[Regular Paper]

Experimental Investigation into Effects of Crude Oil Acid and Base Number on Wettability Alteration by Using Different Low Salinity Water in Sandstone Rock

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In the last two decades, the level of investigation into low salinity water (LSW) flooding has sharply increased. LSW with adequate composition and salinity is injected into the reservoir, which changes the properties of the reservoir rock, thus improving oil recovery. However, for certain reservoirs there might be no improvement at all. This has led for further studies on what exactly governs the lack of improvement upon LSW injection in different reservoirs. Focus was placed on acidic and basic components present in crude oil. They are characterized by measuring total acid number (TAN) and total base number (TBN). This study investigates the wettability alteration of Berea sandstone rock with different crude oil acid and base numbers at specific LSW concentrations and compositions. For this purpose, the sessile drop method was used in specified times. Moreover, the experiments were conducted in two stages, which included crude oil A and B. The experimental results revealed that a significant change in wettability was experienced by the LSW composition of cations K^+ and Na^+ for the crude oil composition with lower acid number.

Keywords

Total acid number, Total base number, Low salinity water, Berea sandstone, Sessile drop method

1. Introduction

The LSW injection method has since gained wide attention and interest from many researchers. Nasralla *et al.* (2011), by conducting injection studies with de-ionized water and seawater, demonstrated that the former improved oil recovery significantly rather than the latter¹⁾. With the purpose of enhancing oil recovery, low salinity water (LSW) with adequate composition and salinity is injected into the reservoir, which changes the wetting properties of the reservoir rock into that favoring oil recovery^{2,3)}. There are many theories regarding the mechanisms of wettability alteration in presence of LSW which are fine migration⁴⁾, the pH effects³⁾, multiple-component ion exchange (MIE)⁵⁾, and wettability modification due to double layer expansion^{2,6)}. Ligthelm *et al.* (2009) suggested that the application of LSW results in an expansion of the ionic electrical double layer between the clay and the oil interfaces. This causes the oil to be desorbed from the rock surface thus increasing the water wetness⁶⁾.

Residual oil determination is in part dependent on the wettability of the rock. Wettability measurements are

performed in laboratory by several methods. Among many, there are the sessile drop method and zeta potential, which provide the results in terms of contact angle. For this case, the degree of wetness is determined following certain classifications. According to Fanchi *et al.* (2010), a reservoir rock is considered strongly water wet if the contact angle with reference to water varies between 0–30°; moderately water wet is from 30–70°; neutrally wet 75–105°; moderately oil-wet 105–150° and finally 150–180° for strongly oil-wet⁷⁾.

2. LSW Mechanism

Clay minerals constitute the major percentage of sedimentary rocks and they have negative surface charge⁸⁾. This property causes the positively charged ions to adhere to it as in the case of Ca^{2+} , Mg^{2+} , Na^+ , or K^+ . Ions such as Ca^{2+} and Mg^{2+} are called divalent cations. They are present in great amounts in the formation water and assist in adsorption of oil onto the surfaces of the rock. This eventually creates oil-wet conditions for the rock and thus causing most of it to be trapped within the reservoir⁵⁾. Upon LSW injection (**Fig. 1(a)**) and due to the ion exchange capability of the clay minerals, there is replacement of the divalent cations by the monovalent cations. The divalent cations leave the rock surfaces along with the desorbed oil (**Fig. 1(b)**).

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Fig. 1 MIE Mechanism Process

Table 1 Properties of the Rock from the Company

Formation	K [mD]	ϕ [%]	UCS	Dimension
Kipton	100 mD brine permeability	20 %	6500-8000 psi	1.5 inch (D) $\times$ 12 inch (L)

Table 2 Rock Sample Properties Measured by the Poroperm Machine

$K_{[gas]}$ [mD]	b [psi]	β [ft ⁻⁴]	V_p [cm ³]	V_g [cm ³]	ϕ [%]	V_b [cm ³]	Grain density
161.378	9.18E-06	- 1.28E + 07	16.137	67.911	19.199	84.048	2.632

Since the monovalent ions are one ended, their adsorption to clay surfaces causes no further oil to be adsorbed and thus creating a water wet condition (**Fig. 1(c)**). The aforementioned methods constitute what is called the multiple-component ion exchange (MIE) mechanism⁵.

3. Materials

3.1. Rock

The reservoir rock was obtained from the KOCUREK Co. (USA). Its additional properties as received from the company are displayed in the **Table 1**. Some of these properties were confirmed by performing mineral analysis by the X-ray diffractometer (XRD) and basic routine core analysis by the poroperm machine. **Table 2** shows results obtained from the poreperm machine. The XRD analysis was conducted with the main aim of identifying the silica oxide based minerals and its pattern is displayed in the **Fig. 2**. Moreover, the **Table 3** further summarizes results for mineral composition from XRD.

The values of permeability from **Tables 1** and **2** are different. This is because of the different methods used for measuring it. The company permeability (**Table 1**) is brine measured while that of the poreperm is gas measured (**Table 2**). For gas-based permeability, the Klinkenberg effect is present which contributes to the high value of the permeability.

3.2. Crude Oil

The effectiveness of LSW is in part influenced by the

Fig. 2 Auto Indexing Pattern for Silicon Oxide-SiO₂

presence of polar components within the crude oil. No effect has been observed by the use of oil free from polar components². Total acid number (TAN) and total base number (TBN) are important parameters for characterizing the polar components within the crude oil. TAN is defined as the total amount of potassium hydroxide (KOH) in milligrams, required for neutralizing 1 g of the petroleum acid in the crude oil; whereas, TBN is defined as the total amount of KOH in milligrams per 1 g of the crude oil⁹. Two samples of crude oil designated as crude oil A and B were used. Their specific total acid and base numbers were measured with reference to the TAN ASTM D 664 and TBN ASTM D 2896 standard methods by the use of METTLER TOLEDO Potentiometric Titrator. The results are summarized in the **Tables 4** and **5**. From the tables, it is patent that crude oil A has higher TAN and

Table 3 Mineral Composition from the XRD

[%]

SiO ₂ (silica)	Al ₂ O ₃ (alumina)	FeO/Fe ₂ O ₃ (iron oxide)	TiO ₂ (titanium oxide)	CaO (calcium oxide)	MgO (magnesium oxide)	Alkalies	H ₂ O (water)	Undetermined	Total
86.47	7.31	1.14	0.70	1.21	0.11	1.65	1.20	0.21	100

Table 4 Total Acid and Base Number of the Crude Oil A

TAN [mgKOH/g]	TBN [mgKOH/g]	Density [g/cm ³]	Viscosity [cP at 25 °C]	Viscosity [cP at 85 °C]
0.667	0.146	0.821	3.761	1.464

1 cP = 1×10^{-3} Pa s.

Table 5 Total Acid and Base Number of the Crude Oil B

TAN [mgKOH/g]	TBN [mgKOH/g]	Density [g/cm ³]	Viscosity [cP at 25 °C]	Viscosity [cP at 85 °C]
0.174	0.750	0.813	3.085	1.233

1 cP = 1×10^{-3} Pa s.

lower TBN as compared to crude oil B that has lower TAN and higher TBN.

3.3. Brine Properties

The brines for LSW were prepared by mixing controlled amounts of K⁺, Na⁺, Ca²⁺, Mg²⁺, and Cl⁻ salts with distilled water. Initial connate water saturation for the rock was obtained by the use of formation water prepared with high concentration of Ca²⁺ and Mg²⁺ cations with other amounts of HCO₃⁻, SO₄²⁻, K⁺, Na⁺, and Cl⁻. **Table 6** summarizes the properties of all brines.

4. Methodology

Figure 3 shows a schematic drawing of the experimental procedures. The first step consisted in extracting 62 core slices from the core plugs. Then the slices were polished and cleaned with toluene. Next, they were dried in oven at 105 °C for about 24 h. The second step entailed the saturation of the core slices with formation water (FW) by using a vacuum pump for 2 to 4 h. After saturation, the slices were put in the oven for nearly 72 h at 80 °C. Next, they were aged in crude oil (31 slices for crude oil A and 31 for crude oil B) at stabilized temperature of 80 °C for 72 h. Finally, the slices were immersed in respective LSW enumerated from one to 30 LSW₁-LSW₃₀ and formation water (**Fig. 4**)

Wettability measurement was performed by the sessile drop method using the IFT 700 equipment (**Fig. 5**). Here, results were recorded after each 0, 6, 12 and 24 h by taking photos of the water drop shape on the slices (**Figs. 6-11**). This procedure was repeated for all the compositions. In addition, the experiments were performed in two stages each of which focused on each type of oil (crude oil A and B). The methods described above, follow a modification from the standard

ones by Shabib-Asl *et al.* (2014)¹⁰.

5. Results

Figures 12 through **16** represent changes of contact angle with time for the NaCl, KCl, MgCl₂, CaCl₂, FW compositions and the mixture of all of these components for the crude oil A and B. The concentrations of LSW are varying from 500 to 6500 ppm. Here, for all LSW compositions and concentrations, the contact angle is changing throughout the time except for the formation water composition that depicts no appreciable changes.

5.1. KCl Composition

Figure 12 depicts changes of contact angle for the KCl composition. Both crude oil A and B create different effects for the set intervals of time. Furthermore, the changes of contact angle for crude oil B are larger and faster than crude oil A. For instance, at the end of 24 h, the change of contact angle for LSW-20 for crude oil B is greater than that of LSW-20 for crude oil A (approximately 110 to 55° versus 110 to 65°, respectively) as shown in **Fig. 12**.

In **Fig. 12A**), the contact angle decreases steadily and rapidly from zero to 12 h. However, from 12 to 24 h, the decrease in contact angle is slow. For the interval of 24 h, the composition LSW-19 creates maximum change of contact angle (112 to 62°). Conversely, LSW-24 creates least changes of contact angle (111 to 89°). For the **Fig. 12B**), the contact angle decreases rapidly from zero to 6 h. From 6 to 12 h the decrease in contact angle is slow. From 12 to 24 h, the contact angle continues to decrease slowly. For the whole interval of 24 h, still the composition LSW-19 causes largest changes of contact angle (112 to 53°). In contrast, LSW-24 causes smallest changes of contact angle (111 to 82°).

Table 6 Composition and Concentration of Different LSW

	Na ⁺ [mg/L]	Ca ²⁺ [mg/L]	Mg ²⁺ [mg/L]	K ⁺ [mg/L]	HCO ₃ ⁻ [mg/L]	SO ₄ ²⁻ [mg/L]	Cl ⁻ [mg/L]	TDS [mg/L]	pH at 24 °C	pH at 80 °C	Ionic strength [mole/L]
LSW-1 (NaCl)	196.7	0	0	0	0	0	303.3	500	6.81	6.71	0.008548
LSW-2 (NaCl)	393.4	0	0	0	0	0	606.6	1000	6.75	6.68	0.017096
LSW-3 (NaCl)	983.5	0	0	0	0	0	1516.5	2500	6.69	6.67	0.042740
LSW-4 (NaCl)	1376.8	0	0	0	0	0	2123.2	3500	6.63	6.60	0.059835
LSW-5 (NaCl)	1770.3	0	0	0	0	0	2729.7	4500	6.62	6.58	0.076931
LSW-6 (NaCl)	2557.1	0	0	0	0	0	3942.9	6500	6.59	6.50	0.111201
LSW-7 (CaCl ₂)	0	180.6	0	0	0	0	319.4	500	7.23	7.16	0.013517
LSW-8 (CaCl ₂)	0	361.2	0	0	0	0	638.8	1000	7.09	7.03	0.027034
LSW-9 (CaCl ₂)	0	903	0	0	0	0	1597	2500	6.91	6.81	0.067584
LSW-10 (CaCl ₂)	0	1264	0	0	0	0	2236	3500	6.85	6.80	0.094611
LSW-11 (CaCl ₂)	0	1625	0	0	0	0	2875	4500	6.82	6.78	0.121638
LSW-12 (CaCl ₂)	0	2347	0	0	0	0	4153	6500	6.80	6.76	0.175691
LSW-13 (MgCl ₂)	0	0	127.6	0	0	0	372.4	500	7.08	7.01	0.015755
LSW-14 (MgCl ₂)	0	0	255.3	0	0	0	744.7	1000	6.99	6.96	0.031516
LSW-15 (MgCl ₂)	0	0	638	0	0	0	1862	2500	6.91	6.87	0.078773
LSW-16 (MgCl ₂)	0	0	893	0	0	0	2607	3500	6.89	6.85	0.110268
LSW-17 (MgCl ₂)	0	0	1149	0	0	0	3351	4500	6.85	6.82	0.141831
LSW-18 (MgCl ₂)	0	0	1659	0	0	0	4841	6500	6.81	6.79	0.204822
LSW-19 (KCl)	0	0	0	262.2	0	0	237.8	500	6.91	6.84	0.006710
LSW-20 (KCl)	0	0	0	524.4	0	0	475.6	1000	6.89	6.83	0.013422
LSW-21 (KCl)	0	0	0	1311	0	0	1189	2500	6.85	6.79	0.033554
LSW-22 (KCl)	0	0	0	1835.4	0	0	1664.6	3500	6.81	6.76	0.046976
LSW-23 (KCl)	0	0	0	2359.8	0	0	2140.2	4500	6.79	6.72	0.060397
LSW-24 (KCl)	0	0	0	3408.6	0	0	3091.4	6500	6.78	6.71	0.087241
LSW-25 (Mix)	19.7	18.1	12.8	183.5	0	0	265.9	500	6.83	6.77	0.008483
LSW-26 (Mix)	39.4	36.2	178.6	52.4	0	0	693.4	1000	6.87	6.79	0.027816
LSW-27 (Mix)	98.6	632	63.8	131.1	0	0	1574.5	2500	6.98	6.96	0.062825
LSW-28 (Mix)	965.3	126.4	89.3	183.5	0	0	2135.5	3500	6.91	6.88	0.067201
LSW-29 (Mix)	88.8	731.3	516.8	118	0	0	3045.1	4500	6.74	6.71	0.125424
LSW-30 (Mix)	511.5	821.6	580.6	341	0	0	4245.3	6500	6.83	6.80	0.164191
FW most (Ca ²⁺ & Mg ²⁺)	5923.4	18310.1	12941.3	6818.2	472.2	1318.6	84216.2	130000	7.12	7.03	3.412470

Fig. 3 Schematic Drawing of the Experimental Procedures

Fig. 4 Core Slices before and after Saturation with Brine

Fig. 5 IFT 700 Equipment for Contact Angle Measurements

Fig. 6 Contact Angle Changes in Presence of Formation Water (FW-130,000 ppm)—Crude Oil A

Fig. 8 Contact Angle Changes in Presence of LSW-19 (KCl-500 ppm)—Crude Oil A

Fig. 7 Contact Angle Changes in Presence of LSW-13 (MgCl₂-500 ppm)—Crude Oil A

Fig. 9 Contact Angle Changes in Presence of Formation Water (FW-130,000 ppm)—Crude Oil B

5.2. NaCl Composition

For this case, still both crude oils create different effects on contact angle changes. As shown in the **Fig. 13A**), in the first 12 h, the contact angle decreases rapidly. From 12 to 24 h, the contact angle decreases slowly. Here, for the whole span of 24 h, the LSW-01 composition causes greatest change of contact angle (112 to 73°). In addition, the LSW-06 composition causes minimum change (110 to 85°). In the **Fig. 13B**), it is clear that the same compositions and

concentrations create different effects on contact angle. There is a steady and rapid decrease in contact angle from zero to 6 h. However, from 6 to the end of 24 h, the decrease in contact angle is slow and not steady. Moreover, changes of contact angle are greater than in the **Fig. 13A**). Moreover, for crude oil B, the LSW-01 composition presents greatest changes of contact angle (112 to 65°), while LSW-06 causes least changes (111 to 80°).

Fig. 10 Contact Angle Changes in Presence of LSW-13 (MgCl_2 -500 ppm)—Crude Oil B

Fig. 11 Contact Angle Changes in Presence of LSW-19 (KCl-500 ppm)—Crude Oil B

5.3. CaCl_2 Composition

The third case consists in verifying the changes of contact angle with time for the CaCl_2 brine composition. As displayed in the **Fig. 14**, the contact angle is decreasing with time. For both **Figs. 14A)** and **14B)**, the contact angle decreases rapidly for the first 6 h. However, from 6 to 24 h the decrease in contact angle is slow. In the interval of 24 h, both crude oil A and B create distinct effects on the contact angle. **Figure 14A)** shows changes of contact angle for the crude oil A. Here, at the end of 24 h, the composition LSW-07 causes maximum changes of contact angle (112 to 78°). In contrast, LSW-12 causes minimum change (112 to 94°). **Figure 14B)** displays contact angle changes for crude oil B. As shown, at the end of 24 h, still the LSW-07 composition creates maximum changes of contact angle (110 to 71°) and LSW-12 creates least changes (111 to 90°). Furthermore, from the **Fig. 14**, it is patent that changes of contact angle for crude oil B are greater than of crude oil A for the whole

A) Contact Angle versus Time for KCl Composition and Crude Oil A

B) Contact Angle versus Time for KCl Composition and Crude oil B

Fig. 12 Contact Angle Changes for KCl Composition

A) Contact Angle versus Time for NaCl Composition and Crude Oil A

B) Contact Angle versus Time for NaCl Composition and Crude Oil B

Fig. 13 Contact Angle Changes for NaCl Composition

span of time.

5.4. MgCl_2 Composition

The fourth case relates changes of contact angle with time for MgCl_2 composition. The contact angle is decreasing with time for the set intervals of time. Both crude oil A and B create different changes of contact

A) Contact Angle versus Time for CaCl_2 Composition and Crude Oil A B) Contact Angle versus Time for CaCl_2 Composition and Crude Oil BFig. 14 Contact Angle Changes for CaCl_2 Composition

A) Contact Angle versus Time for Mix Composition and Crude Oil A

B) Contact Angle versus Time for Mix Composition and Crude Oil B

Fig. 16 Contact Angle Changes for Mix Composition

A) Contact Angle versus Time for MgCl_2 Composition and Crude Oil A B) Contact Angle versus Time for MgCl_2 Composition and Crude Oil BFig. 15 Contact Angle Changes for MgCl_2 Composition

angle. Moreover, as shown in the **Fig. 15**, the changes of contact angle for crude oil A are slower and relatively smaller than of crude oil B. Additionally, for both crude oils, within the first 6 h, the changes of contact angle are rapid. From 6 to 24 h, the changes are slow. From **Fig. 15A**) and interval of 24 h, the composition LSW-13 causes maximum changes of contact angle

(112 to 85°). In contrary, LSW-18 creates minimum changes (110 to 96°). For crude oil B (**Fig. 15B**)), the composition LSW-13 creates maximum changes of contact angle (110 to 75°). In contrast, LSW-18 creates minimum changes of contact angle, that is, from 110 to 93°.

5.5. Composition

The last case is representative of contact angle changes for the mix composition. As shown in the **Fig. 16**, at each time interval, the contact angle is decreasing. Similar to the previous cases, the effects created by both crude oil A and B are different. Here, contact angle changes for the crude oil A are lesser and slower for each composition than of crude oil B.

From **Fig. 16A**), there is a rapid decrease of contact angle for the first 6 h. From 6 to the end of 24 h the decrease is slow with exception to the LSW-25 that presents quite fast decrease. Here, the LSW-25 creates maximum changes of contact angle (110 to 75°). On the other hand, the LSW-30 composition creates least changes of contact angle (112 to 98°). For crude oil B (**Fig. 16B**)), still the contact angle decreases rapidly in the first 6 h. However, from 6 to 24 h, the decrease is slow with the LSW-25 showing slightly faster decrease. The LSW-25 composition causes maximum changes of contact angle (110 to 63°), while LSW-30 causes minimum changes (112 to 91°).

6. Discussion

We observe that at fixed concentration of LSW, the changes of rock wettability in presence of crude oil B (with higher TBN and lower TAN) are greater than those in presence of crude oil A (with higher TAN and lower TBN). Skauge *et al.* (1999) has reported similar results. The author used core samples of sandstone lithology and verified that the wettability changed to more oil wet conditions at higher acid number and to more water-wet conditions at higher base number¹¹. There are several mechanisms behind this behavior. First, the polar components present in the oil phase may be ionized depending on the pH value. The ionization of basic material results in positively charged crude oil components; while the ionization of acidic material results in negatively charged crude oil components¹². These hypotheses are important in understanding the system described below.

We examine an oil/brine/rock system where the formation brine consists mostly of divalent cations, before LSW injection. The presence of these ions results in formation of a water film between the rock and the crude oil¹³. The water film is positively charged, thus it attracts more the negatively charged crude oil acidic components than does for the positively charged crude oil basic components. Therefore, if we consider the crude oil as characterized with higher TAN and lower TBN, it would have established more oil-wet conditions (say system A); whereas, that with crude oil of higher TBN and lower TAN would have created less oil-wet conditions (say system B). Since the system A is more oil wet, we do not expect significant changes of this wettability state upon LSW injection because the acidic components are tightly held through the water phase. However, for the system B, there will be significant change of wettability because the basic components are loosely held through the water phase. This explains the least changes in wettability for crude oil A as compared to crude oil B.

From the results it is also observed that for both crude oils A and B, the KCl composition causes the greatest changes of wettability followed by NaCl, then $MgCl_2$, next $CaCl_2$, and finally the mix composition. Thus if we assume the MIE mechanism, then the easiness of replacement of Ca^{2+} and Mg^{2+} cations from the rock surfaces is of order K, Na, Ca and finally Mg. That is, a LSW composed of KCl at appropriate concentration, will readily replace most of Mg^{2+} or Ca^{2+} cations than would Na^+ , Ca^{2+} and Mg^{2+} . This is true and possibly explained based on the reactivity series (RS). From the reactivity point of view, K^+ is the most reactive and thus it would easily react and bond to the clay surfaces. The second most reactive is Na^+ , then Ca^{2+} and finally Mg^{2+} . Therefore, from the results, their reactivity series for a LSW flooding can be

reformulated as follows: $K^+ > Na^+ > Ca^{2+} > Mg^{2+}$. Regarding the aforementioned, it becomes necessary to add one more condition for the MIE mechanism. This condition supports that the replacement of the divalent cations follows the reactivity series termed as MIE-RS.

7. Conclusions

The changes in wettability of a certain reservoir are affected by the polar components present in the crude oil. Their characterization is performed through measurements of the crude total acid number (TAN) and total base number (TBN). The magnitude of the change of wettability is dependent on the concentration of the components. Here, there is much change of wettability when there is high TBN and low TAN.

The changes of rock wettability are not only dependent on the concentration of crude oil polar components, but also on the composition and concentration of the brine. From the experiment, greatest changes of wettability for both crude oil A and crude oil B were experienced for KCl composition at 500 ppm followed by the NaCl at 500 ppm.

Furthermore, a new mechanism, MIE-RS, is proposed which could open the gates for future studies on wettability alteration upon LSW flooding. However, it has no laboratory basis; therefore, it is only well suited for future work.

Nomenclatures

LSW	: low salinity water	
TAN	: total acid number	
TBN	: total base number	
TDS	: total dissolved solids	
MIE	: multiple-component ion exchange	
KOH	: potassium hydroxide	
IFT	: interfacial tension	
RS	: reactivity series	
b	: slip factor	[psi]
K	: liquid permeability	[mD]
K_g	: gas permeability	[mD]
V_b	: sample bulk volume	[cm ³]
V_g	: grain volume	[cm ³]
V_p	: pore volume	[cm ³]
<Greeks>		
β	: inertial resistivity	[ft ⁻¹]
ϕ	: sample porosity	[%]

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