Investigating Synergistic Effects Of Surfactants And Nanoparticles On Emulsion Stability

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Abstract. A challenge of making oil production viable is emerging with time because most of the oil reserves have been exploited using primary and secondary recovery methods. Chemicals such as surfactants have been used to increase oil production through a method called chemical enhanced oil recovery. However, the application of this method is experiencing difficulties because of excessive use of surfactants that not only has negative effect on the economics of the project, but also poses severe environmental concerns. Another method that is being widely proposed is to apply emulsion flooding to enhance oil production. In order to maximize the impact of this method and as a result achieve higher oil recovery factor, it is vital to maintain the stability of the emulsion used. One of the claimed methods to improve the stability of emulsion is the application of synergistic effect between nanoparticles (NPs) and surfactants. This article aims on investigating the stability of emulsion using bottle test when applying the synergistic effect between NPs and surfactants with varying concentrations of the NPs, and surfactant charge. An anionic surfactant - sodium dodecyl sulphate (SDS) and a cationic surfactant - cetyltrimethylammonium bromide (CTAB) have been utilized in this study. Nano-silica was selected as the NPs used in this study. It was found that synergistic effect is more prominent between SDS and nano-silica with decrease in emulsion phase height percentage (measure of emulsion stability) going down to 22% compared with 43% for combination of CTAB and nano-silica, and 51% in case of using only surfactants. This has proved that the synergistic effect is beneficial in enhancing the emulsion stability, which can be implemented in the application of emulsion flooding where the stability of the emulsion is crucial.

1. Introduction

Natural methods are losing the ability to produce oil from a majority of the reservoirs that have reached maturity level. Hence, it becomes economically unviable to recover hydrocarbons by the primary drives. To maintain production at the targeted level, additional support is usually supplied such as injecting gas or water into the reservoir (namely gas/water flooding), which are known as the secondary recovery methods. Nevertheless, through a combined efforts of the primary and the secondary recovery techniques only 35 – 50 % of oil in place can only be recovered [1].

After using primary and secondary recovery methods, the role of enhanced oil recovery (EOR) comes into action to recover the oil left behind. Rather than that only providing the external energy to push the oil, through applying EOR techniques, few of the physical and/or chemical properties of either/both the oil left behind or/and the displacing fluid might be altered in favor of increasing the
production level. EOR methods are categorized according to the operating mechanism they are functioning to enhance the oil recovery. The three main types are thermal EOR, miscible EOR, and chemical EOR.

Chemical EOR is subdivided based on the chemical substances involved in the modification of the equilibrium of the reservoir. This method involves the usage of surfactants, polymers, and other chemicals such as alkali. It can also be implemented through a suitable combination of those chemicals, as in surfactant-polymer, polymer-alkaline and alkaline-surfactant-polymer (ASP) flooding mixtures [2].

Emulsion consists of mutually two insoluble and immiscible fluids, like water and oil. It is a disperse colloidal system that is thermodynamically unstable. One of the two constituents is the continuous phase that surrounds the dispersed phase that is present in the form of finely dispersed spherical droplets. Emulsions in petroleum systems is generally comprised of oil and water as the interacting fluids [3-9].

Microemulsion possesses special characteristics that enable it to be considered as a promising technology to be utilized in significant application in areas like EOR [3, 10, 11]. Due to the ultra-low interfacial tension (IFT) values that could be achieved between the contacting oil and water microphases of emulsions, water-in-oil (W/O) emulsions stabilized with NPs or surfactants could be considered as an option to look after for applying EOR in harsh condition reservoirs [2, 12, 13].

Emulsion flooding could achieve the purpose of its utilization in enhancing the oil recovery through several mechanisms. First, it can alter the rock wettability towards wettable by oil by modifying the capillary forces. Second, it has the effect of partially blocking the larger pores in the rock by the dispersed emulsion phase. Third, it can assist in the modification of the relative permeability, which results in reduction of water mobility [11]. In order for the emulsion to maintain the effectiveness of its function and fulfill the target of its usage as an EOR flooding method, it is essential to maintain the stability of the emulsion for as long as possible; as it is noted that emulsions are thermodynamically unstable systems.

Emulsions could be formulated with the mixture of oil and another phase, which can be either water or any other immiscible aqueous solution with the supply of sufficient energy by mechanical shear. In general, the small tiny droplets of the dispersed phase, formed in the emulsification process like water in W/O emulsion, tend to coalesce after some time to self-resolve the emulsion [14]. In order to reduce the mechanical energy required for the dispersion process, which is quite high, emulsifiers are used to lower the interfacial free energy and interfacial tension (IFT) [3]. Moreover, emulsifiers function as stabilizers once the dispersed phase has been formed against coalescence [7]. They are aimed to accumulate at the interface of both phases and create a protective layer in the form of tough elastic film which remains unbroken with the collision of the droplets [6, 8]. Emulsifiers can be surfactants, polymers, or even solid particles. In brief, in order for emulsion to be created, there is a need of oil, water, and emulsifier, and energy [9].

Surfactants stabilize emulsions by adsorbing at the interface and reducing the IFT, decreasing the size of the dispersed phase droplets, increasing surface elasticity, and possibly increasing surface viscosity. These effects make oil-water separation difficult and enhances their stability, which is favored in EOR application [3, 6, 15].

Emulsion system could be stabilized by adsorbing particles at the water-oil interface and are known as pickering emulsions [10, 16-20]. There are many particles that are used for stabilizing pickering emulsions such as clays, silica, etc. Droplets of the dispersed phase are hindered from coalescence through the steric hindrance via the adsorption of monolayer film of the solid at the interface [10, 20]. The uniqueness of the form of emulsion resides in the fact that the adsorption energy is relatively irreversible and the particle to particle interactions are considered strong [19]. Harsh subsurface conditions requires excellent physical and chemical stability which are possessed by the NPs. They adsorb on the fluid/fluid interface, which leads to the formulation, rheological enhancement and improvement in their stability [13].
Low IFT is usually needed for the adsorption of NPs to the liquid/liquid interface. One way to achieve that is the addition of surfactants to lower the IFT and therefore attract particles to the interface. Thus, it is advised to exploit the synergy between NPs and surfactants to lower the IFT and increase the adsorption energy. As a matter of fact, the synergistic effect of adding surfactant to the nanoparticle dispersion is considered to be an effective way of spontaneously generating emulsions for the EOR application [21]. Hence, the stability of emulsion could be improved by applying this method.

There are many methods which are used to investigate the stability of the emulsions. Among them are the phase separation methods. These methods depend on measuring the amount of phases that have been separated from the emulsion and resolved; which can be either by natural separation induced by gravity segregation or by external forcing like centrifugation. Bottle test is an example that is implementing phase separation method to investigate emulsion stability [22]. This test is widely used in the industry to measure and monitor emulsion stability [23, 24].

The amount of the dispersed phase separated from the emulsion is measured as a function of time. This can be used as a measure of emulsion stability. For example in the case of W/O emulsion, the amount of water separated is measured as a function of time. The higher the amount of dispersed phase separated from the emulsion is an indication of how loose the emulsion is (higher possibility to separate into oil and water), and smaller the “resolved” volume of dispersed phase (with respect to the original volume), the more stable the emulsion is. This is usually reported as Emulsion Separation Index (also referred to as Emulsion Stability Index and abbreviated as ESI) [22, 25]. ESI is defined as follows:

$$ESI = \frac{\sum \text{Water separation at a given time} \text{(%)} } {\sum \text{number of experiments}}$$ (1)

In some case of stable emulsions, where the separation process takes a longer time, a single experiment could also be used to indicate the stability of the emulsion.

2. Experimental
This study focused on the utilization of anionic (SDS) and cationic (CTAB) surfactant. Nano silica was selected as the NPs to implement the synergy effect. A basic schematic diagram for emulsion preparation is shown in figure 1. Energy was supplied to the emulsion system via a high speed homogenizer (figure 2). Furthermore, since stability of the emulsion is an important aspect to be studied, the main theme of this study was to investigate the stability of the emulsion through phase separation method using bottle test.

After the emulsion preparation process ended and the foam settled down, bottle test was started. The emulsion samples were transferred into test graded bottles with capacity of 25 mL each (figure 3) and preserved stagnant throughout the allocated time for the investigation. The height of the emulsion and amount of the separated dispersed phase were recorded continuously for the first 150 minutes. Then the same recording procedure was followed for one month. The stability of the emulsion were reported as the emulsion phase height decrease percentage in comparison with the initial reading of the emulsion height. The emulsion samples were kept at room temperature and ambient pressure.
Figure 1. Schematic showing emulsion formation.

Figure 2. High speed homogenizer.
3. Results and Discussion

Light mineral oil and brine with salinity of 10,000 ppm were used to prepare the emulsion samples. Critical micelle concentration (CMC) analysis was conducted to decide the amount of surfactants needed to form emulsions. The amount of surfactant added into the emulsion samples were 1.0 g and 0.2342 g for the SDS and CTAB, respectively.

Different samples were prepared with varying NP concentrations (by weight) to perform stability analysis by bottle test. Based on previously conducted experiments on viscosity analysis by Abdul-Razzaq et al. (2019), an optimization set of NP weights was used for the bottle tests [26]. Based on the viscosity analysis, three ratios for each surfactant, which has resulted with peak and two tail viscosity values, were selected to conduct the stability bottle test through measuring emulsion phase height decrease percentage in comparison with the initial reading of the emulsion height. This can be inferred to the amount of water droplets being separated from the W/O emulsions as the height of the emulsion decreases with time. The higher the percentage, the higher the amount water separated and hence less stable the emulsion is. The weight ratios of nano-silica added to the SDS emulsion samples were 0.15, 0.5, and 2 respectively. In the case of using CTAB the weight ratios investigated were 0.5, 1.75, and 2.25 respectively.

Figures 4 and 5 show the emulsion phase height decrease percentages with time in the case of using anionic surfactant (SDS) and negatively charged particles nano-silica. It is clearly shown that the amount of separated water (dispersed phase) from W/O emulsions as the height of the emulsion decreases with time. However, there are inherent differences in case of the samples with different weight ratios of nano-silica to SDS. However, it can be inferred that with the addition of nano-silica, the stability of the emulsion was enhanced. Figure 4 depicts the emulsion stability behaviour in case of SDS and nano-silica in the initial 150 minutes. It was observed that most of the water dispersed phase had resolved in this period for all NPs concentration used in comparison with figure 5, which is a continuation of figure 4 but for the time step of the subsequent 1 month.
Figure 4. Emulsion phase height decrease % as a function of time for different weight ratios of negatively charged NPs (nano-silica) to anionic surfactant (SDS) for the initial 150 minutes.

Figure 5. Emulsion phase height decrease % as a function of time for different weight ratios of negatively charged NPs (nano-silica) to anionic surfactant (SDS) for the subsequent 1 month.

As described in figures 4 and 5, the sample without the addition of any nano-silica has experienced the maximum water separation with a decrease of emulsion phase height up to 51%. In comparison with addition of nano-silica of 0.5 times the SDS used by weight, it has resulted with a decrease in the emulsion height percentage up to 22% only. This can be inferred from the fact that SDS surfactant tends to lower the IFT between emulsion’s constituents (water and oil) which aids the
adsorption process of nano-silica on the interface and thus form emulsion droplets [20]. The adsorption of nano-silica on the dispersed phase droplets supports the droplet with additional steric hindrance which stops their coalescence in the incidents of collision [10, 20].

The stability of emulsions prepared using cationic surfactant (CTAB) and negatively charged particles nano-silica is shown in figures 6 and 7. It is illustrated as a function of the emulsion phase height decrease percentage with time. It is clearly evident that the amount of separated water dispersed phase from W/O emulsion prepared with CTAB increased with time as well. However, the degree of the disassociation of oil and water differed with usage of different weight ratios of nano-silica to CTAB. In general, the stability of the emulsion was enhanced with the addition of nano-silica with a lower weight ratio of nano-silica to CTAB. Figure 6 illustrates the stability trend achieved with the utilization of CTAB and nano-silica in the first 150 minutes of the bottle test. In comparison with figure 7, which is a continuation of figure 6 but for a period of the following 1 month, most of the dispersed water phase got separated during the first 150 minutes (figure 6) for all the NPs used ratios, similar to the case of SDS.

![CTAB Stability Chart](image_url)

*Figure 6. Emulsion phase height decrease % as a function of time for different weight ratios of negatively charged NPs (nano-silica) to cationic surfactant (CTAB) for the initial 150 minutes.*
Figure 7. Emulsion phase height decrease % as a function of time for different weight ratios of negatively charged NPs (nano-silica) to cationic surfactant (CTAB) for the subsequent 1 month.

Figure 6 and 7 demonstrate that the maximum dispersed water phase was separated from emulsion sample without nano-silica, which went up to 51%. The addition of nano-silica slightly improved the stability of the emulsion with the decrease in the emulsion height phase going down to 43% with weight ratio of nano-silica to CTAB being 1.75. This enhancement in the stability could be related to the fact that CTAB reduces the IFT between the water and the oil (emulsion’s constituents), which helps the nano-silica to be adsorbed at the interface and hence form the emulsion’s droplets [20]. As a result, steric hindrance was provided to the emulsion dispersed phase droplets that helps in reducing the coalescence process upon collision [10, 20].

It is noticeable from figures 6 and 7 that when the weight ratio of negatively charged nano-silica to cationic surfactant CTAB was 2.25, the stability of the emulsion degraded to the same level as without addition of any NPs. The reason behind this is with excess amount of negatively charged nano-silica being presented to the system, there was a build-up in the negative charge. Together with the presence of the positive charged cationic surfactant (CTAB) in excess, the electrostatic attractive forces started to overrule and promote the coalescence of the water dispersed phase droplets. This finding was backed up with low value of viscosity achieved with this ratio which indicates that droplets started to coalesce and hence resulted with a less stable emulsion [26].

Our preliminary results also corroborate with the findings from the study conducted by Pei et al. (2015). They found that by adjusting the concentration of NPs, the synergy between NPs and surfactant could be proven and a desirable high viscosity can be achieved for emulsion samples [27]. In their study, they have used CTAB as the surfactant and silica NPs. This is directly related to the findings of this study that CTAB and nano-silica are improving the stability of the emulsion samples. However, with the application of higher concentration, the electrostatic attractive forces dominate the system and inhibit the stability.

Moreover, the viscosity of the emulsion plays a vital role in stabilization process [3, 6, 15, 20, 28]. When the viscosity of the sample is high, it provides resistance to diffusion, hence the possibilities of collisions decreases. This helps in declining the coalescence rate, reducing separation of the water dispersed phase in the W/O emulsion, and enhancing the stability of the emulsion. According to Abdul-Razzaq et al. (2019) the viscosity of the emulsion sample with a 0.5 weight ratio of nano-silica...
to SDS was higher than other weight ratios, which indicates why this ratio presented a better stability behaviour in comparison with the other weight ratios of nano-silica to SDS [26]. In a similar case, the CTAB with a weight ratio of 1.75 exhibited higher viscosity than the other two weight ratios of nano-silica to CTAB under consideration. This explains the reason of getting better stability with 1.75 weight ratio of nano-silica to CTAB [26].

It is observed that the rate of separation of the dispersed phase was decreasing with time. At the start, the separation rate was quite high (figures 4 and 6), which changed after 5 minutes from the commencement of the test to a lower rate. Then, the rate have slowed down again after 1 hour from initiating the test. This is inferring that the amount of the water dispersed phase in the emulsion was decreasing with time, which affected the tendency of the droplets to collide, coalesce, and separate out. After two days there was almost no water separating out from the emulsion and the height of the emulsion phase remained almost constant (figures 5 and 7). Figures 8 and 9 are showing snapshots of the bottle tests conducted for emulsion samples with the application nano-silica with a different weight ratios to SDS and CTAB respectively at the beginning of the bottle test as well as the above mentioned time steps.

In a comparison between figures (4 and 5) and figures (6 and 7), it is clear that in general the synergistic effect of nano-silica was more evident with anionic surfactant (SDS) rather than with the cationic surfactant (CTAB). The charge of the surfactant and electrostatic forces developed in the system plays pivotal role with the stability of the emulsions [21]. With the emulsion formulation with SDS and nano-silica, the prominent electrostatic force in the system is the repulsive forces. This repulsive forces prevents the water droplets of the dispersed phase to collide and coalesce due to the increase in the thickness of the diffusive layer in the electrical double layer (EDL) system developed around the droplets [29].

On the other hand when CTAB was used, the resultant electrostatic force in the system is the attractive forces between negatively charge nano-silica and CTAB. The contraction of the diffusive layer in the EDL system developed surrounding the water dispersed phase droplets is induced by the attractive forces prominent in the system. This reduction in the diffusive layer thickness give way to water droplet in W/O emulsion to collide, coalesce and separate out [29]. Therefore, the stability for this system is not as high as in compassion with system where the repulsive forces are the prominent such as with case of using anion surfactant (SDS) and negatively charged nano-silica. In a study conducted by Tikekar et al. (2013), they have discussed that the aggregation of emulsion droplets could be attributed to partial neutralization of electrostatic negative charge on silica particles [29]. This fact can be related to the current study where the CTAB plays the exact role on neutralizing the negative charge of the nano-silica. Hence, reducing the effectiveness of the nano-silica in stabilizing the emulsion droplets. In another study conducted by Qiu (2010), synergy was proven between NPs and surfactants in enhancing the emulsion’s viscosity [30]. However, the mentioned study utilized different surfactant category. Nonionic Triton X-100 surfactant was used together with CAB-O-SIL TS 530 NPs. It was found that with increasing NPs concentration, the viscosity of the emulsion sample was increasing as well. This improvement in the viscosity with application of the NPs and surfactants, can be directly related to the enhancement of the emulsion stability which is investigated in the current study.
Figure 8. Bottle test of emulsions formulated with different weight ratios of negatively charged nano-silica to anionic surfactant (SDS) (a) 0, (b) 0.15, (c) 0.5, (d) 2 at several time steps.
Figure 9. Bottle test of emulsions formulated with different weight ratios of negatively charged nano-silica to cationic surfactant (CTAB) (a) 0, (b) 0.5, (c) 1.75, (d) 2.25 at several time steps.

4. Conclusion
The synergy effect is clearly observed between surfactants and nanoparticles in enhancing the stability of emulsion samples. Anionic (SDS) and cationic (CTAB) surfactants were used to investigate the stability of the emulsion with the use of negatively charged nano-silica via bottle test. It has been observed that SDS performed better due to their electrostatic behaviour and interaction with nano-silica in forming emulsion dispersed phase droplets and providing the repulsive forces to prevent their coalescence. The emulsion samples’ stability tested in this study have shown promising improvement with the usage of model light mineral oil whereby providing the basis for synergistic use of nano-silica and charged surfactants in enhancing the oil recovery through emulsion flooding.

Acknowledgement
The authors would like to thank the Ministry of Higher Education, Malaysia, and Universiti Teknologi Malaysia for supporting this research through Fundamental Research Grant Scheme (FRGS) number: FRGS/1/2018/TK07/UTM/02/2/5F030.
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