EXPERIMENTAL INVESTIGATION OF OIL COMPOSITIONAL AND SURFACTANT EFFECTS ON WETTABILITY AT RESERVOIR CONDITIONS

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ABSTRACT

Much of the existing literature on wettability deals with ambient condition measurements, and hence the information on fluid compositional effects on wettability is scanty at best. This study is an attempt to fill this information gap. In this study, Dual-Drop Dual-Crystal (DDDC) contact angle measurements have been made using dolomite rock and fluid samples from the Yates reservoir (West Texas) and in the presence of an anionic (ethoxy sulphate) surfactant. The experiments have been conducted at Yates reservoir conditions (82º F and 700 psi) and using live crude oil to provide realistic measurements of in-situ reservoir wettability. Stocktank crude oil has also been used at reservoir conditions to study the oil compositional effects on wettability.

An advancing contact angle of 152º measured for both Yates and outcrop dolomite rocks using Yates stocktank oil and synthetic reservoir brine showed the strong oil-wet nature. However, experiments with Yates live synthetic oil resulted in an advancing contact angle of around 55º on both Yates dolomite and outcrop dolomite substrates, indicating weakly water-wet behavior. In the rock-fluids system consisting of Yates stocktank oil, the surfactant altered the wettability to less oil-wet by decreasing the advancing contact angle to 135º. For rock-fluids system with Yates live synthetic oil, the surfactant altered the wettability from weakly water-wet to strongly oil-wet by increasing the advancing contact angle from 55º to 165º. The oil-wet behavior observed with Yates live oil due to the surfactant indicates the significant wettability altering capability of the surfactant.

The mechanisms for surfactant-induced wettability alterations observed were explained using a four-region surfactant adsorption model. This study recognizes the need for wettability measurements at actual reservoir conditions using live reservoir

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fluids and demonstrates the wettability altering capability of low cost surfactants at low concentrations for possible improved oil recovery applications in the field.

1. INTRODUCTION

Although the surfactants are widely used as surface-active agents in several areas such as chemical, biochemical and pharmaceutical industries, the use of surfactants has been limited in petroleum industry for improved oil recovery due to uneconomical field applications. This is mainly due to restricting the use of surfactants only to reduce oil-water interfacial tension in enhanced oil recovery applications. However, the other possible beneficial aspect of surfactants, namely alteration of wettability has largely been ignored. If this surfactant-induced wettability alteration mechanism is properly explored and exploited, it would be especially beneficial to petroleum industry.

The interfacial properties; wettability (contact angle) and interfacial tension, and the fluid flow characteristics (namely velocity and viscosity) are correlated to oil recovery through the capillary number (N_c),

$$N_c = \frac{v\mu}{\sigma \cos\theta} \qquad \text{……………………………………………………………(1)}$$

where v and μ are the velocity and viscosity of the displacing phase, respectively and θ is contact angle between the fluid-fluid interface and the solid surface, and σ is interfacial tension between the fluid phases.

The greater the capillary number, the lower is the residual oil saturation and hence higher the oil recovery [1]. For significant enhancements in oil recovery, four to six orders of magnitude reduction in interfacial tension is required. The surfactants capable of providing such large reductions in interfacial tension are expensive, rendering them uneconomical in conventional field applications. However, the contact angle term in this equation was largely ignored in the past. If the wettability is altered by some means to near intermediate-wet (≈ 90º), for example using surfactants, the capillary number would become large resulting in very low residual oil saturations or high oil recoveries. Although such ideal alteration is unlikely possible, the combination of lowered IFT and lowered cosθ caused by the surfactants may result in optimum oil recovery improvement.

Therefore, in this paper, we examine the feasibility of wettability alteration mechanism due to an anionic surfactant (ethoxy sulfate) in Yates fractured dolomite reservoir of West Texas. Normally, surfactant-induced wettability alterations occur in porous media due to the surfactant adsorption on to the rock surface. The charged behavior of rock surface and surfactant ions play an important role in deciding which part of the surfactant molecule (hydrophobic or hydrophilic) should preferably adsorb on the rock surface. For ionic surfactants at low concentrations, there are electrostatic interactions between the head groups of the surfactants and charged sites on the rock surface. These electrostatic attractions can be typically described in terms of the interaction of the charged surfactant ions with the electrical double layer of the solid surface [2]. The carbonate surface is positively charged near neutral pH [3]. Hence the anionic surfactant, in which the surface-active portion of the molecule bears a negative charge, has been chosen in this study to enhance surfactant adsorption on the inversely charged rock surface.

Several experimental studies reported in the recent literature related to wettability alteration of carbonates by anionic surfactants is reviewed and discussed below.

Austad and Standnes [4] conducted several experiments involving the spontaneous imbibition of aqueous surfactant solutions into the oil-wet carbonates. They observed the adsorption of anionic surfactants like ethoxylated sulfonates onto the hydrophobic chalk surface resulting in a double layer to create a hydrophilic surface even at low imbibition rates. Babadagli [5] compared the oil recovery for four different rock types (sandstone, limestone, dolomite and chalk), a wide variety of oils (light and heavy-crude, kerosene, and engine oil) and different types (non-ionic and anionic) and concentrations of surfactants by conducting laboratory experiments. Except for light oils such as kerosene and light crude oil in sandstones, in all the other cases the non-ionic surfactant solution yielded a higher ultimate recovery at faster recovery rates. This is attributed to wettability alterations due to the surfactant. In chalks, higher anionic surfactant concentrations yielded higher recovery, but lower surfactant concentrations resulted in even lower oil recovery than that observed with normal brine. Hirasaki and Zhang [6] reported the wettability alterations of the calcite plate to preferentially water-wet in the presence of an alkaline-anionic solution. Seethepalli et al. [7] reported that anionic surfactants can change the wettability of the calcite to an intermediate/water-wet condition and were found to be better than a cationic surfactant with a West Texas crude oil. Cuiec [8] summarized the influence of various thermodynamic conditions on wettability measurement and reported that the effect of temperature and pressure on wettability is unique for each reservoir case.

Chilingar and Yen [9] reported that among 161 carbonate cores studied, 15% of them were strongly oil-wet (θ=160-180°), 65% were oil-wet (θ=100-160°), 12% were intermediate-wet (θ=80-100°) and 8% were water-wet (θ=0-80°). The original wettability of carbonates needs to be examined carefully before using surfactants. Rao [10] summarized the wettability effects in thermal recovery operations and reported the wettability alterations of the carbonates to more water-wet as the temperature is increased. Similar findings were reported by Austad and Standnes [4]. Since the wettability is strongly influenced by all the compositional effects of rock and fluids at the reservoir conditions, simulating similar conditions in laboratory is essential to determine in-situ reservoir wettability. However, most of the experimental studies reported in the literature on wettability consist of stocktank crude oils at ambient conditions of temperature and pressure. Furthermore, there is a lack of enough experimental data in literature concerning the effects of crude oil composition i.e. light ends (methane to pentane) present in live crude oil on wettability. Therefore, the main objectives of this study are: (1)

to determine the effect of oil composition on wettability and (2) to explore the wettability altering capability of the surfactants. For this purpose, the fractured Yates dolomite reservoir live and stocktank crude oils, dolomite rock substrates and an anionic ethoxy sulphate surfactant have been used. Synthetic brine matching the Yates reservoir brine composition was prepared and used in the experiments. All the experiments have been conducted at Yates reservoir conditions of 700 psi and 82°F. The contact angles have been measured using the dual-drop dual-crystal (DDDC) technique [11].

2. EXPERIMENTAL APPARATUS AND PROCEDURE

2.1 Rock and Fluids

Analytic grade reagents are used in the experiments. The salts that were used for synthetic brine preparation are from Fisher Scientific, all having a purity of 99.9%. Deionized water, from the Water Quality Laboratory at Louisiana State University, is used. The dolomite rock substrates are from Ward Scientific. Few rock samples obtained from the Yates reservoir fractured core are also used for comparison. The rock samples were cut into small pieces (12 mm × 10 mm × 0.8 mm in size) and then polished using a series of sandpapers ranging from 240 grits to 2000 grits. The sample is then cleaned by deionized water and aged in the Yates brine for 24 hours before use. Yates stocktank crude oil and anionic surfactant (ethoxy sulphate) are supplied by Marathon Oil Company. Yates stocktank crude oil has been well protected under nitrogen blanket to prevent oxidation. Yates live oil has been prepared by adding appropriate amounts of light ends (methane to pentane) to the Yates stocktank crude oil, according to the live oil composition provided by Marathon Oil Company. Since the outcrop dolomite displayed the same native wettability state as Yates reservoir rock with both Yates stocktank and live crude oils, the outcrop rock samples were used in all subsequent contact angle measurements.

2.2 Apparatus

A high-pressure and high-temperature apparatus has been built to measure contact angles at elevated pressures and temperatures. The schematic flow chart of the system is shown in Figure 1. The central part of this system is an optical cell, which has a design rating of 20,000 psi at 400°F. Four adjustable arms make this cell unique. The top one and a side one are used to hold rock crystals, while the other side arm is used to hold a calibration ball, and the bottom arm has a needle tip to place an oil drop on the rock surface. All these arms can rotate as well as move in and out during the course of the experiments. The other accessories include an oven, which is used to control the temperature, back-pressure regulator to maintain pressure, some high-pressure vessels and valves to hold and transport fluids, and an image capturing system. The image capturing system includes a high-quality digital camera and a light source. It is connected to the computer, monitor and video recorder. The contact angles are measured using a goniometer and the image capturing system is meant for oil-water interfacial tension measurements.

2.3 Procedure

Our previously developed new experimental procedure [12] was used in this study to simulate the matrix-fracture interactions taking place in a fractured reservoir at reservoir conditions. After measurement of wettability using DDDC technique for Yates crude oil-Yates brine-dolomite rock system at reservoir temperature and pressure, the oil drop was kept in original oil-occupied place between two crystals. Then the dilute surfactant solution of different concentrations was slowly injected into the cell from the bottom. Enough surfactant containing brine was pumped into the cell to make sure that all the normal brine was replaced. The entire process was recorded using video camera and a computer. During injection, the angle between the oil/water interface and the lower crystal surface as well as the three phases contact line (TPCL) were continuously monitored. The detailed experimental procedure is given in reference [13]. After injection, two crystals were moved closer to mingle the oil drops, if and when possible. The time required for mingling the two drops varied for different surfactant concentrations. If drops did mingle, the advancing contact angles were then measured, which represent the wettability after exposure to the surfactant.

3. RESULTS AND DISCUSSION

3.1 Initial Reservoir Wettability

3.1.1 Yates Stocktank Oil-Brine-Dolomite System

The initial wettability was measured using the DDDC technique for Yates stocktank crude oil-brine-dolomite system at reservoir conditions of 700 psi and 82ºF, before exposing the system to the surfactant. The sessile drop receding angles measured initially on the lower crystal surfaces were nearly the same, 23-26 degrees for all the experiments conducted. After 24 hours of aging, the equilibrium sessile drop receding angles were either almost unchanged or increased slightly, but the drop contact diameters increased by about 20%. Once the lower surface was turned upside down, part of the oil drop floated away leaving 20-30% of oil on the surface. After the two oil drops were mingled, the lower crystal was shifted laterally to measure the dynamic advancing and receding angles. The advancing angle was about 152 degrees for all the experiments conducted at reservoir conditions, showing a strong oil-wet nature for Yates stocktank crude oil-brine-dolomite system. This is in good agreement with the other published data on wettability interpretations obtained for the same rock-fluids system at ambient conditions [14].

3.1.2 Yates Live Oil-Brine-Dolomite System

The initial wettability was measured using the DDDC technique for Yates live crude oil-brine-dolomite system at reservoir conditions. The sessile drop receding angles measured initially and after 24 hours on both the upper and lower crystal surfaces were nearly the same as the stocktank oil case, but the drop contact diameters on the crystal surface increased only by about 5%. When the lower surface was flipped, the oil drop completely floated away. The oil drop on the upper crystal was now brought down to contact with the initially oil occupied area on the lower crystal. The advancing angle measured was about 55º-60º for all the experiments conducted using Yates live oil-brine-dolomite system at reservoir conditions, indicating a weakly water-wet nature.

Thus, the initial wettabilities are significantly different for Yates live oil and Yates stocktank crude oil systems. This appears to be solely due to the difference in oil composition between stocktank and live crude oils. Therefore, it can be concluded that the wettability measurements using live reservoir fluids at reservoir conditions are necessary to determine true in-situ reservoir wettability.

3.2 Wettability Alterations Due to Surfactant

3.2.1 Yates Stocktank Oil-Brine-Dolomite System

With the drop in the equilibrium position, surfactant-containing brine at a specified concentration was injected into the cell at reservoir conditions of temperature and pressure. During the injection of surfactant, the equilibrium drop moved and floated to the upper crystal very soon even at the low concentrations of 500 ppm. Significant three-phase contact line (TPCL) movement with a constant advancing angle on the lower crystal was observed during the drop movement (Figure 2). The TPCL movement was monitored with reference to the decrease in drop diameter on lower surface. The advancing angle measured during injection was 135-139°, which was lower than the initial advancing angle (152°) measured before surfactant injection. This clearly indicates wettability alteration due to the surfactant. For higher surfactant concentration of 3500 ppm, the same characteristics as observed at 500 ppm concentration were seen. The measured advancing angle during the injection was 141°. For both these cases, there was about a 16º decrease in the advancing contact angles when compared with the initial advancing angle before injection. This indicates reservoir wettability alterations from strongly oil-wet to weakly oil-wet state by the anionic surfactant, thereby displaying the potential to increase oil recovery by wettability alteration mechanism.

About an hour after surfactant injection, two crystals were moved closer to mingle the two oil drops at the equilibrium position. Overnight or longer aging periods in excess of 16 hours were needed to mingle the two oil drops. Advancing angle was measured by shifting the lower crystal laterally. The advancing angle measured after 500 ppm surfactant injection was about 139°, almost the same as that obtained during the surfactant injection. For higher surfactant concentrations, the two oil drops could not be mingled into one to facilitate the contact angle measurements due to the repulsion between the charged hydrophilic heads of the adsorbed surfactant molecules on the oil drops. The reduction in IFT and the surface charge repulsions observed might indicate the formation of emulsions, which could not be always beneficial for oil recovery in flow through porous media.

3.2.2 Yates Live oil-Brine-Dolomite System

As in the stocktank oil system, with the drop in the equilibrium position, surfactant-containing brine at a specified concentration was injected into the cell at reservoir conditions of temperature and pressure (700 psi and 82°F). During the injection of anionic surfactant, the equilibrium drop moved towards the upper crystal due to lowering of the IFT at low concentrations of 500 ppm. However, significant wettability alteration (from water-wet to oil-wet) with a continuous increase in advancing angle, and TPCL movement were observed (Figure 3). The advancing angle increased from 55° to about 140° within about 20 minutes. The advancing angle approached to a maximum value of 165° at 1000 ppm surfactant injection. For surfactant injection at 1500 and 3500 ppm concentrations, similar characteristics were seen but with a slightly lower advancing contact angle of 120°-135°.

Figure 4 summarizes the advancing contact angles as a function of surfactant concentration for both stocktank and live oil systems. It can be seen that for strongly oil-wet Yates stocktank oil system, the addition of anionic surfactant altered the wettability to less strongly oil-wet. However, for the weakly water-wet Yates live oil system, the addition of anionic surfactant altered the wettability to strongly oil-wet at low concentrations and less strongly oil-wet at high surfactant concentrations. Such a strong dependence of wettability on surfactant concentration can be expected to play a significant role on oil recoveries since the injected surfactant solution would invariably be diluted in the reservoir to various concentration levels.

3.3 Mechanisms of Surfactant-Induced Wettability Alteration

A number of factors affect the interaction of surfactants with the solid surface of porous rock and consequently alter wettability. Some of these more obvious factors include surfactant structure, surfactant concentration, kinetics, pore surface composition, surfactant stability, electrolytes and pH, temperature, rock roughness and reservoir structure [15].

The phenomena of surfactant-induced wettability alterations can be well understood by using the widely accepted four-region surfactant adsorption isotherms [2, 15-17]. The four-region surfactant adsorption model recently proposed by Somasundaran and Zhang [17] was utilized to explain the surfactant-induced wettability alterations observed in this study and is shown in Figure 5.

For the initially water-wet Yates live oil system, the thin water film gradually becomes unstable and was replaced with surfactant-containing brine. Region I corresponds to low surface coverage by individual surfactant molecules with the absence of surfactant aggregate, showing weakly water-wet behavior. In Region II, with the increase of surfactant concentration, the surfactant aggregates (called admicelles or hemimicelles) are formed, resulting in the sharp increase in the slope of the adsorption isotherm. The surfactant molecules are adsorbed on the rock surface with their hydrophilic head groups, while the surfactant molecules are oriented with their hydrophobic tails on the oil drop. Oppositely charged behavior of surfactant and the rock substrate cause the random adsorption to become well arranged. This results in strong oil-wet characteristics. In Region III, sufficient accumulation of aggregates result in aggregates to be attracted with each other and the hydrophilic head of one surfactant molecule tends to be connected with the hydrophobic tail of the other. This causes electrostatic attraction between surfactant molecules. Hence, a weaker oil wetting was observed in this region. Region IV begins at the critical micelle concentration (CMC) of the surfactants and is described by complete bilayer coverage of the surface.

In the case of oil-wet stocktank oil, Region I was absent since the surface was already covered with natural surface-active polar components (such as asphaltenes). Hence, it is reasonable to assume surfactant adsorption kinetics directly from Region II. At low surfactant concentrations, the rock surface was strongly oil-wet. At higher concentrations, it became less oil-wet (Region III).

3.4 Surfactant Implications in EOR Field Applications

Although anionic surfactant was effective in altering wettability, it altered the wettability of initially weakly water-wet live oil to strongly oil-wet, which was less beneficial for water imbibition and hence for improved oil recovery. However, there is a possibility to develop a special kind of heterogeneous wettability known as “mixed-wettability” using this surfactant, which may make it a potential EOR choice for field practice. In mixed-wettability state, strongly oil-wet paths are generated in the reservoir at those parts of the pore surface in contact with crude oil, while the remainder stays strongly water-wet [18]. The oil would flow continuously through these well-connected oil-wet paths resulting in very high oil recoveries. Sometimes, the strong oil-wet characteristics rendered on the pore surface due to surfactants may result in continuous oil-wet paths for mixed-wettability development. The corefloods conducted by Ayirala and Rao [19] using the same anionic surfactant in Berea sandstones at ambient conditions with Yates stocktank oil and synthetic brine resulted in very high oil recoveries. This substantiates the ability of this surfactant to develop mixed-wettability in specific reservoir rock-fluids systems for significant oil recovery enhancements. However, the practical development of mixed wettability in oilfield scale needs further investigation.

Improper determination of original reservoir wettability can lead to poor decisions for improved oil recovery field applications using surfactants. Hence, the surfactant must be carefully chosen depending on initial reservoir wettability to maximize the benefit. Thus, an accurate in-situ reservoir wettability characterization and its alteration under in-situ reservoir conditions are essential for the success of any improved oil recovery process using surfactants in the field.

4. CONCLUSIONS

- The effect of crude oil composition and surfactants on dynamic contact angles has been investigated using Yates reservoir rock and fluids at Yates reservoir conditions.
- Significant differences observed between the wettabilities of Yates stocktank and live oils clearly indicate the need to measure wettability using live oil at reservoir temperature and pressure for determination of true in-situ reservoir wettability.

- The anionic surfactant has altered the wettability of the initially strongly oil-wet stocktank oil to less strongly oil-wet, while the wettability of the initially weakly water-wet live oil has been altered to strongly oil-wet by the anionic surfactant used in this study.
- The oil-wet behavior observed with Yates live oil due to the surfactant indicates the possibility to develop mixed wettability using this surfactant in a specific reservoir, which can enable preferential draining of oil phase for significant oil recovery improvements.
- The mechanisms for the surfactant-induced wettability alterations observed in this study at different surfactant concentrations for both stocktank and live oils appear to correlate well with a four-region surfactant adsorption model.

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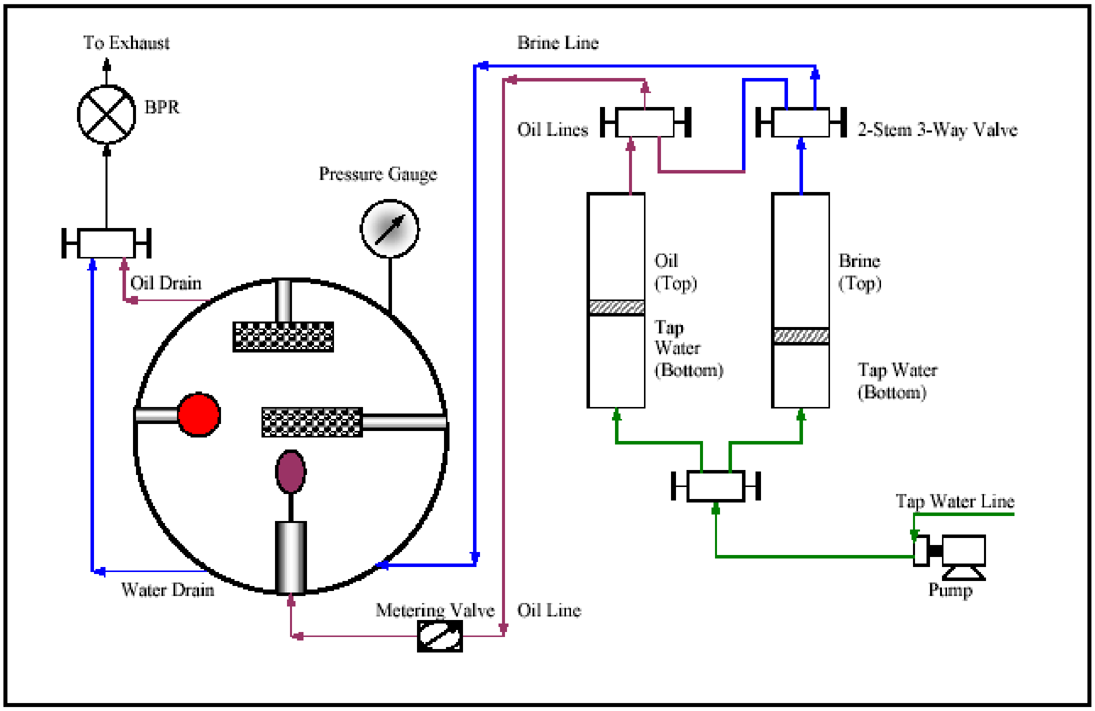


Figure 1. Schematic Flow Diagram of High-Pressure High-Temperature Dual-Drop Dual-Crystal Apparatus Used

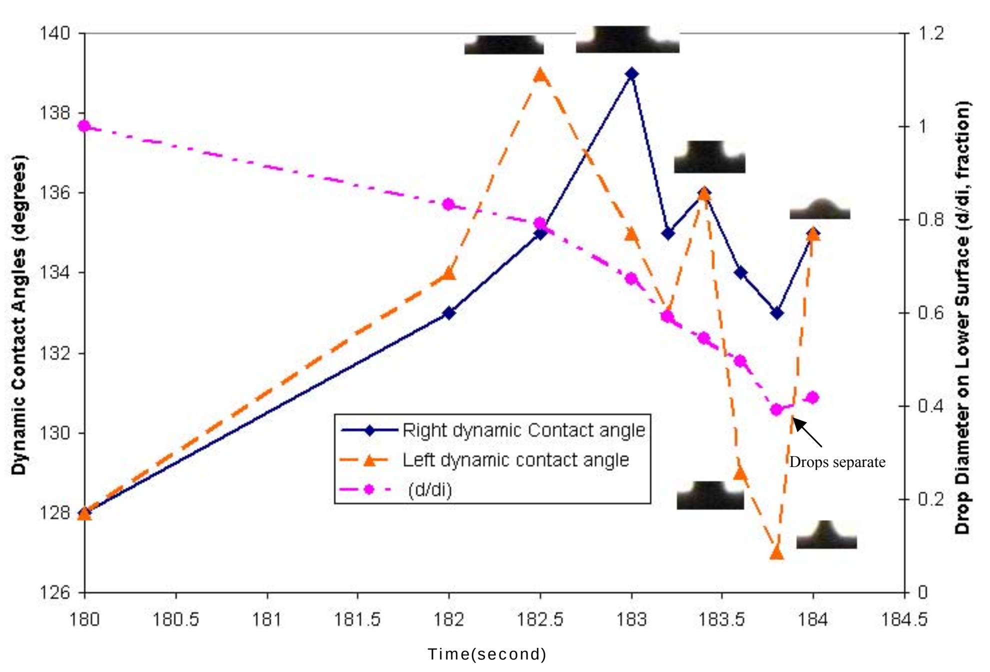
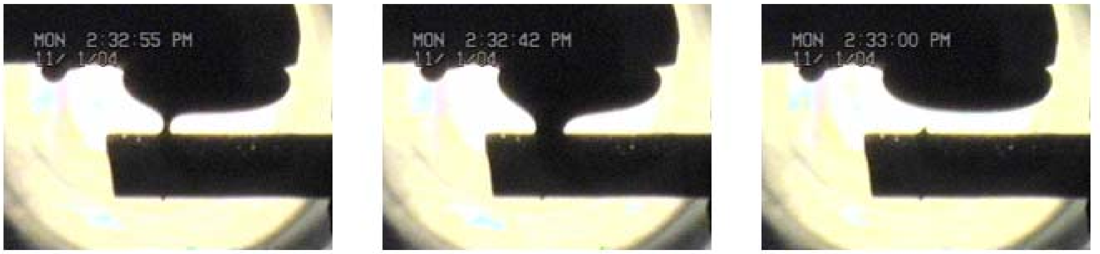


Figure 2. Dynamic Contact Angle and TPCL Movements in Yates Stocktank Oil-Yates Brine-Outcrop Dolomite System during 500 ppm Surfactant Injection (700 psi and 82 ^{o}F)

Before injection θa 49°-53°

Injection 0 min.

Injection 19 min. θa 78°-80°



Injection 20min. 19sec.
θa 130°

Injection 20 min.10 sec.
θa > 90°

Injection 20min. 29sec.
θa 140°

Figure 3. Depiction of Drop Movement during 500 ppm Surfactant Injection for Yates Live Oil-Yates Brine-Outcrop Dolomite System (700 psi and 82 ˚F)

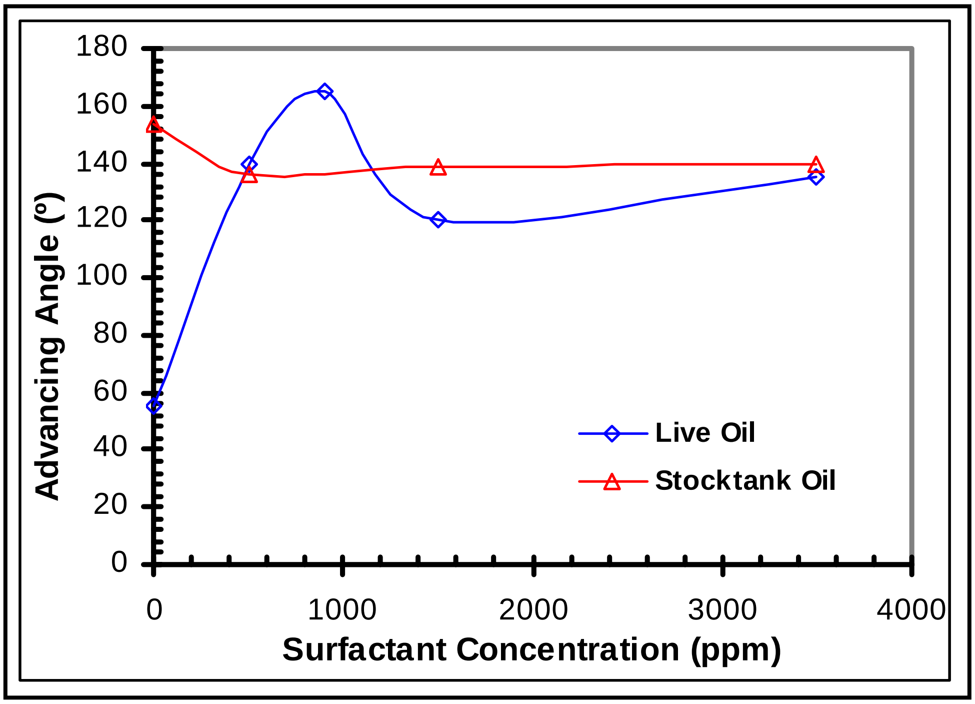


Figure 4. The Effect of Surfactant Concentration on Advancing Contact Angles for Yates stocktank oil and Live Oils (82ºF and 700 psi)

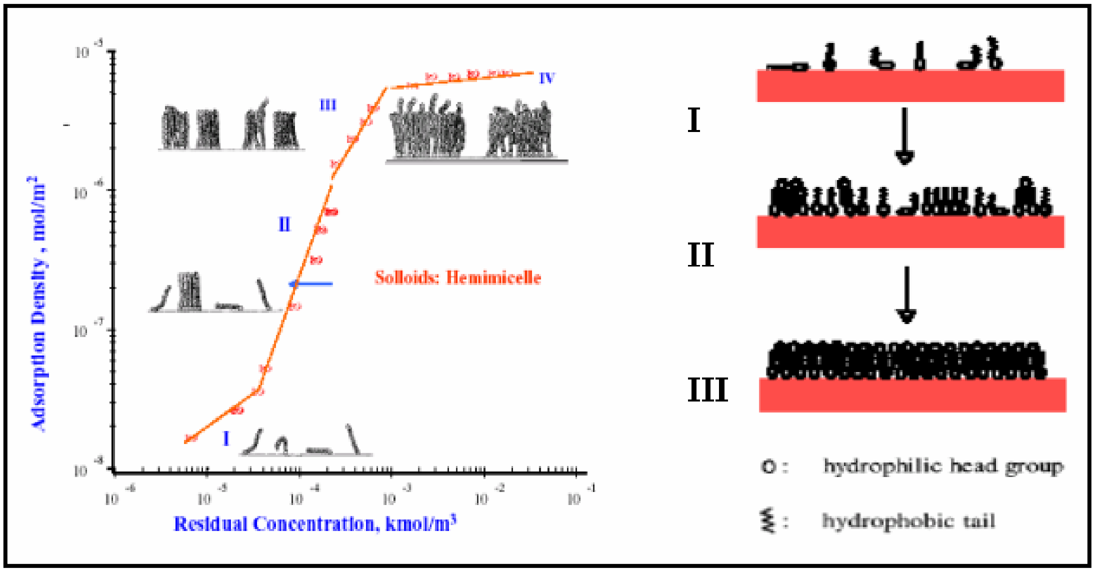


Figure 5. The Growth of Surfactant Aggregates for Various Regions of the Adsorption Isotherm (After Somasundaran and Zhang, 2004; Ref. 17)