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EXPERIMENTAL STUDY OF CRUDE OIL EMULSION STABILITY BY SURFACTANTS AND NANOPARTICLES

by

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ABSTRACT

Surfactants and nanoparticles are materials being widely used for enhanced oil recovery (EOR). Surfactants are mainly used to reduce the interfacial tension between oil and water which could form a stable film of emulsion between these two immiscible fluids. Also, nanotechnology is a potential candidate to offer another solution to improve oil recovery. Nanoparticles can form a solid layer at the droplet's interface and stand as resistant stabilizer under harsh reservoir conditions.

This research investigates whether the combination of surfactants and nanoparticles can provide a more stable emulsion than surfactants only. Two parts of experimental study have been done in this research. First part was implemented using three types of surfactants; nonionic, anionic, and cationic surfactant. The commercial names of these surfactants are IGEPAL Co-530, dodecyl sulfate sodium (SDS), and cetyltrimethyl ammonium bromide (CTAB). Second part was conducted using combinations of same surfactants mentioned above with nanoparticles. These nanoparticles are aluminum oxide (Al_2O_3) and silicon dioxide (SiO_2). These two parts of experimental work were done using different ratios of water to oil. The crude oils used in these experiments were from southeast Kansas, U.S., from an oilfield operated by Blue Top Energy LLC.

Results showed that IGEPAL Co-530 with water continuous phase and CTAB with oil continuous phase could give a stable emulsion in room temperature (25°C) while SDS could break the emulsion within a few hours. More emulsion stability is achieved by adding Al_2O_3 and SiO_2 nanoparticles to IGEPAL Co-530 while adding them to SDS and CTAB depend on the water to oil ratio.

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1. INTRODUCTION

During the life of an oil field, it might pass through three oil recovery methods for hydrocarbon production. Primary recovery is due to the natural forces that move the oil through the reservoir rock to the wellbore by the pressure differential between high pressure in the rock formation and the low pressure in the producing wellbore that may recover less than 30% of total oil in place. The secondary recovery (water injection) can recover about 30 - 50% of oil in place and it is the most common method to maintain the reservoir pressure and to increase the sweep efficiency by oil displacement (Kokal and Al-Kaabi, 2010). The third method is tertiary recovery or enhanced oil recovery (EOR) which can be chemical flooding or thermal recovery (steam injection) or gas methods. Enhanced oil recovery seeks to alter the oil-rock properties to help gain additional production; the techniques of enhanced oil recovery can produce more than 50% and up to 80% of oil in place (Kokal and Al-Kaabi, 2010).

Emulsions are dispersions of one liquid phase in the other. Thus, there is the dispersed phase and a dispersion medium called the continuous phase. The dispersion is called a macroemulsion if the dispersed phase is in from of droplets of the order of millimeters or less. Below 100 nm, the droplets are colloidal and show Brownian motion and diffusivity (Miller and Neogi, 2008). Emulsion can be found in electronics, biomedical, aerospace, pharmaceutical industries as well as in oil production. It is very important to break the emulsion into two continuous phases to be able to use the pure crude oil without any chemical additives. The demulsification can often be very difficult particularly in presence of additives used in EOR methods.

In order to classify crude oil emulsion system, Schubert and Armbruster (1992) have set three main criteria:

- Two immiscible liquids must be in contact (oil and water).
- Surface active component must be present as an emulsifier.
- Agitating effect should be used to disperse one phase liquid in another.

Kokal (2002) has mentioned in a review of crude oil emulsions that an emulsion is unstable due to the natural tendency for a liquid – liquid dispersion to separate and reduce its interfacial area and hence its interfacial energy. Emulsion kinetic stability (i.e., they are stable over a period of time) can be obtained by forming an interfacial film around water droplets involving active surface agents that may present in the crude oil (i.e., asphaltenes) or by adding stabilizers as surfactants. Surfactants serve as a major factor to mobilize more of the residual oil saturation by reducing interfacial tension between water and oil in order to reduce the capillary pressure. Therefore, this strategy allows water to displace additional oil.

Nanoemulsions, have offered well results for field characteristics that attracted the oil producers worldwide. These nanoemulsions, with droplets ranging from 1–200 nm, have good injectivity and penetration without filtration. In addition, these nanoemlsions particles are very stable over time and resistant to coalescence and exchange of the dispersed phase between droplets (Kong and Ohadi, 2010).

Nanoparticles can increase oil recovery by improving both the injected fluid properties (viscosity, density, emulsification improvement, and surface tension) and fluid rock interaction properties (wettability alteration and heat transfer coefficient) (Ayatollahi and Zerafat, 2012).

This thesis represents a general study of the influence of non–thermal substances on the crude oil emulsion stability. This research was done using different types of surfactants and different types of nanoparticles. As one of the enhanced oil recovery techniques, surfactants and nanoparticles were evaluated by their capability to form stable crude oil emulsions among different ratios of water to oil and under various temperatures.

1.1. OBJECTIVE OF THESIS

This work is an evaluation of emulsion stability using crude oil and brine with some chemical compounds such as surfactants and nanoparticles. The main objective of this work is to investigate the most optimum chemical solution to reach a stable or unstable emulsion at different specific temperatures. It is known that surfactant flooding is one of the tertiary recovery techniques that have been used widely to enhance oil recovery of petroleum reservoirs.

This study emphasizes oil/water and water/oil macro-emulsions which usually have a range of 0.5 -50 µm. Macro-emulsion (normal emulsion) is kinetically unstable system which is destroyed by coalescence of droplets and Ostwald ripening (transfer of material from small droplets to large ones).

This thesis addresses two ways in which chemical additives were used. First part was using surfactants with crude oil and the second part is using a mixture of surfactant, nanoparticle, and crude oil. Both parts had been done with different ratios of water to oil. Three types of surfactant were involved in this study: nonionic surfactant, ionic surfactant, and cationic surfactant. The target of using surfactants was to find which mixture of surfactant with crude oil would give more stable emulsion over time and then which one could be separated easily by using various temperatures. Second part was done by using two types of nanoparticles aluminum oxide (Al_2O_3) and silicon dioxide (SiO_2). The objective of the second part was to find if adding nanoparticles to aqueous surfactant solution would give more stable emulsion compared to the use of surfactants alone.

2. LITERATURE REVIEW

This section provides a brief introduction on the fundamental concepts involved in the petroleum field. It includes the EOR concept and its importance in improving oil production by the improvement of the microscopic displacement efficiency and the macroscopic sweep efficiency. This section provides a brief discussion on crude oil and its properties. Finally, it includes the important concepts related to this research work and including the explanation of the emulsion stability mechanisms.

2.1. BACKGROUND

2.1.1. EOR Concept. Enhanced oil recovery has been successfully applied to mature fields in an effort to extract the oil that is left behind in the oil reservoir. One of EOR targets is to create favorable mobility ratio between the injected fluid and the displaced fluid (oil). However, the majority of EOR processes used today were first proposed in the early 1970s at the time of relatively high oil prices (Muggeridge et al., 2013).

Conventional recovery targets mobile oil in the reservoir following a natural progression of oil production from the beginning until the point where no longer is economical to produce from the petroleum reservoir while EOR targets immobile oil (oil cannot be produced due to the capillary and viscous forces) and attempt to recover hydrocarbons beyond secondary method.

Kong and Ohadi (2010) reviewed the EOR techniques that focus on three major categories: (1) thermal recovery which involves the use of heat such as injection of steam in order to reduce the viscosity of the heavy oil and improves its ability to flow through reservoir; (2) gas injection which often uses natural gas, nitrogen, or carbon dioxide. Some gases come out of the solution in the reservoir to push the additional oil to production well, and some gases will dissolve in the oil to reduce its viscosity and then improve its flow rate; and (3) chemical injection that involves the use of long-chained molecules such as polymers to increase water viscosity or involves the use of active surface agents –like surfactants to lower the surface tension between the rock surface and the oil and to lower the interfacial tension (IFT) between two immiscible fluids present in the reservoir (e.g. oil and water).

Muggeridge et al., (2013) recalled the purpose of EOR techniques, which is to improve both the microscopic displacement efficiency and the macroscopic sweep efficiency over that obtained from water flooding. These techniques traditionally involved adding chemicals to the injected water to either change its viscosity (polymer) or reduce its interfacial tension IFT (surfactants).

2.1.2. Crude Oil. Crude oil is a complex mixture that has many organic. These compounds include gases, liquids, and solid hydrocarbon particles. Crude oil is expected to supply 20% to 25% of the world's energy by 2035 as the International Energy Agency published in 2011 (Muggeridge et al., 2013).

The crude oil composition depends on its location over the world and varies with depth within a well. The fluid compositions are also different from one reservoir to another for both aqueous phase (brine that is comprised of different minerals in the form of ions) and the oily phase (hydrocarbon type fluids composed of very light molecules to solid phase asphaltene type), which could affect solid–fluid boundaries such as wettability and interfacial tension (Ayatollahi and Zerafat, 2012).On the other hand, crude oil has natural emulsifiers which include high boiling fractions, like asphaltenes (high molecular weight polar components).These compounds are considered to be the main components that form the interfacial films around water droplets in an oilfield emulsion. Some studies (Kokal and Al-Juraid, 1999) demonstrated that the higher amount of asphaltenes in the crude oils the tighter the emulsion. Asphaltenes have a stabilizing effect on emulsions, which causes very tight emulsions.

2.1.3. Emulsion Stability Mechanisms. An emulsion is a heterogeneous liquid system that consists of two immiscible liquids with one of the liquids dispersed in another. It has an external (continuous) phase with an internal (dispersed) phase with small portion of droplets.

Crude oil and water emulsions can be classified into one of the following:

- Water-in-oil (W/O) emulsions consist of water droplets in a continuous oil phase.
- Oil-in-water (O/W) emulsions consist of oil droplets in a continuous water phase.
- Multiple emulsion (W/O/W) or (O/W/O), consists of tiny droplets suspended in bigger droplets that are suspended in a continuous phase.

Produced oilfield emulsions are classified on the basis of their degree of kinetic stability.

- Loose emulsions separate in a few minutes, and the separated water is free water.
- Medium emulsions separate in tens of minutes.
- Tight emulsions separate (sometimes only partially) in hours or even days.

Whereas, forming stable emulsion is very interested subject, demulsification is another topic that oil producers worry about. Demulsification is the process of breaking crude oil emulsion into oil and water phases. There are many factors work to enhance the emulsion breaking, such as increasing temperature, solids removal, and controlling the amount of emulsifying agents. The mechanisms that are involved in breaking emulsions illustrated in (Figure 2.1) have been categories into the following three processes:

- Flocculation (aggregation): during flocculation the droplets clump together and may get close to each other, even touching at certain points without losing their properties. The rate of flocculation depends on the water cut, temperature, oil viscosity, and the density difference between the oil and water. In flocculation, the van der Waals attraction is weak.
- Coalescence: during coalescence the droplets fuse together to form a large drop. This irreversible process leads to a reduction in the number of water droplets and leads to complete demulsification. Coalescence is enhanced by a high rate of flocculation, the absence of strong films, high interfacial tensions, low oil and interfacial viscosity, a high water cut, and a high temperature (Kokal, 2002).
- Creaming and Sedimentation: This process is produced by external forces (typically gravitational or centrifugal). When such forces exceed the thermal motion(kinetic theory) of the droplets (Brownian motion), a concentration gradient builds up in the system with the larger droplets moving faster to the top (if their density is lower than that of the medium) or to the bottom (if their density is larger than that of the medium) of the container.

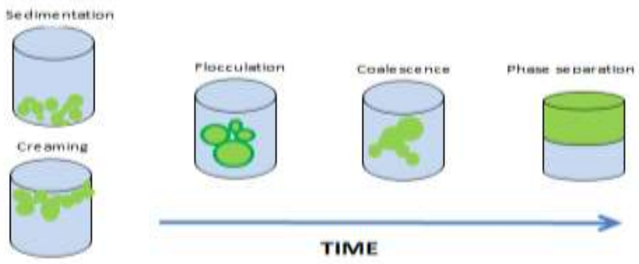


Figure 2.1. Steps toward phase separation in oil-water emulsions

2.1.4. Surface Active Agents (Surfactants). Surfactants work as surface active agents that are composed of organic compounds: a polar (hydrophilic) head part and a non-polar (hydrophobic) tail part. Figure 2.2 illustrates the technique of these surface active agents to create a homogenous phase between oil and water. Surfactant slug must first achieve an ultralow IFT to mobilize residual oil and create an oil bank where both oil and water flow as one continuous phase (Bourrel and Schechter, 1988).

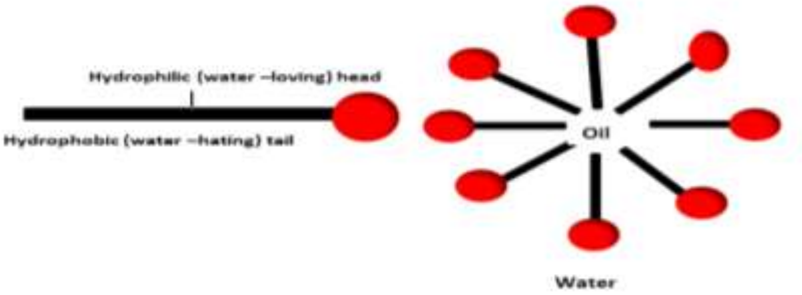


Figure 2.2. Surfactant has hydrophobic tail (water-hating) and hydrophilic head (water-loving)

Surfactants are classified into four types according to the ionic nature of the head group and they are defined as follows:

I. Anionic: This surfactant carries a negative charge. This surfactant is the most widely used as part of EOR process because it has restively low adsorption on sandstone rock whose surface charge is also negative. The molecular structure of sodium dodecyl sulfate (SDS) and ammonium lauryl sulfate (ALS) is shown in Figure 2.3 as examples of anionic surfactants**.**

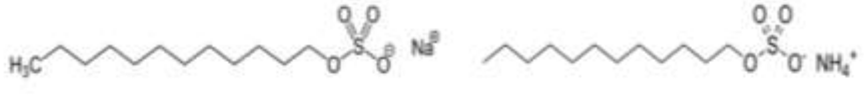


Figure 2.3. The molecular structure of SDS on the right and ALS on the left

II. Cationic: This surfactant carries a positive charge and can strongly adsorb on the sand stone rocks; therefore they are not used in sandstone reservoir, but they can be used in carbonate reservoirs to change wettability from oil wet to water wet. The molecular structure of cetyltrimethyl ammonium bromide (CTAB) is shown in Figure 2.4 as an example of cationic surfactant.

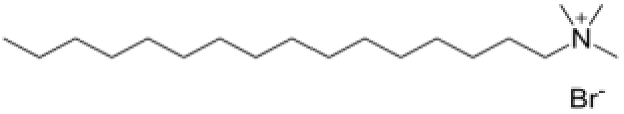


Figure 2.4. The molecular structure of CTAB

III. Nonionic: A nonionic surfactant does not carry any ionic charge (natural) and primary serve as surfactant to improve system phase behavior.

IV. Zwitterionic: A zwitterionic can have both a positive and a negative charge. It can be nonionic-anionic, nonionic-cationic, or anionic-cationic.

3. PREPARING SURFACTANT AND NANOPARTICLE SOLUTIONS FOR CRUDE OIL EMULSION STABILITY SCREENING TESTS

This section focuses on the interaction that occurs between surfactants with crude oil and a mixture of surfactants, and nanoparticles with crude oil. It includes a discussion on how emulsions can form when chemical compounds consolidate with crude oil. It also includes a description of the affect that temperature has on the separation of crude oil emulsion. The purpose of this study is finding the best chemical compounds that may form a stable emulsion. This emulsion mixture should be breakable at higher temperatures.

3.1. INTRODUCTION

Stable emulsion can significantly improve oil recovery. Emulsion can be encountered in almost all phases of oil production and processing. For example, it can be found inside reservoirs, well heads, well bores, transportation through pipelines, crude oil storage, and finally could be found in drilling fluid processing. This section includes a discussion on a method that can be used to prepare crude oil emulsion for EOR processing. Three types of surfactants were used to conduct the first part of experiments: nonionic surfactant, IGEPAL CO-530, a cationic surfactant, cetyltrimethyl ammonium bromide (CTAB), and anionic surfactant, sodium dodecyl sulfate (SDS).

Nanoparticles, aluminum oxide (Al_2O_3) and silicon dioxide (SiO_2) were used to conduct the second part of experiments. Nanoparticles stabilize emulsions droplets which are small enough to pass typical pores, and flow through the reservoir rock without much retention and also remain stable under hard conditions in the reservoirs due to irreversible adsorption of the nanoparticles on their droplet surface (Zhang et al., 2010).

3.2. EXPERIMENTAL MATERIALS

- Surfactants: Three types of hydrophilic surfactants were used for a crude oil emulsion stability experiment with concentrations at 1wt. % in water containing 1 wt. % of NaCl. This solution was then added to oil at different water–to–oil ratios. Their properties are listed in Table 3.1.
- Nanoparticles: Two types of hydrophilic nanoparticles were received from Sigma Aldrich. Each was used to enhance the crude oil emulsion stability .The nanoparticle concentration was always at 1 wt. % in brine. A third type of nanoparticle was obtained from Phosphorex. This is very expensive nanoparticle. It is polymeric, polymethyl methacrylate, which is hydrophobic. The mean size was 25nm.Unfortunatly, the amount supplied (approximate 5ml) was very low and thus could not be used in these experiments. The nanoparticle size and description are given in Table 3.2.

Table 3.1. Properties of the surfactants used

No.	Name of Surfactant	Type of Surfactant	Description	Supplier
1.	IGEPAL CO-530	Non-ionic	Liquid/Colorless	--------------
2.	Cetyltrimethyl ammonium bromide (CTAB)	Cationic	White Powder	CALBIOCHEM®
3.	Sodium dodecyl sulfate (SDS)	Anionic	White Powder	Aldrich

Table 3.2. Properties of the nanoparticles used

No.	Type of nanoparticle	Particle size (nm)	Description
1.	Aluminum Oxide (Al_2O_3)	<50nm	White Liquid (Suspension)
2.	Silicon Dioxide (SiO_2)	12nm	White Powder

- Brine. A 1.0 wt. % of sodium chloride (NaCl) was used to prepare all of the surfactant /nanoparticle aqueous solutions.
- Graduated tubes with plug seal caps were used to distinguish the different ratios of water to oil.
- A water bath with a heater was used to maintain the system at a fixed temperature.
- Distilled water was used to prepare the aqueous solutions.
- Crude Oil. A heavy crude oil was obtained from an oilfield operated by Blue Top Energy LLC in southeast Kansas. Two samples of A-Hauser were received at different times. A-Hauser (1) had a viscosity of 650 cp and an API gravity of 19.9 API°. It was used with all of the series that included either the surfactants only or surfactants with Al_2O_3 nanoparticles. There was not enough amount of A-Hauser (1) to complete the rest of the experiments. A-Hauser (2) was the second sample received. It was from the same lease as A-Hauser (1). It was used with all of the series that included SiO_2 nanoparticle. It had high viscosity about 3000 cp. The crude oil may have increased in viscosity because some wax may have precipitated at low temperatures, and was not dissolving out easily at room temperatures. Thus, oil was placed in the oven at 80°F for 30 minutes so that it could reach its melting point. It was then cooled and measured again. A-Hauser (2) reaches an average viscosity of 750 cp, and the API gravity was 23° API°.

- Viscometer. A Brookfield viscometer was used to measure the oil viscosity with 34 spindle size. Both viscometer and the spindle were used to measure the oil viscosity are shown in Figure 3.1.

Figure 3.1. The left side is the viscometer used to measure the crude oil viscosity and on the right is the 34 spindle

3.3. EXPERIMENTAL PROCEDURE

3.3.1. Emulsion with Surfactants and Crude Oil.

Three different aqueous solutions were prepared as follows:

- First mixture: 100g water, 1 wt. % NaCl, and 1 wt. % IGEPAL CO-530
- Second mixture: 100g water, 1 wt. % NaCl, and 1 wt. % cetyltrimethyl – ammonium bromide (CTAB)
- Third mixture: 100g water, 1 wt. % NaCl, and 1 wt. % sodium docecyl sulfate (SDS).

A magnetic stirrer bar was used to mix each aqueous solution. The mixtures agitated overnight to obtained homogenous solutions. Each surfactant´s aqueous solution was added to the graduated tubes according to the ratios listed in Table 3.3.

Table 3.3. Water- to- oil ratios

Water	Oil
1	9
2.5	7.5
5	5
7.5	2.5
9	1

A-Hauser (1) crude oil was added to the aqueous solutions in the graduated tubes according to the ratios listed on Table 3.3. The tubes were then sealed and agitated vigorously both by hand and digital mixer (see Figure 3.2) for approximately three minutes until each mixture appeared to be a homogeneous phase. The water bath was readied, and the temperature was fixed at 24˚C.Tubes that contained the mixtures were placed in a water bath for 24 hour at 24˚C. changes and observations were recorded. If the mixtures were still one phase that exhibited emulsion during 24 hours, then the temperature was increased to 40˚C for another 24 hours. If separation did occur and no emulsion phase was left, then it was not necessary to increase the temperature. Every set of surfactants/crude oil emulsion stability tests was conducted over a single day.

Figure 3.2. The digital mixer was used to mix the graduated tubes to obtain an emulsion phase

3.3.2. Emulsion with Surfactants / Nanoparticles and Crude Oil. Five different ratios were used for each surfactant/nanoparticle and crude oil tests. This study was conducted to investigate whether or not the addition of the nanoparticle to a surfactant solution would reach more stable crude oil emulsion. Procedures used to conduct this work are illustrated below.

3.3.2.1 Emulsion with surfactants and aluminum oxide (Al_2O_3). Three different aqueous solutions were prepared as follows:

- First mixture: 100g water, 1 wt. % NaCl, 1 wt. % IGEPAL CO-530, and 1 wt. % Al_2O_3
- Second mixture: 100g water, 1 wt. % NaCl, 1 wt. % cetyltrimethyl ammonium bromide (CTAB), and 1 wt. % Al_2O_3
- Third mixture: 100g water, 1 wt. % NaCl, 1 wt. % sodium dodecyl sulfate (SDS) and 1 wt. % Al_2O_3

3.3.2.2 Emulsion with surfactants and silicon dioxide (SiO_2). Three different aqueous solutions were prepared as follows:

- First mixture: 100g water, 1 wt. % NaCl, 1 wt. % IGEPAL CO-530, and 1 wt. % SiO_2
- Second mixture: 100g water, 1 wt. % NaCl, 1 wt. % cetyltrimethyl ammonium bromide (CTAB), and 1 wt. % SiO_2
- Third mixture: 100g water, 1 wt. % NaCl, 1 wt. % sodium dodecyl sulfate (SDS) and 1 wt. % SiO_2

3.3.2.3 Preparing the mixture of the aqueous solutions and crude oils. A magnetic stirrer bar was used to mix each aqueous solution. The mixture was agitated overnight to obtain a homogenous solution. Each surfactant´s aqueous solution was added to the graduated tubes according to the ratios listed in Table 3.3. The A-Hauser (1) crude oil was added to the aqueous solutions of Al_2O_3 in the graduated tubes also following the ratios listed in Table 3.3. The A-Hauser (2) crude oil was added to the SiO_2 aqueous solutions in the graduated tubes following the ratios listed in Table 3.3. The tubes were then sealed and agitated vigorously both by hand and digital mixer (see Figure 3.2) for approximately three minutes until each mixture appeared to be homogenized as one phase. The water bath was readied, and the temperature was fixed at 24˚C. Tubes that contained the mixtures were placed in a water bath for 24 hour at 24˚C. Changes and observations were recorded. If the mixtures were still one phase that exhibited emulsion during 24 hours, then the temperature was increased to 40˚C for another 24 hours. If separation did occur and no emulsion phase appeared, then it was not necessary to increase the temperature. Every set of surfactants with nanoparticles and crude oil emulsion stability tests was conducted in a one day period.

4. SURFACTANT AND NANOPARTICLE EMULSION STABILITY RESULTS AND DISSCUSION

Surfactants are long chain compounds comprised of a long hydrocarbon (aliphatic) molecule that ends in a polar head group. This group can be ionic. Thus, it dissociates in water. If the group is sodium sulfate as in sodium dodecyl sulfate (SDS), the sulfate is negatively charged and bonded to the tail. Hence, SDS is an anionic surfactant; the sodium is positively charged. Surfactants with either divalent or trivalent cations have very limited solubility and precipitate as “bathtub rings.”

Cetylrimethyl ammonium bromide (CTAB) is a cationic surfactant. The counter ion is the bromide ion that carries a negative charge, and the surfactant ion is positively charged in the amine group. The cationic surfactants are more expensive. Nonionic surfactants are also possible when the head groups are comprised of bulky ethoxy groups. These groups are hydrophilic as they form hydrogen bonds with water. These bonds break, however as the temperature increases.

Surfactants are surface active. Thus, they accumulate at the oil – water interface. Both SDS and CTAB, however, are also preferentially water soluble and will stay in the aqueous phase as well. A nonionic surfactant (e.g., $C_{12-14}E_{20}$) will become oil soluble at high temperatures when its surface active properties disappear. (Hydrogen bonds cannot be made at higher temperatures).

Nanoparticles are less than 30nm in diameter. They can be either hydrophilic or hydrophobic. Hydrophobic particles are very expensive and difficult to handle. Alumina (Al_2O_3) and silica (SiO_2) particles are hydrophilic particles that are easily obtained. However, alumina tends to charge positively, and silica is charged strongly negatively. These particles by themselves are not surface active.

4.1. EMULSION STABILITY IN CRUDE OIL AND SURFACANTS

4.1.1. Emulsion Stability in A-Hauser (1) Crude Oil with 650 cp and IGEPAL CO-530. The IGEPAL CO-530 should stabilize emulsion only when it is water continuous. Emulsion should become very unstable at a higher temperature. The emulsion samples of A-Hauser (1) crude oil and IGEPAL CO-530 were placed in a 25˚C water bath for 24 hours to test this theory. The results were unstable emulsion for samples with a 1:9, 5:5, and 2.5:7.5 ratio of water to oil (in which oil was the continuous phase). These samples became unstable within a few hours; forming two phases of separation (see Figure 4.1 a, b, and c). However, samples with 7.5:2.5, and 9:1 ratios of water to oil (in which water was the continuous phase) became stable for several nights without exhibiting separation (see Figure 4.1 d and e).

Samples that did not exhibit separation were returned to the water bath at a higher temperature so that their ability to break emulsion as the temperature increased could be tested. The temperature was increased to 40˚C for another 24 hours. The results remained stable for the 7.5:2.5 ratio of water to oil (see Figure 4.2d). The 9:1 ratio of water to oil became unstable and exhibited two phases (see Figure 4.2e).

Figure 4.1. The result after IGEPAL CO-530 was combined with A-HAUSER (1) at 25˚C

Figure 4.2. The result of ratio 7.5:2.5, and 9:1 water to oil after the temperature increased to 40˚C

4.1.2. Emulsion Stability in A-Hauser (1) Crude Oil with 650 cp and Dodecyl Sulfate, Sodium (SDS). The SDS should stabilize an emulsion only when it is water continuous and the effects are not dependent on temperature. For testing this theory, all samples of the combination of A-Hauser (1) crude oil and SDS were placed in a water bath at 25˚C. The results were unstable for all samples. Each sample exhibited two phases: an oil phase and a water phase. The SDS water phase had a good, clear quality (see Figure 4.3). These samples did not need to be tested at a higher temperature due to emulsion instability at room temperature.

Figure 4.3. Unstable emulsion results of SDS at 25˚C. From left to right: 1:9, 2.5:7.5, 5:5, and 7.5:2.5, 9:1

4.1.3. Emulsion Stability in A-Hauser (1) Crude Oil with 650 cp and Cetylrimethyl Ammonium Bromide (CTAB). The CTAB should stabilize emulsion only when water is oil continuous and the effects are not dependent on temperatures as expectations. Samples of A-Hauser (1) crude oil that were combined with CTAB placed in a water bath at 25˚C for 24 hours. The 9:1 water to oil ratio formed both a brown phase and a black phase (see Figure 4.4a). The 7.5:2.5, 5:5, and 2.5:7.5 water to oil ratios formed a dark brown phase and a black phase (see Figure 4.4b, c, and d). The (1:9) water to oil ratio, however, formed one phase of emulsion without separation (Figure 4.4e). The samples were kept in the water bath for an additional 24 hours. No break in emulsion occurred during this 48 hour period. The CTAB samples were then placed in a water bath at higher temperatures (40˚C, 50˚C, and 60˚C). No break in emulsion occurred, indicating the CTAB was unaffected by temperature.

Figure 4.4. The results of CTABs immersed in A-Hauser (1) crude oil. From left to right: (9:1, 7.5:2.5, 5:5, and 2.5:7.5, 9:1)

4.2. EMULSION STABILITY IN CRUDE OIL, SURFACTANTS AND ALUMINUIM OXIDE (Al_2O_3) NANOPARTICLES

4.2.1. Emulsion Stability in A-Hauser (1) Crude Oil with 650 cp, IGEPAL CO-530, and Al_2O_3. Adding Al_2O_3 nanoparticle to IGEPAL CO-530 should let the IGEPAL CO-530 adsorb on alumina. Both water continuous emulsion and oil continuous emulsion should be more stable than IGEPAL CO-530 alone. In contrast, only water continuous emulsion becomes unstable when the temperature increases. In order to test this theory, Samples of A-Hauser (1) crude oil combined with IGEPAL CO-530, and Al_2O_3 were placed in a 25˚C water bath for 24 hours. The samples had an unstable emulsion at ratios of 7.5:2.5 and 5:5. The remaining samples 2.5:7.5 and 1:9 formed stable emulsion and remained oil continuous phase as pictured in Figure 4.5.The (9:1) water to oil showed light separation considered as a stable emulsion. The aqueous phases that had water to oil ratios of 7.5:2.5 and 5:5 were brown. Thus, they must have contained oil droplets. The addition of Al_2O_3 nanoparticles to the IGEPAL CO-530 stabilized emulsion more than the IGEPAL CO-530 alone.

Figure 4.5. The results after IGEPAL CO-530 was combined with Al_2O_3 at 25˚C (24 hours)

All samples were next placed in a 40˚C water bath for an additional 24 hours so that the behavior of crude oil, IGEPAL CO-530, and Al_2O_3 emulsion could be investigated at higher temperatures. The 9:1 ratio of water to oil broke emulsion within a few hours (see Figure 4.6e). The remaining samples`s properties did not change as the temperature increas (see Figure 4.6a, b, c, and d).

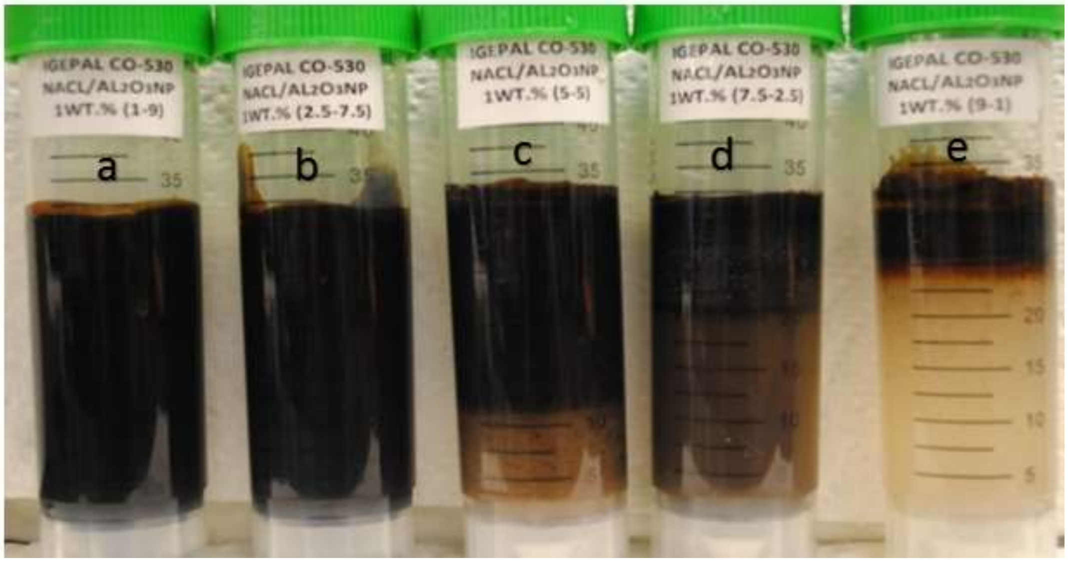


Figure 4.6. The results after IGEPAL CO-530 was combined with Al_2O_3 at 40˚C

4.2.2. Emulsion Stability in A-Hauser (1) Crude Oil with 650 cp, SDS, and Al_2O_3. The SDS will adsorb on alumina. Such particles will be partially hydrophobic and, hence, surface active. Water continuous emulsions are expected to be more stable than SDS alone. To test these expectations, samples A–Hauser (1) crude oil combined with SDS and Al_2O_3 were placed in a 25˚C water bath for 24 hours. The 1:9 ratio of water to oil (oil continuous) had stable emulsion after 4 hours as pictured on (see Figure 4.7a). The remaining samples exhibited rapid separation as pictured on (see Figure 4.7b, c, d, and e).

Samples of 2.5:7.5, 5:5, 7.5:2.5, and 9:1 water to oil ratios were completely separated into two phases (see Figure 4.8b, c, d, and e) after 24 hours. The (1:9) water to oil did not exhibit a phase separation as pictured (see Figure 4.8a).

Figure 4.7. The results after SDS was combined with Al_2O_3 at 25˚C (four hours)

Figure 4.8. The results after SDS were combined with Al_2O_3 at 25˚C (24 hours). From left to right: 1:9, 2.5:7.5, 5:5, 7.5:2.5, and 9:1.

4.2.3. Emulsion Stability in A-Hauser (1) Crude Oil with 650 cp, CTAB, and Al_2O_3. The CTAB will neither adsorb on alumina nor exhibit small adsorption. Water continuous emulsions are expected to be as stable as those with CTAB alone. To test this theory, samples of CTAB and Al_2O_3 crude oil emulsion were placed in a 25˚C water bath for 24 hours. The results of this immersion are pictured in Figure 4.9. Samples of 1:9, and 2.5:7.5water to oil formed a stable emulsion (see Figure 4.9a, and b). Samples with ratios of 5:5, 7.5:2.5 and 9:1 water to oil formed unstable emulsion (see Figure 15c, d and e). The 9:1 water to oil ratio formed both a yellow phase and a black phase. The 7.5:2.5 water to oil ratio had a light brown phase and a black phase. The (5:5) water to oil formed a dark brown phase and a black phase. The 1:9 and 2.5:7.5water to oil formed one phase of emulsion without separation. Samples (1:9 and 2.5:7.5) water to oil were next placed in a 40˚C water bath for an additional 24 hours so could be investigated at higher temperatures. Samples (1:9 and 2.5:7.5) water to oil remained stable at higher temperature.

Figure 4.9. The results after CTAB was combined with Al_2O_3 at 25˚C (24 hours)

4.3. EMULSION STABILITY IN CRUDE OIL, SURFACTANTS AND SILICON DIOXIDE (SiO_2) NANOPARTICLES

4.3.1. Emulsion Stability in A-Hauser (2) Crude Oil with 750 cp, IGEPAL CO-530, and SiO_2. If IGEPAL CO–530 adsorbs on silica, then both water continuous emulsion and oil continuous emulsion should be more stable than they are with IGEPAL CO–530 alone. Only the water continuous emulsion would become unstable when the temperature is increased. To test this theory, the emulsion samples of A-Hauser (2), IGEPAL CO-530, and SiO_2 were placed in a 25˚C water bath for 24 hours. All of samples were stable, and no phase separation accrued (see Figure 4.10).

The temperature had to be increased every 24 hour to (40˚C, 50˚C, and 60˚C) to break the emulsion. Eventually, the temperature had no effect on the emulsion of IGEPAL CO-530 and SiO_2 crude oil samples.

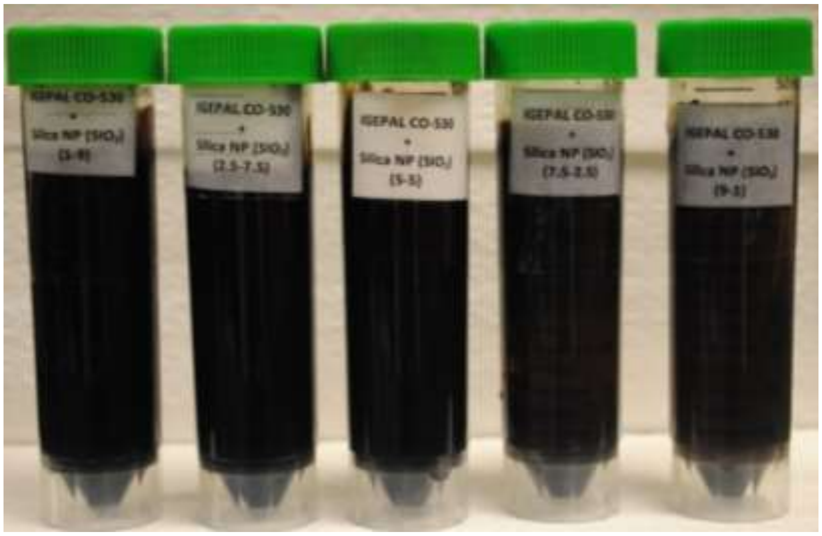


Figure 4.10. The results of adding SiO_2 to IGEPAL CO-530 at 25° C (24 hours)

4.3.2. Emulsion Stability in A-Hauser (2) Crude Oil with 750cp, SDS, and SiO_2. The SDS will neither adsorb on silica nor exhibit small adsorption. Water continuous emulsions are expected to be as stable as those with SDS alone.

Prepared samples of the combination of A-Hauser (2) crude oil, SDS, and SiO_2 were placed in a 25°C water bath for 24 hours. The (1:9) water to oil ratio (oil continuous) had a stable emulsion phase (see 4.11a). The (2.5:7.5) water to oil ratios separated slightly with some drops of oil stuck on the surface (see Figure 4.11b). The 5:5, 7.5:2.5, and 9:1 water to oil ratios (water continuous) exhibited three phases, dark gray particles in the bottom, a milky solution in the middle and a black color on the top (see Figure 4.11c, d, and e).

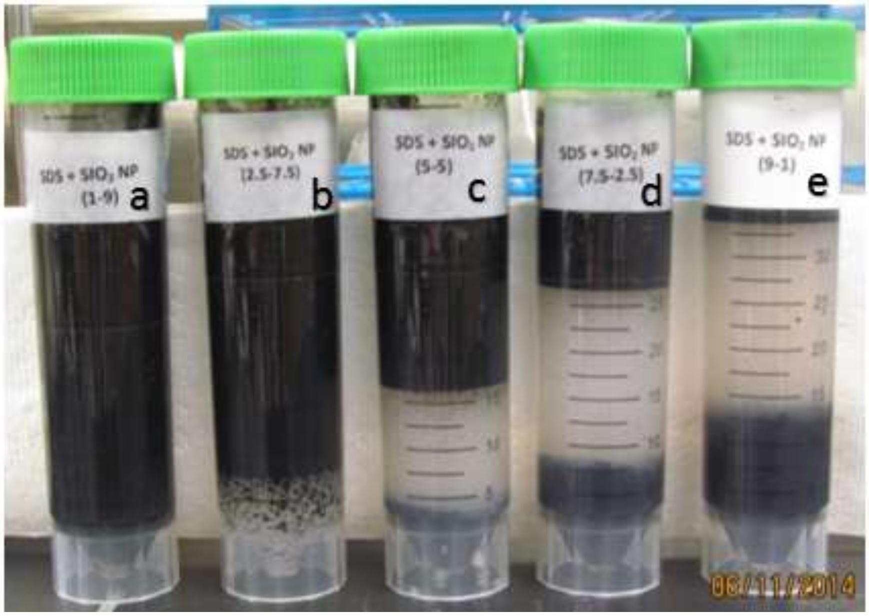


Figure 4.11. The results of adding SiO_2 to SDS at 25° C (24 hours)

4.3.3. Emulsion Stability in A-Hauser (2) Crude Oil with 750cp, CTAB, and SiO_2. The CTAB will adsorb on silica. Such particles will be partially hydrophobic and, hence, surface active. Water continuous emulsions are expected to be more stable than those with CTAB alone.

To examine these expectations; samples of the combination of CTAB and SiO2 were placed in a 25°C water bath for 24 hours. The 1:9 and 2.5:7.5 water to oil ratios had an oil continuous phase that formed a stable emulsion (see Figure 4.12a, and b). The (9:1) water to oil ratio formed three phase: a yellow phase in the middle, a black phase on the top and some gray particles at the bottom (see Figure 4.11e). The 7.5:2.5 and 5:5 water to oil ratios formed two phases: a milky brown phase at the bottom and a black phase on the top (see Figure 4.12c and d). There were not an effect of temperature on CTAB and SiO2 crude oil emulsion after increasing temperature for the 1:9 and 2.5:7.5water to oil ratios.

Figure 4.12. The results of adding SiO_2 to CTAB at 25˚C (24 hours)

4.3.4. Summary. This section described a study that was conducted to investigate a surfactant influence on crude oil emulsion stability. It also described the influence of adding nanoparticles into surfactant solutions to achieve more emulsion stability. Both studies had done using different ratios of water to oil, and under different temperatures. Nonionic surfactants may be affected by differences in temperature. Anionic and cationic surfactants, however, may not. A summary of the results of surfactants and crude oil emulsion is shown in Table 4.1.

Several of the samples exhibited a stable emulsion. Thus, some surfactants may have the ability to reduce the interfacial tension (IFT) that exists between the crude oil and the aqueous phase. The additional of nanoparticles would help increase crude oil emulsion stability. A summary of the results of surfactants with Al_2O_3 nanoparticles and crude oil emulsion is shown in Table 4.2. Different types of nanoparticles with different sizes should lead to different results. A summary of the results of surfactants with Si_2O nanoparticles and crude oil emulsion is shown in Table 4.3.

Table 4.4 summarizes the expectations and the results for each system of chemicals products used in these experiments.

In this section also, there are important facts need to be recalled:

1. SiO_2 is negatively charged, Al_2O_3 is positively charged, SDS is negatively charged and CTAB is positively charged.
2. Nonionic loses surface activity on heating.
3. Systems with flocculation without coalescence can break on heating.
4. Systems with larger droplets are unstable.

Table 4.1. Stability results of surfactants at 25˚C and 40˚C.

Water to Oil ratio	Nonionic (IGEPAL CO-530)	Anionic (SDS)	Cationic (CTAB)
1-9	Unstable at 25˚C Fig 4.1a	Unstable at 25˚C Fig 4.3	Stable at 25˚C Fig 4.4e
2.5 -7.5	Unstable at 25˚C Fig 4.1c	Unstable at 25˚C Fig 4.3	Unstable at 25˚C Fig 4.4d
5-5	Unstable at 25˚C Fig 4.1b	Unstable at 25˚C Fig 4.3	Unstable at 25˚C Fig 4.4c
7.5-2.5	stable at 25˚C Fig 4.1d stable at 40˚C Fig 4.2d	Unstable at 25˚C Fig 4.3	Unstable at 25˚C Fig 4.4b
9-1	Stable at 25˚C Fig 4.1e Unstable at 40˚C Fig 4.2e	Unstable at 25˚C Fig 4.3	Unstable at 25˚C Fig 4.4a

Table 4.2. Stability results of surfactants with Al_2O_3 at 25°C and 40°C

Water to Oil ratio	Nonionic (IGEPAL CO-530)+Al_2O_3	Anionic (SDS)+Al_2O_3	Cationic (CTAB)+Al_2O_3
1-9	Stable at 25°C Fig 4.5 Sable at 40°C Fig4.6a	Stable 25°C Fig 4.8a	Stable 25°C Fig 4.9 Sable at 40°C
2.5 -7.5	Stable at 25°C Fig 4.5 Sable at 40°C Fig4.6b	Unstable 25°C Fig 4.8b	Stable 25°C Fig 4.9 Sable at 40°C
5-5	Unstable at 25°C Fig 4.5	Unstable 25°C Fig 4.8c	Unstable 25°C Fig 4.9
7.5-2.5	Unstable at 25°C Fig 4.5	Unstable 25°C Fig 4.8d	Unstable 25°C Fig 4.9
9-1	Slightly stable at 25°C Fig 4.5 Show more separation at 40°C	Unstable 25°C Fig 4.8e	Unstable 25°C Fig 4.9

Table 4.3. Stability results of surfactants with SiO_2 at 25°C and 40°C

Water to Oil ratio	Nonionic (IGEPAL CO-530)+SiO_2	Anionic (SDS)+ SiO_2	Cationic (CTAB)+ SiO_2
1-9	Stable at 25°C Fig 4.10 Sable at higher temperatures	Stable at 25°C Fig 4.11a Sable at higher temperatures	Stable at 25°C Fig 4.12 Sable at higher temperatures
2.5 -7.5	Stable at 25°C Fig 4.10 Sable at higher temperatures	Unstable 25°C Fig 4.11b	Stable at 25°C Fig 4.12 Sable at higher temperatures
5-5	Stable at 25°C Fig 4.10 Sable at higher temperatures	Unstable 25°C Fig 4.11c	Unstable 25°C Fig 4.12
7.5-2.5	Stable at 25°C Fig 4.10 Sable at higher temperatures	Unstable 25°C Fig 4.11d	Unstable 25°C Fig 4.12
9-1	Stable at 25°C Fig 4.10 Sable at higher temperatures	Unstable 25°C Fig 4.11e	Unstable 25°C Fig 4.12

Table 4.4. Chemical additives system expectations and results.

System	Expectations	Results
IGEPAL CO-530	- o/w stable because of charge effect, temperature sensitive: becomes unstable at high temperature as it loses surface activity - w/o unstable	- o/w stable at 25°C - w/o unstable - o/w unstable at 40°C Fig. 4.1, 4.2 A.1
SDS	- o/w stable because of charge effect, but not temperature sensitive. - w/o unstable	- All unstable Fig. 4.3 A.2
CTAB	- o/w stable because of charge effect, but not temperature sensitive - w/o unstable	- w/o stable - no effect of higher temperatures Fig. 4.4
IGEPAL CO-530 + Al_2O_3	- Adsorption on Al_2O_3 - Stable than IGEPAL CO-530 alone	- w/o stable at 25°C - o/w unstable Fig. 4.5
SDS + Al_2O_3	- Adsorption of SDS on Al_2O_3 - Al_2O_3 is surface active.	- w/o stable (1 water: 9 oil) - o/w unstable Fig. 4.8
CTAB + Al_2O_3	- No adsorption of CTAB on Al_2O_3	- w/o stable - o/w unstable Fig 4.9
IGEPAL CO-530 + SiO_2	- adsorption on SiO_2	- All stable - Stable to temperature rise up to 60°C Fig. 4.10
SDS + SiO_2	- No adsorption of SDS on SiO_2, so SiO_2 is not surface active	- w/o stable Fig. 4.11
CTAB + SiO_2	- Adsorption of CTAB on SiO_2 so, SiO_2 is surface active.	- w/o stable Fig. 4.12

5. CONCLUSION

These sections summarize the conclusions drawn from two major experiments were described in sections three and four. It discusses also the final results have been extracted from this work.

5.1. INTERACTION BETWEEN SURFACTANTS AND CRUDE OIL EMULSION

- IGEPAL CO–530 is a nonionic surfactant and could form stable emulsion for the heavy oil in Kansas and synthetic brine. The emulsion can be separated into two phases within a few hours at higher temperature without adding any de-emulsifier.
- IGEPAL CO–530 with (9-1) water to oil ratio could form a stable emulsion and be separated into two phases with higher temperature.
- SDS is anionic surfactant and could not form stable emulsion and breaks into two phases within a few hours.
- CTAB is a cationic surfactant which could form emulsion for long term and cannot be separated with higher temperature.
- CTAB with (1-9) water to oil ratio could form stable emulsion and cannot be separated with higher temperatures.

5.2. INTERACTION BETWEEN SURFACTANTS, NANOPARTICLES, AND CRUDE OIL EMULSION

- IGEPAL CO–530 could form more emulsion stability with Al_2O_3 and SiO_2
- IGEPAL CO–530 with SiO_2 form stable emulsion and it is not breakable with higher temperatures.
- SDS could form an emulsion by adding Al_2O_3 and SiO_2 nanoparticles when it is oil continuous phase.
- CTAB could stabilize the emulsion more with samples have oil as continuous phase and no effect of temperature increasing.

5.3. RECOMMENDATION

Based on the present study, future research studies are recommended in the following areas:

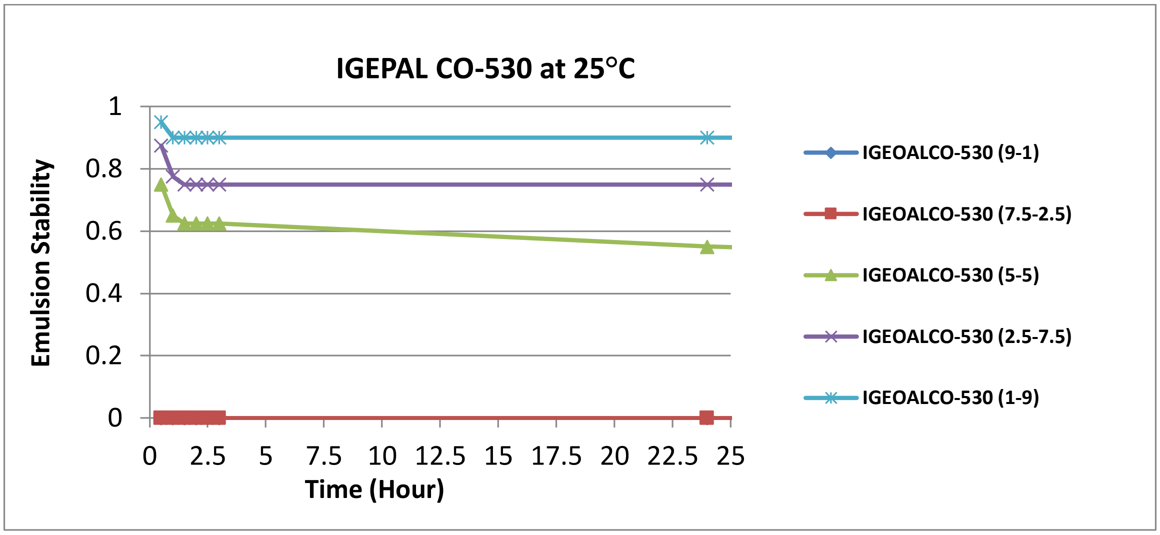
- Explore these experiments in different concentrations of brine, and surfactants.
- Conduct these experiments in low concentrations of nanoparticles.
- Conduct core flooding experiments with applying reservoir conditions to obtain more representative data
- Examine these experiments by using nanoparticles alone with no additives of surfactants
- Examine emulsion phases under a microscope to find their droplet size distribution and based on that find their emulsion stability

APPENDIX

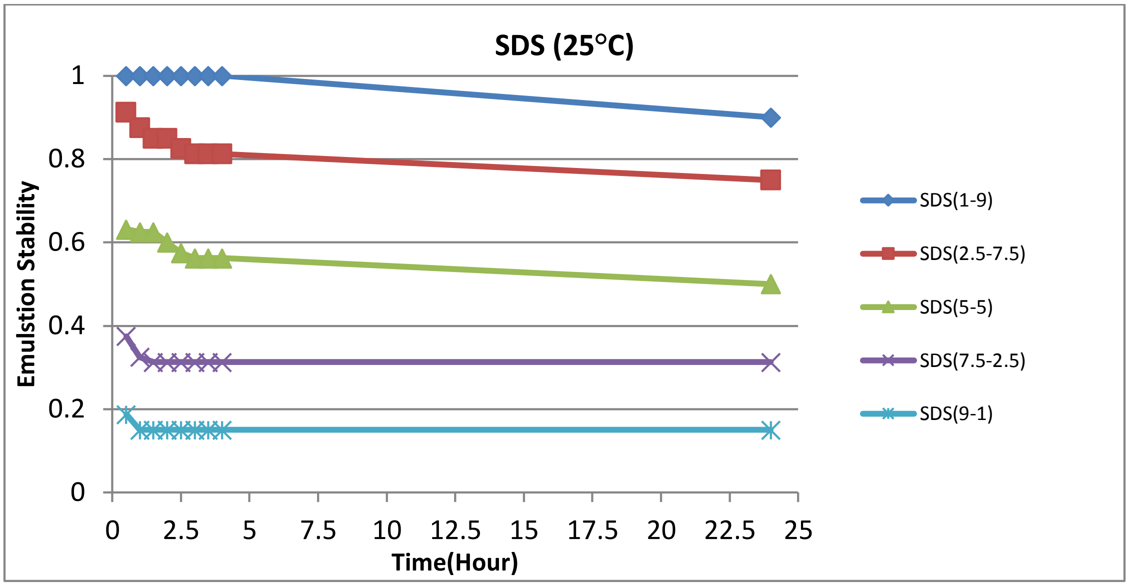
THE BEHAVIOR OF EMULSION STABILITY BY SURFACTANTS AND NANOPARTICLES WITH A-HAUSER CRUDE OIL VS.TIME

This appendix has the main results of these conducted experiments. They present the behavior of each chemical products with A-Hauser Kansas crude oil. Each product is showing five differernt ratios of water to oil and all of them were implemented in a 25°C. These graphic results were accomplished using this equation below.

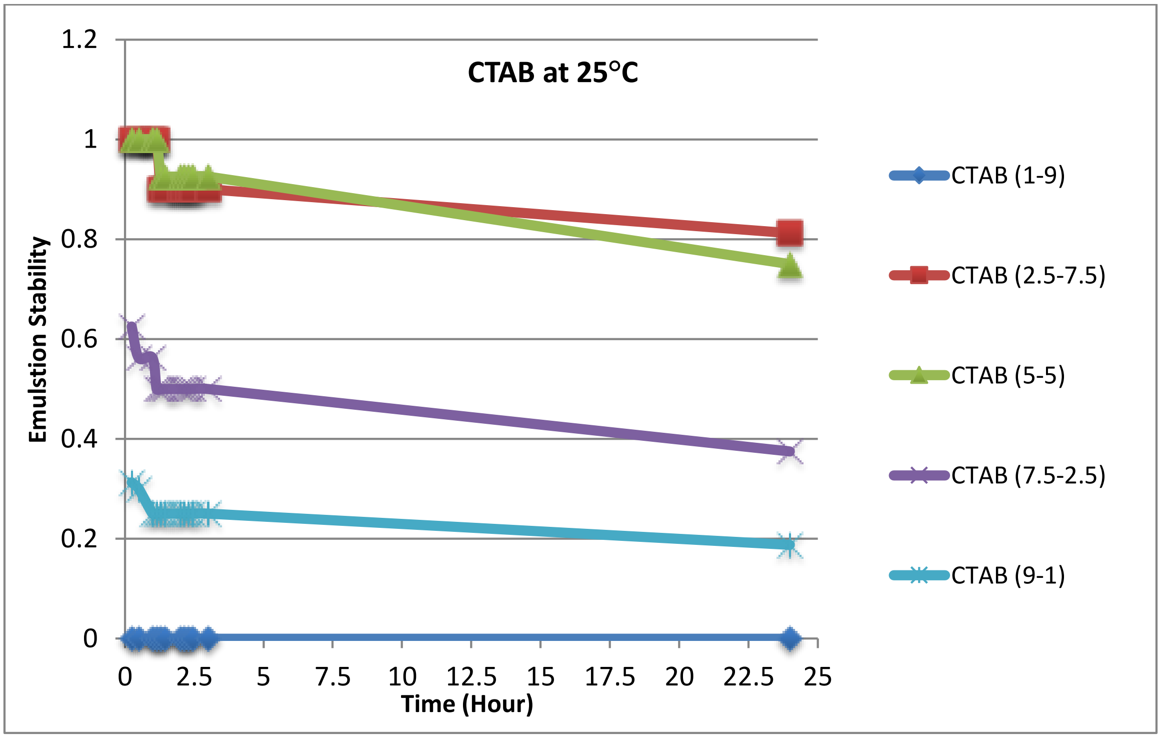
$$EmulsionStability = \frac{H_{\max} - H_{brine}}{H_{\max}}$$



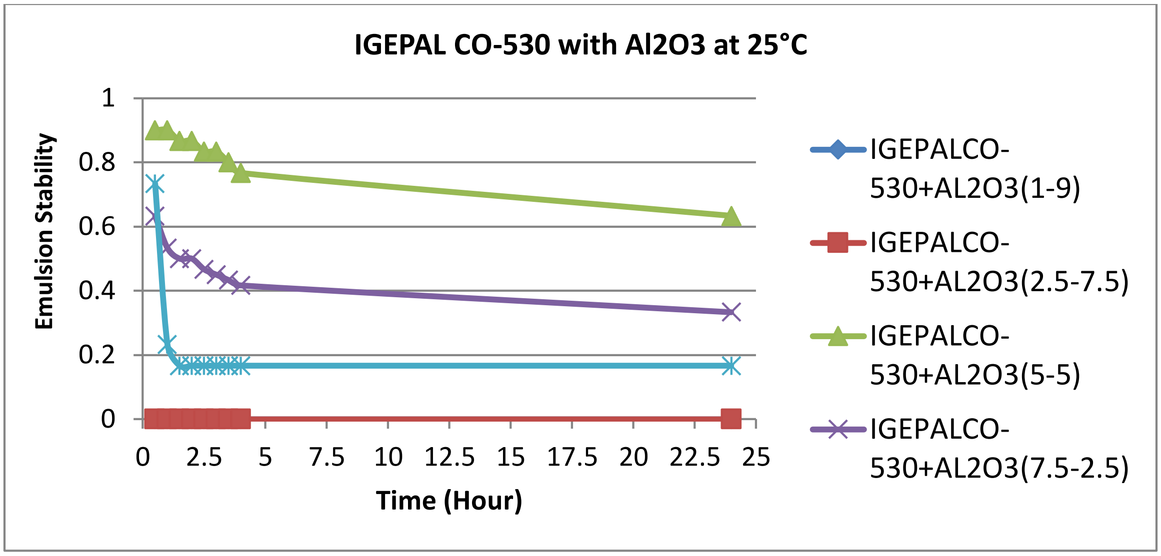
Crude oil emulsion stability vs. time of a nonionic surfactant (IGEPAPI CO-530)



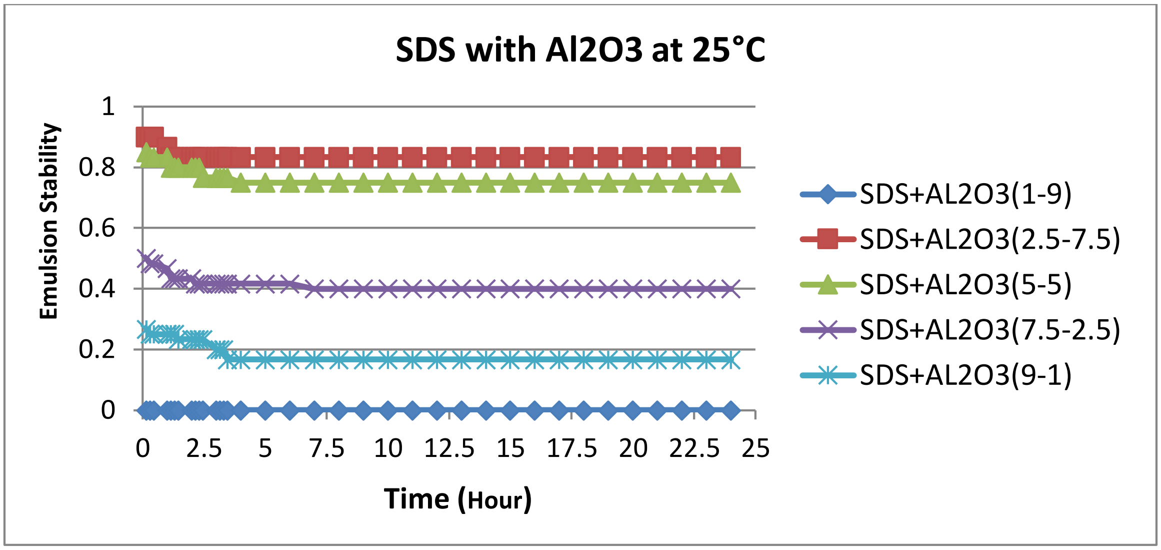
Crude oil emulsion stability vs.time of anionic surfactant (Dodecyl sulfate sodium, SDS)



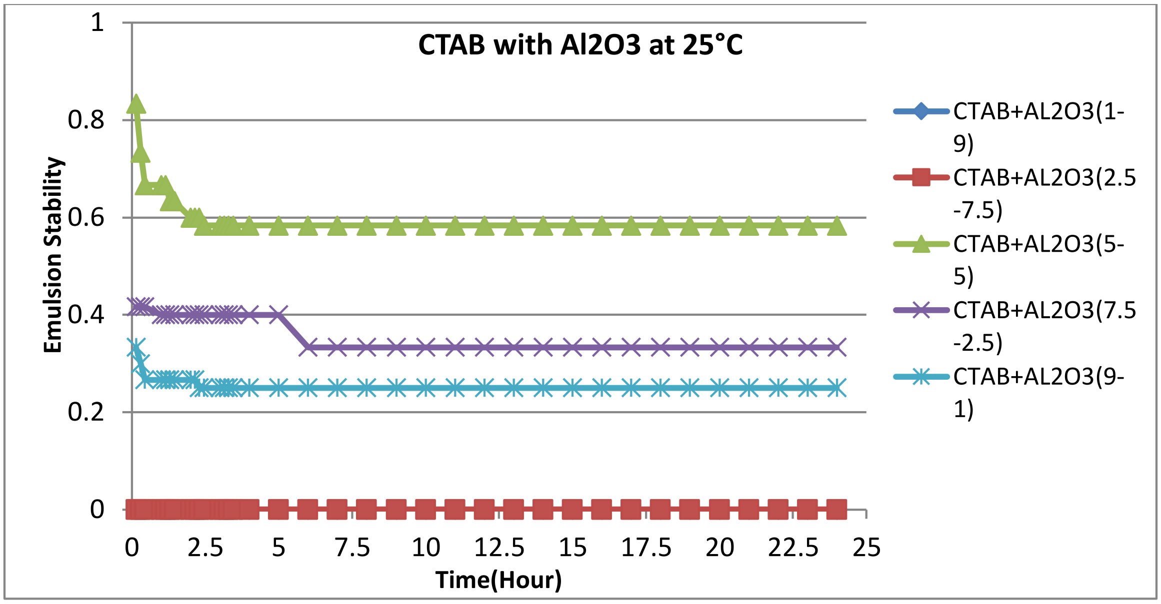
Crude oil emulsion stability Vs.time of a cationic surfactant (Cetylrimethyl ammonium bromide, CTAB)



Crude oil emulsion stability Vs.time of IGEPAL CO-530 with Al_2O_3



Crude oil emulsion stability Vs.time of dodecyl sulfate sodium with Al_2O_3



Crude oil emulsion stability Vs.time of cetylrimethyl ammonium bromide with Al_2O_3

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VITA

Zainab A Albdulmohsen is from Alhassa, Saudi Arabia. Zainab received her B.S. in summer 2013 in Petroleum Engineering as a major study and in Geology as a minor study from Missouri University of Science and Technology (S&T), Rolla, MO, USA. Zainab was interested in completing her education and decided again to join S&T for her master study. She was working in the lab as a researcher and her studies were focusing on the stability of crude oil emulsion using chemical products such as; surfactants and nanoparticles. In May 2015, Zainab received her Master’s degree in Petroleum Engineering from Missouri University of Science and Technology (S&T). For her future plan, she wishes to work as a professional researcher in one of the biggest research centers in the world.