

CONTRADICTIONARY TRENDS FOR SMART WATER INJECTION METHOD: ROLE OF PH AND SALINITY FROM SAND/OIL/BRINE ADHESION MAPS

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ABSTRACT

Smart Water Injection Methods (SWIM) requires a good understanding of the evolution of wettability not only as a function of salinity but also of pH. In order to have a better understanding of this parameter, adhesion tests were performed on crude oil/sand/brine systems. These tests were developed according to an in-house experimental protocol which guarantees full control of pH during its various stages and when crude oil/brine/rock are in equilibrium. Fontainebleau and Ottawa sands were used to understand the effect of pH and salinity on wettability. The brine was composed of a mixture of NaCl and CaCl₂ (ratio 9/1). The salinity varies from 7.9 to 785 mM and pH from 2 to 12.

Firstly, our adhesion tests are in good agreement with previous adhesion maps from the literature, although the experimental techniques are very different. Moreover our tests incorporate the effect of divalent ions and long term interactions between oil/water and the solid, which were not accounted for in previous studies. Our results confirm that at low salinity and acidic pH (pH < 7), oil adhesion increases for both sands, although quite more importantly for Fontainebleau. In that respect, our adhesion maps highlight contradictory results compared to the recent literature regarding the effect of salinity on wettability.

Secondly we find three distinct oil adhesion areas (for salinity from 0.5 to 50 g/L) which vary with equilibrium pH: from pH = 2.5 to 6 and pH = 8.5 to 12 showing oil wettability, pH = 6 to 8.5 showing water wettability. Interestingly it is found that critical pH thresholds exist in the range pH = 6 to 8.5 at which large variations in the adhesion between oil and sand are seen. This critical pH might be rock dependent: these three areas are specifically pronounced for Fontainebleau sand compared to Ottawa sand where the water wet area is more important (pH = 5.5 to 10). Considering the fact that both sands are mainly composed of quartz, it would imply that the predominant mineral species is not an impacting factor on wettability and therefore on SWIM.

INTRODUCTION

SWIM (Smart Water Injection Methods) have been widely investigated in the last 15 years. This is an emerging EOR (Enhanced Oil Recovery) technique highlighted by Tang and Morrow [1] in Berea sandstone, which is more and more studied due to its

economical aspect and feasibility. There are in the literature, numerous papers on this topic in order to understand the mechanism, but at this time, the real mechanism or combination of mechanisms which lead to the increase of the oil recovery have not been clearly defined. Several authors have various approaches to explain this effect as fine migration [2] controlled by clay particles, alkaline-flooding behavior which relies on interfacial tension reduction caused by pH increase [3], Multiple-Ion-Exchange (MIE) process [4], pH elevation [5] and formation of water micro-dispersions in the oil phase [6]. None of these mechanisms on its own can be retained to explain the increase in oil recovery: rather it is nowadays accepted that a combination of several factors and a mix of all these mechanisms is behind the SWIM effect [7]. A general consensus is that low salinity water may induce a change in the wettability towards a more water wet condition, which would somehow help in releasing additional oil from the pore space. But this mechanism has not yet been visualized or demonstrated in a convincing manner.

Since wettability is an important parameter which determines the fluids distribution and has an effect on oil recovery, several authors performed adhesion experiments in order to understand the interaction between crude oil, brine and rock [8-12]. Usually the reservoir sandstone rock is represented by crystalline quartz or various glass surfaces and classically two kinds of experiments are performed: contact angle [9, 10, 12] measurement and adhesion of a drop of crude oil on the surface [8, 10, 12]. In both cases, crude oil is surrounded with a brine composed of sodium chloride at various salinities and with a pH which varies from 2 to 10 and several parameters are normally tested as pH, Temperature, I (ionic strength), nature of oil, amongst others. From these results, adhesion maps are built which give a rapid and semi-quantitative means for characterizing crude oil interactions with a brine/solid system. Most researches have highlighted three areas in the adhesion map as a function of pH, brine salinity [9, 10] whose extension depends on crude oil's nature, brine's ionic composition, temperature and nature of solid:

- Adhesion at low pH ($\text{pH} < 6$) and low salinity ($\text{TDS} < 5.8 \text{ g/L}$)
- Non-adhesion for pH comprised between 6 and 9 at moderate salinity ($\text{TDS} \approx 50 \text{ g/L}$)
- Adhesion at high pH ($\text{pH} > 9$) and high salinity ($\text{TDS} > 50 \text{ g/L}$)

Lebedeva and Fogden [13] have shown similar results for kaolinite. It is seen that pH has a non negligible effect on adhesion. These results also highlight that there is more adhesion at lower salinity, which is difficult to reconcile with the claim that at low salinity, the system become more water wet. Nevertheless all these previous experiments were performed in a very short time for the contact between oil and glass (less than 10 min) which means that the system equilibrium may not have been reached. Moreover, no divalent ions were present in surrounding brine and for some authors [14, 16, 24-26], their presence in the brine is essential to observe a low salinity effect.

In this paper, we developed adhesion tests with a home-made protocol which allow the equilibrium of the system for various pH and salinities. Simple solids were used as pure

sands. This study allows us to evaluate the combined effect of salinity and pH on oil adhesion to the rock grains.

EXPERIMENTAL PROCEDURES

Materials

Minerals

For this study, two pure sands were used, Fontainebleau (from VWR) and Ottawa sands (from Fischer Chemical) in order to mimic sandstone reservoirs. These samples have been chosen for their purity in terms of mineralogy. Both sands are comparable in terms of mineralogy and are mainly composed of quartz (SiO_2) (Table 1).

Table 1: Mineralogy of sands from XRF measurements

Sands	SiO_2	CaO	K_2O	P_2O_5	Fe_2O_3	TiO_2	Ba
Fontainebleau	99.914	0.070	0.006	0.014	0.017	0.017	0.045
Ottawa	99.896	0.071	0.013	0.019	0.021	0.007	0.000

These samples were also characterized with a binocular magnifier to evaluate their external structure (Figure 1). We can remark that the sands have completely different structure. These sands were also characterized by laser granulometry. Ottawa sand has bigger particles with a size of 880 μm compared to Fontainebleau which has a particle size of 260 μm .

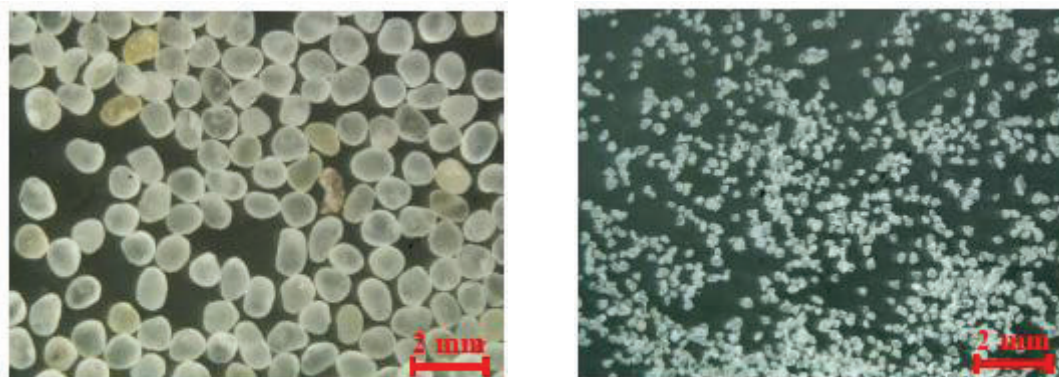


Figure 1: Sand observations with the binocular magnifier, Ottawa sand (left) and Fontainebleau sand (right)

Crude Oil

The same crude oil was used for all the adhesion tests. The oil has been filtered at 40 μm before use. Table 2 highlights its properties.

Table 2: Analysis of crude oil

Property	Units	Crude oil
TAN	mg/g KOH	0.2
TBN	mg/g KOH	1.77
Density @ 40 °C	kg/m^3	834
Density @ 60 °C	kg/m^3	820
Viscosity @ 40 °C	cSt	6.26
Viscosity @ 60 °C	cSt	3.94
% Asphaltenes	weight %	2.9
TCC*	°C	35

*TCC: Critical Temperature of Crystallization

Brines

Synthetic brine solutions were prepared at various salinities with 90 wt% of NaCl and 10 wt% of CaCl₂. NaCl(s) and CaCl₂(s) were manufactured by Sigma Aldrich® with a purity higher than 99.9%. Demineralized water was used. Brine solution pH was adjusted with HCl(aq) or NaOH(aq) solutions at 0.1 or 1 M. After stirring, all the solutions were filtered at 0.45 μm, pH varies from 2 to 12 and it was controlled during manipulation. Tested TDS (Total Dissolved Salts) were 0.5, 1, 3, 7, 11, 15, 25 and 50 g/L. The properties of the brine used in this study are summarized in the Table 3.

Table 3: Salinity, molar concentration and ionic strength of used brines

TDS(g/L)	0.5	1	3	7	11	15	25	50
C (mM)	7.85	15.7	47.1	11	17.3	23.6	39.3	78.5
I*	0.019	0.036	0.11	0.25	0.40	0.55	0.91	1.82

*: Ionic Strength

Experimental Protocol

Sand Cleaning

In order to eliminate all contaminations, mainly by iron, the sand was washed beforehand with an acid solution (HCl(aq), 1M), then neutralized with sodium bicarbonate (NaHCO₃(aq), 1M), rinsed thoroughly with demineralized water and finally oven dried at 60°C for 24 h.

Protocol of Adhesion Tests

The test tube experiment is a fast screening of sand/oil/brine interactions permitting to focus on the effect of one parameter at the time. These batch experiments use a static procedure which is described below.

1. Prepare brine at the chosen salinity.
2. Place a known mass of cleaned sand in contact with a known volume of brine. The brine has to be in excess compared to the sand.
3. Add HCl(aq) at C = 0.1 M or 1 M or NaOH(aq) at C = 0.1 M to adjust the pH to the chosen value.
4. Leave the solution at 60°C during 24h for equilibration; regularly shake manually to homogenize the sand/brine system.
5. Measure the pH.
If the target pH is not reached, repeat the steps 3 and 4 to eliminate the buffering effect of sand.
6. When the target pH is reached and is seen to be stable, mark this pH as *initial pH* (pH_i). Now add a known volume of oil to the system.
7. Shake manually the glass vial to free the oil that is trapped under the weight of the sand and not actually adhering to it.
8. Leave the system at 60°C during 48 h under continuous stirring.
9. Observe wetting state at equilibrium at T = 60°C. The wetting state is the evaluation of the oil quantity which is in adhesion on and in the sand compared to the oil which stays above the brine phase (Figure 4 for example). Three states were defined; adhesion, transition and non-adhesion. When the oil quantity in the sand is higher than in the brine, then there is adhesion. If there is

quite the same quantity of oil in the sand and in the brine phase, then it is transition state. Finally when no oil adheres in the sand, then it is non-adhesion state.

10. Shake manually, wait for equilibrium and observe the wetting state. As previously, manual shake makes the system more homogeneous and helps to estimate visually the actual sand and oil adhesion by freeing the oil that is trapped under the weight of the sand.
11. Measure the *final pH* (pH_f) after filtration at $0.45 \mu\text{m}$ of the brine in the system.
12. Report the observed wetting state at fixed pH in an adhesion map
13. Perform this protocol for another initial pH until the adhesion map is complete with all the tested salinity and initial pH

Remark: Equilibrium was estimated with tests at various experiment durations. It was estimated that after 48h of stirring, the interaction between oil, sand and brine stays the same even after more hours of stirring at temperature.

This protocol allows removal of the buffering effect of the sand (which can be quite important) and to control the initial and final pH of the system. The key parameter in this study is the control of pH. The final pH could be estimated with the pH_i - pH_f diagram of the used crude oil (Figure 2). We can remark in this graph that the evolution of pH is the same at low salinity (1 g/L) as at high salinity (50 g/L). For this studied oil, when initial pH is comprised between 4 and 10, the final pH is between 6 and 8. This oil is mainly a basic one.

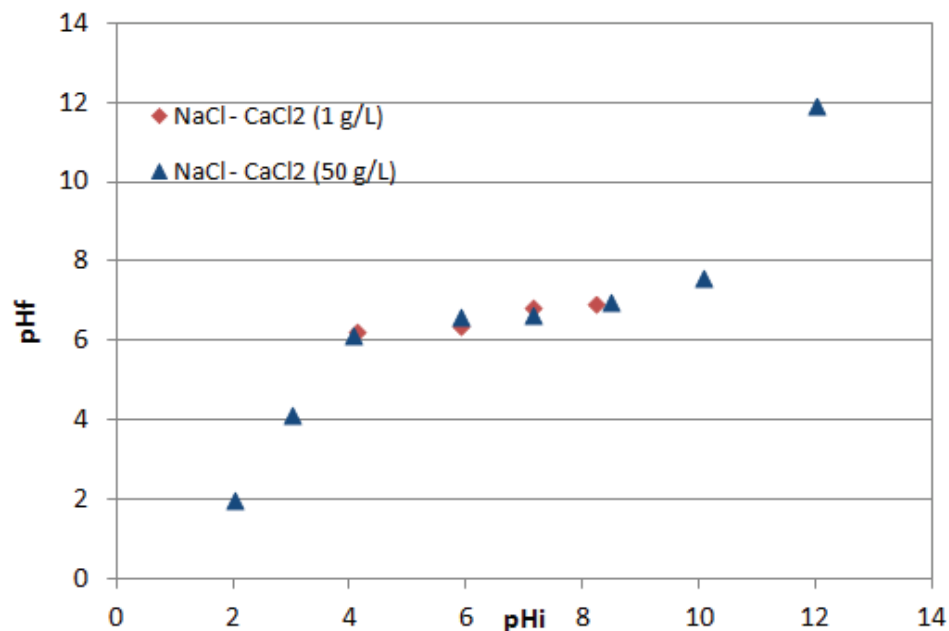


Figure 2 : pH_i - pH_f diagram of the tested crude oil at 45°C in NaCl/ CaCl_2 brine at low and high salinity (TDS = 1 and 50 g/L)

RESULTS AND DISCUSSIONS

The adhesion map of Fontainebleau sand is displayed in Figures 3a and 3b.

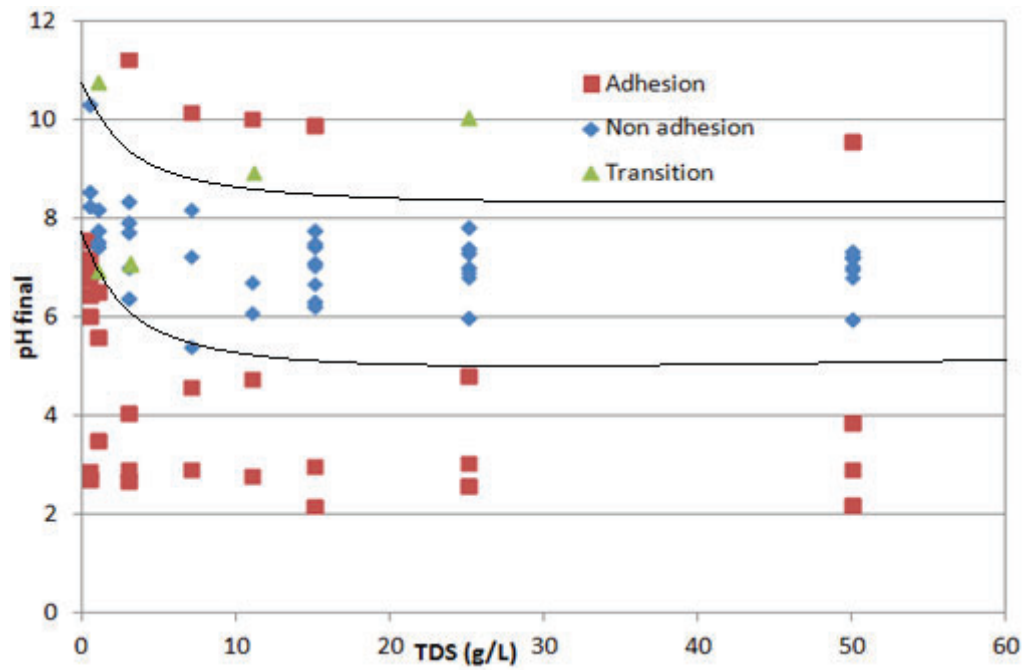


Figure 3a: Adhesion map of Fontainebleau sand

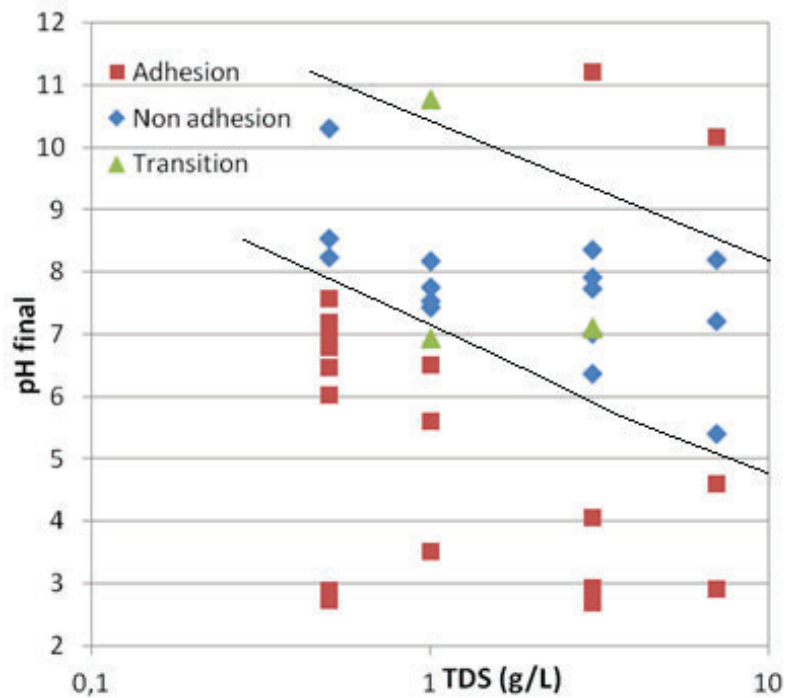


Figure 3b: Adhesion map of Fontainebleau sand (zoom on low salinity (TDS<10 g/L))

The information in this map can be approximated by three main adhesion areas roughly situated at:

- $2 < \text{pH} < 6$: oil adheres to grains
- $6 < \text{pH} < 8$: oil does not adhere to grains
- $8 < \text{pH} < 12$: oil adheres to grains

An example of test tube experiments is displayed in Figure 4 to illustrate this adhesion map at 7 g.L^{-1} . In this example, adhesion occurs at final pH equal to 5.42 and at higher pH_f (10.17), whilst non-adhesion is seen for $\text{pH}_f = 7.23$.

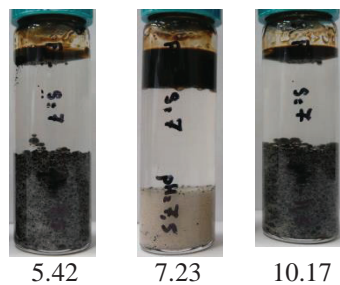


Figure 4: Glass vial adhesion map results at fixed salinity (7 g.L^{-1}) and for three pH (pH_f) values. From left to the right: adhesion, non-adhesion, adhesion.

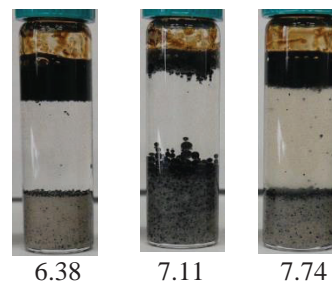


Figure 5: Glass vial adhesion map results at fixed salinity (3 g.L^{-1}) and for three pH (pH_f) values. From left to the right: non-adhesion, transition, non-adhesion.

By decreasing the pH, whatever the salinity, a greater adhesion of oil on the Fontainebleau sand is visible, a result in agreement with the work of Yang et al. [10] and Drummond et al. [9]. Oil adhesion is systematic for pH below 5 as well as for pH higher than 10. In addition, at low salinity ($\text{TDS} < 3 \text{ g/L}$), the zone of oil adhesion extends towards higher pH. In this area ($6 < \text{pH} < 8$) small variations of salinity for a given pH_f can create very different adhesion results (Figure 5).

This observation is very important since pH values expected at reservoir conditions would cover the interval $5 < \text{pH} < 7$ [22]: wettability being so sensitive in these pH-salinity ranges, could create an important variability of outcomes and would render SWIM performance not trivial to assess.

In conclusion, the result obtained for Fontainebleau shows that the rock becomes more oil wet when salinity decreases, in contradiction to comments in literature on smart water techniques ([14-17]) where when decreasing the salinity, the water wetness of the solid increases, leading to the increase of oil recovery.

In Figure 6 the adhesion map for Ottawa sand is displayed. There are three main areas of adhesion, mainly controlled by pH, as for Fontainebleau, but here there is much less influence of salinity. The oil adheres when pH decreases ($\text{pH} < 5$) and for low salinity ($\text{TDS} < 10 \text{ g/L}$) as well as for basic pH (> 11) for all salinities. The last area in this adhesion map shows water wet condition.

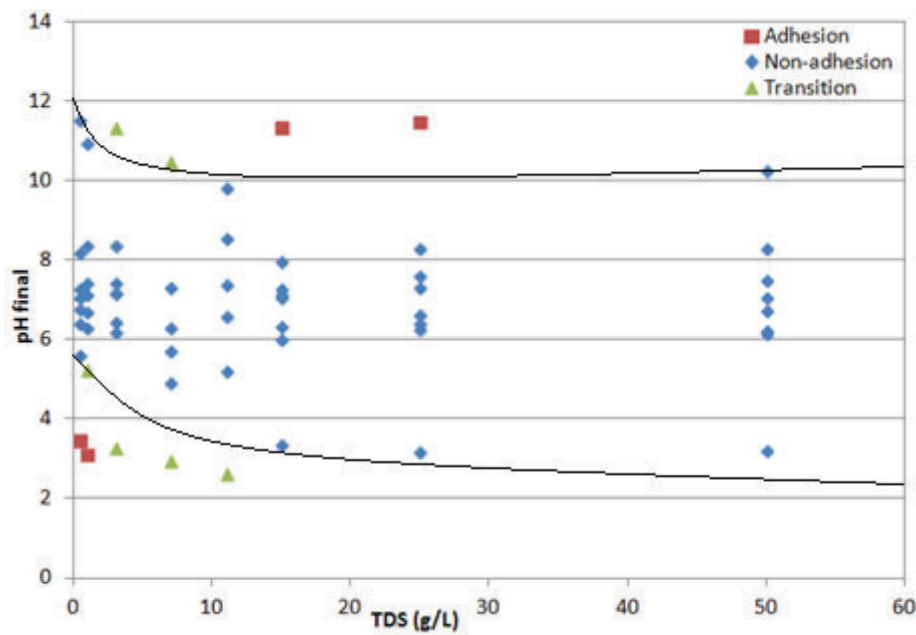


Figure 6: Adhesion map of Ottawa sand

Since both sands share a very similar mineralogy, then we would conclude that for this case sample mineralogy does not have an important effect on wettability. The three areas in the adhesion maps of Fontainebleau and Ottawa sands could be partially explained by adsorption of oil components on sand and electrostatic repulsion, at least at low pH (Figure 7).

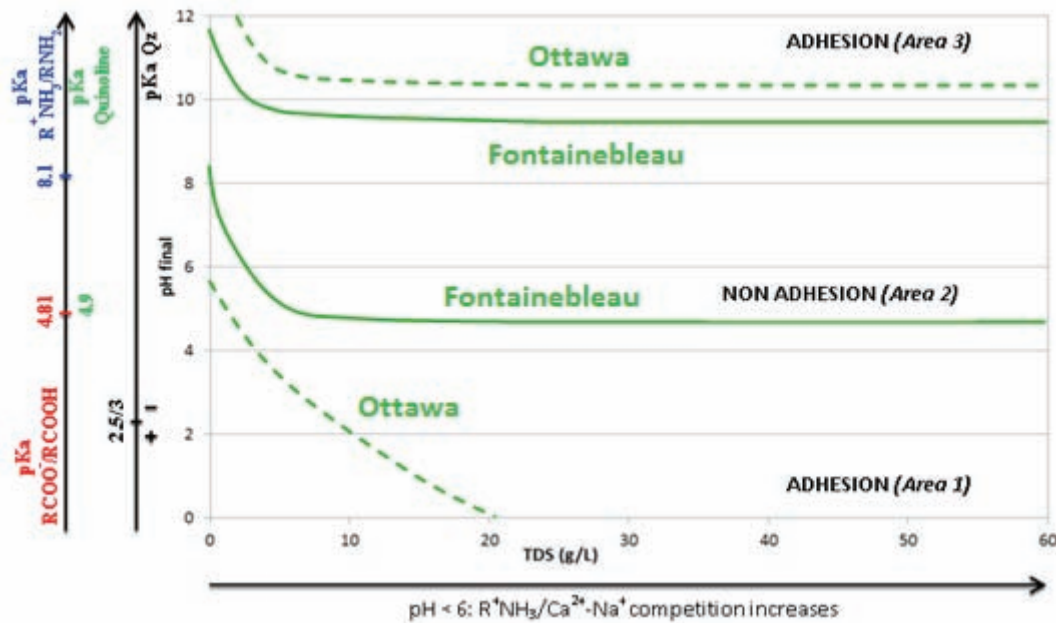


Figure 7: Interpretation of adhesion maps. Continuous and dotted green curves show the transition from water-wet to oil-wet for Fontainebleau and Ottawa sand respectively

The results of this study could be compared with those from Bondino et al. [18], where the authors used similar experimental setup to evaluate oil adhesion on sand. They highlighted also oil adhesion at high and low salinity conditions (TDS = 1 and 50 g/L)

and for acidic pH ($pH_i < 5$). Water wetness is observed at basic pH ($pH > 7$) and for low salinity. Nevertheless the main difference between the two studies is that in [18], the pH was not controlled and measured at the end of the experiment (therefore the pH discussed in the paper happens to be the initial one). Regarding our results, we demonstrate pH is a key parameter in wettability variation and has to be controlled and measured all along the experiment.

These results are comparable with those from the literature [8-12] where adhesion maps highlight also three areas which depend on pH and salinity range. Oil adhesion occurs also for low pH (< 6) and high pH (> 9) and non-adhesion for intermediate pH ($6 < pH < 10$) and salinity (≈ 50 g/L). Transition zone for low salinity (TDS < 5.8 g/L) is located for pH close to 6 and 8, as observed in this paper. This pH area is therefore critical and pH seems to be a key parameter in oil adhesion. This study confirms these previous contradictory results where oil adhesion increases for low salinity range. Nevertheless the test tube experiments presented in this paper seem to be more representative of SWIM compared to the previous adhesion tests. Indeed, compared to the results of [8-12], duration of experiments is longer and led to equilibrium within the system oil/brine/sand. Moreover brines were composed of mono and divalent ions as Ca^{2+} which is favourable for low salinity effect [4, 16] compared to brines with only monovalent ions in the cited literature.

Adhesion maps (Figures 3 and 6) could be explained by electrostatic interaction between oil and sand. The isoelectric point of quartz is estimated to be close to 2.5 ([19, 20]) which means that quartz (Qz) is negatively charged for pH higher than 2.5 (Figure 8). Usually, acid and base components of oil are simplified by RNH_2 , $RCOOH$ and Quinoline. pKa of these redox couples are illustrated in Figure 8. At low pH (< 4.9), oil components are essentially positive and inversely at basic pH (> 8.1).

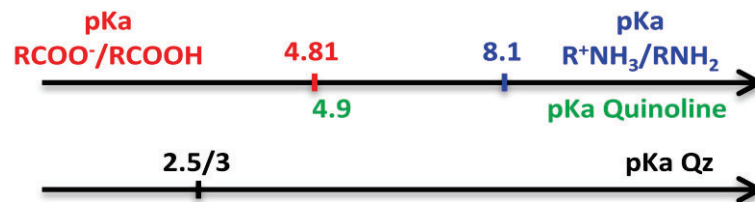


Figure 8: pKa of oil components (RNH_2 , $RCOOH$, Quinoline) and isoelectric point of Quartz (Qz)

Figure 7 summarizes adhesion maps with adhesion areas for Fontainebleau and Ottawa sands. In *Area 1*, adhesion occurs at low pH ($2.5 < pH < 5$) due to reduction of electrostatic interaction between sands negatively charged and oil components positively charged (R^+NH_3 , Quinoline positively charged). In *Area 2*, non adhesion occurs at higher pH ($pH > 4.9$) due to repulsion between sands still negatively charged and neutral (RNH_2 , Quinoline) and negative (CH_3COO^-) oil components. However, when salinity increases in *Area 1*, oil adhesion occurs at lower pH for Fontainebleau sand but not for Ottawa sand. One hypothesis is the influence of roughness and granulometry which could increase specific surface area of sand grains and therefore increase the probability of adhesion. The roughness of Fontainebleau sand seems higher than Ottawa's, therefore its specific surface area could be higher and this will increase the probability of oil adhesion. Similar observations were already highlighted

in the literature [21]. In the *Area 3*, oil adhesion at very basic pH ($\text{pH} > 10$) is more difficult and complex to explain: further investigations are ongoing.

This variation of adhesion with pH suggests also that not only the final pH is necessary to know, but also the initial pH of the system. If there is a pH evolution during the waterflooding experiment in the system rock/brine/oil, we could have a change of wettability which could have an impact on the recovery.

This study could be useful for understanding spontaneous imbibitions tests, as adhesion maps were elaborated with test tube experiments without dynamic aspect. In the literature, some authors highlight high response variability for spontaneous imbibitions tests with various carbonates rocks [22] or various type of oil on Berea sample [23] for example. Romanuka et al. [22] show various wettability modification in function of rock samples or ionic composition of injected water. Suijkerbuijk et al. [23] found very different oil recovery during spontaneous imbibitions with ten different oils on Berea sandstones. Adhesion tests as described in this study could be useful screening tests for understanding the variability in response of SWIM spontaneous imbibitions experiments.

CONCLUSIONS

In this paper, test tubes experiments were performed in order to implement adhesion map for crude oil/sand/brine system. A home-made protocol allows the system to reach equilibrium at temperature with full control of the pH. Indeed, pH appears to be an essential parameter in wettability, more than the salinity. Adhesion map of Fontainebleau sand highlights oil adhesion at low salinity ($\text{TDS} < 3 \text{ g/L}$) in reservoir pH area ($6 < \text{pH} < 8$) and non adhesion for a salinity higher than 3 g/L in this pH area. Moreover, this domain is very sensitive to pH variation. A little variation in pH will change drastically the wettability. This highlight that pH has to be well monitored during waterflooding experiments.

The fact that low salinity increases the oil wettability is contradictory to what is expected in the literature for SWIM, where low salinity waterflooding is seen to increase water wetness and then the oil recovery. In light of these results, we think that it would be more appropriate to state that oil recovery depends from the transition from some initial to some final state, and not simply from a system becoming more water wet.

It was rather interesting to note that Ottawa and Fontainebleau highlight different adhesion maps despite the fact that their mineralogy is quite similar. Therefore the mineralogy may not always be a determinant parameter in low salinity effect. Moreover both sands have various roughness, which could be an important parameter for adhesion behavior. The effect of surface roughness and granulometry is currently under investigation. Finally electrostatic interactions between acid and base components of the oil seem to control the adhesion on the solid.

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NOMENCLATURE

SWIM	Smart Water Injection Methods	I	Ionic Strength
EOR	Enhanced Oil Recovery	wt%	Weight %
TCC	Critical Temperature of Crystallization	C	Molar Concentration
TDS	Total Dissolved Salts	M	mol/L

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