

## Article

# Effects of Surfactant and Hydrophobic Nanoparticles on the Crude Oil-Water Interfacial Tension

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**Abstract:** Surfactants and nanoparticles play crucial roles in controlling the oil-water interfacial phenomenon. The natural oil-wet mineral nanoparticles that exist in crude oil could remarkably affect water-oil interfacial characteristics. Most of recent studies focus on the effect of hydrophilic nanoparticles dispersed in water on the oil-water interfacial phenomenon for the nanoparticle enhanced oil recovery. However, studies of the impact of the oil-wet nanoparticles existed in crude oil on interfacial behaviour are rare. In this study, the impacts of Span 80 surfactant and hydrophobic SiO<sub>2</sub> nanoparticles on the crude oil-water interfacial characteristics were studied by measuring the dynamic and equilibrium crude oil-water interfacial tensions. The results show the existence of nanoparticles leading to higher crude oil-water interfacial tensions than those without nanoparticles at low surfactant concentrations below 2000 ppm. At a Span 80 surfactant concentration of 1000 ppm, the increase of interfacial tension caused by nanoparticles is largest, which is around 8.6 mN/m. For high Span 80 surfactant concentrations, the less significant impact of nanoparticles on the crude oil-water interfacial tension is obtained. The effect of nanoparticle concentration on the crude oil-water interfacial tension was also investigated in the existence of surfactant. The data indicates the less significant influence of nanoparticles on the crude oil-water interfacial tension at high nanoparticle concentration in the presence of Span 80 surfactant. This study confirms the influences of nanoparticle-surfactant interaction and competitive surfactant molecule adsorption on the nanoparticles surfaces and the crude oil-water interface.

**Keywords:** nanoparticle; surfactant; interfacial tension; crude oil; hydrophobic



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## 1. Introduction

The surface-active substances that exist in crude oil-water systems, such as chemical surfactants and nanoparticles, could contribute to generating a stable emulsion [1–3]. The massive ‘small droplets’ in water-oil emulsion lead to the high viscosity of reservoir fluids [4] and cause the problem of water-oil separation [5]. The development of exploitation of many oil fields has recently entered the second and tertiary recoveries. The water injection in the crude oil has been further increased during the second recovery. The presence of surfactant in chemical flooding in the tertiary recovery and the natural mineral particle or the addition of nanoparticles causes severe emulsion formation problems. Nanoparticles combined with surfactants have been recently used and investigated in emulsification [6] and enhanced oil recovery processes [7–12].

It is well known that emulsions could be stabilized by particles called Pickering emulsions [13]. A wide range of particles was investigated for emulsion stability [14–17]. Recently, the surfactant-nanoparticle combined impact on emulsion stability has been highlighted [18–20]. In order to reveal the impacts of surfactants and nanoparticles on the emulsion properties, the roles of surfactants and particles at the oil-water interface should

be fully understood. The interfacial tension of the oil-water system could be regarded as the one of the crucial factors affecting emulsion stability. Powell and Chauhan studied the impact of the adsorbed carbon black particles on water-oil interfacial behavior and measured the dynamic water-oil interfacial tensions using the pendant drop method [21]. Soleimani et al. reported the influence of ZnO nanoparticles on the oil-water interfacial tension and oil recovery factor, and obtained the optimum concentration of ZnO nanofluid for the decrease of water-oil interfacial tension [22]. Panahpoori et al. measured the water-oil interfacial tensions in the system containing hexadecyl trimethyl ammonium bromide (CTAB) and TiO<sub>2</sub> nanoparticles [23]. They found that higher concentrations of nanoparticles contributed the higher interfacial tension without surfactant [23]. The nanoparticle concentration increase lead to a primary increase and a subsequent reduction of the water-oil interfacial tension when the surfactant existed [23]. Rezaei et al. revealed the impacts of alkali and nanoparticles on the water-oil interfacial tension and wetting alteration in the system containing surfactant [24]. Pichot et al. found different effects of silica hydrophilic particles on the vegetable oil-water interfacial tension with the addition of W/O and O/W surfactants [25]. Biswal et al. investigated the silica nanoparticle effect on the n-hexane-water interfacial tensions, considering the influences of four types of surfactants, namely, SDS, CTAB, Tween 20 and TX-100 [26]. They found the decrease of water-oil interfacial tension caused by the silica nanoparticles in the presence of ionic surfactants, but the water-oil interfacial tension was increased in the nonionic surfactants-silica particle system. They also confirmed the non-impact of sole silica nanoparticles on the decrease of interfacial tension. Zhang et al. presented that the water-decane interfacial tension was reduced by the increase of the nanoparticle hydrophobicity [27]. Emadi et al. studied the nano-surfactant flooding by evaluating the effect of nano silica particles on the kerosene-surfactant aqueous solution interfacial tension [28]. Nesterenko et al. studied the combined influence of surfactant emulsifier and particles on oil-water emulsion stability [29]. Through interfacial tension measurements, the strong interactions between particles and surfactant molecules were confirmed [29]. Saien and Fadaei measured the water-kerosene interfacial tension, considering the impacts of silica nanoparticles with various sizes and CATB surfactant [30]. Jafarnejhad et al. investigated the nanofluids on the oil recovery, and found the reduction of interfacial tension caused by the SnO<sub>2</sub> nanoparticles suspended in brine [31].

The nanoparticles used in the most of studies are water-wet and dispersed in the water phase for enhanced oil recovery (EOR) processes. However, the influence of oil-wet nanoparticles existing in crude oil on interfacial behavior has been often neglected, such as clay particles in crude oil. Natural clay particles existing in crude oil in the presence of surfactants has an impact on the interfacial characteristics [32]. Hong et al. highlighted the significance of particles existing in the oil on the stabilization of a complex emulsion system, and performed an interfacial rheological work on the effect of hydrophobic clay particles on the stability of emulsion in the presence of surfactant [6].

Additionally, the oil used in most of the relevant studies on the effects of surfactants and nanoparticles is 'pure' oil such as alkanes [20,21,26,27], vegetable oil [25], kerosene [28,30], and paraffin oil [19,29]. Comparatively, the studies on crude oil-water interfacial tension are less sufficient. Nanoparticles could be commonly found in the recovered crude oil due to the clay existing in the reservoir and the nanoparticle EOR processes. They play a significant role on the water-oil interfacial phenomena and stabilization of emulsion [6,25,33]. Since the nanoparticles are usually aged in crude oil for long time, most of the nanoparticles could be oil-wet. In addition, owing to the presence of natural surfactant existing in crude oil and the added surfactant from EOR processes, the combined influence of nanoparticles and surfactants on the oil-water interfacial behaviors should be studied. Hence, the impacts of hydrophobic nanoparticles and surfactants on the crude oil-water interfacial tension are examined in this study.

## 2. Materials and Methods

### 2.1. Chemicals

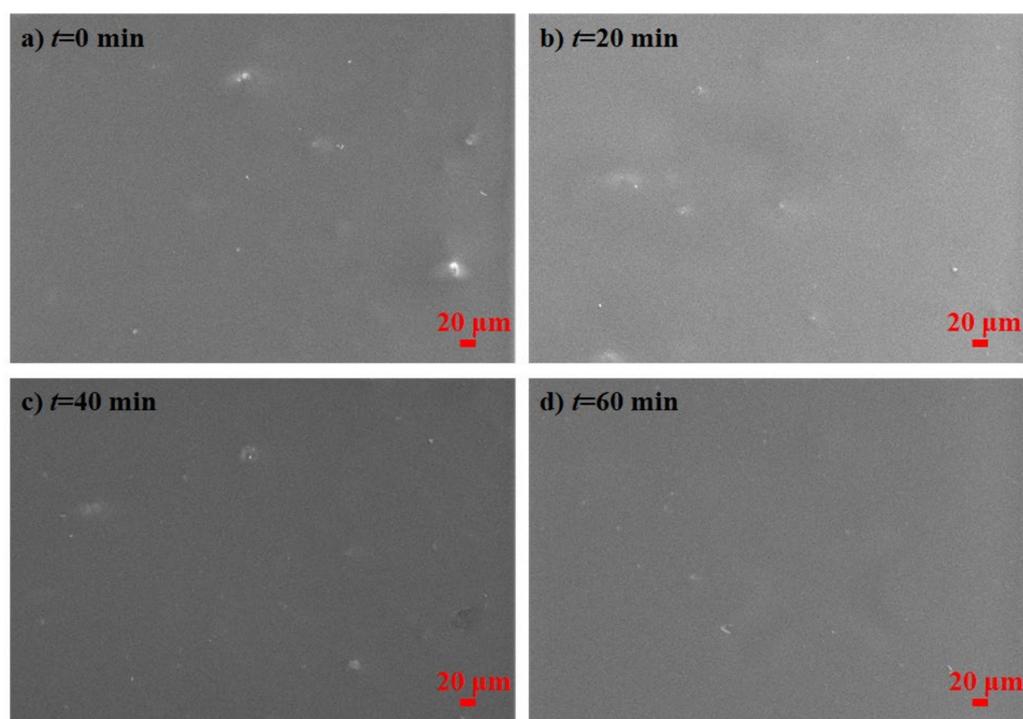
The crude oil used in this work is Xinjiang heavy crude oil (density: 0.9343 g/cm<sup>3</sup> at 25 °C). Table 1 indicates the saturates, aromatics, resins, asphaltenes (SARA), and analysis of crude oil. Table 2 shows the chemical element analysis of crude oil. Deionized water was used as the water phase. The nanoparticles used are hydrophobic SiO<sub>2</sub> nanoparticles (Shanghai Yuanjiang Chemical Co., Ltd., China, nanoparticle size: 30 nm). The SiO<sub>2</sub> content of nanoparticles is 98%. The hydrophobic SiO<sub>2</sub> nanoparticles were made by the modification of hydrophilic SiO<sub>2</sub> nanoparticles with a silane coupling agent. The nanoparticles were first added to toluene to make a uniform suspension. Silane coupling agent was then added to the suspension for ultrasonic mixing and constant-temperature reaction. The modified SiO<sub>2</sub> nanoparticles were finally obtained by vacuum drying. The nanoparticles are not easy to disperse in the oil because the heavy crude oil is highly viscous. Thus, SiO<sub>2</sub> nanoparticles were gradually added and continually stirred under a stirring rate of 2000 r/min until the white SiO<sub>2</sub> nanoparticles entirely disappeared. The dispersion system was then ultrasonically stirred for 30 min by an ultrasonicator (Zhongkeqili Technology Co., Ltd., China, model: ZK-300W, ultrasonic power: 0.15 kW, ultrasonic frequency: 40 kHz) and continuously stirred for 1 h. Span 80 (Tianjin, Guangfu Fine Chemical Research Institute, China) was used as the surfactant. In this work, the concentrations of Span 80 were used are from 200 ppm to 20,000 ppm. a scanning electron microscope (SEM) (Hitachi, SU3500, Japan) was used to characterize the nature of the emulsified nanoparticles in crude oil. Figure 1 shows the SEM images of the emulsified nanoparticles in crude oil at the 10,000 ppm nanoparticle and the 2000 ppm Span 80 at different times. As shown in Figure 1, the nanoparticle distributions were similar in crude oil at different times, and no significant nanoparticle aggregation and deposition were observed. This indicates the high stability of the nanoparticle suspension.

**Table 1.** SARA analysis of crude oil.

Component	Mass Fraction (%)
Saturates	61.09
Aromatics	16.77
Resins	11.12
Asphaltenes	1.96

**Table 2.** Chemical element (C, H, O, N, S) analysis of crude oil.

Test Name	Mass Fraction (%)
C	83.64
H	12.51
O	3.92
N	0.71
S	0.06



**Figure 1.** SEM images of the emulsified nanoparticles in crude oil at the 10,000 ppm nanoparticle and 2000 ppm Span 80 at (a)  $t = 0$  min, (b)  $t = 20$  min, (c)  $t = 40$  min and (d)  $t = 60$  min.

## 2.2. Experimental Method

In this study, the Biolin Attension Theta was employed to measure the interfacial tensions by pendant drop method. The experimental temperature was 20 °C. The DI water was filled in the transparent sample cell as the surrounding phase. The droplet of crude oil could be precisely generated in the water phase from the U-shaped needle (outer diameter = 0.8 mm) of the microsyringe. The volume of droplet varies with the interfacial tension measurement. For instance, the droplet volumes are around 40–60  $\mu\text{L}$  at the interfacial tension of around 30 mN/m. For the measured interfacial tension of around 4 mN/m, small volumes of droplets were generated, which were around 6–8  $\mu\text{L}$ . To achieve the interfacial tension data, the shape of crude oil droplets was then captured, recorded and analyzed by the image analysis system (OneAttension, Theta, Finland). The frames of recording were 138 fps. The image resolution was  $1984 \times 1264$ .

The measurement period was set to 3600 s for the measurements of dynamic interfacial tensions. Equilibrium interfacial tension data was regarded as the average values of the dynamic interfacial tensions from the last 60 s. Each experimental run was repeated three times. The presented data was the average value.

## 3. Results and Discussion

### 3.1. Effect of Surfactant Concentration on the Crude Oil-Water Interfacial Tension

The Span 80 was used as the water-oil surfactant to study the surfactant concentration effect on the crude oil-water interfacial tension. A wide surfactant concentration range was studied, in this case from 200 ppm to 20,000 ppm. Figures 2 and 3 show the crude oil-water dynamic interfacial tensions and equilibrium interfacial tensions at various Span 80 concentrations, respectively. From Figure 2, the dynamic interfacial tension data shows that the crude oil-water interfacial tension decreases as the time increases. This results from the dynamics of Span 80 surfactant adsorption on the crude oil-water interface. The variations of dynamic interfacial tensions from  $t = 0$  s to  $t = 3600$  s are 3.4 mN/m, 4.9 mN/m, 6.9 mN/m, 7.2 mN/m, 5.7 mN/m, 1.4 mN/m and 0.8 mN/m at the surfactant concentrations of 200 ppm, 500 ppm, 1000 ppm, 2000 ppm, 5000 ppm, 10,000 ppm and 20,000 ppm, respectively. From Figure 3, it can be seen that the crude oil-water interfacial

tension remarkably decreases with the Span 80 concentration from 200 ppm to 2000 ppm, by 15.3 mN/m. Beyond 10,000 ppm Span 80 concentration, the crude oil-water interfacial tension does not decrease significantly by the increase of Span 80 concentration. Thus, the critical micellar concentration (CMC) of this system could be between 10,000 ppm and 20,000 ppm, and close to 10,000 ppm.

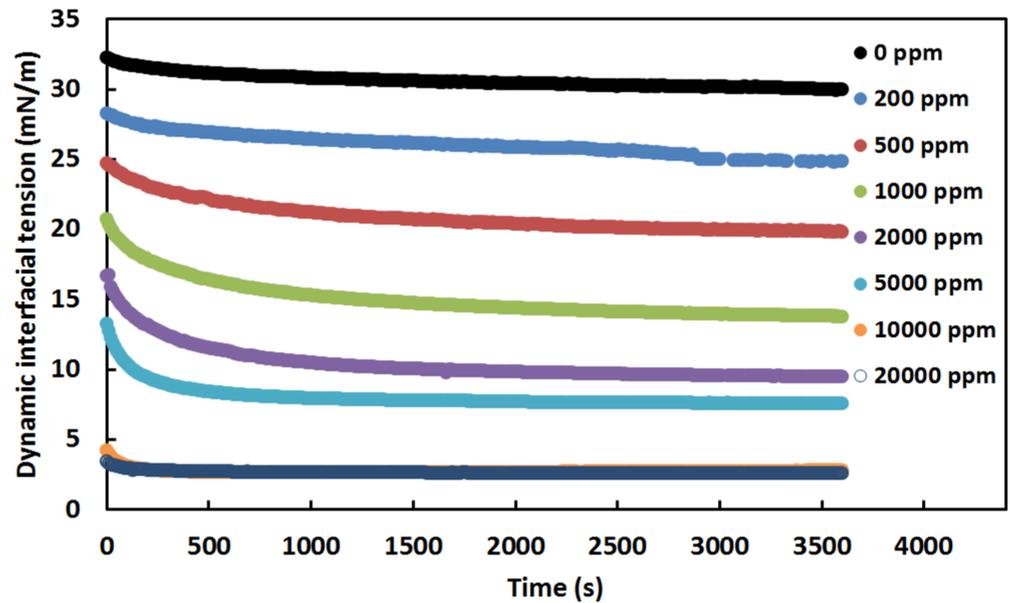


Figure 2. Dynamic crude oil-water interfacial tensions at various Span 80 concentrations.

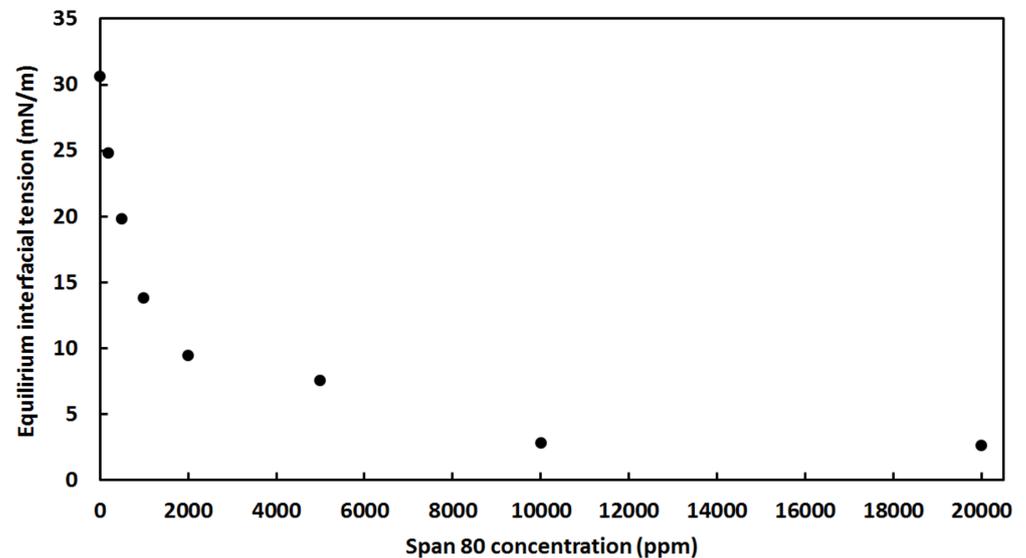


Figure 3. Equilibrium crude oil-water interfacial tensions at various Span 80 concentrations.

### 3.2. Effect of Surfactant Concentration on the Crude Oil-Water Interfacial Tension in the Presence of Nanoparticles

Since nanoparticles exist in crude oil-water mixtures in the enhanced oil recovery, it is essential to investigate the influence of surfactant on the interfacial tension of crude oil-water systems containing nanoparticles in order to reveal the combined effect of surfactants and nanoparticles on the oil-water interfacial phenomenon. Herein, different Span 80 concentrations of surfactant, which were 200 ppm, 500 ppm, 1000 ppm, 2000 ppm, 5000 ppm, 10,000 ppm and 20,000 ppm were used to investigate the influence of Span 80 concentration

on the crude oil-water interfacial tension at 10,000 ppm hydrophobic nanoparticle. Figure 4 shows the dynamic crude oil-water interfacial tensions at various Span 80 concentrations in the presence of nanoparticles. The overall trends are similar to those without nanoparticles. The crude oil-water interfacial tension is reduced as time increases, which reaches the equilibrium state in the end. However, compared with the data in Figure 2, it can be clearly seen that the dynamic crude oil-water interfacial tensions by the addition of nanoparticles (Figure 4) are quite different from those in the absence of nanoparticles. The presence of nanoparticles increases the magnitude of dynamic crude oil-water interfacial tensions and possibly changes the CMC value.

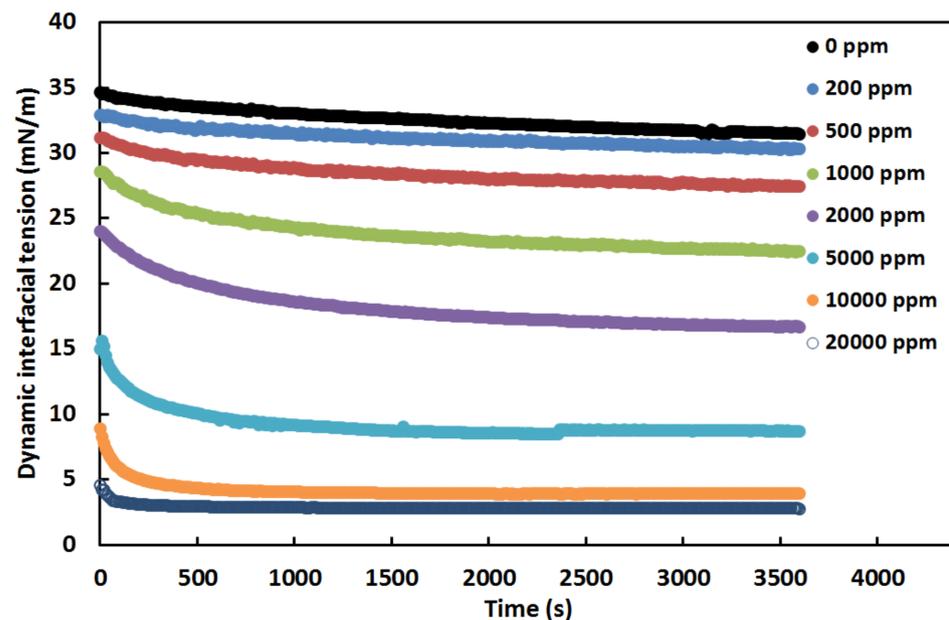
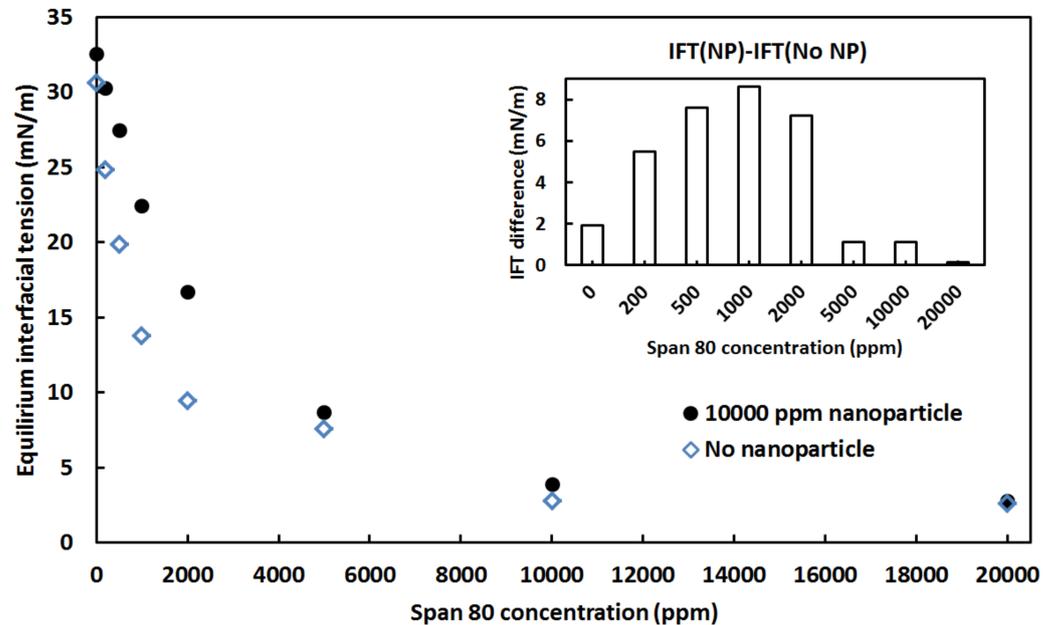


Figure 4. Dynamic crude oil-water interfacial tensions in the presence of nanoparticles at different Span 80 concentrations.

Figure 5 indicates the equilibrium crude oil-water interfacial tensions at different surfactant concentrations in the presence of nanoparticles compared with those in the absence of nanoparticles. The results indicate that the crude oil-water interfacial tensions at the surfactant concentrations from 200 ppm to 10,000 ppm in the presence of nanoparticles are obviously larger than those without nanoparticles. This could result from the steric hindrance created from the nanoparticles by preferential adsorption with the polar component in crude oil, such as asphaltenes [34,35]. This could be also caused by the surfactant adsorption on the nanoparticles owing to the large specific surface area of nanoparticles [36]. This causes the surfactant concentration reduction at the crude oil-water interface [29], and finally increases the interfacial tensions compared with the ones from pure surfactant system. The interfacial tension (IFT) differences are also indicated in Figure 5. The interfacial tension difference is related to the concentration of surfactant. When the Span 80 concentration is 200 ppm, the crude oil-water interfacial tension is 30.3 mN/m in the presence of nanoparticles, which is larger than the one without nanoparticles, by around 5.5 mN/m. The interfacial tension difference becomes more remarkable as the Span 80 concentration increases. At 1000 ppm surfactant concentration, the interfacial tension difference is the most significant. The crude oil-water interfacial tension with nanoparticles is larger than the one without nanoparticles by approximately 8.6 mN/m. This could confirm the strong interaction between surfactant and nanoparticle. These results indicate the surfactant molecule adsorption on the nanoparticles with large specific area [29,36]. This further leads to the reduction of surfactant concentration on the water-oil interface and in the bulk phase. This might also indicate the adsorption of nanoparticles on the crude oil-water interface, reducing the coverage of surfactant molecules and thereby contributing

to the higher interfacial tension. This has also been presented in other literature [25]. In addition, the presence of nanoparticles could also influence the adsorption behavior of the natural amphiphiles in crude oil. The nanoparticle reduces the surfactant efficiency at the relatively low surfactant concentrations.



**Figure 5.** Equilibrium crude oil-water interfacial tensions in the presence of nanoparticles at different Span 80 concentrations and interfacial tension (IFT) difference between the systems with nanoparticles and without nanoparticles.

During this surfactant concentration range from 200 ppm to 1000 ppm, the nanoparticles adsorbed at the nanoparticle surfaces lead to the reduction of surfactant-covered interfacial area. Beyond the surfactant concentration of 1000 ppm, the interfacial tension difference turns out to be less significant. When the surfactant concentration reaches 20,000 ppm, the interfacial tension difference is almost diminished due to surplus amount of surfactant. The quick surfactant molecule adsorption at the interface resulted from the high surfactant concentration weakens the nanoparticle adsorption. In other words, the sufficient surfactant molecules could diminish the nanoparticle impact on the oil-water interfacial characteristics. The results also might indicate that the critical micellar concentration (CMC) with nanoparticles could be higher than the one without nanoparticles. This finding is similar to the one from the literature [29]. Nesterenko et al. presented the lower CMC value in the sole surfactant system compared with the one with silica nanoparticles [29]. These could be caused by the interaction between nanoparticles and surfactant molecules, and competitive surfactant adsorption on the nanoparticle surfaces and the oil-water interface at low surfactant concentration [29]. This study focuses on the effect of hydrophobic nanoparticles dispersed in crude oil on the crude oil-water interfacial tension in the presence of surfactant. The obtained results might be inconsistent from the ones in the studies that used hydrophilic nanoparticles dispersed in the water. Pichot et al. reported that the hydrophilic silica nanoparticles have no impact on the oil-water interfacial tension in the presence of W/O surfactant [25]. The oil-water interfacial tension could be affected by nanoparticles combined with O/W surfactant [25]. Moghadam and Azizian dispersed ZnO nanoparticles in water with CTAB, and found that the synergistic influence of nanoparticle and surfactant lead to oil-water interfacial tension reduction due to the adsorption of surfactant and nanoparticles on the interface [37].

### 3.3. Effect of Nanoparticle Concentration on the Crude Oil-Water Interfacial Tension in the Presence of Surfactant

In this section, the impact of nanoparticle concentration on the crude oil-water interfacial tension in the presence of surfactant is revealed. The surfactant concentration of 1000 ppm is used, since the nanoparticle-surfactant interaction phenomenon would be significant at this concentration. The significant nanoparticle adsorption could not be diminished. The dynamic and equilibrium crude oil-water interfacial tensions at the nanoparticle concentrations of 100 ppm, 1000 ppm, 2000 ppm and 4000 ppm were measured at the 1000 ppm Span 80 surfactant (Figures 6 and 7). As shown in Figure 6, the trends of dynamic crude oil-water interfacial tensions with nanoparticles are similar to the one without nanoparticles. However, the crude oil-water interfacial tensions with nanoparticles are larger than the one without nanoparticles. More importantly, the reduction rate of dynamic interfacial tension with nanoparticles is lower than the one without nanoparticles. In addition, the lower reduction rate is measured at the higher concentration of nanoparticles. This suggests a nanoparticle effect on the dynamic adsorption behavior on the crude oil-water interface. Figure 7 shows that the influence of nanoparticles on the crude oil-water interfacial tension is insignificant at high nanoparticle concentration in the presence of surfactant.

In addition to the significant effect of nanoparticles on the oil-water interfacial tension, the oil-water interfacial tension could be also affected by pH [38–40], salinity [38,41–43] and other components, such as asphaltenes and resin [39,41,42]. Akhlaghi et al. presented that the increase of pH could cause the water-oil interfacial tension reduction [38]. This impact was more remarkable when the concentration of surfactant was lower than the CMC [38]. High salinity could result in the water-oil interfacial tension reduction because of the increase of ionic strength [38]. The effects of pH, salinity and oil components on the interfacial phenomena on the water-oil system would be involved in our future work.

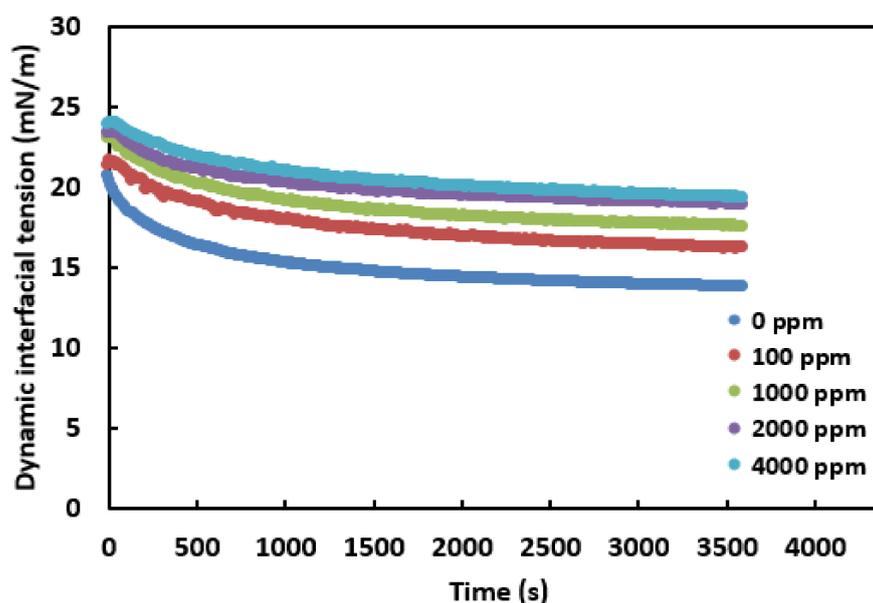


Figure 6. Dynamic crude oil-water interfacial tensions in the presence of Span 80 at different nanoparticle concentrations.

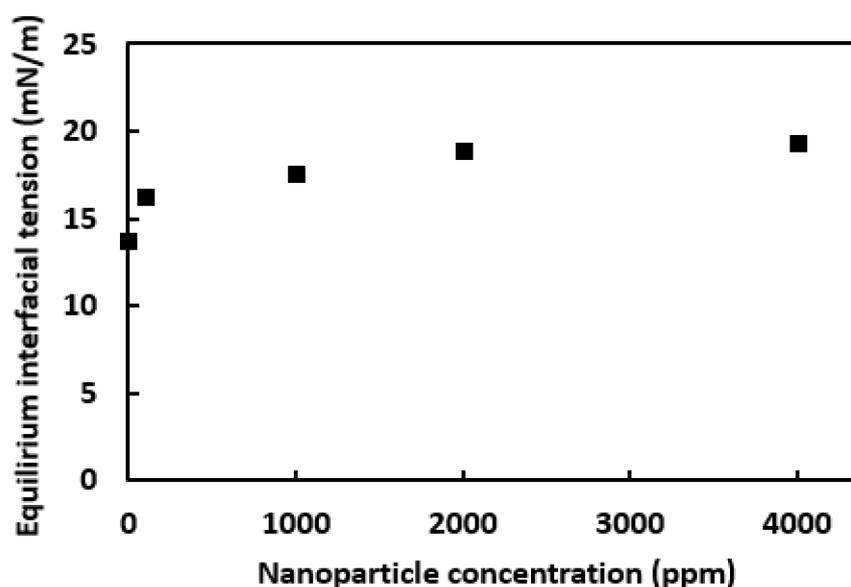


Figure 7. Equilibrium crude oil-water interfacial tensions in the presence of Span 80 at different nanoparticle concentrations.

#### 4. Conclusions

Because of the significance of the crude oil-water interfacial behavior in the presence of nanoparticle and surfactant during enhanced oil recovery and water-oil separation processes, the effects of the Span 80 surfactant and hydrophobic SiO<sub>2</sub> nanoparticles on crude oil-water interfacial tensions were investigated, specifically in terms of the impact of concentrations of Span 80 surfactant and hydrophobic SiO<sub>2</sub> nanoparticles and the surfactant-nanoparticle interactions. The results confirm a strong interaction between Span 80 surfactant and hydrophobic SiO<sub>2</sub> nanoparticles, and imply the adsorption of surfactant molecules on the nanoparticles with a large specific area leading to the surfactant concentration reduction on the oil-water interface. This thus results in the higher crude oil-water interfacial tension than the one without hydrophobic SiO<sub>2</sub> nanoparticles. The data reveals the effect of the concentration of Span 80 surfactant on the crude oil-water interfacial tension with hydrophobic SiO<sub>2</sub> nanoparticles. The effect of hydrophobic SiO<sub>2</sub> nanoparticles on the of interfacial tension is more significant at the lower Span 80 surfactant concentration. The higher Span 80 surfactant concentration could diminish this effect. The influence of hydrophobic SiO<sub>2</sub> nanoparticle concentration on the crude oil-water interfacial tension in the presence of Span 80 surfactant is studied. Results indicate that the effect of hydrophobic SiO<sub>2</sub> nanoparticles on the crude oil-water interfacial tension becomes less remarkable at higher nanoparticle concentration. This study could improve the understanding of the combined effects of surfactants and nanoparticles on the crude oil-water interfacial characteristics for enhanced oil recovery and water-oil separation processes.

**Author Contributions:** Conceptualization, X.J., X.L. and L.W.; methodology, X.J. and X.L.; validation, M.L.; formal analysis, X.L. and S.L.; investigation, M.L. and S.L.; resources, X.J., L.W. and X.G.; data curation, M.L. and S.L.; writing—original draft preparation, X.J.; writing—review and editing, X.L.; visualization, X.J., X.L. and L.W.; supervision, X.L.; project administration, X.J. and X.L.; funding acquisition, X.L. All authors have read and agreed to the published version of the manuscript.

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