APPLICATINON OF AN OPTIMIZATION METHOD FOR RESTORATION OF CORE SAMPLES FOR SCAL EXPERIMENTS

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ABSTRACT

An important aspect of preliminary core preparation for SCAL experiments is the restoration of core sample to its original wettability. The prevalent method for restoration of core sample to either strongly oil-wet or weakly oil-wet is largely dictated by increasing or decreasing the time of aging at reservoir temperature. There is no consistent or reliable method ascribed for a specific sedimentary core sample to restore to its original state which is obtained from preserved core samples. In this study we have identified three important parameters, brine salinity, restoration temperature, and restoration time (age in number of days) as contributing to wettability. The objective of this study was to determine the optimum level of these independent variables (brine salinity, temperature, and age) for restoring wettability. In this paper we use a Box Behnken model of surface response methodology to analyze the effects of brine salinity, temperature and age of conditioning on wettability restoration. The samples for this study include Berea sandstone and Silurian dolomite. The whole aging process was carried out using overburden coreholders and the wettability was experimentally validated using contact angle measurements. A regression model was developed and the adequacy of predicting the output variable (wettability) to nearly all conditions was verified. The study showed a strong influence of brine salinity and temperature towards wettability restoration, which was further confirmed by the regression model. Further 2-D and 3-D surface plots were generated to show the interaction between aging time, temperature, and brine salinity on contact angle.

INTRODUCTION

Specialized Core Analysis (SCAL) data are important for reservoir characterization and secondary and tertiary production optimization. An important consideration for ensuring quality and reliability in SCAL experiments is the restoration of wettability of core samples. Incomplete core sample restoration may lead to unrealistic estimates of residual oil saturation which further affects relative permeability and capillary pressure measurements. Reservoirs are generally considered to be oil-wet and can be achieved either from native or from restored states (Hirasaki 1979). 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 (Anderson 1986). Hence laboratory experiments are routinely performed on core samples from restored state. The restoration process typically involves cleaning the core sample with various solvents rendering it water wet. The cleaned samples are 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 inundated with various aging strategies, but the conundrum is the lack of a commonly accepted aging process for either sandstones or carbonates. In this paper we try to address the effect of three parameters that are generally agreed upon as contributing to the aging process (Anderson 1986; Morrow 2000): brine salinity, aging time, and aging temperature.

According to Anderson (1986) for an oil-brine-rock system, the rock is classified as water-wet if the contact angle between a water droplet and rock is $< 75^o$, intermediate wet if the contact angle is between $75\text{-}100^o$ and oil wet if the contact angle is $105 - 180^o$. This wettability characterization using contact angle method has so far been successfully applied on carbonates (Alotaibi 2010), sandstones, and shales (Alvarez 2016).

Aging time and temperature are generally accepted to be the two most important factors that contribute to the aging (wettability alteration) process. Anderson (1986) indicated that 1000 hours (40 days) of aging at reservoir temperature is sufficient for wettability equilibrium based on experiments performed using Berea plugs where wettability changed from water to wet to moderately oil wet in 40 days. Morrow (1995, 2000, 2013) in various water flooding and imbibition studies on Berea samples observed lowest wettability index values with water when the samples were aged at $80 - 90^oC$ for 10 days. Torsæter (1995) identified that short term imbibitions were significantly reduced by shortened aging time but aging at high temperature (90^oC) yielded more recovery. Carbonates, being natural slightly oil wet, were shown to display oil-wet attributes when aged for only 2 days at 90^oC (Alotaibi 2010).

During the aging process, it is important to saturate the core with brine prior to oil to ensure brine chemistry is not ignored. Numerous studies have described the effect of brine salinity on aging by changing the concentration and composition. Tang and Morrow (1999) identified that increasing brine salinity from 15,000 to 35,000 ppm had a significant effect on altering wettability in Berea sandstone where oil production decreased during water flooding. Vijapurapu et al. (2003) investigated the effect of various dilutions of brine (2000 - 20,000 ppm) on oil wettability and found that there was a threshold brine concentration at 4500 ppm for wettability alteration. Chattopadhyay (2002) varied the concentration of brine (40,000 - 200,000 ppm) and found increasing wettability altering behavior with increasing NaCl concentration. Similar trends were observed by Jadhunandan and Morrow (1995) on Berea exhibiting more oil-wet characteristics when aged with increasing brine salinity from 40,000 to 60,000 ppm.

EXPERIMENTAL METHODODLOGY

The objective of this work is to optimize the aging process for wettability restoration of reservoir rocks for SCAL experiments. Statistical design of experiments (DOE) provides an understanding of the parametric effects controlling a process so as to decrease the number of experiments, time, and resources. In this paper we chose to apply the Box Behnken response surface method to optimize the input parameters i.e. aging time, temperature and brine salinity for the desired output, i.e. wettability (contact angle). For a complex experiment involving multiple parameters of interest, response surface modeling provides an estimate of the interaction of the input parameters and the quadratic effects associated with it, the result a shape of the response surface under investigation. Box Behnken requires only 15 trials for a three factor experiment and providing maximum efficiency for a surface model. Tables 2 and 3 summarize the experimental plan. 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 optimization (brine salinity, aging time, and aging temperature).

Three brine concentrations (80,000; 100,000; 120,000 ppm) were used to saturate the core plugs and carry out the wettability experiments. In addition to the sodium and chloride ions, divalent calcium and magnesium ions were also added to the brine. Lichaa (1992) demonstrated that these divalent ions affect wettability. The brine salinity used is high compared to literature, but it represents many reservoirs globally, including offshore Newfoundland. Hibernia (offshore Newfoundland) dead crude oil was used with a measured viscosity of 5.9 cP and a density of 878 kg/m^3 at room temperature. The oil was vacuumed for 48 hours to prevent any gas production during the drainage and aging processes. The core samples were saturated with oil in its unfiltered original state to include light and heavy components in the restoration process. Berea sandstone and Silurian dolomite core samples were used. The Berea samples came from Cleveland Quarries, Ohio with porosity from 18 to 22 % and gas permeability of 350 mD. The Silurian dolomite was obtained from Kocurec Industries, Texas. Porosity of the dolomites varied from 10 to 14% and the gas permeability was measured to be 120 mD.

The core samples for testing were initially cut to 1 - 2” long and sonicated for 45 minutes to remove any fines from the pores. The samples were then dried in an oven for 24 hours at 100^{o}C. The dried samples were weighed and loaded in a pressure cell and vacuumed to 100 microns. Brine was injected to the vacuumed pressure cell and samples were saturated with brine for 24 hours. The wet weight of the samples were measured to calculate the pore volume and porosity. The saturated core samples were then loaded in a core holder for centrifuge. The overburden pressure was set to 3000 psi using silicon oil.

A Vinci Rotosilenta 630RS refrigerated centrifuge was used to bring the brine saturated core samples to connate water conditions. A calibrated receiving tube in the coreholder allows for collection of the displaced fluid and a high resolution camera with a glass window in the centrifuge allows for fluid level measurement in the receiving tube. A drainage test with oil displacing brine was carried out on all the samples with the receiving tube filled with water and couple of pore volumes of crude oil. During the initial set-up the coreholder inlet was vacuumed and pressurized with crude oil to ensure circulation of the crude oil from the receiving tube to the core samples to displace any dissolved gases in the oil. Centrifugation was carried out in eight steps from 500 to 3000 rpm with 3 hours 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 reduced to ~1500 psi to compensate for pressure build up due to heating the samples to aging temperature. Overburden pressure was adjusted to 3000 psi after the pressure stabilized. Once the aging process was completed, the samples were removed from the coreholders and thin slices (5 mm) cut from the core sample (without using water to avoid any contamination). The cut samples were smoothened to obtain a flat surface and 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 sandstone and dolomite samples are listed in Tables 2 and 3. Figure 1 shows representative contact angles for the brine sessile drop in the presence of air.

RESULTS AND DISCUSSION

Box Behnken response surface methodology, with three factors, was chosen to investigate and optimize core restoration (wettability) based on brine salinity, temperature for aging and time of aging. The models are then used to suggest optimal aging conditions for the sandstone and dolomite. The experimental sequence was randomized in order to minimize bias and variability in contact angle measurements. The experimental results, shown in Tables 2 and 3, were analyzed using Design Expert ® Software. The optimal aging conditions were predicted using different mathematical models (linear, quadratic, and polynomial) which were fitted to correlated relationships between brine salinity, aging temperature and time (input variables) and contact angle (response). The experimental data was analyzed 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 visualize the relationship between the three input variables and the contact angle.

The statistical model developed from the experimental data for both sample types were checked for adequacy to determine if the results are reliable. The linear empirical model was chosen as best to fit the experimental results obtained from the Box Behnken design. Figure 2 shows a plot of experimental versus predicted contact angle values for both the sandstone and dolomite where a reasonable agreement was observed as most of the data points lie close to the diagonal line. The resulting linear regression equations for Berea sandstone and Silurian dolomite are indicated in Equations 1 and 2 using the actual values of input parameters.

$$Contact\ Angle\ (Berea) = 47.95 + 1.56\times10^{-4} Salinity\ (ppm) - 0.06 Temp\ (^{\circ}\mathrm{C}) + 8.56 Age\ (Weeks) \tag{1}$$

$$Contact\ Angle\ (Dolomite) = 56.49 + 8.75\times10^{-5} Salinity\ (ppm) + 0.22 Temp\ (^{\circ}\mathrm{C}) + 1.68 Age\ (Days) \tag{2}$$

A diagnostic test was carried out to compare the experimental data with the model predicted results. Sequential model sum of squares and model summary statistics were applied on various higher order polynomial models to tests the adequacy. The results of the analysis of variance (ANOVA) tests for the Berea sandstone and Silurian dolomite are presented in Tables 4 and 5 and show the adequacy or fitness (F-Test) of a developed model using multiple regression through partial least squares method. The analysis of variance (ANOVA) shows that the linear models are indeed significant for both the sandstone and dolomite, as shown by the overall model p-values of 0.0087 and 0.0073, respectively. These p-values are significantly less than the significance cut-off value of 0.05. The higher F-values and low p-values for age (sandstone); as well as age and temperature (dolomite) indicate the significance of these input parameters respectively. Conversely, the ANOVA tables show that salinity and temperature do not have significant influence on wettability alteration for Berea sandstone, likewise, salinity does not significantly alter the wettability for Silurian dolomite. Wettability alteration towards oil-wet is mostly due to age for sandstone as well as age and temperature for dolomite.

From the developed statistical models we were able to produce 3-D response surface plots and 2-D contour plots for the three input parameters against contact angle. Figures 3ai, aii and aiii represent the contour plot and 3-D RSM for Berea sandstones. The 2-D and 3-D plots provide an understanding of the interaction between two input parameter, while keeping the third parameter constant. These plots reiterate the results found in the ANOVA (Tables 4 and 5) showing that aging time is the most significant factor impacting wettability alteration for Berea. We see this directly from Figures 3aii and 3aiii where the slope of the contact angle varies significantly with age but there is little change in contact angle with respect to temperature or salinity. As the aging period was extended from 2 to 6 weeks, the contact angle measurement increased indicating a shift from intermediate oil-wet to strongly oil-wet characteristics. A similar trend was observed by Morrow (2000) where Berea samples displayed less water-wet behavior at longer aging periods. The observation from this study is also in strong agreement with Anderson's (1991) threshold of 40 days to attain intermediate oil-wet conditions. Figures 3ai, ii, and iii show that varying temperature (60 - 90^{o}C) and brine salinity (80,000 – 120,000 ppm) are insignificant in altering wettability for sandstone. This is highly likely due to the input range for temperature and brine salinity being too narrow and hence there is no significant impact on wettability alteration. The brine samples used for this work are near neutral pH of ~6.5. At this pH, silica particles in Berea tend to become negatively charged. This enables more adsorption of organic acids in the crude oil on the silica surface and making it more oil wet. Since the pH of all three brines were almost same (6.39 to 6.57), the brine salinity effect on wettability alteration is rather muted. This is in in contrast to Chattopadhyay (2002) work on wettability alteration in sandstone when the brine concentration was drastically increased from 3000 to 200,000 ppm.

The response surface plots for Silurian dolomite are shown in Figures 3bi, bii, & biii. Although the aging process was carried out for only 6 days, aging time and temperature were found to be the significant input parameters contributing to wettability restoration.

The aging time for dolomites was reduced due to their inherent oil-wet characteristics and commonly cited time in literature. In this study, we were able to restore dolomite to intermediate oil-wet conditions by increasing aging time and temperature. The 3-D response surface in Figure 3 indicates a strong change in contact angle with change in temperature and time. Brine salinity was found to be less significant in affecting the wettability. The brine samples used for this work have a neutral pH rendering the calcites in carbonates positively charged (Anderson 1991) resulting in a strong bond with negatively charged crude oil. Hence the interaction between oil and carbonate rock particles dominated over various concentrations of brine on calcite particles (Adamson 2000). A similar behavior in carbonates was reported by Alotaibi (2000) where lower contact angle was produced when analyzed with sea water of 54,680 ppm salinity.

Alternatively, a reduced linear model was tested for both Berea sandstone and Silurian Dolomite by ignoring temperature and salinity for the Berea and salinity for the dolomite. The resultant ANOVA tests for Berea are shown in Tables 6 and 7. The results show that the models are significant for the reduced parameters considered. These results confirm that there is little significance of salinity and temperature on the wettability alteration of Berea sandstone and little significance of salinity on the wettability alteration of Silurian dolomite for the range of values investigated.

Optimization: The regression models developed in this study were utilised for optimising the contact angle measured beyond a certain value to validate aging. As previously mentioned the wettability criteria for an oil-rock-brine system, a contact angle >105° is considered oil wet. The Derringer's desirability function in Design expert searches for a combination of input parameters to maximise the contact angle. Applying the Derringer's desirability function, the optimum aging conditions for Berea wettability restoration were found to be 99,165 ppm, 80°C and 5.7 weeks. The results are graphically represented via 3-D response surface in Figure 4, where the surface plateaus (reaching the desired contact angle) after reaching the optimum value. The optimum aging conditions to restore intermediate oil wettability in Silurian dolomite were found to be 103,000 ppm, 88°C and 5.8 days. As the optimization methodology is limited to highest value of contact angle observed in the experiment, the Silurian dolomites were optimized for intermediate wettability. The optimized response surface models for dolomites are shown in Figure 6.

CONCLUSIONS & RECOMMENDATIONS

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

1. 15 Berea sandstone and Silurian dolomite samples were aged according to Box Behnken design of experiments measuring contact angle to confirm wettability.
2. The experimental results were analyzed statistically using a regression model and analysis of variance (ANOVA). The ANOVA results for both Berea and Silurian

Dolomite showed high coefficient of determination values, ensuring a satisfactory fit of the developed mathematical model with the experimental data.

3. Response surface models and 2-D contour plots were generated to analyze the interaction between the three input parameters on contact angle measurement. The results for both samples were in strong agreement with proven results.
4. The ANOVA and surface plots indicate the significance of aging time on wettability alteration for Berea sandstone and Silurian dolomite. Brine salinity (at the concentrations tested) did not prove significant in altering wettability for either rock sample. Aging temperature was shown to affect wettability alteration of the dolomite only. A wider range of salinity (and pH) and temperature may show that there is more of an effect on aging and should be further studied.
5. Applying the optimization methodology, the optimum value of input parameter for restoring oil-wet conditions in Berea samples were successfully calculated as brine salinity at 99,165ppm, temperature at 80°C and 5.7 aging weeks.
6. Intermediate oil wettability in Silurian dolomite suggests optimum aging conditions using 103,000 ppm brine salinity, 88°C, and 5.8 aging days.
7. Both samples should be aged even longer to confirm the optimum aging time.
8. We are in the process of confirming wettability alteration using Amott-USBM tests as well as SEM imaging. We are hopeful that the SEM imaging may further elucidate the rock-fluid ionic interactions.

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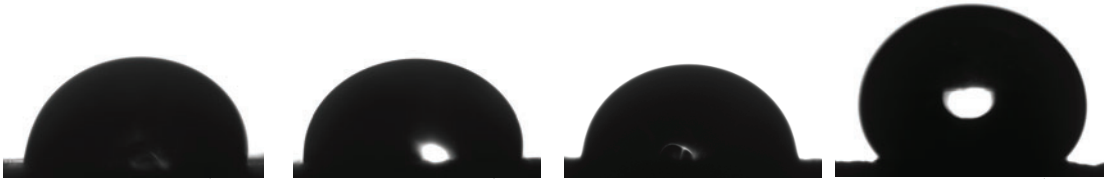


Dolomite 1 (84^{O}) Dolomite 7 (90^{O}) Berea 4 (85^{o}) Berea 12 (122^{O})

Figure 1. Contact angle measurements for Berea and Silurian Dolomite Samples.

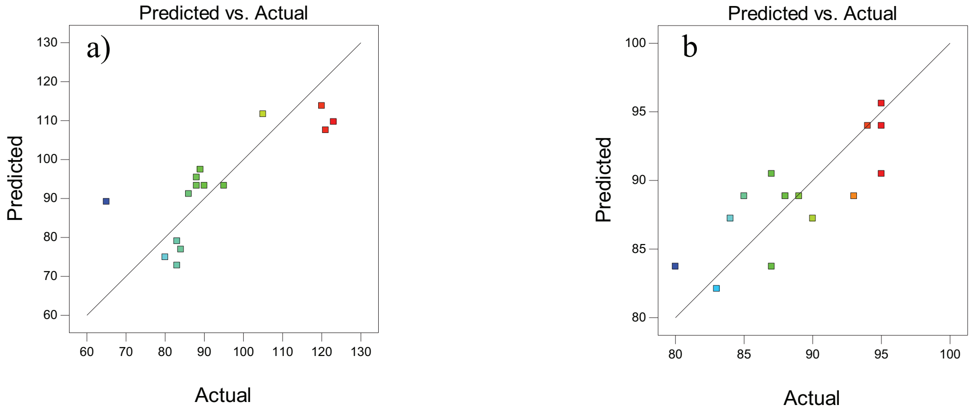
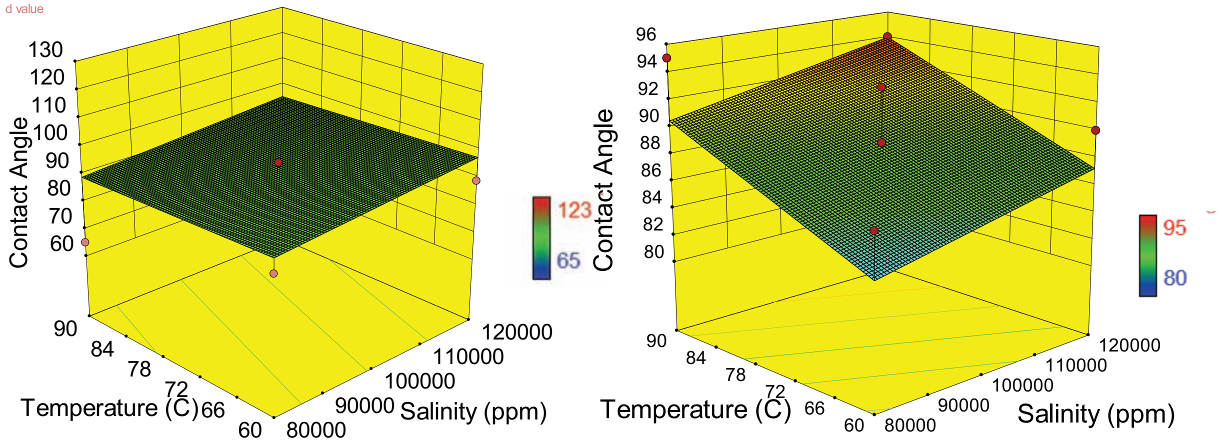
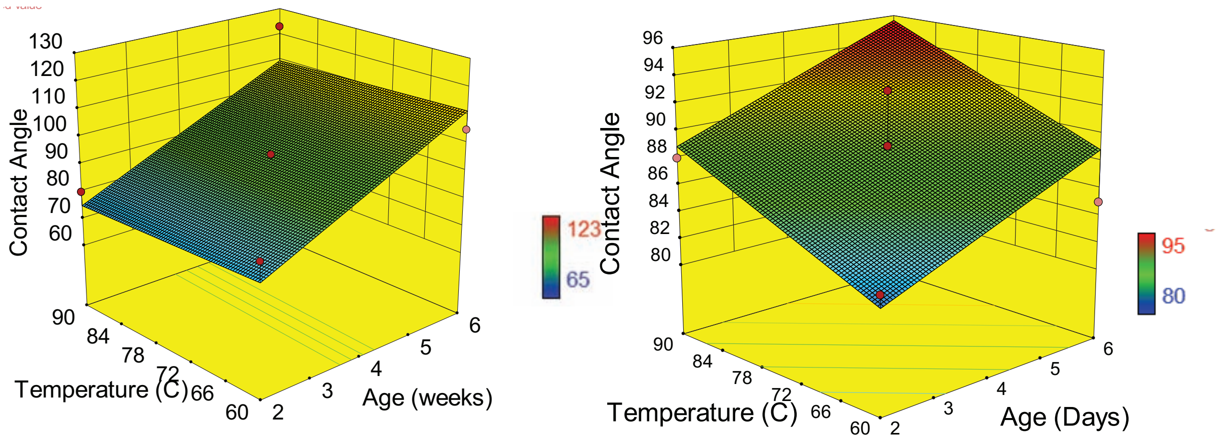


Figure 2. Predicted vs Actual for (a) Berea Sandstone and (b) Silurian Dolomite



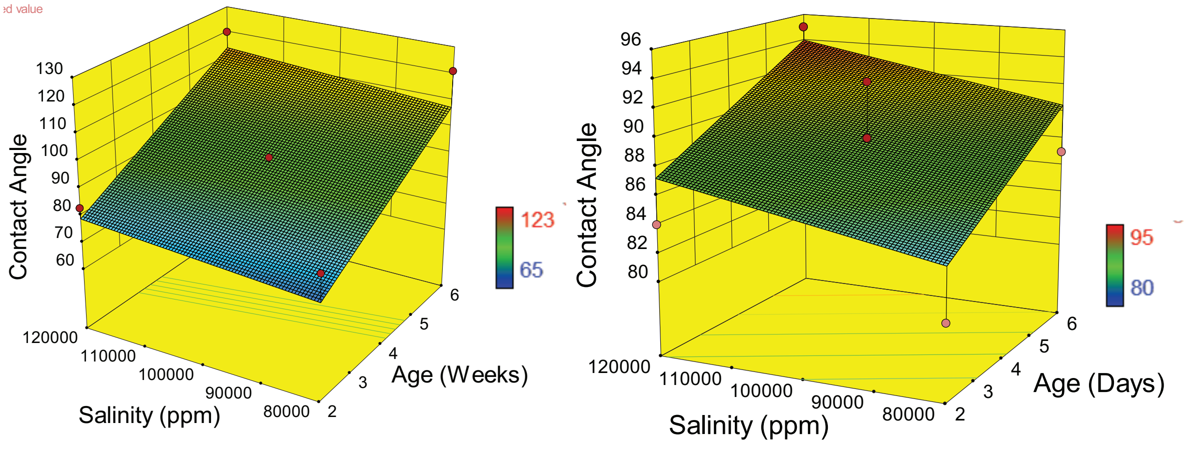
ai) Effect of Temperature & Salinity on Berea

bi) Effect of Temperature & Salinity on Dolomite



aii) Effect of Temperature & Age on Berea

bii) Effect of Temperature & Age on Dolomite



aiii) Effect of Salinity & Age on Berea

biii) Effect of Salinity & Age on Dolomite

Figure 3. Contact Angle 3D RSM & 2D Contour Plots of Temperature, Salinity & Age

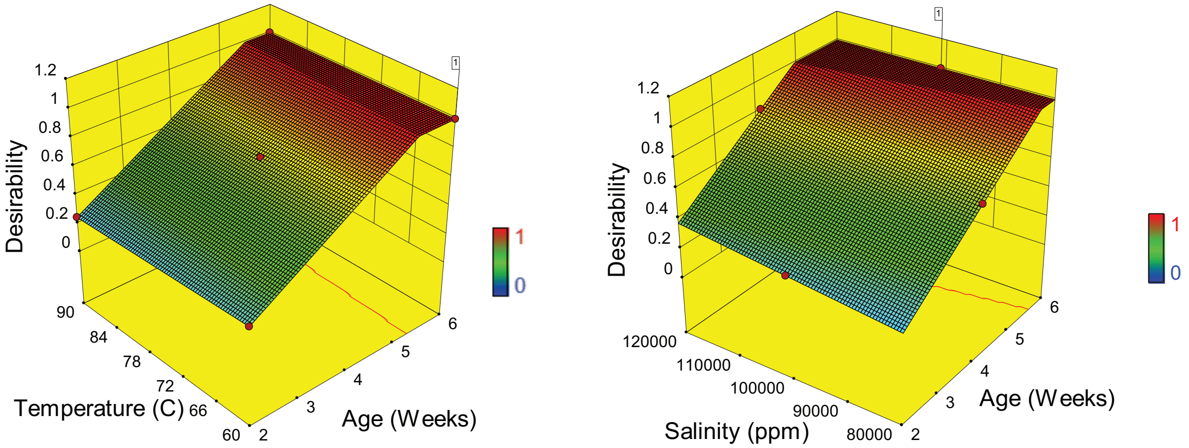


Figure 4. Optimum Aging Conditions for Berea (5.7 weeks, 80°C, 99,165 ppm Salinity)

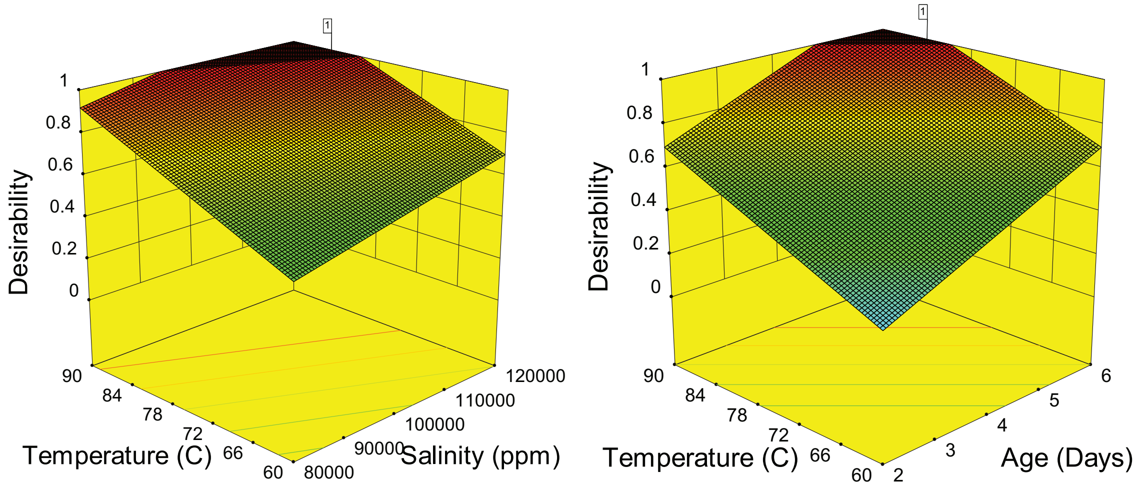


Figure 5. Optimum Aging Conditions for Silurian Dolomite (5.8 days, 88.5°C, 103,000ppm Salinity)

Table 1. Brine Properties

Brine Salinity (ppm)	Density (kg/m^3)	Viscosity (cP)	IFT with oil (Nm/m)	pH
80,000	1080	1.13	32.8	6.39
100,000	1070	1.21	22.4	6.45
120,000	1060	1.38	10.56	6.51

*Brine Composition: NaCl 84.38%; $CaCl_2$*$2H_2O$ 12.32%; $MgCl_2$*$6H_2O$ 2.57%; KCl 0.4%; Na_2SO_4 0.32%*

Table 2. Contact Angle Measurement for Berea Sandstone

Berea	Porosity (%)	Salinity (ppm)	Temperature (°C)	Time (weeks)	Contact Angle (°)
Sample 1	21.21	80,000	60	4	86
Sample 2	21.21	80,000	90	4	65
Sample 3	19.29	120,000	60	4	89
Sample 4	18.97	120,000	90	4	88
Sample 5	21.08	80,000	75	2	83
Sample 6	20.98	80,000	75	6	121
Sample 7	18.81	120,000	75	2	83
Sample 8	19.17	120,000	75	6	120
Sample 9	19.86	100,000	60	2	84
Sample 10	20.00	100,000	60	6	105
Sample 11	19.96	100,000	90	2	80
Sample 12	20.45	100,000	90	6	123
Sample 13	20.93	100,000	75	4	88
Sample 14	20.38	100,000	75	4	95
Sample 15	20.55	100,000	75	4	90

Table 3. Contact Angle Measurement for Silurian Dolomite

Silurian Dolomite	Porosity (%)	Salinity (ppm)	Temperature (°C)	Time (Days)	Contact Angle (°)
Sample 1	12.69	80,000	60	4	80
Sample 2	14.20	80,000	90	4	93
Sample 3	13.26	120,000	60	4	95
Sample 4	12.69	120,000	90	4	87
Sample 5	13.43	80,000	75	2	95
Sample 6	13.03	80,000	75	6	84
Sample 7	12.00	120,000	75	2	90
Sample 8	10.82	120,000	75	6	87
Sample 9	12.69	100,000	60	2	88
Sample 10	10.89	100,000	60	6	95
Sample 11	14.20	100,000	90	2	94
Sample 12	14.06	100,000	90	6	89
Sample 13	12.55	100,000	75	4	83
Sample 14	13.22	100,000	75	4	85
Sample 15	14.12	100,000	75	4	88

Table 4. ANOVA Test Results for Berea Sandstone (age, temperature, salinity)

Analysis of variance table [Partial sum of squares - Type III]					
Source	Sum of Squares	df	Mean Square	F Value	p-value (Prob > F)
Model	2501.25	3	833.75	6.48	0.0087
A-Salinity	78.12	1	78.12	0.61	0.4524
B-Temperature	8.00	1	8.00	0.062	0.8077
C-Age	2415.13	1	2415.13	18.76	0.0012

Table 5. ANOVA Test Results for Silurian Dolomite (age, temperature, salinity)

Analysis of variance table [Partial sum of squares - Type III]					
Source	Sum of Squares	df	Mean Square	F Value	p-value (Prob > F)
Model	206.75	3	68.92	6.83	0.0073
A-Salinity	24.50	1	24.50	2.43	0.1475
B-Temperature	91.12	1	91.12	9.03	0.012
C-Age	91.13	1	91.13	9.03	0.012

Table 6. ANOVA Test Results for Berea Sandstone (age only)

ANOVA for Response Surface Reduced Linear model					
Source	Sum of Squares	df	Mean Square	F Value	p-value (Prob > F)
Model	2415.13	1	2415.13	20.90	0.0005
C-Age	2415.13	*1*	2415.13	*20.90*	*0.0005*

Table 7. ANOVA Test Results for Silurian Dolomite (age & temperature, no salinity)

ANOVA for Response Surface Reduced Linear model					
Source	Sum of Squares	df	Mean Square	F Value	p-value (Prob > F)
Model	182.25	2	91.12	8.07	0.006
B-Temperature	91.12	1	91.12	8.07	0.0149
C-Age	91.13	1	91.13	8.07	0.0149