

# Potential of Low Salinity Brines in Altering Wettability and Improving Oil Recovery from Liquid Rich Shale by Spontaneous Imbibition

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## **Abstract**

Low salinity brines were tested to be effective at altering wettability in sandstone and carbonate reservoirs while their impact on shale has not been examined so far. Such studies could help analyze the effectiveness of monovalent and divalent salts which could further be used to formulate affordable injection fluids that economically recover additional oil compared to conventional injection fluids. This paper aims to investigate the effect of concentration of brines of monovalent sodium and divalent calcium on altering the wettability and oil-water IFT in liquid rich shale with heterogeneous lithological composition that includes carbonate, quartz and clays. Contact angle, IFT and zeta potential tests were conducted to analyze rock-brine and oil-brine interactions while spontaneous imbibition was conducted at an optimum salinity derived from wettability tests. Computed Tomography was used to study fluid penetration into rock matrix and its effect on the resultant oil recovery.

## **Keywords**

*Wettability Alteration; Unconventional Reservoirs; Liquid Rich Shale; Low Salinity; Rock Fluid Interaction*

## **Introduction**

Craig (1971) defined wettability as the tendency of one fluid to preferentially spread on the surface of a solid in the presence of other immiscible fluids. In a water-wet state, the rock has higher tendency to stay in contact with water whereas in an oil-wet state, the rock prefers to stay in contact with the oil phase. Various factors affecting the wettability include surface charge of the rock, surface active agents in crude oil, composition of the brine in contact with the rock and temperature (Anderson, 1986, Part 1).

Wettability alteration coupled with reduction in interfacial tension (IFT) at the crude oil – water interface have been the focus of various imbibition studies as they affect the imbibition profile and consequent oil recovery (Alvarez and Schechter, 2016) (Babadagli, 2003). Most studies in understanding the interplay of these factors on oil recovery were conducted on high permeability/porosity sandstone or carbonate rocks, which have also been the focus of experimental work to study the effect of brine composition on wettability alteration. Martavaltzi et al. (2012) performed experiments on limestone outcrop samples to analyze the change in wettability with various brine solutions. Ca<sup>2+</sup> was found to influence the wettability of rock significantly as Mg<sup>2+</sup> was found to have little effects on the rocks examined. AlShaikh and Mahadevan (2014) studied the effect of chloride and sulfate salts of sodium, magnesium and calcium on wettability on aged calcite mineral surfaces and concluded that chloride salts have the maximum impact on changing the contact angle of the surface to water-wet state. Quan et al. (2012) conducted tests on clay and quartz samples with three chloride salts and observed sodium chloride promoted better water-wet state compared to calcium and magnesium salts. On the contrary, Standal et al. (1999) investigated the contact angles of isooctane oil phase and water solutions consisting of NaCl and CaCl<sub>2</sub> on silicate glass and  $\alpha$ -alumina as solid phases and concluded that increasing salt concentration increases oil wetness. Zekhri, Ghannam and Almehaideb (2003) measured the contact angles of oil/rock samples obtained from UAE oilfields at 0, 1,000, 10,000 and 50,000 ppm salinities of NaCl and found out that the maximum reduction in contact angle, hence maximum water wetness, was achieved at 10,000 ppm (1 wt%) as the contact angle decreased from 48° to 29°. This helped them conclude there is

an optimum salinity which is different than the formation salinity that would change the contact angle of a flooded area during water injection towards more water-wet.

The underlying mechanisms of wettability alteration due to addition of salts is unclear as various works attribute the changes to a multitude of factors as discussed by Myinth and Firozabadi (2015), who relate the effect of monovalent and divalent ions on wettability to double layer expansion and multicomponent ion exchange. For lower salinities the aqueous film thickness on the rock surface increases due to reduced shielding of the repulsive forces between negatively charged crude and negatively charged quartz/clay/silica surfaces (Hirasaki, 1991). This effect is more pronounced with monovalent ions as there is less shielding and more repulsion. The mechanism of double layer expansion may be different for carbonates as they are positively charged in aqueous solutions. Introduction of cations which compete with the host rock surface to attach to the negatively charged crude surface active agents leads to the detachment of oil from the rock surface.

Another mechanism of wettability alteration by ions is the multicomponent ion exchange through the thin water film. Divalent ions such as calcium form complexes with the negatively charged/acidic crude components as they are adsorbed onto the negatively charged sites in clays/silica. These complexes can be broken by introducing unbridged monovalent/divalent cations that interact with the negative sites in oil and rock separately as proposed by Lager et al. (2008). Besides, there is also a high possibility of reversal of surface charge due to the addition of cations, which reduces the ability of oppositely charged crude components to interact with the rock surface.

IFT changes with brine salinity are complex. Some studies report increase in IFT with salt concentration (Cai, Yang and Guo, 1996) while others conclude IFT does not necessarily increase with salinity (Abdel-Wali, 1996; Vijapurapu and Rao, 2003; Alotaibi and Nasr-El-Din, 2009). Various phenomena have been attributed to the impact of salts on IFT. Price (1976) argued that solubility of hydrocarbon species reduces with increasing salinity due to salting out effect while Bai et al. (2010) suggest salts can also accelerate the diffusion of surface active agents in crude from the bulk solution to the interface, thereby improving their activity. The effect of salts on IFT also depends on the type and the amount of surface active components present in the crude oil/brine system (Moeini et al. 2014).

All the literature reviewed on improved oil recovery by rock fluid interactions thus far is limited to conventional rocks as there is limited evidence of such studies in unconventional reservoirs. Some of the early attempts include investigations by Wang et al (2011), Alvarez et al. (2014), Alvarez and Schechter (2017), and Alvarez and Schechter (2016) who focused on the effect of wettability alteration and IFT reduction in unconventional reservoirs using surfactants. Their results suggest altering wettability to water-wet and moderately reducing the IFT improve recovery from the ultra-low permeability and porosity rocks, which validates the importance of these studies in unconventional. Early studies of the impact of brine salinity on recovery from unconventional rocks were conducted by Morsy and Sheng (2014) who evaluated the stability and oil recovery from Mancos and Bakken shale samples when imbibed with NaCl and KCl brines of salinities varying from 5 wt% to 30 wt%. Mancos shale samples were the most sensitive to distilled water and brine solutions as they were observed to be fragmented at salinities lower than that of formation water (13.8 – 21.2 wt% salinity). However, the oil recovery factor went up to as high as 59% at 5 wt% salinity, which might be due to the opening and propagation of fractures. In addition, low salinity brine was also found effective in improving recovery from Bakken shale samples. No visible cracks were observed in Bakken at low salinity of 15 wt% and hence clay swelling was not the dominant mechanism here. The increase in recovery was attributed to wettability alteration by decreasing brine salinity from 30 wt% to 15 wt% which marginally reduced the contact angle from 81° to 74° though such a minor reduction in contact angle does not alter the wettability of the sample significantly. Despite the aforementioned efforts, the impact of varying brine concentration and the associated surface changes in unconventional reservoirs remains unexplained.

Liquid rich shale reservoirs are responsible for increasing United States' oil production in the last five years and positioned it as one of the world's top oil producers (Doman 2015). However, OLRs low porosity and ultralow permeability lead to current recovery factors that do not exceed more than 10% of the original oil in place (OOIP) with average values of 5 to 6% (Alharthy et al. 2015). In liquid rich shale reservoirs fracture treatment performance and oil recovery can be improved by adding low salinity brines to stimulation fluids to promote imbibition by

wettability alteration and interfacial tension (IFT) reduction and consequently improved oil recovery by maximizing well performance after stimulation. This study includes a workflow that aims to characterize the interaction of brine with rock and oil surface that leads to the alteration of wettability of reservoir rock as well as the effect of such alteration on the imbibition and penetration of brine into the rock matrix. To our knowledge, this comprehensive study, using low salinity brines, has not been done in liquid-rich shale reservoirs. Finally, the impact of rock wettability alteration on oil recovery is analyzed by measuring the oil recovery from spontaneous imbibition experiments which gives insights into the application of sodium and calcium chloride brines as potential injection fluids for improved oil recovery.

## Experiments

### *Rock and Fluid Samples*

The rock samples received were from a well of a liquid rich shale play in South Texas. Samples were taken at different depths of the same well and were named S1-1 and S1-3 that had a difference of 120 feet. Further description of the lithology of these samples is provided in Table 1. The core samples obtained included trims and plugs that were aged in reservoir oil. The plugs had a porosity of 12-13% and a permeability of 250 nD. Oil samples obtained were taken from the same well and had a density of 0.72 g/cc (52.6 oAPI) at testing conditions of atmospheric pressure and 180oF. The two salts used for preparing brines, sodium and calcium chloride, were provided by Fischer Scientific. Distilled water was used as solvent. Salt concentrations of 1, 2, 3, 5, 10 and 15 wt% of sodium chloride and calcium chloride were used for contact angle measurements. Concentrations of 1, 2, 3 and 5 wt% of both salts were used for zeta potential measurements.

TABLE 1 LITHOLOGICAL COMPOSITION OF ROCK SAMPLES

Sample	S1-1	S1-3
Calcite %	56.3	60.2
Quartz %	15.3	15.4
Clays %	20.2	15.8
Pyrite %	4.7	4.1
Plagioclase %	2.8	2.6
Marcasite %	0	1.2

### *Aging Process*

Unaged trims were cut into smaller pieces, typically measuring 0.25 in. x 0.25 in. and were aged in the obtained crude oil at 180oF in an attempt to reach the temperature closest to that of the reservoir within the operational constraints of the equipment used. Whole core samples used for spontaneous imbibition experiments were obtained in a preserved state. These samples were taken out from wax and aged for a period of 3 months at 180oF to account for the loss of fluids due to flashing and gas entry during coring process.

### *Contact Angle Measurements*

Captive bubble method was used to measure the contact angle between and liquid and a solid surface. In this case, a drop of crude oil is injected from a needle pointing upwards onto the shale rock surface that is also in contact with the stimulating fluid. More details on the experimental setup can be found in Alvarez et al. (2014). The measured contact angle is then subtracted from 180o to yield contact angle on water on the rock surface. Contact angles ranging from 0o to 60o, 60o to 120o and 120o to 180o are employed for water, intermediate and oil-wet nature respectively after Wang and Gupta (1995). All the measurements were performed at 180oF.

### *Interfacial Tension Measurements*

The OCA 15 Pro apparatus described earlier was used for IFT measurements. Pendant drop – bottom up method was used since the dispensing phase was oil, which is less dense compared to the aqueous solution and the released drop moves up from bottom. All the experiments were conducted right after the contact angle measurements when

the aqueous phase was equilibrated at 180oF.

### Zeta Potential Measurements

The procedure for measuring zeta potential was developed by modifying the procedure described by Zhang (2005). Samples for zeta potential measurements required core trims to be crushed into fine powder using a mortar and pestle and passing the resulting powder through an ASTM E-11 325 sieve. The brine solutions prepared were triple filtered and rock was added in the ratio of 1:10 (< 1 mg per 10 mL of filtered brine) and sonicated using a QSonica ultrasonic probe at 40 Hz for one minute with at least half the tip immersed into the solution. The sonicated mixture is then allowed to stand for a while leading to the settling down of heavy particles. A small amount (typically 1.5 mL) of the mixture was added to a cuvette that was later attached to an electrode, which was then inserted into the zeta potential measurement device. NanoBrook™ ZetaPALS device, manufactured by Brookhaven instruments, was employed for zeta potential measurements. Phase Analytical Light Scattering (PALS) technique was used to measure the electrophoretic mobilities of solutions and correlate them to their zeta potentials. All the measurements were performed at 77oF.

### Spontaneous Imbibition

Modified Amott cells were used for carrying out the spontaneous imbibition experiments. The cell consisted of upper and lower chambers made from glass that are held together by aluminum rings with the help of clamping screws. The lower part or the base holds the core sample on a plastic stand designed to hold 2 – 3 inch long cylindrical core samples. The upper part or top has a graduating scale attached which is used to monitor the volume of oil recovered in the process. The modified Amott cell setup was placed in a Memmert U1060 oven and maintained at a temperature of 180oF. The setup was periodically scanned using a Toshiba Aquilion TSX – 101A CT scanner at the Chevron Petrophysical Laboratory at Texas A&M University. Images obtained from the CT scanner were analyzed using ImageJ, an open source image processing software. Further details can be found in Alvarez and Schechter (2017). Core plugs from well S-1 were aged for a period of three months at a temperature of 180oF. Once the sample was taken out from the oil for the imbibition experiments, the length, weight and contact angle with distilled water were measured. Reading from these measurements as well as porosity values provided by the core supplier are shown in Table 2.

TABLE 2 PROPERTIES OF CORE SAMPLES USED FOR SPONTANEOUS IMBIBITION EXPERIMENTS

Fluid	Core sample properties				
	Diameter (in)	Length (in)	Porosity	Initial Water Saturation (post aging)	Initial Contact Angle (degrees)
Water	0.997	2.288	12.3%	13.9%	104.9
2.5% NaCl	0.997	2.004	13.0%	10.4%	97.0
5% CaCl <sub>2</sub>	1.0015	1.723	13.0%	14.5%	99.4

The core is subsequently placed in the modified Amott cell that is filled with imbibing fluids prepared by adding 4 wt% KI which served as a dopant to increase the contrast between the oil and aqueous phases. The effect of KI on wettability of the samples was determined to be negligible to be considered for comparative analyses. The filled cells were then placed into the oven which was preheated to 180oF. Oil recovered is monitored periodically and the percentage recovery is calculated by dividing the measured oil volume at any instant by the product of pore volume and oil saturation of the core sample. Porosity ( $\phi$ ) information for the samples was provided by the sponsoring company and was used to calculate the pore volume ( $V_{pore}$ ) deduced from the diameter (d) and length (L) of the sample.

$$V_{core} = \pi d^2 L / 4$$

$$V_{\text{pore}} = V_{\text{core}} * \phi$$

$$\text{Oil recovery (\%OOIP)} = \{V_{\text{oil recovered}} / (S_{\text{oi}} * V_{\text{pore}})\} * 100$$

**Computed Tomography**

Computed Tomography (CT) is a non-destructive imaging technique that can be utilized for observing porous interior of a rock as wells as the flow of fluids through it. CT scanners using the petrographic analysis differ from the conventional X-ray techniques in that they generate cross sectional images of the object by measuring the attenuation of a beam of X-rays as it is rotated around the object at angular increments form a single plane (Akin and Kovscek, 2003). These images are then converted into a cross sectional image using back projection algorithms and Fourier transforms.

Modified Amott cells assembled with cores were scanned periodically and the scans taken were retrieved and processed using ImageJ to analyze the changes in CT number, measured in Hounsfield Units (HU), and fluids profile in the core sample. A parameter called penetration magnitude was then calculated to quantify the movement of fluid into the core sample with the progression of time based on the initial average CT number CT<sub>base</sub> and the average CT number at a time ‘t’ CT<sub>t</sub> as defined by Alvarez et al. (2014):

$$\text{Penetration Magnitude} = \text{CT}_t - \text{CT}_{\text{base}}$$

The difference in density between aqueous and oil phases was dependant on the loading of the dopant. As mentioned earlier, 4wt% KI was used which resulted in a CT number of 1000 HU for the pure brine while the oil had a CT number of -240 HU. This difference in CT number was due to the difference in densities of both the phases which led to a difference in attenuation of thre X-ray beams and hence, different CT numbers. Helical scans were taken with power and current set at 135 kV and 350 mA with a rotation time of 1 second. The slice thickness was 0.5 mm and the interval between each slice was 0.3 mm.

**Results and Discussion**

**Contact Angle Results**

Initial contact angles were measured in distilled water to understand the natural wetting state of the rocks. Both the samples were found to be intermediate wet with the contact angles being 75.7o and 73.3o for S1-1 and S1-3 respectively. The results for both the samples are as shown in Figures 1 and 2.

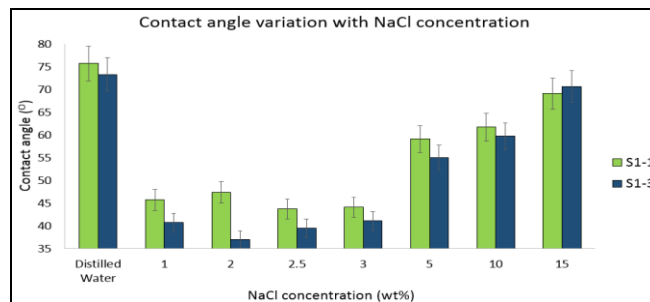


FIG. 1 CONTACT ANGLE CHANGE WITH SODIUM CHLORIDE CONCENTRATION FOR BOTH SAMPLES

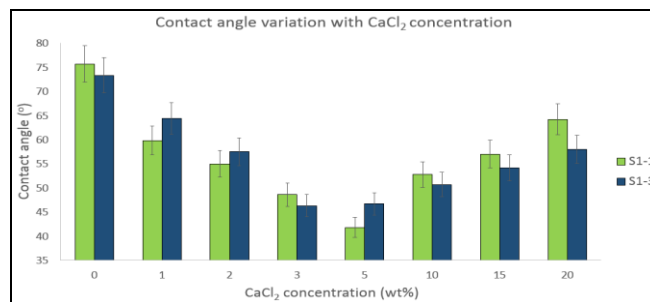


FIG. 2 CONTACT ANGLE CHANGE WITH CALIUM CHLORIDE CONCENTRATION FOR BOTH SAMPLES

Addition of salts decreased the contact angle to water wet and interesting trends were observed with increasing salinity. Experiments with NaCl brine on S1-1 suggested that low contact angle were obtained at low salt concentrations up to 3 wt %. The contact angle increased beyond this point indicating that lower salt content favored the promotion of water wetness. CaCl<sub>2</sub> produced similar trend with S1-1 with the contact angles decreasing to a minimum at low salt concentrations and further increasing as the salt content increased. The lowest contact angle produced by NaCl with S1-1 was found to be 43.7° at 2.5 wt% and that for CaCl<sub>2</sub> was determined to be 41.8° at 5 wt%. It was also observed that the contact angles were lower for NaCl at lower salinities and CaCl<sub>2</sub> at higher salinities. Experiments on S1-3 confirmed the trends obtained with S1-1. The lowest contact angles produced were 37° at 2 wt% of NaCl and 46.3° at 3 wt% of CaCl<sub>2</sub>.

### 1) Comparison of Results for Na<sup>+</sup> vs Ca<sup>2+</sup>

Figures 3 and 4 represent the variation in contact angles for S1-1 and S1-3 with varying sodium and calcium ion contents in the solution.

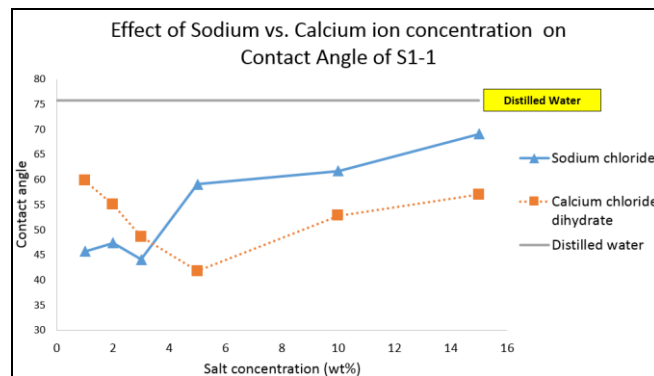


FIG. 3 CONTACT ANGLE CHANGES FOR S1-1

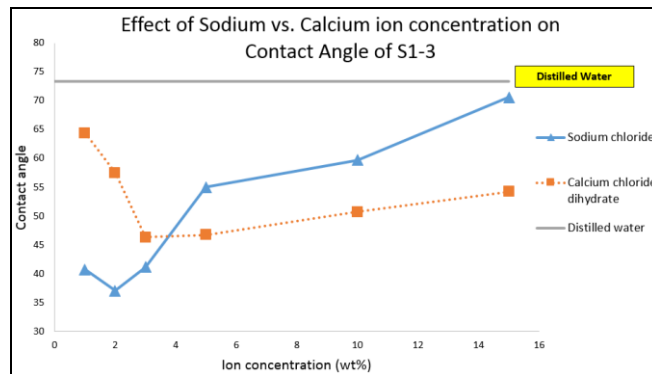


FIG. 4 CONTACT ANGLE CHANGES FOR S1-3

It was observed for both the cases that NaCl was more effective at promoting water wetness compared to CaCl<sub>2</sub> while this was reversed for higher salinities. Hence the question arises as to how these ions alter wettability, which could further be explained by zeta potential results in the coming sections. One possible explanation is that these cations compete with cationic components of crude for the negatively charged sites of clays and quartz in the rock surface as well as interact with the anionic components of crude that are adsorbed onto the positively charged carbonate and strip these components off from the rock surface. Hence the more positively charged Ca<sup>2+</sup> ions have a better effect than Na<sup>+</sup> at higher salinities as the interaction with anionic surface active groups in crude is stronger for the former. Dissolution of calcite in the presence of lower concentration brine also contributes to altering wettability. The loss of calcium by diffusion from the rock surface due to difference in rock and bulk Ca<sup>2+</sup> concentration leads to release of oil molecules associated with the calcite surface which promotes water wetness. However, the contact angle increases with increasing salinity due to salting out effect as the region around oil droplets is dehydrated and the oil surface active agents to be adsorbed at oil-rock interface as suggested by Standal et al (1999). However, cationic adsorption or exchange does not explain the performance of Na<sup>+</sup> at low salinities which could be

understood by zeta potential measurements.

**Interfacial Tension Results**

Interfacial tension was measured at 180oF for the oil brine interface using the pendant drop method. Initial oil/water IFT was found to be 34.03 mN/m. Figure 13 shows the variation in oil-brine IFT with increasing ion concentration for Na+ and Ca2+.

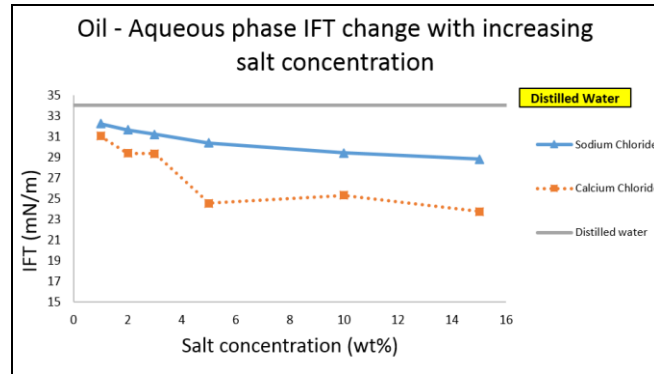


FIG. 5 CHANGE IN INTERFACIAL TENSION OF CRUDE OIL-BRINE INTERFACE WITH VARYING SALT CONCENTRATION

IFT reduced with increasing concentration for both the salts and the reduction was higher in the case of Ca<sup>2+</sup> compared to Na<sup>+</sup>. Though most IFT experiments involving the crude oil – brine interface report an increase in IFT with increasing concentration, as discussed earlier, experiments also suggested a decrease in IFT which as indicated by our experiments (Standal et al, 1999; Lashkarbolooki and Ayatollahi, 2014). The reason for this trend could be the fact that salts accelerate the diffusion of surface active components of crude from the bulk phase to the interface (Bai et al, 2010). The results were also consistent with the findings of Lashkarbolooki and Ayatollahi (2014) who suggested divalent chlorides produced lower IFT values than monovalent chlorides with acidic crudes.

**Zeta Potential Results**

Figures 6 and 7 illustrate the zeta potential results for S1-1 and S1-3 respectively. It can be inferred from these results that the zeta potential becomes less negative with increasing salt concentration as more cations surround the rock particles that are initially negatively charged as indicated by zeta potential values with distilled water, which were found to be -17.73 mV and -22.45 mV for S1-1 and S1-3 respectively. Zeta values of +/- 10 mV or above are assumed to be marginally stable and those below are instable with the stability increasing with increasing magnitude. Hence by this definition, the stability of brine films decreases as more salts are added to solution though NaCl was found to produce better stable film with S1-1 at low salinity (1 wt %) compared to distilled water. Stability of films formed by CaCl<sub>2</sub> were much lower compared to those formed by NaCl at all the concentrations examined. However, the increase in magnitude of the positive zeta potential with increasing Ca<sup>2+</sup> concentration implies an improvement in film stability, which might be a possible explanation for lower contact angles at higher salinities when compared to Na<sup>+</sup>.

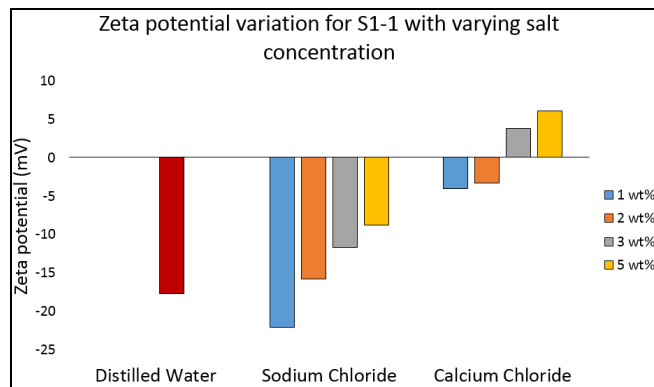


FIG. 6 ZETA POTENTIAL CHANGES FOR S1-1 WITH VARYING BRINE SALINITY

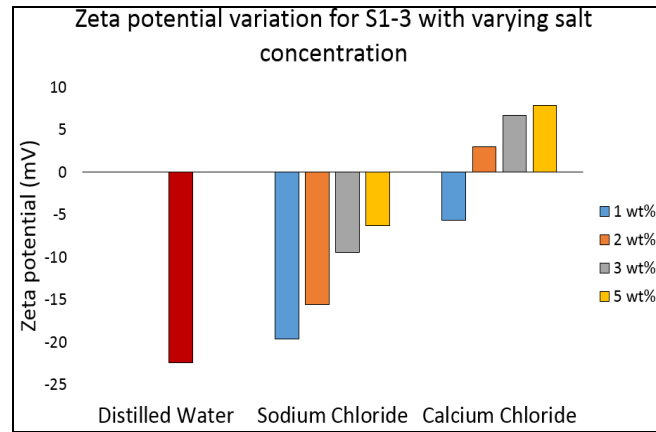


FIG. 7 ZETA POTENTIAL CHANGES FOR S1-3 WITH VARYING BRINE SALINITY

This stability of NaCl brine films at low salinity is consistent with results obtained by Quan et al (2012) who suggested NaCl formed stable films on silica and clays at low salinities. Similar results were also observed by Shehata and Nasr-El-Din (2015) who tested sandstone, quartz, clays and micas and suggested 0.5 wt% NaCl produced more negative zeta potentials compared to CaCl<sub>2</sub> brines. The correlation of stability and negativity of NaCl films to the low contact angles at low salinities can be understood by electrical double layer expansion. More negative the zeta potential, more electronic repulsion with oil – brine interface which is usually negatively charged and stable wetting. However, the results obtained for CaCl<sub>2</sub> suggest double layer expansion/ repulsion is not the only mechanism at play in wettability alteration of the rocks examined as was discussed earlier. These can be cationic adsorption at negatively charged sites and desorption of anionic polar compounds from oil by divalent cations which is the case with Ca<sup>2+</sup>. Positive zeta potentials of the rock surface with increasing Ca<sup>2+</sup> also verify this fact as the surface changes its nature and starts to desorb previously adsorbed cationic components from crude.

Hence it can be understood that a multitude of factors determine the effect of monovalent/divalent salts in shaping the wettability of shale rock surface. In addition to this, the rocks examined were heterogeneous in lithological composition as indicated in Table 1, which implies the surface charge at a particular site depends on the type of mineral, i.e. carbonate or clay and sandstone that is in contact with the aqueous solution which further adds to the uncertainty in characterizing the rock-fluid interaction and wettability.

### Spontaneous Imbibition Results

This section presents the imbibition results for water and brines which include wettability changes, penetration of the fluids into cores and the associated oil recovery. The order of discussion of results same as listed before with wettability and IFT changes used to understand the penetration of fluids and oil recovery.

Aging the cores at 180oF led to natural imbibition of oil which displaced the trapped gaseous phase that is assumed to have entered the core during coring and preservation. Flashing of in situ oil phase also leads to generation of gas which may be lost from the cores during the preservation process. The cores were used after a period of 3 months when bubbling of the gaseous phase stopped. The following equations were used to calculate changes in oil saturation based on imbibed oil volumes ( $V_{oil,im}$ ) and core properties.

$$W_{post-aging} - W_{pre-aging} = W_{oil,im}$$

$$V_{oil,im} = W_{oil,im} / \rho_{oil @ 180F}$$

$$\Delta S_o = V_{oil,im} / V_{pore}$$

Table 4 provides data on imbibition volumes of oil into different core samples during the aging process. The density of oil ( $\rho_{oil}$ ) used for calculations was measured at 180oF and found to be 0.7176 g/cc. The data provided for this well suggests that the reservoir rock had an initial oil saturation of 45.8%. This is added to the change in oil saturation due to aging to yield the initial oil saturation in the sample prior to conducting the spontaneous imbibition tests,  $S_{oi}$ , which is presented below.



TABLE 4 FLUID FORMULATIONS FOR SPONTANEOUS IMBIBITION TESTS

Sample	W <sub>oil,im</sub> (g)	V <sub>oil,im</sub> (ml)	V <sub>pore</sub> (ml)	ΔS <sub>o</sub>	S <sub>oi</sub>
1	1.04	1.44	3.59	40.3%	86.1%
2	1.05	1.46	3.33	43.9%	89.6%
3	0.82	1.15	2.89	39.8%	85.5%

Table 5 is a summary of results for distilled water and brine imbibition studies. Figures 8 and 9 show the penetration magnitude and oil recovery plotted for water and brines with time respectively.

TABLE 5 SUMMARY OF SPONTANEOUS IMBIBITION RESULTS

Fluid	Sample	IFT (mN/m)	Final contact angle (o)	Final penetration magnitude (Hounsfield Units)	Oil Recovery (%OOIP)
Water	1	34.03	85.3	8	1.9
2.5 wt% NaCl	2	31.57	41.1	14	8.1
5 wt% CaCl <sub>2</sub>	3	29.33	44.8	12	7.3

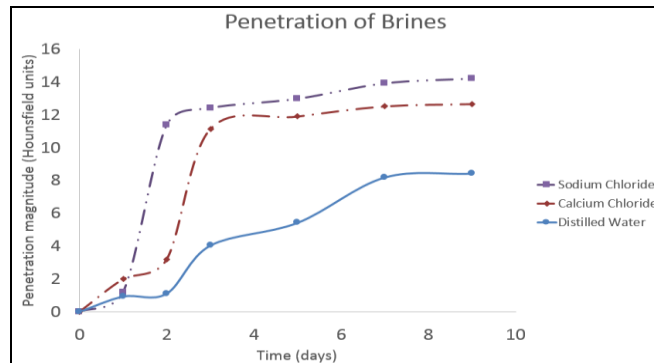


FIG. 8 PENETRATION MAGNITUDE OF FLUIDS USED FOR SPONTANEOUS IMBIBITION TESTS

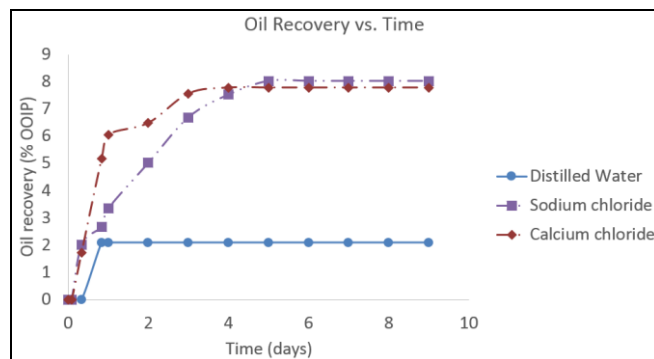


FIG. 9 OIL RECOVERY RESULTS

1) *Distilled Water*

Distilled water was used to serve as a reference point for imbibition studies. Water slightly altered the wettability of the rock though the final state remained intermediate wet (Figure 10). It can be observed from the Figure 8 that the

penetration was steady over the course of the experiment though the magnitude was low. The ultimate penetration magnitude was found to be 8 HU meaning the overall CT number of the sample increased by 8 HU as the lower density oil phase was replaced by high density aqueous phase.

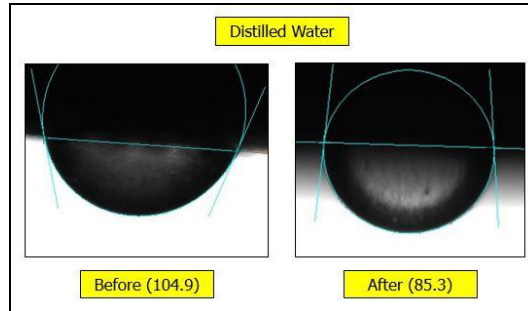


FIG. 10 CONTACT ANGLES EMASURED BEFORE AND AFTER SPONTANEOUS IMBIBITION WITH WATER

Movement of water was studied using the CT scans taken at various times (Figure 11). The purple portions in the circled region are the bedding planes along which the most noticeable changes occur. Hence it is safe to say water imbibed along the bedding planes to displace oil although the change between initial and final states is not significant. The oil recovery curve shown in Figure 25 denotes that oil was recovered over a period of 1 day beyond which no additional oil could be recovered. This is an indication that water was not able to penetrate deeper into the matrix thus recovering only oil from the periphery. Though penetration was increasing over time, the change in density is low implying the imbibing fluid could not displace the oil from pore throats due to high IFT and intermediate wet state. The ultimate recovery was 1.9% of the oil originally in place.

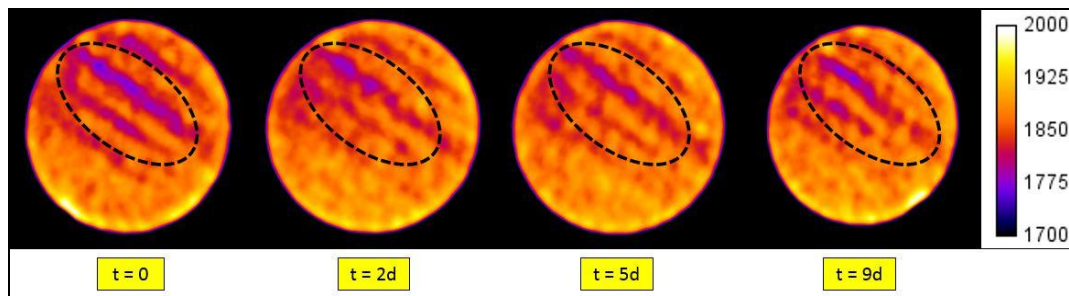


FIG. 11 CT SCAN IMAGES SHOWING PENETRATION OF WATER DURING SPONTANEOUS IMBIBITION

## 2) Brines

The objective of brine experiments was to understand effectiveness of two brine solutions, 2.5 wt% sodium chloride and 5 wt% calcium chloride on imbibing into the rock matrix and displacing oil. These concentrations were chosen to be the optimum concentrations for promoting the maximum water wetness from contact angle experiments as discussed earlier. Sodium chloride produced a more water wet state while calcium chloride produced a lower IFT compared to sodium chloride brine as established earlier. Contact angle and IFT values in Table 4 also verify this trend. Thus the imbibition results from these experiments were intended to determine the dominant mechanism that aids fluid penetration into the samples. Figures 12 and 13 show the contact angles measured on the flat side the core sample used for imbibition tests.

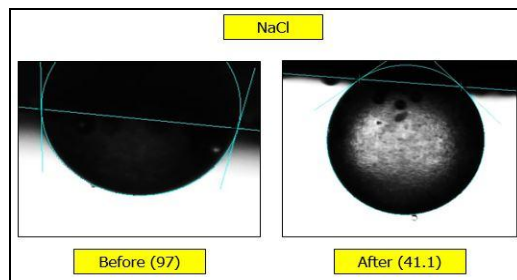


FIG. 12 CONTACT ANGLE CHANGES FOR SODIUM CHLORIDE BRINE IMBIBITION

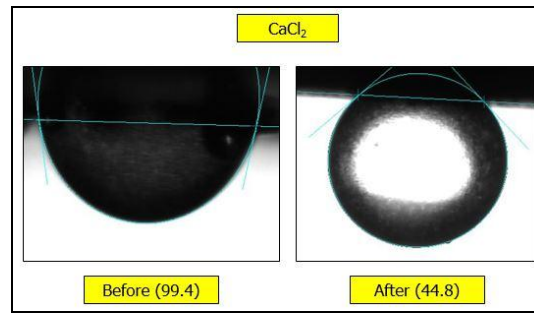


FIG. 13 CONTACT ANGLE CHANGES FOR CALIUM CHLORIDE BRINE IMBIBITION

As can be observed, both the salts altered the wettability from an initial intermediate wet to a water wet. Sodium chloride produced a slightly lower contact angle meaning more water wetness. As is evident from the penetration magnitude plot, sodium chloride penetrated the most into the rock followed by calcium chloride. The final penetration for sodium chloride was almost twice as that of water. Brines penetrated deeper into the cores than water implying alteration of wettability plays a major role in improving the penetration of fluid into the rock matrix. Calcium chloride penetrated faster initially compared to sodium chloride. This is probably due to the lower oil/brine IFT values produced by calcium chloride which help the fluid to penetrate faster into the rock matrix. However, as time progressed, sodium chloride penetrated faster suggesting capillary forces dominated imbibition. The low penetration of water at the same time signifies the importance of altering wettability for improved imbibition. CT scan images (Figures 14 and 15) indicated that imbibition occurred along the bedding planes shown in the circled regions similar to frac water. However, salts were able to alter the wetting nature of the rock thereby imbibing better along these planes and displacing more oil as low density purple zones are replaced by higher density orange and yellow zones. It is important to note that these images do not completely reflect the imbibition profiles of fluids as the assigned color is an average value calculated for a pixel while mechanisms such as clay swelling and rock dissolution take place at the micro level that may not be captured by the CT scanner. Clay swelling and osmotic fluid movement at the interface of clays and bulk brine phase lead to the development of microcracks which serve as additional conduits for flow. The impact of these phenomena on oil recovery cannot be ignored as the rocks contained a sizeable amount of clay minerals.

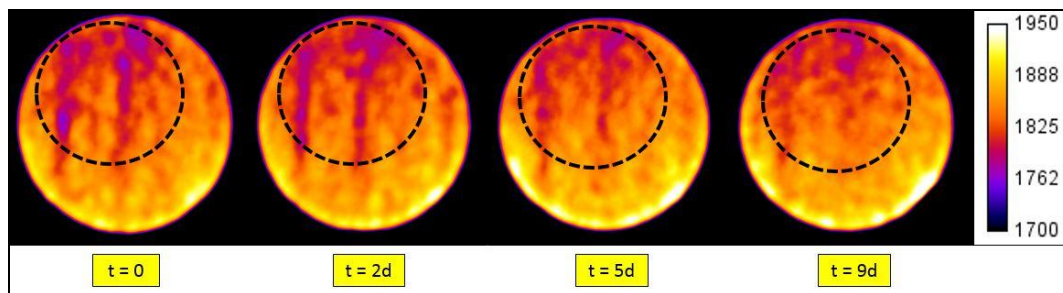


FIG. 14 CT SCAN IMAGES SHOWING PENETRATION OF SODIUM CHLORIDE BRINE DURING SPONTANEOUS IMBIBITION

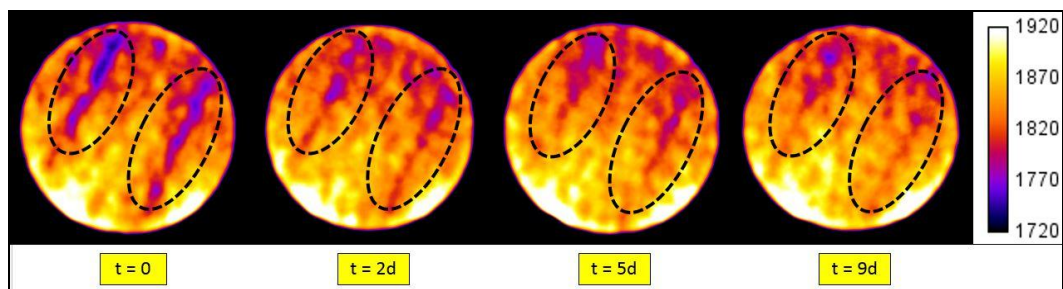


FIG. 15 CT SCAN IMAGES SHOWING PENETRATION OF CALIUM CHLORIDE BRINE DURING SPONTANEOUS IMBIBITION

As suggested by the recovery curves in Figure 15, both the salts were able to recover more oil than frac water. Besides, the recovery curves for the salts did not flatten out as fast as the curve for water implying the brine were

able to penetrate deeper in to the rock matrix and drive more oil out as time progressed. Thus as the wettability of the rock is altered, the fluid penetrates deeper into the rock and displaces the oil in place. Reduction in oil/brine IFT seems to improve the penetration in the early stages while capillary imbibition seems to dominate the ultimate recovery. This is understandable as moderate to high IFT values improve the recovery from ultra-low permeability reservoirs where capillary imbibition plays a significant role.

## Conclusions

The effect of sodium and calcium chloride brines on the wettability alteration of liquid rich shale samples and IFT reduction at the crude oil/brine interface as well as their impact on oil recovery from imbibition were studied. The following conclusions were reached based on our observations and analysis of the results obtained

1. Both sodium and calcium chloride were effective in promoting water wetness on the rock samples used with the maximum water wetness observed at 2.5 wt% for sodium chloride and 5 wt% for calcium chloride.
2. Increasing salt concentration beyond the optimum point shifted the rock nature towards more oil-wet which meant lower salinities promoted water wetness better compared to higher salinities.
3. Sodium chloride was found to promote higher water wetness and form stabler aqueous films on rock surface compared to calcium chloride at low salinities while this reversed at higher salinities examined.
4. Double layer expansion mechanism alone cannot explain wetting changes which may be a result of a multitude of other factors such as desorption of organic components, ion exchange with clays and divalent ion complexing with polar organic molecules in oil phase.
5. Crude oil/brine IFT reduced marginally with increasing salinity for both the salts and was found to be lower for calcium chloride at all concentrations examined.
6. Brines penetration magnitude was higher than that of water which also resulted in higher oil recovery. Brines were also able to penetrate deeper into the core and displace oil as opposed to water which only recovered oil from the periphery. Sodium chloride recovered more oil compared to calcium chloride which suggests that moderate IFT reduction coupled with a strong water-wet state favored capillary imbibition in the unconventional rocks used.

## ACKNOWLEDGMENT

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Dr. Schechter was the author and Principal Investigator for a \$13 million Department of Energy/National Petroleum Technology Office Class III Field Demonstration CO<sub>2</sub> project in the naturally fractured Spraberry Trend Area. In addition, he worked with CEO Mark Fischer from Chaparral Energy and with \$1.75 million dollars, formed the Chaparral-Fischer, CO<sub>2</sub> Enhanced Oil Recovery Center in 2011. Dr. Schechter has taught 75 short courses and consulted in more than 20 countries in collaboration with over 30 companies and he has 35 years of experience with EOR including all aspects from basic laboratory studies to development, implementation and analysis of full field performance. Dr. Schechter is a SPE Distinguished Member since 2014.

# Towards Physico-Chemical Characteristics of Micro Glass Bubble-based Fluids

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## Abstract

One of the most important and controversial issues throughout drilling operations in Iranian Southern Oil Fields is drilling fluid loss. To overcome this problem, an under balance drilling method has been carried out in a few wells. However, controlling damage in the reservoir due to fluid loss has been considered as a critical issue. The aim of this study is to investigate the effect of glass bubbles materials to control fluid losses at drilling fluids used in drilling operations in the Asmari reservoir. As a result, several laboratory experiments were performed on Asmari's drilling fluids by glass bubbles with 4000 type from the 3M Company. The effects of adding glass bubbles and other materials used in Parsi oil field, Iran, were investigated specifically. The results obtained illustrated that glass bubbles decreases the weight of water-based emulsion fluid from the lightest, which was 55.5 pcf to 43 pcf with no rheological changes in the fluid properties in case of using maximum 40% V/V glass bubbles in the fluid. Effects of different combination of additives were also investigated in this study. Finally, a characteristic plot was drawn for emulsion fluid showing mud weight variation based on the amount of glass bubbles added.

## Keywords

*Naturally Occurring Fractures; Glass Bubble; Loss Circulation; Under Balance Drilling; Temperature*

## 1 Introduction

Drilling operation is the most cardinal phase in exploitation of hydrocarbon reserves. The success of such operation depends to a large extent on appropriate selection of drilling fluid. Drilling fluids, which are also referred to as "drilling mud" in petroleum industry, play a significant role in drilling natural gas, oil, and water wells [1]. Two primary types of drilling fluid, which are extensively being used in petroleum industry, include: water-based and oil-based muds [2]. Although oil-based muds have many excellent features including the temperature stability, lubricity, and wellbore stability, which leads to their higher performance in drilling operations; nevertheless, excessive application of these kinds of fluids could result in irreparable environmental hazards [3-6]. Therefore, water-based drilling muds were developed as more environmentally-friendly fluids aiming to alleviate such toxicity problems.

Drilling fluids were primarily designed to control the subsurface pressure and prevent the formation fluid from entering the wellbore. Aside from subsurface pressure control, minimization of formation damage as well as loss circulation into the formation was among the cardinal factors taken into accounts while designing such fluids. The loss circulation, which is defined as the loss of drilling fluid into the formation, is among the severe problems encountered in drilling operation [1, 7]. Complete loss occurs when all drilling fluid invades into the formation and nothing returns to the surface during circulation. Fluid loss is the entire loss of both continuous and non-continuous components into the formation. Excessive levels of overbalance could result in severe fluid invasion and loss into the formation. Among the problems associated with loss circulation are differential sticking, wellbore instability, and even severe blowouts [8].



One of the methods identified and implemented for improving the recovery of hydrocarbons from low pressure reservoirs is under balanced drilling (UBD). In UBD, lower density drilling fluids with specific gravity less than one (62.4 pcf) such as mist, aerated muds (classic muds with nitrogen), or foam are implemented to provide a pressure lower than that of pore pressure of the formation rock.

Among the drilling fluids used in UBD drilling of depleted reservoirs, drilling operators are not prone to use aerated fluids in UBD operations mainly owing to the complexities associated with their compressibility and two phase condensation [9]. Aside from operational complexities, high operational costs arising from rental of compressors to produce in situ air or nitrogen are among the disadvantages of using aerated muds (Figure 1).

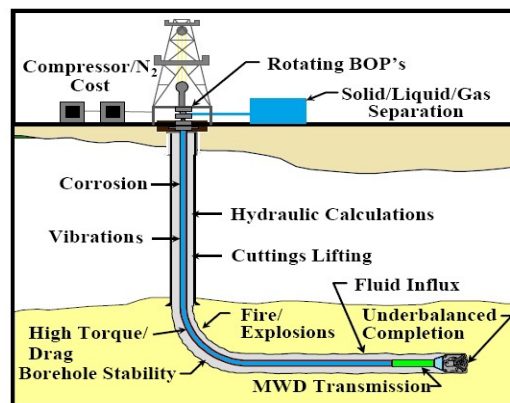


FIGURE 1. HOLE PROBLEMS WITH AERATED DRILLING FLUIDS [9].

In order to alleviate such problems, an alternative approach to produce a low density drilling while maintaining the advantages of aerated muds is to employ the hollow glass bubbles into conventional drilling fluids. In fact, this incredible additive could be added to any type of drilling fluid independent of its nature in order to considerably reduce its weight. Low density drilling fluids containing hollow glass bubbles can lead to an increase in the rate of penetration, prevent any overbalance problems and damage to the formation caused by invasion of fluid solids, or filtered fluid and also improve the longevity of the bit (Figure 2).

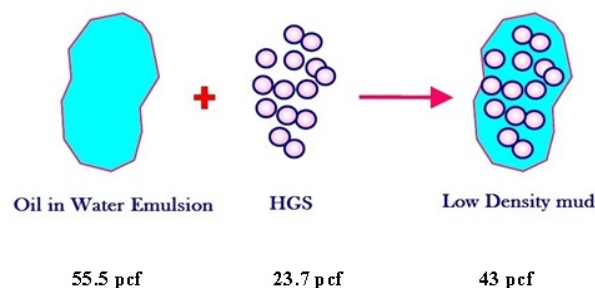


FIGURE 2. THE HOLLOW GLASS SPHERE, HGS LOW DENSITY FLUID CONCEPT [10].

Implementation of light density fluids based on glass bubbles together with loss circulation materials (LCM) have proved to be highly efficient in eliminating the fluid loss problems in fractured reservoirs. One of the most important features of glass bubbles is that they provide an underbalanced or near balanced condition in depleted reservoirs without the use of air or any other kind of gas while implementing the routine standard mud handling equipment [11].

In Guafita field located in southwestern Venezuela, drilling operators were seeking a low density fluid with good rheological properties. Therefore, PDVSA-INTEVEP designed a drilling fluid system referred to as INTEFLOW-2000 for specific use in low pressure depleted zones like that of Guafita field. Addition of density reducing glass bubble agents known as LITEDEN to INTEFLOW-2000 drilling fluid has already proved to be highly successful in minimizing the problems of differential sticking in well GF-136D located in Guafita [10, 11].

In recent years, the problem of drilling through depleted fractured carbonate reservoirs has been encountered in Parsi oilfield. Parsi oilfield is among the most depleted reservoirs located in southwestern part of Iran. This field started to produce with initial pressure of 3800 psia in 1966. In 2001, the reservoir pressure was 3000 psia with approximate 1.48 Billion STB production. By June 2009, an overall 1.68 Billion STB had been produced and the reservoir pressure had reached 2470 psia at depth of 1800 subsea. Complete loss during drilling operations has been a serious challenge in this field. Drilling of the well Parsi-21 was started with a mud weight of 67.5 pcf and extreme loss circulation was observed while drilling the reservoir section. The wellbore fluid was changed with gasoil and the rig was released. Later, a workover operation was conducted and the drilling operation continued with a water-gasoil emulsion of 55-60 pcf weight and again complete loss occurred at the depth of 2228 m. In this respect, National Iranian Oil Company (NIOC) has initiated the UBD technology program since 2005 in depleted southern Iranian fields.

Therefore, the primary objective of this study is to develop a new water-based emulsion fluid with density reducing glass bubble additives aiming to provide a viable solution in response to the concerns raised in well No. 21 of Parsi oilfield. To this end, the optimum concentration of glass bubble additive to reduce the density of water-based emulsion fluid to 43 pcf, as requested in the well No. 21 to provide an under balanced condition, was investigated in this study. Moreover, a number of fluid experiments have been carried out under different temperatures employing different additives to generate a drilling fluid the characteristics of which is similar to the fluid already used in this field. Development of such fluid is intended to obviate the need for under balanced drilling operations already in progress as they have appeared to be highly expensive and less effective.

## 2 Experimental Methods and Materials

Experiments were conducted over different temperatures so that after addition of density reducing glass bubble agents together with other additives, a fluid similar to the drilling fluid implemented in the Parsi oilfield was generated. Characteristics of the water-based emulsion fluid used in the Parsi oilfield are summarized in Table 1.

TABLE 1. CHARACTERISTICS OF PREPARED EMULSION USED IN WELL NO.1 AT PARSIOIL FIELD.

Properties	Value
Fluid weight, pcf	52-55.5
Viscosity, sec	51-57
Plastic viscosity, cp	20-40
Yield point, lb/ 100 ft <sup>2</sup>	4-24
pH	8.5
Fluid Loss, cc	No Control
Salt, ppm	4000
Calcium, ppm	800-1200
Gel 10 sec, lb/ 100 ft <sup>2</sup>	2-3.4
Gel 10 min, lb/ 100 ft <sup>2</sup>	4
Temperature (°F)	100-120

In order to build a water-based gas oil emulsion, different percentages of gasoil and water without addition of any foreign material was investigated. The main components of the water-based emulsion fluid include:

1. Continuous phase: It includes diesel, which its material composition may vary from one refinery to another refinery. However, in this work, the diesel of grade two was used for preparation of the water-based emulsion fluid.
2. Emulsifier: Its chemical type includes non-ionic polymer chains of long type and irresolvable, named DME (Drilling Fluid Emulsifier). DME is insoluble in water and should never be added directly to gasoil, and it should be added to water [12].
3. Diffuser phase: It includes water and usually is piped and pumped from the closest place to drilling well.

In order to find the optimum configuration for the water-based emulsion fluid, different percentages of water and gasoil were mixed in experimental barrels. Each experimental barrel is equivalent to 350 cc in volume; thereby the

percentages are assessed based on this volume. For instance, to build a 50:50 emulsion mud, 1 volume percent of 350 cc emulsion mud (equivalent to 3.5 cc) is allocated to DME (Drilling Mud Emulsifier) and water and gasoil share the remaining volume at 50:50 ratio. Different emulsions based on various gasoil water ratios were generated and the emulsion properties were measured in static condition and temperature of 77 °F. In these experiments, the weights of gasoil and water were 52.5 and 62.4 pcf, respectively. Experiments were repeated at the temperature of 140 °F using the emulsion configurations obtained in previous test. Figures 3 and 4 show the emulsion properties measured for different emulsion configuration at temperatures of 77 and 140 °F, respectively. Water gasoil emulsions with 50:50 and 65:35 (gasoil: water) have shown satisfactory physical properties at 140 °F using only 1 percent of fresh water DME. Increasing the gasoil ratio above 60 results in gradual emulsion height reduction and the consequent increase in viscosity, which in turn, results in a considerable increase in yield point and the gelation. On the other hand, increasing the water ratio in water-based emulsified fluid results in instability of the emulsion and separation of a considerable amount of water, which makes it per se impractical.

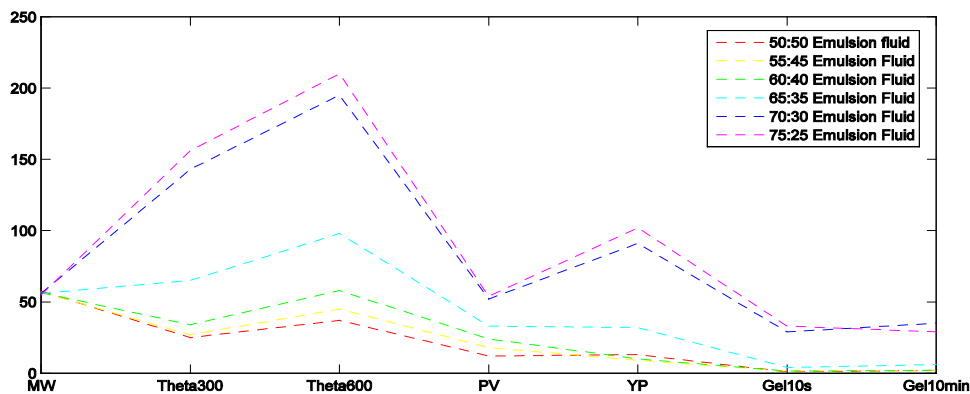


FIGURE 3. WATER-BASED EMULSION FLUID IN DME AT 77 °F.

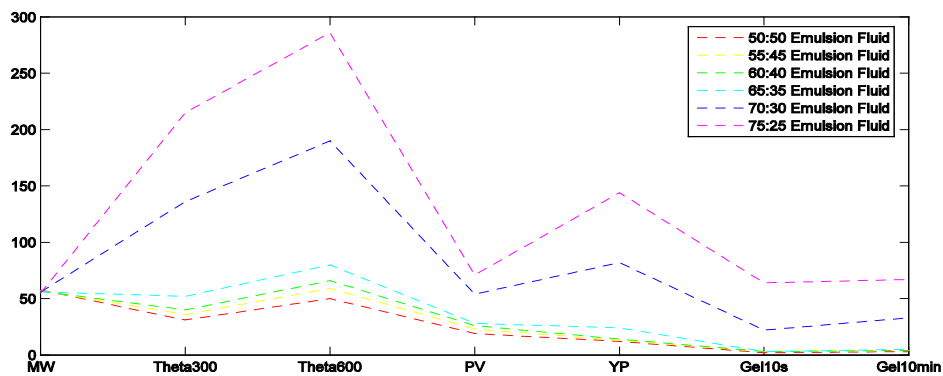


FIGURE 4. WATER-BASED EMULSION MUD IN DME AT 140 °F.

In order to simulate the drilling fluid circulation during drilling operation, before the main experiment, the drilling fluid underwent rolling operation and then its properties was quantified at the temperature of 140 °F in dynamic condition. The results have been summarized and represented in Figure 5. Note that all the experiments so far have been conducted based on fresh water DME. According to Figure 5, all water-based emulsified fluids have preserved their physical properties at 140 °F and no variation could be observed.

Based on the physical properties of 70:30 water-based emulsified fluid used in Parsi oilfield as well as the reservoir pressure conditions, which requires a 43 pcf mud weight, experiments were carried out to create an appropriate water-based emulsion via implementing density reducing hollow glass spheres. The hollow glass spheres (HGS) are about 36 micron in diameter with this particle size distribution fitting appropriately between sand and colloidal particles. Glass bubbles could be implemented as lightening additives for any type of fluid system. In general, any type of liquid including water, salt water, and gasoil could be used to generate a low density drilling fluid without

taking account the nature of liquid phase [11]. The properties of gasoil in water emulsion fluid with the ratio of 70:30 are presented in Tables 2 and 3. Even though, the emulsions with 60:40 and 65:35 ratio appeared to have a better performance based on the test results; nevertheless, these configurations required higher volumes of glass bubble (more than 35 % v/v), which in turn resulted in an adverse increase in rheological properties of the drilling fluid, thereby making them inapplicable in the Parsi oilfield.

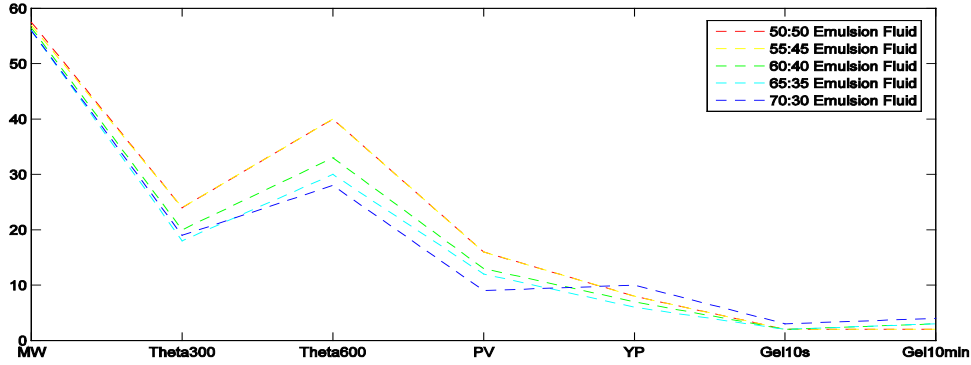


FIGURE 5. WATER-BASED EMULSION FLUID IN DME AT 140 °F IN ROLLING CONDITION.

TABLE 2. PROPERTIES OF HOLLOW GLASS SPHERES [6].

Shape	Unicellular Hollow Sphere
Color	White to Unaided Eye
Composition	Soda-Lime-Brosilicate Glass
Specific gravity, g/cc	0.38
Collapse pressure, psi	4,000
Average particle size, micron	36
Thermal stability, (°F)	1112
Alkalinity, meq/g	Máx, 0.5
pH when dispersed in water	9.5

TABLE 3. CHARACTERISTICS OF PREDICTED OIL IN WATER EMULSION.

Fluid weight, pcf	55.5
Viscosity, sec	±40
Plastic viscosity, cp	25±
Yield point, lb/100 ft <sup>2</sup>	12-10
pH	8.5
Fluid Loss, cc	No Control

In this respect, a 70:30 emulsion fluid was created (Water (30 % v/v) + DME (1% v/v) + Gasoil (70 % v/v)) using salt water DME, which is also applicable in fresh water DME. This is due to the fact that oil wells in Iran mostly implement salt water DME. In order to reduce the mud weight to 43 pcf, glass bubble was added in amount of 40% of the total volume of water-based emulsion fluid according to Equation 1, which was then set under rolling condition for 4 hours at 200°F.

$$\sum \rho \left( \frac{gr}{cc} \right) = \frac{\sum M}{\sum V} \tag{1}$$

This mud was completely homogeneous after 0.5, 1, 2, 4, 8, 12 hours; nevertheless, after 24 hours in static condition at 77°F, the 70:30 43 pcf emulsion mud became segregated and a two-phase condition occurred (Figure 6).

Therefore, 0.1 g Xanthan gum was added to the two-phase mud and it was mixed for 10 minutes at 77 °F. After 3 days in static condition at 77°F, the mud was completely homogeneous. Figure 7, compares the 55.5 pcf water-based emulsion fluid (70:30) to 43 pcf mud weight created after adding 0.1 g Xanthan gum at 77°F. These two fluids are very similar from rheological perspective. Owing to the fact that these experiments were conducted in the

rolling device at a low rpm, shearing process has not occurred completely.

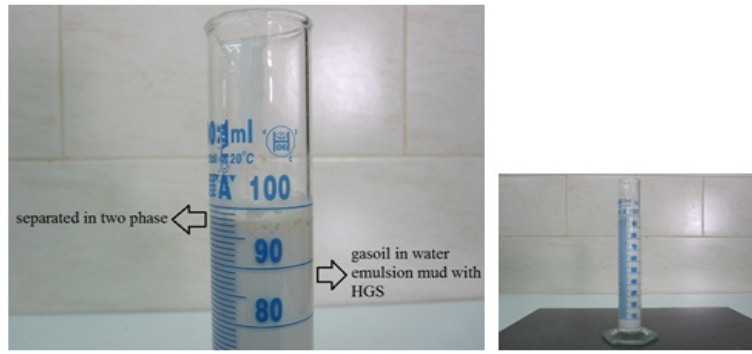


FIGURE 6. SAMPLE OF PREPARED WATER-BASED EMULSIFIED FLUID.

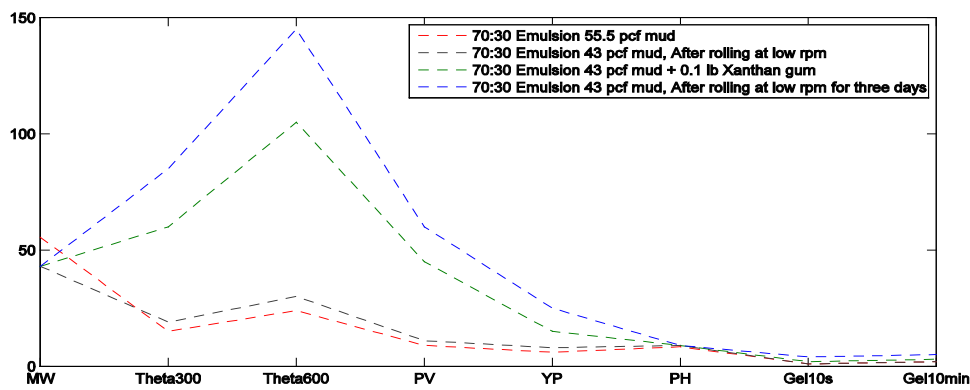


FIGURE 7. COMPARISON BETWEEN WATER-BASED EMULSION FLUIDS, AFTER 24 HRS, AFTER ROLLING OPERATION AT LOW RPM, AND AFTER 3 DAYS AT LOW RPM.

Figure 8 compares the 70:30 water-based emulsion fluids of 55.5 pcf and 43 pcf weight before and after rolling at high rates at 77°F and 140°F. Rolling at high rpm has resulted in a better rheological property, which is much more pronounced at 77°F. The effect of temperature on 43 pcf mud is also presented in Figure 9, which shows the considerable effect of temperature on glass bubble and water-based emulsion fluid.

In later experiments, it was observed that 35% volume of glass bubble added to the water-based emulsified fluid could also provide 43 pcf mud weight. Thus, the water-based emulsified fluid with 35% volume of the glass bubbles was prepared. Fluid properties were again recorded after 4 hours in rolling operation at 200 °F. The water-based emulsified fluid became homogenous and stable after 0.5, 1, 2, 4, 8, 12, 24 hours.

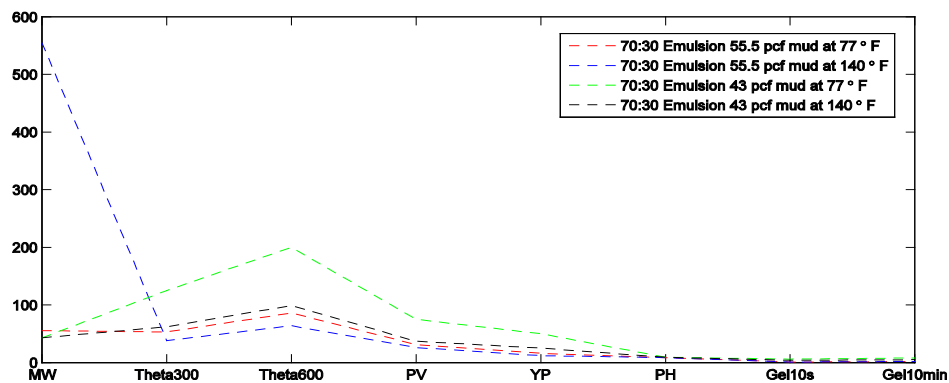


FIGURE 8. COMPARISON BETWEEN WATER-BASED EMULSION FLUIDS WITH AND WITHOUT GLASS BUBBLES AT HIGH RPM ROLLING CONDITION.

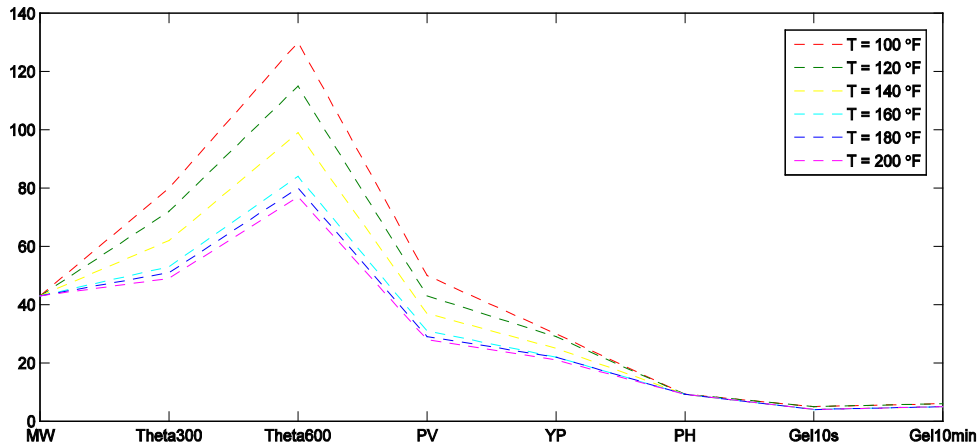


FIGURE 9. EFFECT OF TEMPERATURE ON 43 PCF 70:30 WATER-BASED EMULSION FLUIDS AT ROLLING CONDITION.

### 3 Results and Discussion

HGS is added to the drilling fluid to decrease its density. Through this process, a light weight fluid is acquired, which is still incompressible. This is a possible solution to overcome some problems in underbalance drilling such that the drilling fluid maintains many of its benefits. HGS has been utilized in many industries such as aerospace and automotive, where it is of interest to reduce the weight without influencing the strength. Moreover, HGS is used in buoyancy modules for subsea risers due to its high strength to weight ratio. In underbalance drilling sometimes it is necessary to reduce the density to a base fluid. In this situation, the specific gravity of the additive plays a key role. In addition, an inert additive having high tolerance for isostatic pressure environment is beneficial in a given challenging condition of hydrostatic pressure and temperature. The physical properties of the HGS are also desirable.

Figure 10 compares two 70:30 ratio water-based emulsified fluids with density of 43 pcf, one with 40% and the other with 35% of glass bubbles. From Figure 10, it can be concluded that by increasing the concentration of glass bubbles, the interaction between particles increases, which led to higher rheological properties. In addition, the water-based emulsified 70:30 fluid with 35% volume of glass bubbles was diluted to float the glass bubble particles, the results of which are shown in Figures 11 & 12. The results of prepared fluid by using recycled glass bubbles were recorded after 4 hours rolling operation at 200 °F. The experiment showed that, about 90% of glass bubbles were recycled by floating and reused without problem for further experiments.

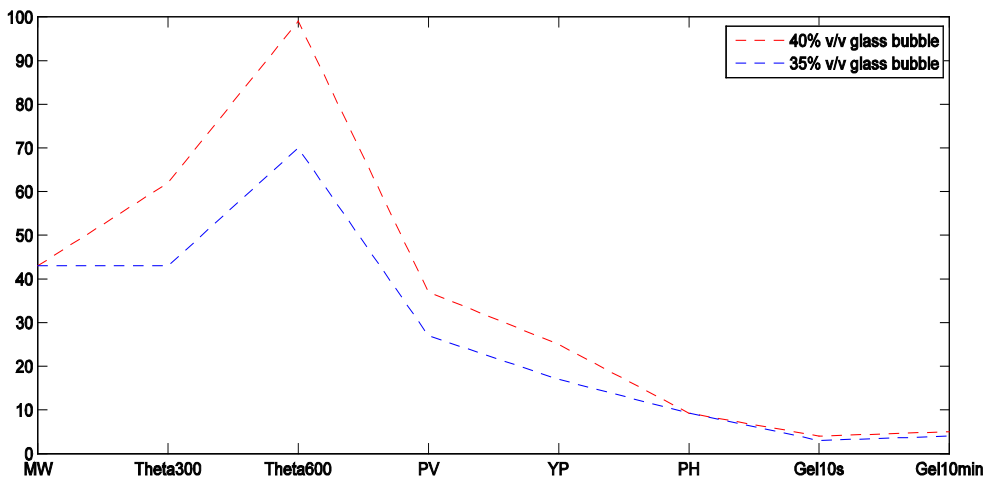


FIGURE 10. COMPARISON BETWEEN 43 PCF 70:30 WATER-BASED EMULSION FLUIDS WITH 40% V/V AND 35% V/V GLASS BUBBLE.

In order to investigate the effect of common mud additives on the 70:30 water-based emulsified mud and glass bubble agents as well as to create an appropriate emulsion fluid having 43 pcf weight, which could meet the

requirements in well No. 1 of Parsi oilfield, multiple experiments were conducted.



FIGURE 11. THE RECYCLED GLASS BUBBLES BY USING FLOATING METHOD IN THE LABORATORY.

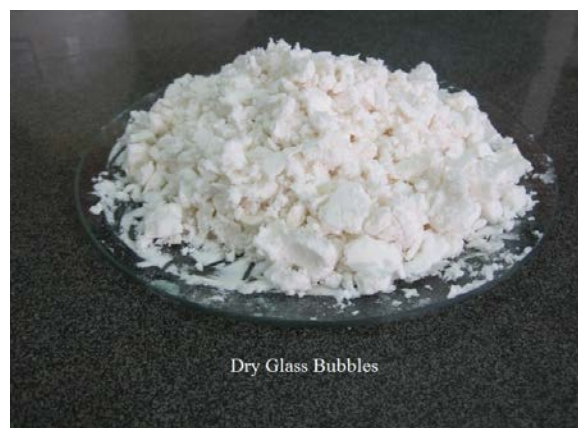


FIGURE 12. THE RECYCLED DRY SPHERES.

First by using 2 pounds of starch and 0.25 pounds xanthan gum at high temperature, two 70:30 ratio water-based emulsified fluids were prepared, one with (43 pcf) and the other without glass bubbles (55.5 pcf). Their properties were recorded after rolling operation at 140 °F. These fluids were homogenous and stable after 0.5, 1, 2, 4, 8, 12, 24 hours and the results are presented in Figure 13. This figure shows the favorable effect of 0.25 pounds of Xanthan gum on the yield point as well as gel strength in the water-based emulsified fluid. Presence of 2 pounds of starch at high temperature together with glass bubbles resulted in a decrease in fluid loss.

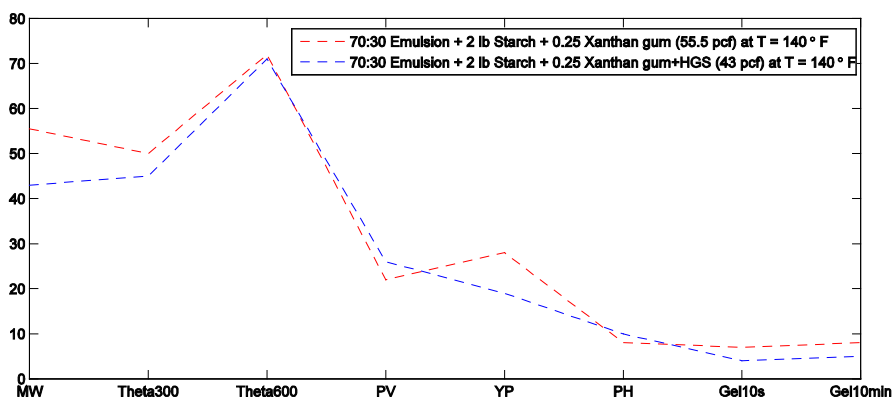


FIGURE 13. COMPARISON BETWEEN 70:30 WATER-BASED EMULSION FLUIDS WITH AND WITHOUT HGS AT PRESENCE OF STARCH AND XANTHAN GUM AT 140 °F.

Since the bentonite reveals the best fluid lost properties, it was combined with the water-based emulsified fluid. In this combination, glass bubbles were added to prepare 70:30 ratio water-based emulsified fluid with density of 43



pcf, and the results were recorded after 4 hours rolling operation at 200°F. This combination was homogenous and stable after 0.5, 1, 2, 4, 8, 12, 24 hours. Figure 14 compares the two water-based emulsified fluids (with and without glass bubbles) each containing 2 pounds of Bentonite. Water-based emulsified fluid with 55.5 pcf mud weight also contained 0.5 of PHPA (Partially Hydrolyzed Polyacrylamide), which provided shale encapsulation and a better rheology behavior. At the end, 2 pounds of bentonite plus 0.5 pounds of PHPA was added to each fluid and the fluids were evaluated. The results of this water-based emulsified fluid after 4 hours at 200 °F were recorded. This combination was homogenous and stable after 0.5, 1, 2, 4, 8, 12, 24 hours. Based on the results obtained, it was concluded to first add PHPA to prevent high rheological properties in the water-based emulsified fluid. Other additives could be added to the fluid after bentonite has been added and thoroughly mixed.

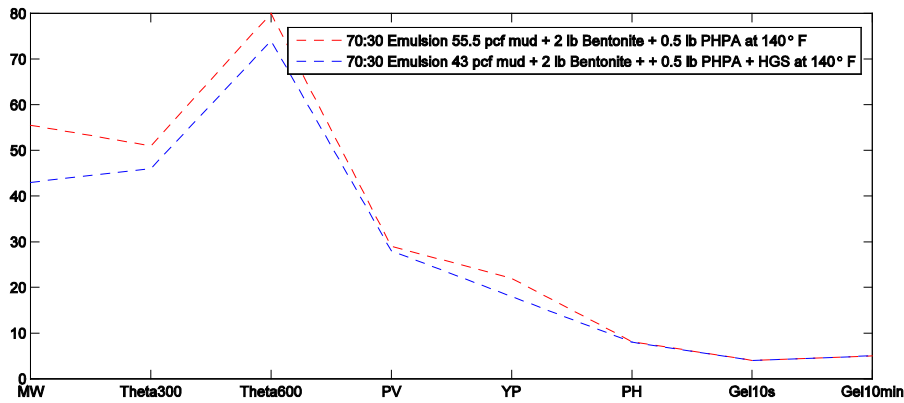


FIGURE 14. COMPARISON BETWEEN 70:30 WATER-BASED EMULSION FLUIDS WITH AND WITHOUT HGS AT PRESENCE OF BENTONITE AT 140 °F.

Since salt may cause in instability and high density problems no salt was considered for the designed fluid. The pH of this fluid was in range of 8-10 at the presence of glass bubbles favoring the stability of emulsified fluid. In water-based emulsion, the higher the percentage of oil, the higher the viscosity, and as a result, it helps the suspension of glass bubbles particles. Figure 15, recommends a general diagram for degree of mud weight reduction for different combinations of glass bubbles in water-based emulsions.

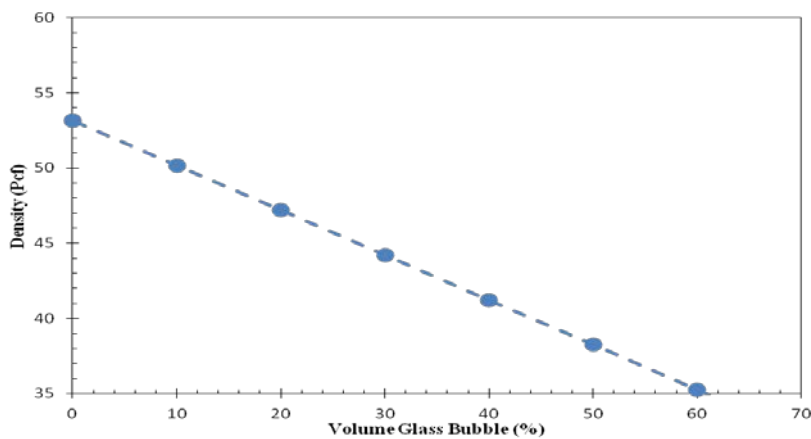


FIGURE 15. THE VOLUMETRIC PERCENTAGE OF GLASS BUBBLES ACCORDING TO THE WEIGHT OF WATER-BASED EMULSIFIED FLUID.

#### 4 Conclusion

The results of using the glass bubbles as the drilling fluid additive were investigated in this paper. Based on the obtained results from this research work, the following conclusions can be drawn:



1. The best appropriate ratio for the water-based emulsified fluid was 60:40 or 65:35 since they exhibited the best rheological properties. In this study, the ratio of 70:30 was used and the amount of 35% (v/v) of the glass bubbles in fluid was applied in order to reduce its weight to 43 pcf. As the ratios of 60:40 and 65:35 could produce high volume of tiny spheres in the fluid, these cases were not considered at this study
2. Viscosity of HGS drilling fluids increase with increased HGS and drill solid concentrations.
3. Lightweight incompressible drilling fluids can be constructed using commercially available hollow glass spheres (HGS). At sphere concentrations below 40% by volume, lightweight muds behave similarly to conventional drilling fluids
4. In an water-based emulsified fluid, the higher percentage of oil the higher viscosity of drilling fluid.
5. PHPA should be added in the final stage of fluid preparation and its appropriate concentration was 0.25-0.5 pounds per barrel. 2 pounds bentonite and 2 pounds starch per barrel should be added in the first stage at high temperature to the water for better rheological properties.

#### ACKNOWLEDGMENT

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#### NOMENCLATURE

M= Mass

Pcf= Pounds per Cubic Foot

Psia= Pounds per square inch absolute

STB= Stock Tank Barrel

V/V= Volume Percent

V= Volume

$\rho$  = Density

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