restoration (wettability) process. The experimental results are shown in Table 2 and Design Expert ® Software was used for statistical analysis. The optimal aging conditions were predicted using a first order polynomial model which was fitted to correlated relationships between brine salinity, aging temperature and time (input variables) and contact angle, wettability index and organic content i.e., oil content (responses). The experimental data was analysed by multiple regression analysis through least squares method. Analysis of Variance (ANOVA) was applied to compute regression coefficients of the linear and higher order (quadratic and polynomial) models with interaction effects. Statistical validation of the model was done using F-test where a “fitted” model is deemed significant if the probability level is low, i.e. p-value ≤ 0.05 . The regression model was used to develop the response surface plots in order to visualise the relationship between the three input variables and responses. Finally, the developed models were used to suggest optimal conditions for aging. The experimental sequence was randomized in order to minimise bias and variability in measurements.

The results of the analysis of variance (ANOVA) tests were carried out individually for the three response factors i.e. contact angle, USBM wettability index and organic content from SEM-MLA and are presented in Tables 3, 4 and 5, respectively. For the contact angle measurement and organic content using SEM-MLA, the ANOVA test indicated only the quadratic model to be significant over other models. This is evidenced by the overall model p-values of 0.0464 and 0.0095, respectively. The resultant quadratic equations for the model developed using contact angle is presented in Equation 1. Aging time was the only significant parameter affecting wettability. Brine salinity and temperature were found to influence wettability only when they were considered with aging time, but independently they were found to be inadequate in influencing contact angle measurement. In comparison with the linear model developed in our previous study [12], the model equation is different but it is still in close agreement with aging time and its contribution to the aging process.

$$\begin{aligned} \text{Contact Angle} = & +65.12 - 3.62 \times 10^{-4} [\text{Salinity (ppm)}] + 0.59 [\text{Temp (}^\circ\text{C)}] + \\ & 13.38 [\text{Age (Weeks)}] + 0.031 [\text{Temp} \times \text{Age}] - 1.843 [\text{Age}^2] \end{aligned} \quad (1)$$

In the case of the USBM wettability index, a linear model was found to be best suited for predicting the experimental outcome which is confirmed from very low p-value of < 0.0001 . Among the three parameters, aging time was found to have the highest F value thereby significantly influencing the restoration process. The linear model equation using USBM wettability index (USBM WI) is shown in Equation 2.

$$\text{USBM WI} = 0.782 - 1.9 \times 10^{-6} [\text{Salinity (ppm)}] + 3.02 [\text{Temp (}^\circ\text{C)}] - 0.179 [\text{Age}] \quad (2)$$

In order to evaluate the significance of each input parameter on the outcome wettability measurement, F-tests were conducted on the three models. In the model based on contact angle measurement, aging time had higher F-values compared to temperature and salinity. Whereas in the model considering wettability by SEM-MLA, temperature and salinity had

higher F-values respectively. For the model developed based on wettability index, aging time was the significant factor contributing to the restoration process. Additionally, p-values for input parameters in all three cases were calculated to be less than the model F-value which is an indication that the statistical models are statistically significant. A diagnostic test was carried out to compare the experimental data with the model predicted results. Figure 4 shows a plot of experimental versus predicted values for both contact angle and USBM wettability index. Both analyses showed strong agreement between the experiment and model data as observed through most data points lying close to or on the diagonal line. A similar trend with predicted and actual values was observed in the organic content using SEM-MLA, but the results are withheld as we are progressing with further analysis.

From the developed statistical model we were able to produce 3-D response surface plots and 2-D contour plots for the three input parameters (aging time, temperature and salinity) against contact angle. Figure 5 represents the contour plot and 3-D RSM. The 2-D and 3-D plots provide an understanding of the interaction between two input parameters (e.g. age and salinity), while keeping the third parameter constant (temperature). The time of aging was found to be the most significant factor in impacting the aging process as observed through contact angle measurement. As the aging period was extended from 2 weeks to 8 weeks, the contact angle increased indicating a shift from intermediate oil-wet to strongly oil-wet characteristics. A similar trend was reported by Morrow (2000) and Anderson (1986) where strong oil wet characteristics was observed when aging time was extended to more than 40 days. Increasing temperature resulted in more oil-wet behavior whereas increasing brine salinity resulted in decreasing contact angle hence more water-wet behavior. This is in close agreement with published work [14, 15] on wettability alteration in sandstone when the brine concentration was increased from 0.3 % to 20%.

Optimisation: The regression models developed using three different wettability characterizations were utilised for optimising the input parameters such as temperature, aging time and brine salinity. As previously mentioned, the wettability criterion for an oil-rock-brine system with a contact angle $>105^\circ$ is considered oil-wet. Applying the optimisation criteria of maximizing the contact angle, numerical solutions were generated to establish the optimum value for Brine Salinity, Temperature and Time to be 104,257 ppm, 95°C & 5.5 weeks, respectively, for restoring wettability. The results are graphically represented via 3D response surface in Figure 6, where the peak points region is observed in both 2-D and 3D plots. In the case of wettability index, the optimal conditions for restoration were found to be aging time of 6.2 weeks, brine salinity of 115,000 ppm and temperature of 99°C . The optimal values were determined by setting a criteria of wettability index in the range of 0 to -0.6.

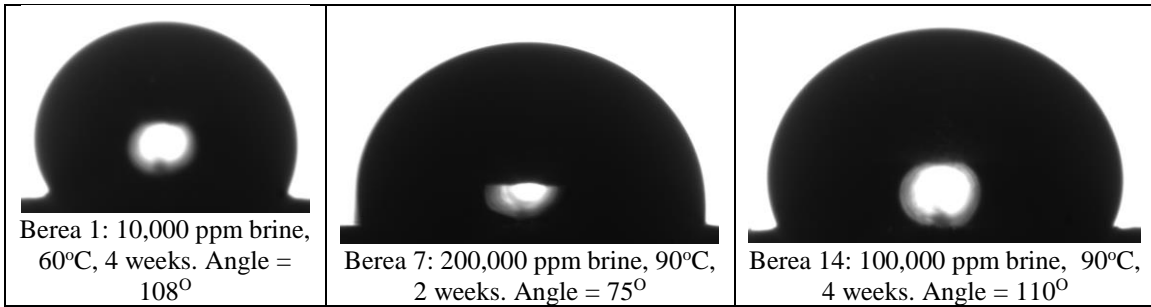


Figure 1. Contact angle measurements for Berea.

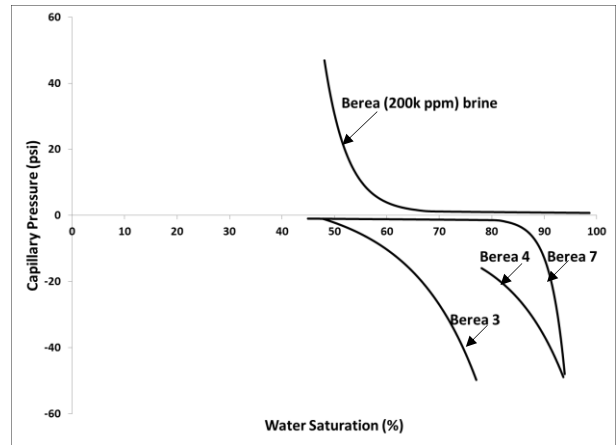
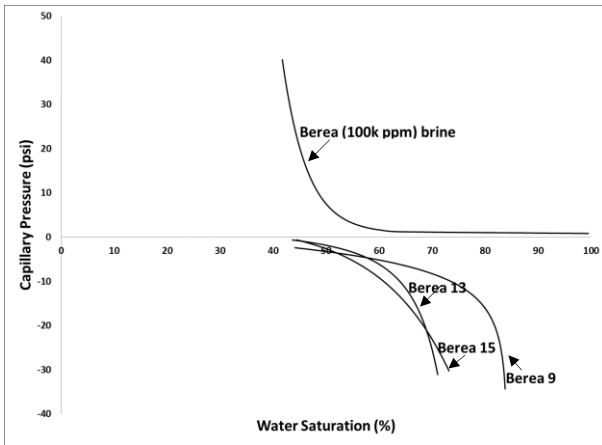


Figure 2. Capillary pressure curves for Berea samples (Imbibition and Drainage) Figure 3. (a) BSEM images of Berea sample 15 (b) Mineral map of Berea sample 15

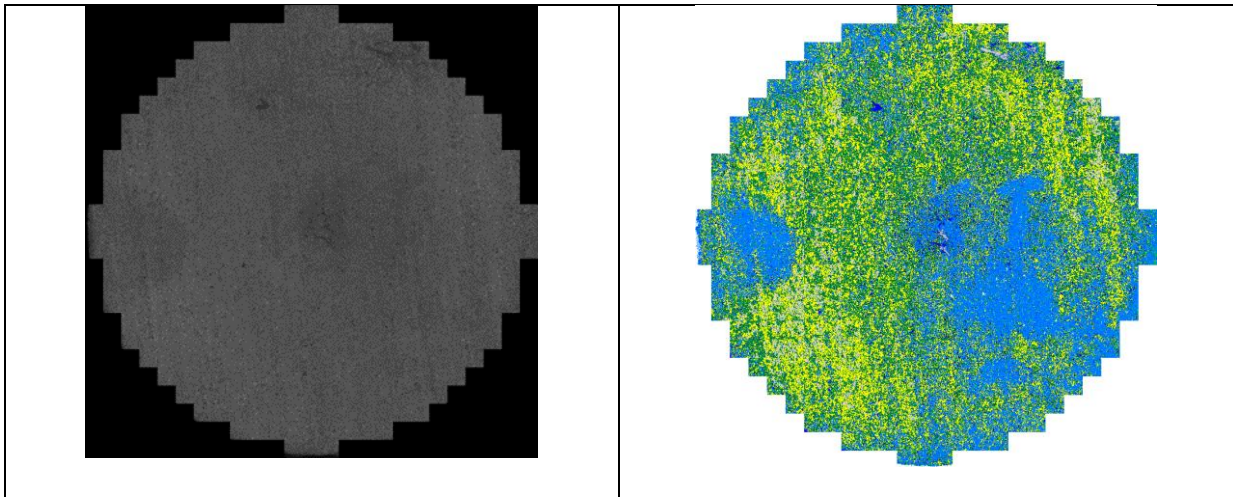


Figure 3. (a) BSEM images of Berea sample 15 (b) Mineral map of Berea sample 15

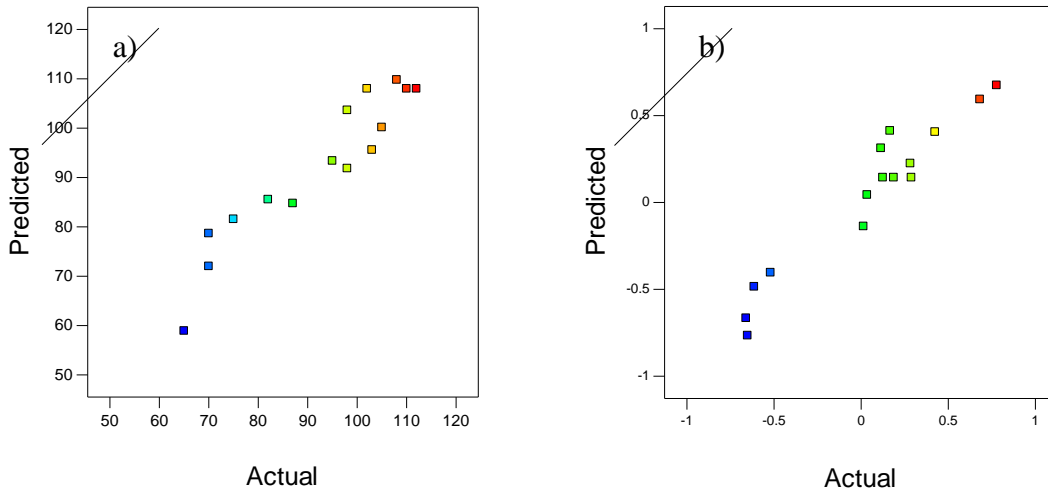
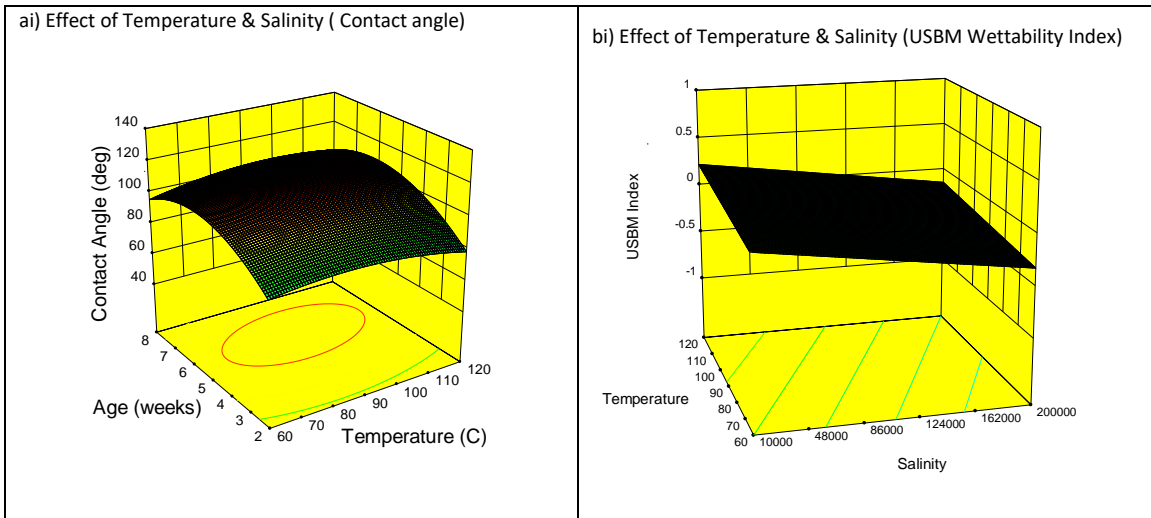


Figure 4. Predicted vs Actual for Berea Sandstone (a) Contact Angle (b) USBM Wettability Index
Figure 6. Optimal Aging Conditions (contact angle, 5.5 weeks, 95°C, 104,257 ppm)



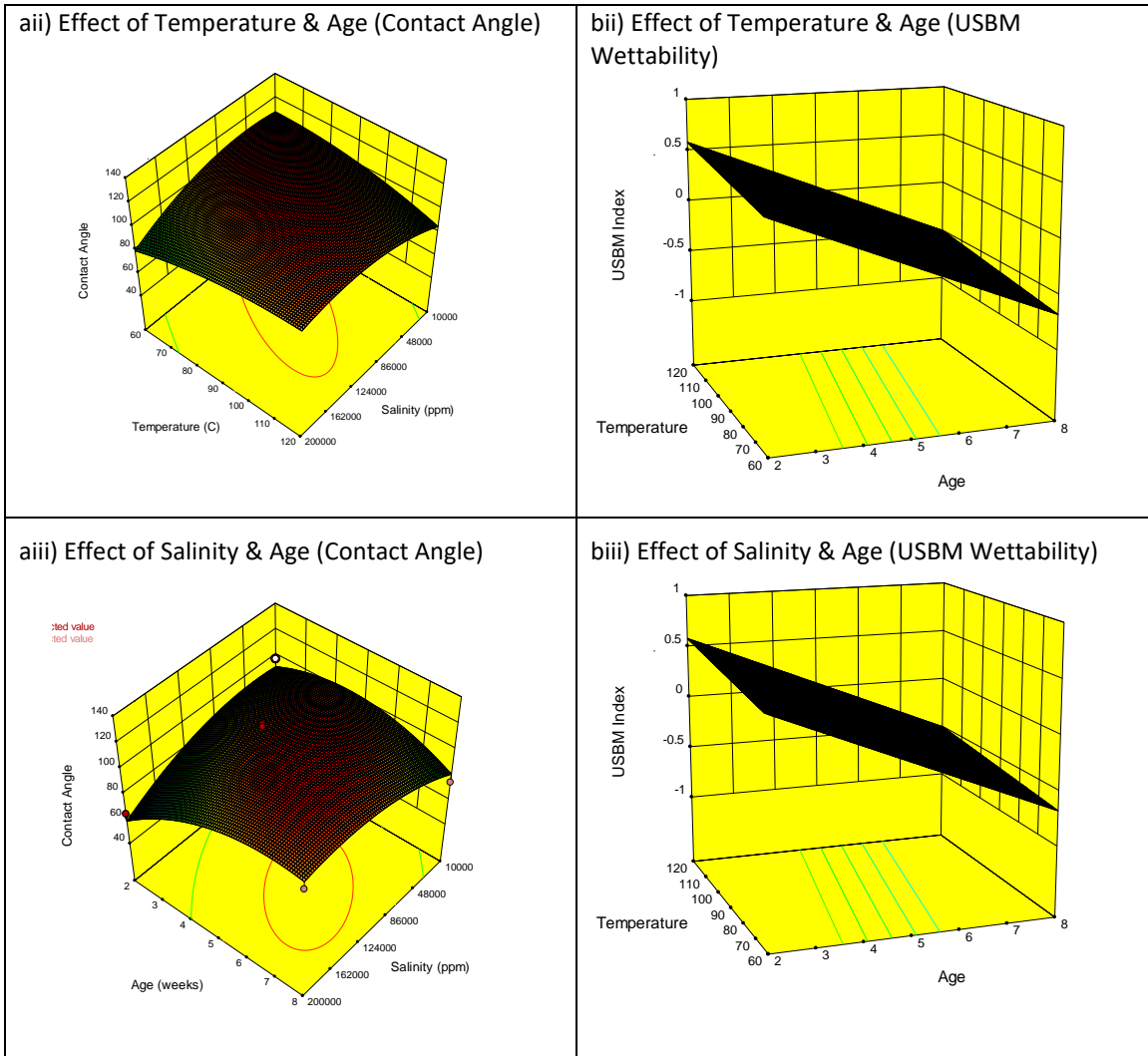


Figure 5. Contact Angle 3D Surface Models & 2D Contour Plots of Temperature, Salinity & Age

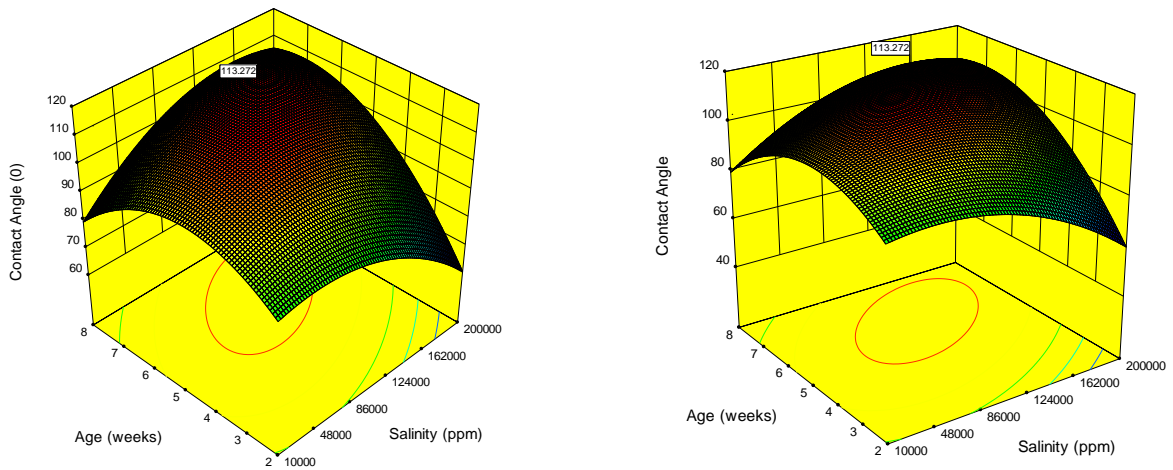


Figure 6. Optimum Ageing Conditions (contact angle, 5.5 weeks, 95°C, 104,257 ppm)

CONCLUSIONS

Response Surface Modelling (RSM) using three factor Box Behnken Design was successfully applied to study and optimise brine salinity, temperature of aging and aging time for wettability restoration in core samples.

1. A suite of 15 Berea sandstones were prepared and aged following Box Behnken design of experiments. Wettability was validated based on three response factors i.e., contact angle, USBM wettability index and organic content using SEM-MLA analysis.
2. The Berea samples used for this study were initially water-wet and under saturation with crude oil with little or no asphaltene content we were able to develop an optimization strategy for restoring core samples to desired wettability conditions found in the reservoir.
3. The experimental results were analyzed statistically using a regression model and analysis of variance (ANOVA). The ANOVA results for the three response factors showed high coefficient of determination values, ensuring a fit of the developed mathematical model with the experimental data.
4. Applying the optimization methodology, the optimum value of input parameters for restoring oil-wet conditions using contact angle measurement was calculated as brine salinity at 104,257 ppm, temperature at 95°C and time of aging at 5.5 weeks.
5. Optimization results for restoring wettability using USBM method provided an optimum brine salinity of 115,000 ppm, temperature of aging at 99°C and aging time for 6.2 weeks.
6. Optimal solution for restoration using SEM-MLA method is under process and the final results will be presented in a future paper.
7. Response surface models and 2-D contour plots were successfully developed for analyzing the interaction between the three input parameters on contact angle measurement. The results for Berea were in strong agreement with proven results.

ACKNOWLEDGEMENTS

The authors would like to thank Chevron Canada, Hibernia Management and Development Company (HMDC), Research and Development Corporation of Newfoundland and Labrador (RDC), Natural Sciences and Engineering Research Council of Canada (NSERC), and the Canadian Foundation for Innovation (CFI) for financial support. We thank our colleagues in the Hibernia EOR Research Group for technical support.

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Table 1. Brine Properties

Brine Salinity (ppm)	Density (kg/m ³)	Viscosity (cP)	IFT with oil (Nm/m)
10,000	1010	1.05	70.8
100,000	1070	1.21	22.4
200,000	1140	1.50	5.32

*Brine Composition: NaCl 84.38%; CaCl₂*2H₂O 12.32%; MgCl₂*6H₂O 2.57%; KCl 0.4%; Na₂SO₄ 0.32%*

Table 2. Experimental Measurement for Berea Sandstone

Berea Sample	Porosity (%)	Salinity (ppm)	Temp (°C)	Time (weeks)	Contact Angle (°)	SEM-MLA Oil %	Wettability Index (USBM)
B1	18.45	10,000	60	4	108	0.1	0.282
B2	18.19	10,000	120	4	70	0.2	0.424
B3	19.71	200,000	60	4	87	1.2	-0.013
B4	20.69	200,000	120	4	95	1.3	0.034
B5	18.24	10,000	90	2	98	0.59	0.778
B6	18.24	10,000	90	8	65	0.07	0.519
B7	18.12	200,000	90	2	75	1.81	0.113
B8	21.13	200,000	90	6	98	0.53	-0.651
B9	18.40	100,000	60	2	82	1.57	0.165
B10	18.45	100,000	60	8	70	0.39	-0.660
B11	18.52	100,000	120	2	103	2.54	0.681
B12	19.46	100,000	120	8	105	1.58	-0.613
B13	18.00	100,000	90	4	112	1.02	0.2881
B14	18.60	100,000	90	4	110	1.39	0.1870
B15	18.47	100,000	90	4	102	1.72	0.1249

Table 3. ANOVA test results for Berea Sandstone (Contact Angle)

Analysis of variance table [Partial sum of squares - Type III]					
Source	Sum of Squares	df	Mean Square	F Value	p-value (Prob > F)
Model	0.42	9	0.047	4.95	0.0464
A-Salinity	8.5E-003	1	8.5E-003	0.90	0.3863
B-Temperature	6.5E-005	1	6.5E-005	6.8E-003	0.9372
C-Age	0.082	1	0.082	8.65	0.0322
AB (Salinity x Temp)	0.070	1	0.070	7.35	0.0422
AC (Salinity x Age)	0.12	1	0.12	12.16	0.0175
BC (Temp x Age)	4.9E-003	1	4.9E-003	0.52	0.5042
A²	0.061	1	0.061	6.45	0.0519
B²	0.013	1	0.013	1.41	0.2890
C²	0.097	1	0.097	10.20	0.0242

Table 4. ANOVA test results for Berea Sandstone (USBM Wettability Index)

Analysis of variance table [Partial sum of squares - Type III]					
Source	Sum of Squares	df	Mean Square	F Value	p-value (Prob > F)
Model	2.77	3	0.92	48.00	<0.0001
A-Salinity	0.26	1	0.26	13.65	0.0035
B-Temperature	0.066	1	0.066	3.41	0.0919
C-Age	2.45	1	2.45	127.00	<0.0001

Table 5. ANOVA test results for Berea Sandstone (SEM-MLA Wettability Analysis)

Analysis of variance table [Partial sum of squares - Type III]					
Source	Sum of Squares	df	Mean Square	F Value	p-value (Prob > F)
Model	6.28	9	0.70	10.38	0.0095
A-Salinity	0.52	1	0.52	7.68	0.0393
B-Temperature	0.18	1	0.18	2.63	0.1659
C-Age	0.14	1	0.14	2.13	0.2039
AB (Salinity x Temp)	1.10	1	1.10	16.39	0.0098
AC (Salinity x Age)	0.17	1	0.17	2.48	0.1758
BC (Temp x Age)	0.40	1	0.40	5.91	0.0593
A²	2.12	1	2.12	31.55	0.0025
B²	0.83	1	0.83	12.42	0.0168
C²	0.25	1	0.25	3.72	0.1118

Table 6. Hibernia crude oil composition

Composition of Hibernia Crude Oil			
Component	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
i-C₅	0.0018	0.0070	0.0026
n-C₅	0.0028	0.0086	0.0040
C₆	0.0054	0.0165	0.0075
C₇₊	0.0163	0.0427	0.0206