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## **Nanoparticle-Stabilized Emulsions for Improved Mobility Control for Adverse-Mobility Waterflooding**

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### **Abstract**

The immense nanotechnology advances in other industries provided opportunities to rapidly develop various applications of nanoparticles in the oil and gas industry. In particular, nanoparticle has shown its capability to improve the emulsion stability by generating so-called Pickering emulsion, which is expected to improve EOR processes with better conformance control. Recent studies showed a significant synergy between nanoparticles and very low concentration of surfactant, in generating highly stable emulsions. This study's focus is to exploit the synergy's benefit in employing such emulsions for improved mobility control, especially under high-salinity conditions.

Hydrophilic silica nanoparticles were employed to quantify the synergy of nanoparticle and surfactant in oil-in-brine emulsion formation. The nanoparticle and/or the selected surfactant in aqueous phase and decane were co-injected into a sandpack column to generate oil-in-brine emulsions. Four different surfactants (cationic, nonionic, zwitterionic, and anionic) were examined, and the emulsion stability was analyzed using microscope and rheometer.

Strong and stable emulsions were successfully generated in the combinations of either cationic or nonionic surfactant with nanoparticles, while the nanoparticles and the surfactant by themselves were unable to generate stable emulsions. The synergy was most significant with the cationic surfactant, while the anionic surfactant was least effective, indicating the electrostatic interactions with surfactant and liquid/liquid interface as a decisive factor. With the zwitterionic surfactant, the synergy effect was not as great as the cationic surfactant. The synergy was greater with the nonionic surfactant than the zwitterionic surfactant, implying that the surfactant adsorption at oil-brine interface can be increased by hydrogen bonding between surfactant and nanoparticle when the electrostatic repulsion is no longer effective.

In generating highly stable emulsions for improved control for adverse-mobility waterflooding in harsh-condition reservoirs, we show a procedure to find the optimum choice of surfactant and its concentration to effectively and efficiently generate the nanoparticle-stabilized emulsion exploiting their

synergy. The findings in this study propose a way to maximize the beneficial use of nanoparticle-stabilized emulsions for EOR at minimum cost for nanoparticle and surfactant.

## Introduction

Recent studies on the nanoparticle application to oil and gas industry showed the promising results in various sectors, such as the drilling fluid that enhances shale integrity (Jung, 2013); virtually water-less fracturing fluid (Xue et al., 2015); CO<sub>2</sub> foams for CO<sub>2</sub> EOR mobility control in harsh-condition reservoir (Aroonsri et al., 2013; Singh et al., 2015; Worthen et al., 2015); enhanced reservoir imaging and sensing (Rahmani et al., 2015); and efficient contaminants removal from oilfield produced water (Ko et al., 2014; Prigiobbe et al., 2015). In particular, research on EOR processes has been steadily advanced due to the continuous development of functionalized nanoparticles which enable their application even at very high salinity and high temperature conditions (Worthen et al., 2016). Success of such EOR application highly depends on the emulsion or foam generation with long-term stability, leading to effective conformance control and improved volumetric sweep.

To enhance oil recovery from mature oil reservoirs, extensive research has been carried out on polymer flooding, surfactant/polymer flooding, CO<sub>2</sub> foam flooding, and a host of other EOR processes; and most of the techniques have been successfully applied in the field. These techniques are, however, difficult to implement at high-temperature, high-salinity reservoirs, because surfactants and/or polymers employed in the processes are usually susceptible to degradation or precipitation (ShamsiJazeyi et al., 2014). Recently, nanoparticle-stabilized foams and emulsions are being considered as an alternative mobility control option to improve volumetric sweep, because the nanoparticles are much less susceptible to the harsh reservoir conditions due to their being solid.

The immense benefits of nanoparticle-stabilized ("Pickering") emulsion have been recognized lately (Binks and Lumsdon, 2001; Melle et al., 2005; Vignati et al., 2003). Because the strong particle adsorption onto liquid-liquid interface greatly enhances the emulsion stability, the application of Pickering emulsion and foam is drawing considerable interest in the areas that long-term emulsion stability is required, such as mobility control for EOR processes. While nanoparticles act like surfactants in stabilizing emulsions and foams, once adsorbed at the interface, however, nanoparticle becomes difficult to desorb, thereby rendering the robust stability of emulsions and foams. In order to draw particles to the interface, one strategy is to add surfactants to lower interfacial tension, modify the nanoparticle surface properties change its contact angle, and increase adsorption energy. In fact, many reported the synergistic effect of adding a low concentration of surfactant to the nanoparticle dispersion in generating emulsions (Binks et al., 2007; Worthen et al., 2014) and foams (Singh and Mohanty, 2015; Zhang et al., 2008). However, most studies were done with a single combination of surfactant and nanoparticle: nanoparticle with cationic surfactant (Cui et al., 2010), nonionic surfactant (Zhang et al., 2008), and zwitterionic surfactant (Worthen et al., 2014), respectively. Therefore, a detailed comparative study employing various combinations of surfactant and nanoparticle is needed to better quantify the synergy between nanoparticle and surfactant. Also, it is necessary to examine the mechanisms for the synergy from consideration of the interactions between surfactant and nanoparticles.

In this study, an experimental setup for oil-in-brine emulsion generation was designed to assess the synergistic effect of different types of surfactant and the negatively-charged silica nanoparticles. To control the energy input for emulsion generation, instead of using sonication (Zhang et al., 2010), co-injecting through a sandpack column was adopted. Four different charges types of surfactants were selected: anionic, cationic, nonionic, and zwitterionic. In addition, the effect of pH on emulsion stability was further examined using the nanoparticles only. For clear comparison of the synergy from different types of surfactants, the nanoparticle size and concentration were kept constant for all tests. In addition, all the tests were carried out with the lowest nanoparticle and surfactant concentrations possible to

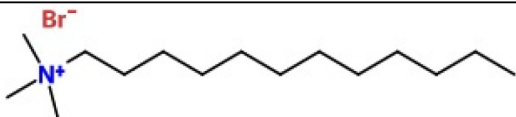
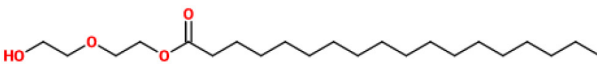
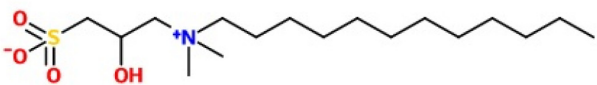
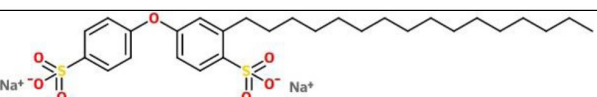
generate emulsions, aiming to determine the minimum required amount of nanoparticles and surfactants. The visual and rheological properties of emulsion were analyzed to examine the emulsion stability.

## Materials and Methods

### Materials

Negatively charged spherical silica nanoparticles were provided by Nissan Chemical (Houston, USA). In a companion paper investigating the effects of different nanoparticle sizes on the emulsion stability, it was found that the stability improved as the nanoparticle size decreased (Kim et al., 2016). Therefore, 5 nm nominal diameter silica nanoparticle which was the smallest available was selected for this study. The details of the nanoparticle characterization can be found in the previous study (Kim et al., 2015). The surfactants in this study were selected on the basis of the charge type as shown in Table 1. The decane (Fisher certified) was used as a representative oil phase, and the aqueous phase was prepared in API brine condition (8% NaCl and 2% CaCl<sub>2</sub>, Fisher Scientific). Deionized (DI) water (Barnstead E-Pure system, Thermo Scientific) with a resistivity of 18.2 MΩ-cm was used for all experiments.

Table 1—Surfactants used in this study.

Name	Synonym	Charge type	Chemical structure
Dodecyl Trimethyl Ammonium Bromide	Lauryl trimethylammonium Bromide	Cationic (+)	
<sup>a</sup> PEG-100 Stearate	Polyoxyethylene 100 Monostearate	Nonionic (no charge)	
MACKAM LHS	Lauryl Hydroxysultaine	Zwitterionic (+ and -)	
<sup>b</sup> DOWFAX 8390	Alkyldiphenyloxide Disulfonate	Anionic (-)	

<sup>a</sup>Polyethylene glycol with an approximate molecular weight of 100 attached to stearic acid

<sup>b</sup>A pair of sulfonate groups on a diphenyl oxide backbone with a linear 16-carbon group

### Experimental Set-up

Figure 1 is the schematic of experimental system. Two influents (oil phase, and nanoparticle/surfactant-containing aqueous phase) were co-injected into a sandpack column to generate oil-in-brine emulsions at room temperature and pressure. Each influent was pumped by a dual piston pump at 10 mL/min, keeping the volume ratio of oil to aqueous phase to be 1:1. Two accumulators were employed to keep oil and aqueous solution separately as well as to prevent direct contact of oil or nanoparticles with the pump. Each accumulator was filled with decane and API brine with the surfactant and/or the nanoparticles, respectively. In the cases with the nanoparticles, they were added to API brine right before the test started to minimize the aggregation of the nanoparticles during the test period (Kim et al., 2015).

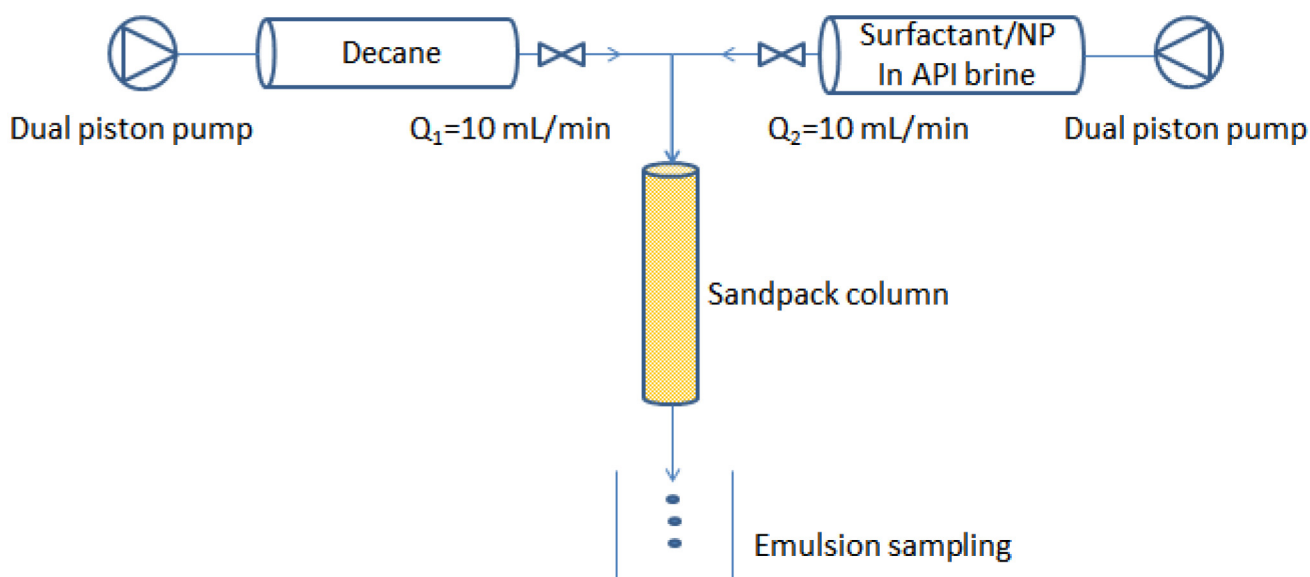


Figure 1—Schematic diagram of oil-in-brine emulsion experimental setup.

For sandpack, a high pressure column of 0.45 cm in diameter and 15.24 cm in length (HiP, Erie, PA) was packed with 350  $\mu\text{m}$  Ottawa sands. The pore volume was 1.02 mL and the porosity was 0.42. Through the effluent line open to the air, the sample was directly collected into a 40 mL sample vial. The sandpack column was flushed with the brine for 50 pore volumes followed by the co-injection with the oil phase for 100 pore volumes. Then, the effluent from the column was sampled for 10 pore volumes in a sample vial. After each test, the sandpack column was cleaned with DI water, air-dried, and refilled with a new sandpack for the next test. For the test with different pHs, the brine pH was controlled by NaOH or HNO<sub>3</sub>. Otherwise, pH was approximately neutral.

### Emulsion Stability Analysis

A series of preliminary emulsion tests was conducted to find out the lowest surfactant and nanoparticle concentrations to generate emulsion when only surfactant or nanoparticles were used. Then, the synergy between surfactant and nanoparticle was examined keeping the lowest surfactant and nanoparticle concentrations.

For the emulsion stability analysis, the emulsion in the sample vial was photographed with digital camera immediately and 10 days after the generation. In-vial photographs were also taken using a portable microscope (Supereyes, Shenzhen D&F Co.). Microscopic images of emulsion samples were also taken immediately after the generation by the microscope (Nikon Labophot-Pol) with a Nikon Digital Sight DS-Fi1 camera. A small amount of sample ( $\sim 100 \mu\text{L}$ ) was loaded on the glass slide and the microscopic images were captured by the imaging software (Nikon NIS-Elements).

### Emulsion Rheology

The rheological properties of the emulsions were measured using rheometer (ARES-LS1, TA Instruments). Strain-controlled steady rate sweep tests were performed using a double wall Couette geometry at the range of shear rates from 0.1 to 800  $\text{s}^{-1}$ . Dynamic sweep strain was carried out to investigate storage modulus and loss modulus in the strain range from 1 to 800 %. For the rheological analysis, 9 mL of emulsion sample was placed into the double wall Couette geometry. All the analysis was done at room temperature.

## Results and Discussion

### Minimum Concentrations of Nanoparticle and Surfactant for Oil-in-Brine Emulsion Formation

When the 5 nm nanoparticles were used only, the lowest nanoparticle concentration to start to generate oil-in-brine emulsions was at around 0.01 wt%. This concentration was one to two orders of magnitude lower than those used in other studies (Cui et al., 2010; Eskandar et al., 2007; Lan et al., 2007). When surfactants were used only, the lowest surfactant concentration to start to generate emulsions was as follows: 0.005% DTAB (cationic), 0.0005% MACKAM LHS (zwitterionic), 0.0005% PEG-100 Stearate (nonionic), and 0.1% DOWFAX 8390 (anionic). At the lowest surfactant concentration, most emulsions were rapidly coalesced. When the nanoparticles and surfactants were mixed to generate emulsions, significantly different stability behavior was observed depending on the surfactant types.

Comparing the emulsion layer height in the sample vial, which varies for different surfactants as shown in Figure 2, almost no synergy was observed with the anionic surfactant in emulsion quantity and stability. However, the cationic and nonionic surfactants with the nanoparticles generated significant synergy in emulsion quantity and stability. In case of the zwitterionic surfactant, immediate synergy was shown with the microscopic photographs (Figure 3), however, the emulsion layer had completely coalesced within a month. Overall, the synergy was observable except for the case of anionic surfactant. For the cases of cationic or nonionic surfactant, the emulsions were very stable for a long-term over three months. Considering that the aqueous phase was quite salty, the nanoparticles at the emulsion interface may have aggregated or self-assembled, preventing the coalescence of the droplets. Due to the high stability, the microscopic images of the strong emulsion samples were available (Figure 4) while others were unavailable due to the quick coalescence.

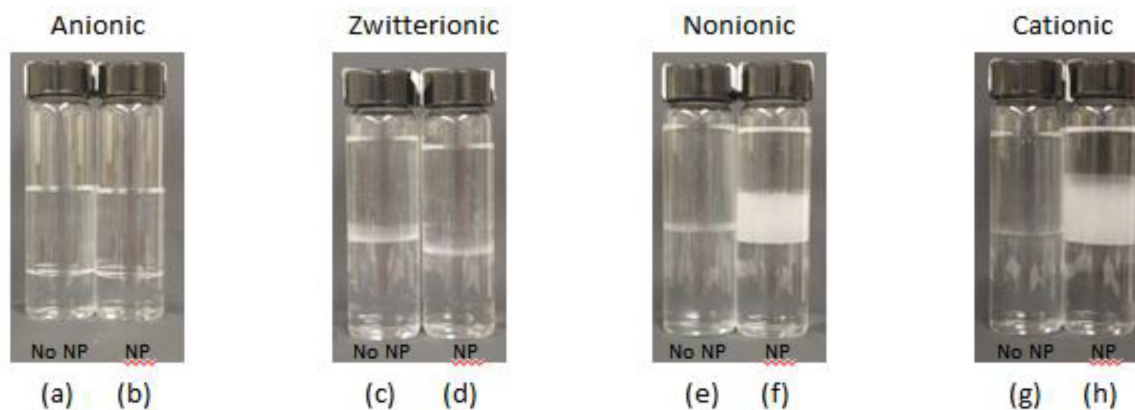


Figure 2—Photographs of the emulsion in the sample vial taken 10 days after generation: (a) and (b) 0.1% DOWFAX 8390, (c) and (d) 0.0005% MACKAM LHS, (e) and (f) 0.0005% PEG-100 Stearate, (g) and (h) 0.005% DTAB.

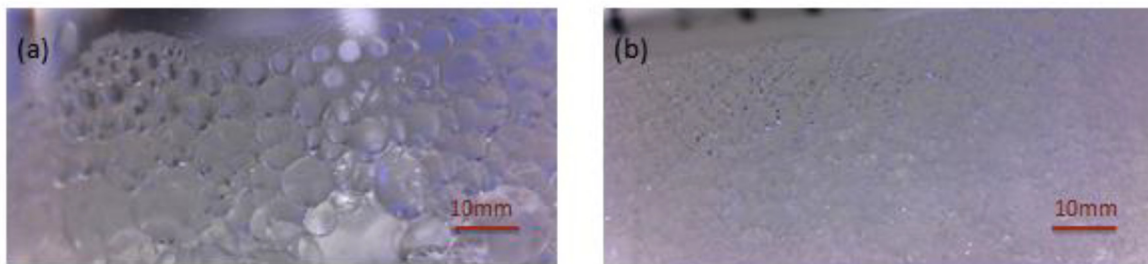


Figure 3—Microscopic photographs of the emulsions taken immediately after generation with the zwitterionic surfactant: (a) 0.0005% PEG-100 Stearate and (b) 0.0005% PEG-100 Stearate with 0.01 wt% nanoparticles.

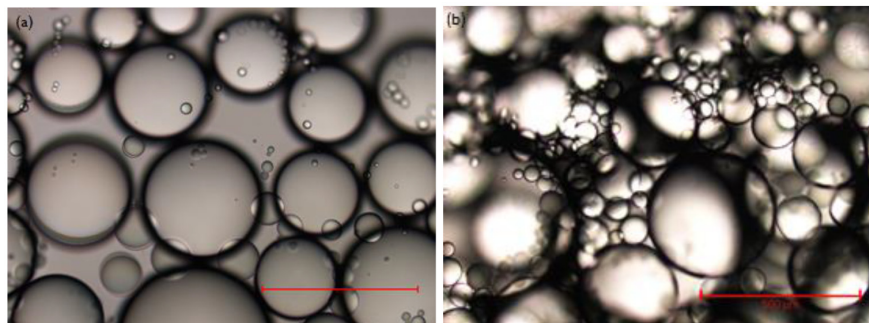


Figure 4—Microscopic images of emulsions generated with (a) the nonionic surfactant and (b) the cationic surfactant with the nanoparticles. The scale bar is 500  $\mu\text{m}$ .

### Emulsion Rheology for Comparison of Nanoparticle-Surfactant Synergy

Further analysis with the rheometer clearly showed the synergistic effect. Figure 5 compares the apparent viscosity of emulsions stabilized with the four surfactants with and without nanoparticles. When stable emulsions were formed from synergy, highly shear-thinning viscosity was observed. Not only the high viscosity at low shear rate attainable with synergy, but also the differences in viscosity between with and without nanoparticles, decreased in the following order: cationic > nonionic > zwitterionic > anionic. For example, the viscosity of the nanoparticles with cationic surfactant was three orders of magnitude higher than that of the cationic surfactant only. Storage modulus ( $G'$ ) and loss modulus ( $G''$ , not shown here) of the emulsion showed the emulsion structure is more elastic (close to solid than liquid) when the surfactant was cationic or nonionic compared to zwitterionic or anionic surfactant (Figure 6).

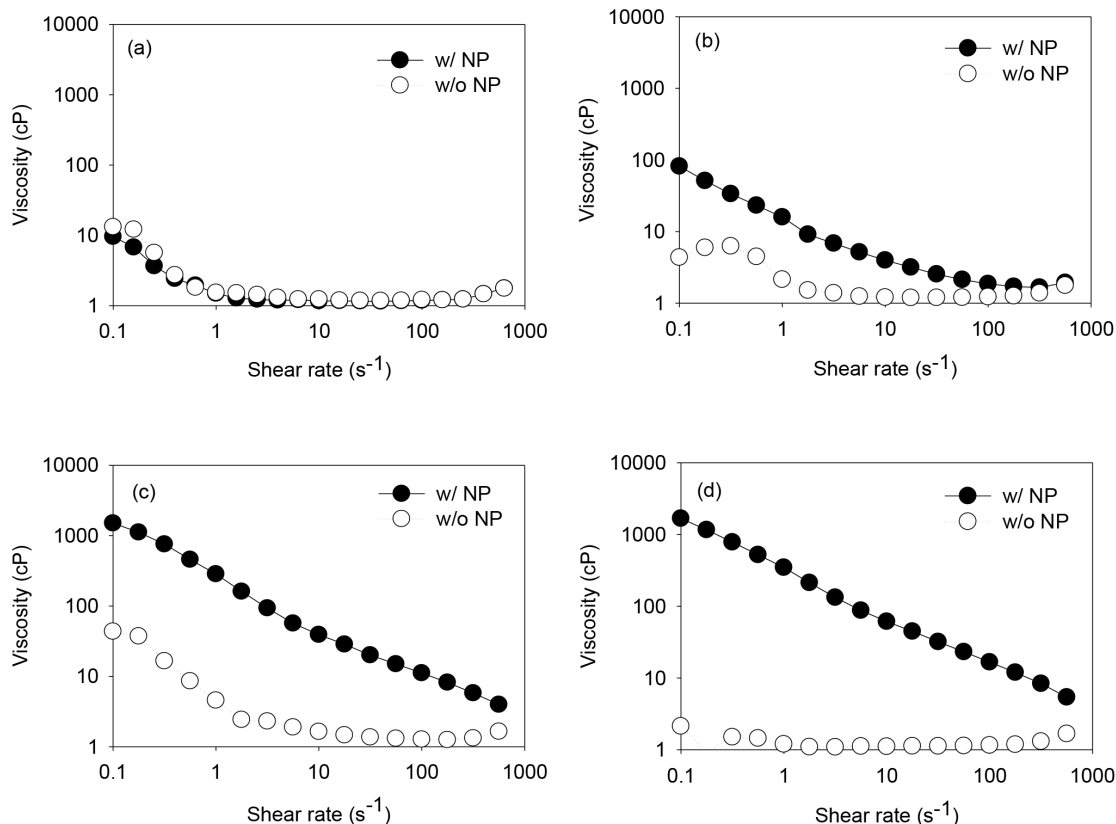


Figure 5—Effects of surfactant type and the presence of nanoparticles on emulsion viscosity; (a) anionic, (b) zwitterionic, (c) nonionic, and (d) cationic surfactant.

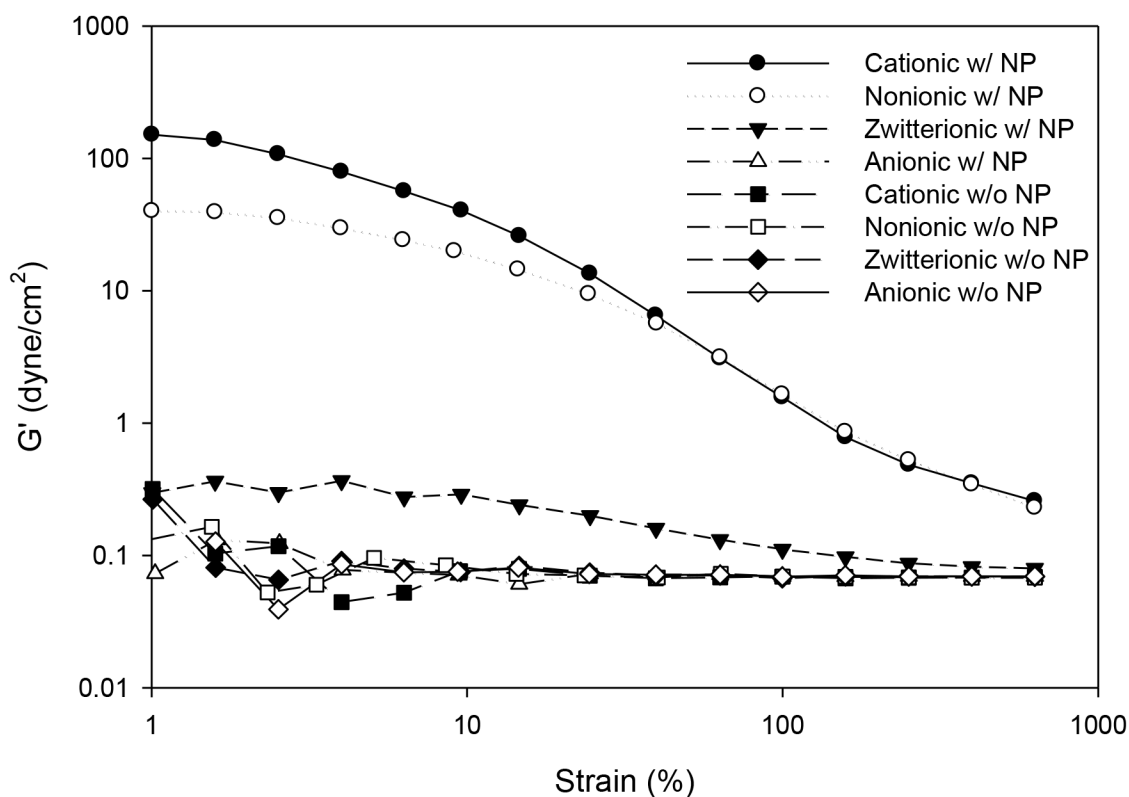


Figure 6—Effect of surfactant charge and the presence of nanoparticle on storage modulus in sweep strain test.

### Discussion on Nanoparticle-Surfactant Synergy

As briefly described in the Introduction, different mechanisms for the synergy have been proposed (Worthen et al., 2014). By employing surfactants with four different charge states with the same nanoparticle, we were able to better understand the mechanism for synergy. To begin, all four surfactants will readily adsorb at the liquid/liquid interface (even before the nanoparticles are adsorbed), lowering the interfacial tension and thereby creating more droplets/interfaces in a transient manner. (Note that the surfactant alone cannot generate stable emulsions at the low concentrations that we tested.)

First, for the case of cationic surfactant, while it is readily attracted to the negatively charged silica surface, their adsorption would expose its hydrophobic chain to the water environment. On the other hand, the nanoparticles will be more favorably attracted to the positively charged liquid/liquid interface (with the adsorbed surfactant); and once attached to the interface, the surfactant molecule will readily adsorb on the oil-exposed portion of the particle while its water-exposed portion will still have the negative charge state, allowing inter-particle stability and also the inter-droplet stability. Such will explain the robust synergy by the cationic surfactant.

At the other extreme, for the case of anionic surfactant, the electrostatic repulsion between the surfactant, the nanoparticle, and the interface will strongly discourage the adsorption of the nanoparticles to the interface which is also negatively charged with the early adsorption of the surfactant.

Similarly for the zwitterionic surfactant, even though its positive charge would be attracted to the nanoparticle's negative charge, the net consequence of its adsorption on the particle could be still the negative charge for the nanoparticle. The surfactant's adsorption at the interface will also likely reveal a negative charge state (see its molecular structure in Table 1) which will strongly discourage the adsorption of the nanoparticles to the interface.

As for the case of nonionic surfactant, because the interactions between the nanoparticles, and between nanoparticle and the adsorbed surfactant layer at the interface, will be governed by the hydrogen bonding

and molecular chain's configurational repulsion, a meaningful comparison of its behavior with other surfactants' is difficult.

Considering the surfactant loss during its transport in sandstone reservoirs whose rock surface is negatively charged, nonionic surfactant can be an alternative to the cationic surfactant, since synergy between nanoparticles and nonionic surfactant was impressive in emulsion stability. Assuming nonionic surfactant is not charged, the adsorption of nonionic surfactant on the nanoparticle surface is likely due to hydrogen bonding between oxyethylene head group of the surfactant and silanol groups of the nanoparticles (Rutland and Senden, 1993). Such adsorption could help bring the nanoparticles for adsorption to the interface, as well as lower the interfacial tension at the interface. Even if nonionic surfactant can be adsorbed to rocks, the selection of surfactant formulation with greater hydrophilic part can help to reduce the surfactant loss (Curbelo et al., 2007).

Because the electrostatic interaction is one of the key parameters controlling the adsorption of surfactant to liquid/liquid interface and to nanoparticle, as well as the adsorption of nanoparticles to the interface, the effect of pH on emulsion stability was further investigated since the amount of hydronium ions available to adsorb on surface can alter the surface charge of the oil-water interface and the nanoparticles. Another set of emulsion test using the nanoparticles only was carried out under different pHs. Figure 7 shows the effect of pH on the emulsion droplet size. The optimum pH for stable emulsion generation was appeared to be in the neutral pH range (6~8). The emulsion stability was drastically deteriorated above pH 9, showing a rapid coalescence right after emulsion generation. As pH decreases, on the other hand, the adsorption of nanoparticle to the interface becomes lower due to the charge neutralization and nanoparticle aggregation. Although slight aggregation of nanoparticles might be helpful in forming the particle network at the interface (Yang et al., 2006), the increase in aggregate size would hinder the close-packed adsorption of nanoparticles at the oil-water interface (Midmore, 1998). When pH increases, emulsions become more vulnerable to coalescence because nanoparticles would be more spread-out at the interface due to the highly negatively charged surfaces (Pichot, 2010); and as a consequence, the interfacial film becomes mobile and the emulsion stability weak.

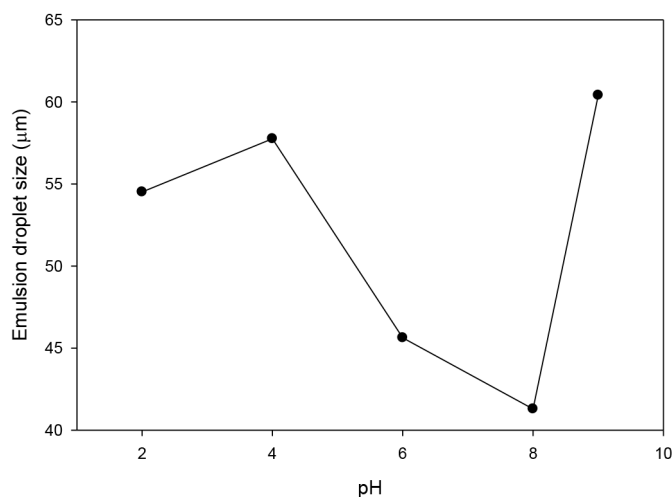


Figure 7—Effect of pH on the emulsion droplet size.

As shown in this study and by others (Griffith et al., 2016; Lotfollahi et al., 2016; Worthen et al., 2015), the benefits of using the nanoparticle-stabilized emulsions and foams for mobility control under harsh reservoir conditions are clear; however, their generation by simple injection of nanoparticle-containing water and CO<sub>2</sub> (or low-cost oil such as natural gas liquids for emulsion) at typical reservoir flow rates would be difficult because the adsorption of nanoparticles to the liquid/liquid would require lower



interfacial tension. Addition of a very small concentration of surfactant, thereby exploiting the synergy between nanoparticle and surfactant, is proven to be an effective way of spontaneously generating emulsions (foams) for the EOR application. Our study is an initial step to find the optimum surfactant and its concentration, in employing nanoparticles for mobility control for EOR from harsh-condition reservoirs.

## Conclusions

The synergy between the silica nanoparticles and four different classes of surfactants on the oil-in-water emulsion stability was experimentally evaluated. The extent of synergy was quantified based on the observation of emulsion height change, microscopic analysis of droplet size, and the emulsion rheology measurements. The synergy decreased in the following order: cationic > nonionic > zwitterionic > anionic. The addition of very small amount of surfactant to the nanoparticles promoted emulsion generation and its stability, except the anionic surfactant. While the synergistic effect was difficult to quantify with visual observations, the measurements of steady shear viscosity and elastic modulus allowed clearer distinction among different nanoparticle/surfactant systems. This result supports that the electrostatic interaction is a decisive factor to bring more nanoparticles to the oil-water interface with aid of the surfactant. Therefore, depending on the surface charges of reservoir rock, appropriate surfactant can be chosen to help bring the nanoparticles to the liquid/liquid interface. For example, for anionically charged sandstone reservoirs, the nonionic surfactant was emerged as a promising candidate in field application, showing synergy with the negatively charged silica nanoparticles.

## Acknowledgements

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