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Measuring Interfacial Tension Between Oil and Nano-Saline Solutions in the Presence of SiO_2 or TiO_2 at Low and Intermediate Pressures

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In this paper, SiO_2 and/or TiO_2 nanoparticles (NPs) were used to enhance the performance of water injection process causing an increase in oil recovery. To this end, several nano saline solutions were prepared from each of the following salt compounds: NaCl, $CaSO_4$, or $MgSO_4$ (1000 ppm) together with different concentrations of SiO_2 and/or TiO_2 NPs from 0.01 to 0.07 wt.%. Interfacial tensions (IFT) between the oil, collected from Sarvestan oil reservoir, and the saline solutions were measured at low and intermediate pressures, *i.e.* 14.5, 72.5, 145, 217.5, 290, 362.5, 435, 507.5, and 1160 Psi using a homemade IFT-device. Results showed that SiO_2 and/or TiO_2 NPs successfully reduced the interfacial tension between the oil and the saline solutions. The maximum reductions of 63.95% and 63.46% were obtained at the pressure of 507.5 Psi for 0.05 wt.% of SiO_2 in the $MgSO_4$-H_2O solution and 0.05 wt.% of TiO_2 in the $CaSO_4$-H_2O solution, respectively. The maximum IFT reductions for NaCl-H_2O-TiO_2, NaCl-H_2O-SiO_2, $MgSO_4$-H_2O-TiO_2, and $CaSO_4$-H_2O-SiO_2 solutions were 58.13%, 49.65%, 36.18%, and 20.88%, respectively.

Keywords: Interfacial tension, SiO_2 nanoparticles, TiO_2 nanoparticles, Enhanced oil recovery, Wettability

INTRODUCTION

Enhanced oil recovery (EOR) methods are generally classified into three groups: chemical, thermal, and miscible recovery methods. Chemical methods generally decrease the oil viscosity, interfacial tension (IFT), and wettability. In thermal methods, the oil recovery factor increases as a result of an increase in the oil thermal expansion. In miscible methods, miscible gases decrease the oil viscosity, and therefore, increase the oil recovery factor [1].

Nanoparticles with the ability of developing oil and gas industries can be used for several purposes including exploration, drilling, production, and recovery. Nanoparticles contribute to efficient, cheap, and eco-friendly technologies [1-3]. The idea of using nanoparticles in EOR methods has been recently introduced by several researchers [4-7]. Kong *et al.* examined nano and micro technologies for the oil and gas industries [8]. Fangda *et al.* used nanoparticles for a surfactant flooding process and searched the optimum conditions of the nano-surfactant emulsion. Results confirmed that the optimum conditions were vastly sensitive to the thermal recovery process [9]. Elkady *et al.* increased oil recovery factor up to 67.16% through nano-polymer flooding [1]. Biyouki *et al.* examined IFT reduction between the oil and water by synthesizing nickel oxide NPs using two different methods: in-situ combustion (ISC) and ex-situ combustion (ESC). In the ISC method, the nickel aqueous solution was dispersed in the heavy oil solution (prepared from asphaltene, toluene, and gasoil) in a Parr reactor unit at certain temperature and pressure. However, a simple precipitation method was implemented in the method of ESC. Additionally, NiO NPs synthesized by the ISC method tend to form more coke

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deposition compared to those synthesized by the ESC method [10]. According to Eq. (1), a decrease in interfacial tension causes an increase in the capillary number, and hence enhances the oil recovery factor [7].

$$N_c = G\frac{\nu\mu}{\delta} \quad (1)$$

where N_c, ν, μ and δ are the capillary number, Darcy velocity, viscosity of displacing fluid, and the interfacial tension, respectively.

Ranjbar *et al.* assessed the influence of γ-Al_2O_3 and MgO NPs on the surface tension and concluded that there is not a certain amount of NPs concentration, which can be considered as an optimum value to reduce surface tension [11]. Amraei *et al.* used a nanofluid including SiO_2 NPs, Span-80, Tween-80, (CTAB), (SDS), Lauric alcohol-3EO, and K-Laurate surfactants in water and subsequently examined the impact of temperature on reduction of the IFT between the oil and water. Their findings revealed that increasing the temperature led to a decrease in IFT between the oil and water [12]. Ziad *et al.* evaluated the role of morphology of aluminum oxide NPs on the reduction of oil IFT. Results showed that changing morphology improved the dispersion of NPs and consequently, increased oil recovery [13].

Although there are many research on the effect of NPs on decreasing IFT, to the best of our knowledge, our work is the first study that examines the impact of SiO_2 and/or TiO_2 NPs on IFT reduction between the oil and Asaline solutions at low and intermediate pressures. As expected, low saline solutions increase oil recovery to an acceptable level [14]. Furthermore, recent researches have shown that adding NPs to saline solutions can promote more desirabe reduction of IFT between the saline solution and oil [10,13-17]. Therefore, both low saline solutions and NPs were used in this study to reduce IFT between the oil and saline solutions. In addition, several saline solutions were first prepared from NaCl, $CaSO_4$, and $MgSO_4$ salts. Then, SiO_2 and/or TiO_2 NPs were separately dispersed into the saline solutions by a sonicating device. Finally, IFT between the oil and saline solutions was measured using a homemade IFT device under low and intermediate pressures.

MATERIALS AND INSTRUMENTS

Sodium chloride (NaCl, 99 wt.%), calcium sulfate ($CaSO_4$, 99 wt.%) and magnesium sulfate ($MgSO_4$, 99 wt.%) were provided from Sigma-Aldrich Chemical Company (Milwaukee, WI) and exploited for the preparation of saline solutions. Silica oxide chemical compound (SiO_2, particle size range of 20-30 nm, true material density of 3.4 g cm^{-3}, specific surface area between 180-600 m^2 g^{-1}, Merck Company) and titanium dioxide chemical compound (TiO_2, particle size range of 20-30 nm, true material density of 3.9 g cm^{-3}, specific surface area between 200-240 m^2 g^{-1}, Merck Company) were used as the nanoparticles. Table 1 shows the NPs characteristics. Distilled water was used as solvent to prepare the solutions. Oil sample with a density of 90 g ml^{-1} and viscosity of 39 cP was supplied from Sarvestan oil-reservoir located in south of Iran. A probe-type sonication (Tomy, UD-201, Japan) was employed to disperse NPs into the saline solutions. A Haskel pump (3L SS-41-H) was used to pressurize samples in the IFT device. The digital balance (Sartorius balance, BP 210 S, German) was measured the weight of salt samples. A digital microscopic camera (Dino-Lite Digital Microscope, Maximum magnification of 220, Taiwan) was utilized to record pictures of appeared bubbles in the IFT device.

A high-pressure and temperature IFT device (Fig. 1) at the Oil and Gas Thermodynamic Laboratory, Shiraz University was calibrated by carbon dioxide-water system and exploited to measure IFT values between Sarvestan oil and saline solutions containing NPs. A detailed explanation of the IFT device has been reported in our previous work [18].

EXPERIMENTS

Saline solution samples (1000 ppm) were prepared from each of these salt compounds: NaCl or $CaSO_4$ or $MgSO_4$. Also, it should be noted that increasing brine salinity causes the agglomeration of NPs due to reduction of the Zeta potential [19].The concentrations of brines was considered 1000 ppm in this study. SiO_2 and/or TiO_2 NPs were separately added to the saline solutions to prepare saline solutions containing NPs with the concentrations of 0.01, 0.03, 0.05, and 0.07 wt.%. The NPs were then sonicated

Table 1. The Characteristics of NPs

Nanoparticle	Particle size range (nm)	True material density ($g\ cm^{-3}$)	Specific surface area ($m^2\ g^{-1}$)
SiO_2	20-30	3.4	180-600
TiO_2	20-30	3.9	200-240

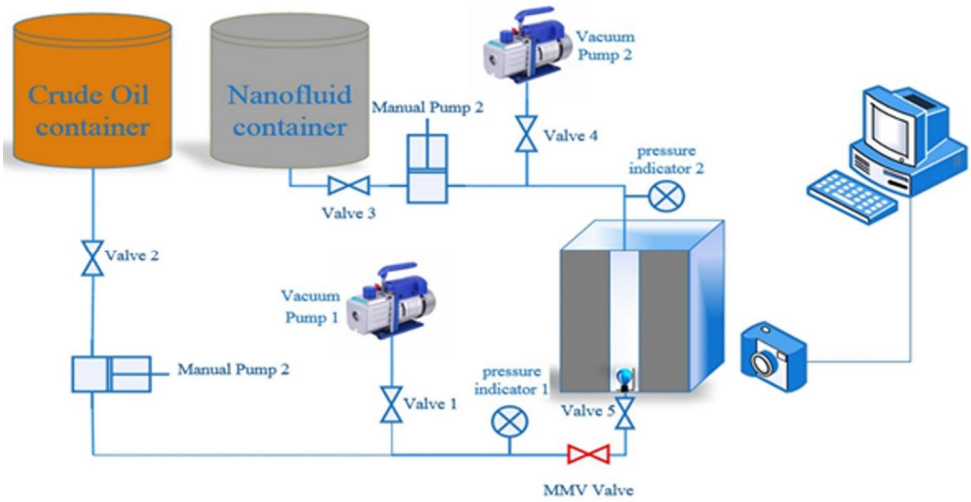

Fig. 1. Schematic diagram of the homemade high-pressure and temperature IFT device.

(100 kHz) for 20 min to adequately disperse them in the saline solution. All the tests were carried out at ambient temperature (25 °C). It is expected that surface tension decreases at higher temperatures, due to reduction of molecular interactions between NPs and rock reservoir.

The accuracy of the IFT device was first checked by carbon dioxide-water system and compared to the experimental data reported in the published literature. To do so, the container was filled with double-distilled water. The surface tension of distilled water at the temperature of 25 °C was measured at different pressures, *i.e.* 72.5, 217.5, 362.5, 507.5, and 580 psia. To this end, a gas bubble was produced at the bottom section of the device at each pressure. The camera was recorded several pictures of the bubble growing into the fluid container. The largest length and width of the bubble, which are on the verge of separation from the ostium, were recorded to calculate the correction factor needed for the IFT measurement. Figure 2 shows the experimented results of water-CO_2 surface tension obtained in this work that is compared to those

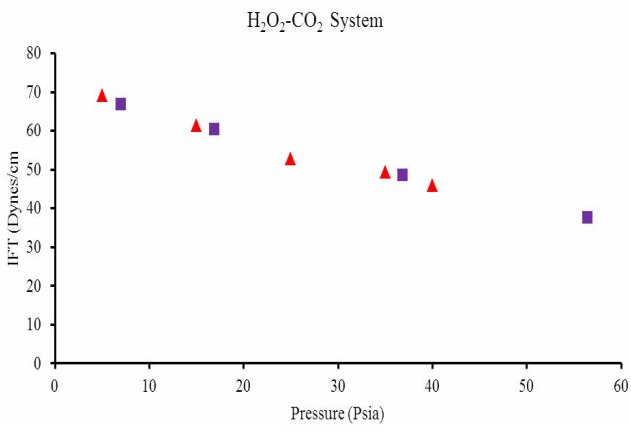

Fig. 2. Water surface tension in carbon dioxide-water system at 25 °C.

reported in the literature. As can be seen, the experimental results of this work were in line with those published in the literature with an average absolute relative deviation (AARD%) of less than 5% [20].

After calibrating the IFT device, the IFT between the oil and different nano saline solutions was measured by filling the container with a certain amount of brine. The oil was pressurized using a Haskel pump. It was then injected into the brine solution as a small drop using a tiny needle at the desired pressure. The digital camera continually recorded the images of the oil drop in the brine, as shown in Fig. 3. Finally, the container was depleted, and this method was repeated for all nano-saline solutions at various concentrations and pressures. The IFT values between the oil and brine were calculated by analyzing the prepared images.

In this study, Andreas empirical relationship [21], Eq. (2), was used to calculate oil interfacial tension.

$$\delta = \frac{g D_e^2 \Delta\rho}{H} \tag{2}$$

where δ, Δρ, D_e, and H are the interfacial tension, density difference between fluids, the highest diameter of the bubble width, and the correction factor, respectively. The correction factor, H, could be calculated from Eq. (3).

$$S = \frac{D_s}{D_e} \tag{3}$$

where D_s and D_e are the highest width and highest length of the bubble, respectively.

RESULTS AND DISCUSSION

In this study, oil interfacial tension was measured in of the presence of saline solutions (NaCl, $CaSO_4$, $MgSO_4$, 1000 ppm) containing various weight percentages of SiO_2 and/or TiO_2 NPs (0.01, 0.03, 0.05 and 0.07 wt.%) at different pressures of 15, 73, 145, 218, 290, 363, 435, 508, and 1160 Psi, and at ambient temperature (25 °C).

The obtained interfacial tensions between the oil and NaCl-H_2O solutions (1000 ppm) containing 0.01, 0.03, 0.05, and 0.07 wt.% of SiO_2 NPs and 0.01, 0.03, 0.05 of TiO_2 NPs are shown in Fig. 4 (a and b). As can be seen, the interfacial tension decreased up to 0.05 wt.% with an increase in concentration of NPs. This is due to increase of Brownian motion resulting from repulsion forces. Increasing the NPs can significantly alter wettability; this indicates that the texture and porosity of the reservoir rock can be modified and improved in the presence of NPs. On the other hand, based on the Zeta potential analysis shown in Table 2, SiO_2 nano-saline solution at the concentration of 0.07 wt.% has lower effect on increasing the IFT compared to that at concentration of 0.05 wt.%; this is due to the coagulation and agglomeration phenomena [7,22-24]. In addition, increasing the pressure caused a decrease in the interfacial tension between the oil and the NaCl-H_2O-SiO_2 solutions. The descending trend of the IFT reduction was continued even at intermediate pressures. Although the changes in IFT were negligible after 600 Psi, the results confirmed that the pressure was a vital factor on the IFT reduction. Therefore, the addition of SiO_2 and/or TiO_2 NPs into the NaCl-H_2O solutions improved the oil recovery factor, especially at intermediate pressures.

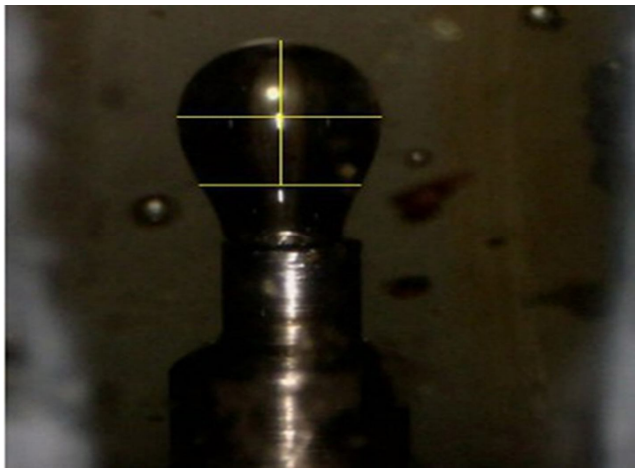

Fig. 3. An image of the oil drop in the high-pressure IFT device.

Table 2 shows the results of Zeta potential analysis after the end of each experiment for nano-saline solutions containing 0.05-0.07 wt.% of SiO_2 NPs and 0.05 wt.% of TiO_2 NPs. To do so, a specific amount of nanofluids with different concentration of nanoparticles was separated and

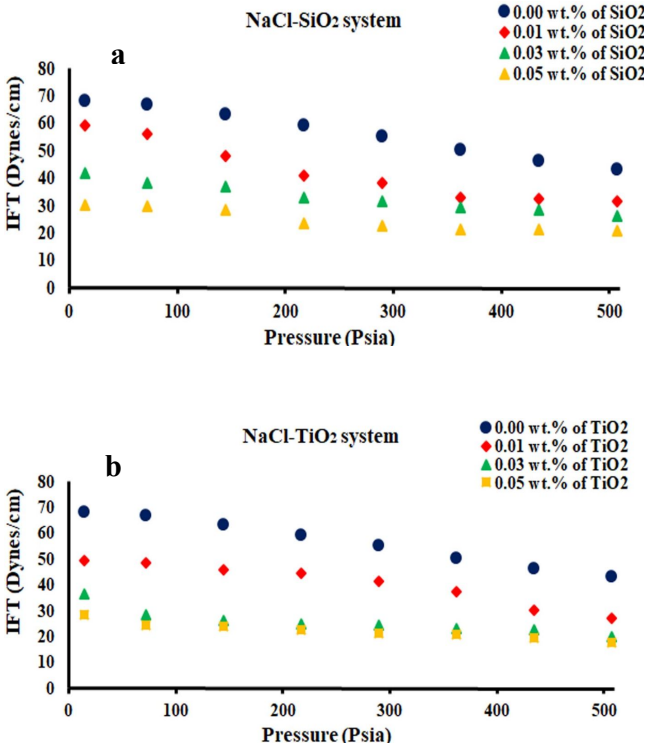

Fig. 4. Experimental interfacial tension data between the oil and NaCl-H_2O solutions (1000 ppm) containing 0.01, 0.03, and 0.05 wt.% of SiO_2 (a) or TiO_2 (b) NPs.

used for the Zeta potential analysis under ambient condition.

The stability of solutions varies with the value of Zeta potential. If the absolute value of Zeta potential is between 0-10 mV, the solution is unstable. Otherwise, the solution is semi-stable when the absolute value of Zeta potential is between 20-30 mV, and the solution is extremely stable when the absolute value of Zeta potential is between 30-60 mV [25-31]. Nano-saline solutions containing 0.05 wt.% of SiO_2 or TiO_2 NPs were stable due to the value of the Zeta potential that was higher than 30 mV. However, nano-saline solutions containing 0.07 wt.% of SiO_2 were semi-stable due to Zeta potential value that was less than 30 mV. It is worth mentioning that we could not measure the IFT of TiO_2-saline solution at 0.07 wt.%, as it was an opaque solution. For this reason, the Zeta potential of TiO_2-saline solution at 0.07 wt.%, was not measured. Therefore, the optimum concentration of SiO_2 NPs was considered 0.05 wt.%. based on the results of IFT measurements in various concentrations (0.01, 0.03, and 0.05 wt.%) and Zeta potential measurements. However, for TiO_2 NPs the optimum concentration could not be obtained; its suitable value for the IFT reduction assessed in this work was considered0.05 wt.%.

Figure 5 shows the experimental interfacial tension data between the oil and $CaSO_4$-H_2O solutions (1000 ppm) containing 0.01, 0.03, 0.05, and 0.07 wt.% of SiO_2 NPs, and 0.01, 0.03, and 0.05 wt.% of TiO_2 NPs. According to our results, the interfacial tension, as the force holding the surface of a particular phase together, decreased with an increase in pressure, and caused an increase in concentration of NPs up to 0.05 wt.% in the $CaSO_4$-H_2O solutions. These observed interfacial tensions can arise from the two opposite effects of the nanoparticles: first, the depletion of the saline solution due to the adsorption of NPs on the particles interface, which leads to an increase in interfacial tension with changes in the particle content. Second, the

Table 2. Zeta Potential of SiO_2 Saline-nanofluid and TiO_2 Saline-nanofluid at Different Concentrations after the End of each Experiment

Nano-saline Solution	Zeta potential (mV)	Nano-saline solution	Zeta potential (mV)
SiO_2-NaCl-nanofluid (0.05 wt.%)	-35.8	TiO_2-NaCl-nanofluid (0.05 wt.%)	-35.4
SiO_2-$CaSo_4$-nanofluid (0.05 wt.%)	-30.2	TiO_2-$CaSo_4$-nanofluid (0.05 wt.%)	-41.3
SiO_2-$MgSo_4$-nanofluid (0.05 wt.%)	-31.6	TiO_2-$MgSo_4$-nanofluid (0.05 wt.%)	-31.5
SiO_2-NaCl-nanofluid (0.07 wt.%)	-26.6	-	-
SiO_2-$CaSo_4$-nanofluid (0.07 wt.%)	-22.0	-	-
SiO_2-$MgSo_4$-nanofluid (0.07 wt.%)	-24.1	-	-

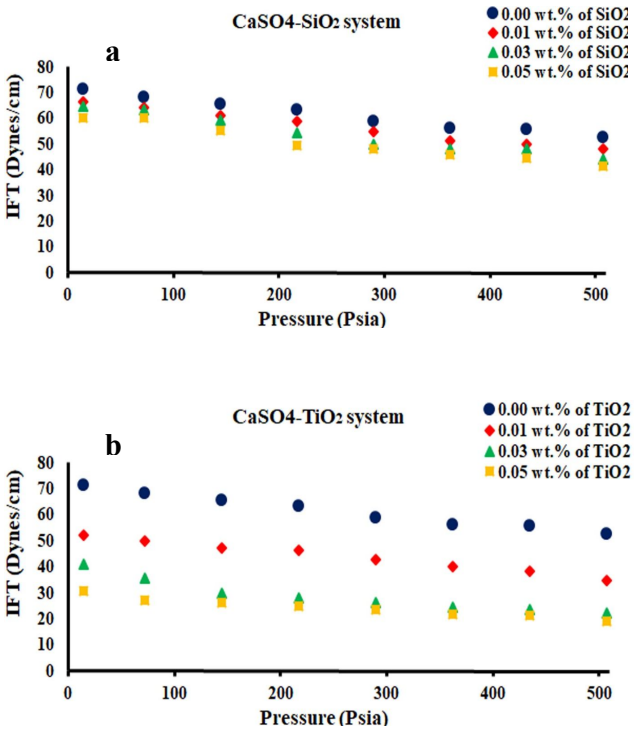

Fig. 5. Experimental interfacial tension data between the oil and $CaSO_4$-H_2O solutions (1000 ppm) containing 0.01, 0.03 and 0.05 wt.% of SiO_2 (a) or TiO_2 (b) NPs.

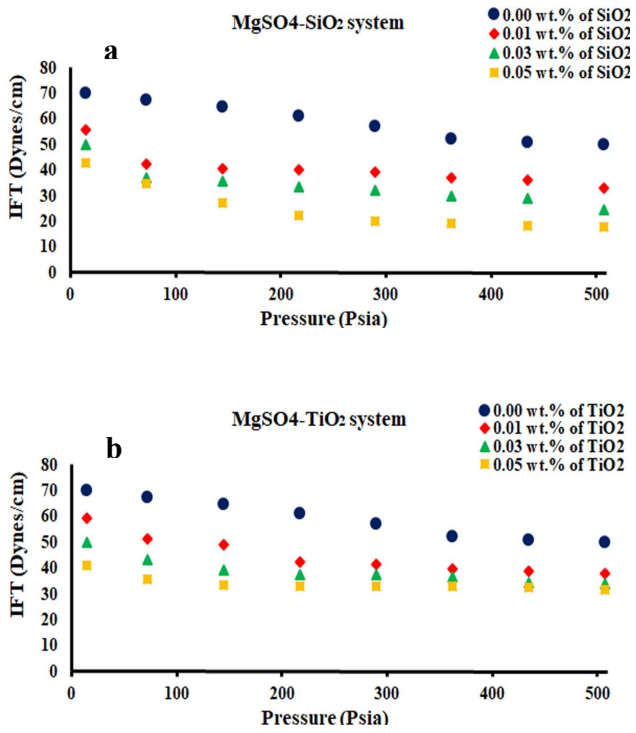

Fig. 6. Experimental interfacial tension data between the oil and $MgSO_4$-H_2O solutions (1000 ppm) containing 0.01, 0.03 and 0.05 wt.% of SiO_2 (a) or TiO_2 (b) NPs.

attachment of particles at the interfacial layers, which decreases the interfacial tension. Therefore, SiO_2 and/or attachment of particles at the interfacial layers, which decreases the interfacial tension. Therefore, SiO_2 and/or TiO_2 NPs play an important role in reducing the IFT in $CaSO_4$-H_2O solutions. Other researchers [32,33] have confirmed the reduction of IFT using NPs.

Figure 6 shows the experimental interfacial tension data between the oil and $MgSO_4$-H_2O solutions (1000 ppm) with the concentrations of 0.01, 0.03, 0.05, and 0.07 wt.% of SiO_2 NPs and 0.01, 0.03, and 0.05 wt.% of TiO_2 NPs. Results revealed that SiO_2 and/or TiO_2 NPs intensified the reduction of the oil interfacial tension. The reason for this decrease in IFT can be attributed to the increase in conductivity, the presence of active ions (charge effect) in the solution. Generally, conductivity of the nanofluids increases linearly with an increasie in concentration. In addition, rising pressure and the concentration of NPs in the $MgSO_4$-H_2O solutions can directly affect the IFT reduction. This confirms that IFT is a function of pressure and the composition of each phase. IFT reduction using NPs has been acknowledged by other researchers in $MgSO_4$-H_2O solutions [33,34].

Figure 7 compares the ability of two different NPs (SiO_2/TiO_2) in reducing the IFT between the oil and different saline solutions at different pressures and different NPs concentrations. Results illustrated that surface active materials have a measurable effect on surface tension. Besides, SiO_2 NPs are more effective in decreasing the oil interfacial tension than TiO_2 NPS. Both physical and/or chemical properties of the adsorbent affect the IFT reduction. The size of NPs plays an important role in their physical properties, as higher repulsion forces are contributed with smaller NPs size. On the other hand, the ultra-small size of NPs causes agglomeration phenomenon. In this study, the size of SiO_2 or TiO_2 NPs was considered

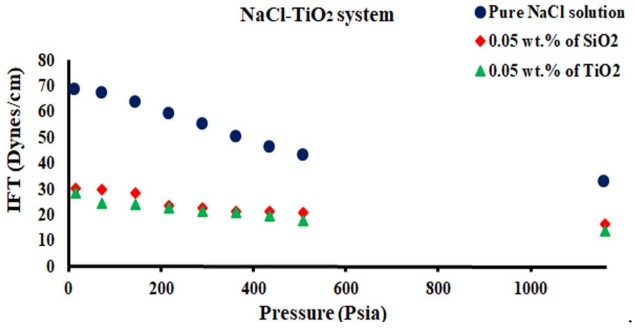

Fig. 7. Comparison results between two different NPs (SiO_2 /TiO_2) in reducing the IFT between the oil and different saline solutions at different pressures and NPs concentrations.

20-30 nm. Since adsorbents were similar in shape and size, the appeared difference between SiO_2 and TiO_2 can be attributed to chemical properties of NPs such as the available functional groups in their structures. The more important difference between Ti-O and Si-O bonds is that silicon is more electronegative than titanium, (χ Ti = 1.54, χ Si = 1.90), which causes the formation of more chemical bonds between Si and available mineral ions leading toformation of a thicker nanolayer, as nanofilm, on the solid surface. However, immethodical increase in the NPs concentration can decrease the porosity and permeability of the reservoir rock because of NPs' agglomeration and high NPs deposition on the rock surface [36].

In this study, the effect of 21 different nano saline solutions on the oil IFT was examined. In all experiments, the IFT reduction in the presence of the NPs was more than that in the absence of the NPs. Although all of the low salinity solutions could reduce IFT, the effect of divalent cations was more than that of monovalent ones. Increasing the pressure and NPs concentration had desirable effects on the oil IFT reduction. SiO_2 NPs was more efficient than TiO_2 NPs in reducing oil IFT. The Zeta potential results indicated that TiO_2-saline solutions were stable up to 0.05 wt.%, and also confirmed that the optimum NPs concentration for SiO_2-saline solutions assessed in this work was 0.05 wt.%.

CONCLUSIONS

In this study, several saline solutions (NaCl; $CaSO_4$; and/or the $MgSO_4$) containing SiO_2 and/or TiO_2 NPs were prepared. The interfacial tension was measured between the oil and the saline solutions containing NPs with the concentration range from 0.01 to 0.07 wt.% for SiO_2 and from 0.01 to 0.05 for TiO_2. The measurmnets were performed at low and intermediate pressures using a homemade high-pressure IFT device. Results revealed that the addition of 0.05 wt.% of the SiO_2 or TiO_2 NPs to the saline solution decreased the oil interfacial tension by a factor of 63.95% and 63.46%, respectively, and therefore increased the oil recovery factor. Additionally, an increase in pressure and NPs concentration caused a decrease in the oil IFT.

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