Improvement in demulsification of Iraqi crude oil with water removal demulsifiers in oil fields

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Abstract. This study is targeted to present an overall survey on the most eminent techniques and methods that could be used in treating petroleum emulsions. Presently, the generally used way for handling petroleum emulsion is the process of chemical additives, known as demulsifiers. Considered nanomaterials are materials used for boosting oil restoration. This project discusses whether the collection of demulsifiers and nanomaterials can supply a much stable emulsion than demulsifiers only to obtain the best method of removal of water phase from crude oil emulsion mixture. Previous studies carried out two sections of experiments in their search. First were performed utilizing three kinds of demulsifiers; non-ionic, ionic, and cationic demulsifier. Trade names of those demusifiers were Poly Ethylene glycol (PEG), sodium dodecyl sulphate (SDS), and cetyltrimethyl ammonium bromide (CTAB). The other section utilized groups of self-same demulsifiers referred to at the top with nanomaterials were aluminium oxide (Al₂O₃) (60nm). These experiments were carried out using laboratory batch of homogenizer stirrer. These two sections were investigated with effect of different parameters such as; the kind of substance and the concentration or dosage of demulsifiers, various proportion of water to oil, stirring temperature, stirring speed of mixing, various of pH, TDS, on the efficiency of water removal. The sample test of case study used Iraqi crude oil (Basra & Kirkuk Oil fields. The results reached efficiency water removal of (86%) by (SDS) at optimum operating parameters, so, it is better than other chemical demulsifiers reaching the efficiency water removal (84%) by (CTAB) and (67%) with (PEG). However, the maximum efficiency improved within the range 88-90% of water separation from oil was obtained with using a blend of SDS with non-materials of (Al₂O₃) at optimum values of parameters.

Introduction

Crude oil originates from underground complex mix, which mostly includes hydrocarbons with some components of Oxygen, Sulphur and Nitrogen. It observed as a viscous liquid, which contains water and sediments content. In the event if both exist then it is required to remove wet from crude oil, which leads to abrasion of piping and fitting while dealing in oil refineries as well as affecting the goodness or quality of petroleum. The specification of crude is seriously affected by the existence of water. Oil seems at the better as it is very viscous or had quite low amount of water, moreover, the determination of water proportion is significant in gauging actual net proportions of real crude oil proportion in gross sales and exchanges (Williams, 1990). In command to reduce the output problems concerning oil emulsions and ecological care, industrial refineries labourers are required to ban the genesis of or destroy these emulsions (Sjoblom et al., 2007; Ramalho et al., 2010). Many techniques are presently available for breaking water-in-oil emulsions, including heating methods (e.g., gravity settling, mixing, ultrasonic, electrical fields, membrane segregation and chemical
demulsification...etc.) (Abdurahman et al., 2007; Djuve et al., 2001; Ekott and Akpabio, 2010; Guzmán-Lucero et al., 2010; Hajivand and Vaziri, 2015; Issaka et al., 2015; Yang et al., 2009). Crude oil is often gives output as a water-in-oil emulsion and the water ought to be extracted (down to a level of <1%), in an operation that is usually called demulsification or dehydration, which includes breaking down of the coalescence of water droplets and producing their segregation by settling. Physico-chemical cases, such as shape (i.e., the nature of the materials, temperature, and composition), are recognized to be attached to the emulsion specifications, especially its stability or constancy. Water-in- oil emulsions is a critical problem in industrial applications like the oil fields and oil refineries sector. Actually, emulsified water can corrode the equipment in refinery and oilfields and the water-emulsion can make many troubles in distillation columns, catalysts in reactor, exchangers... etc. (Issaka et al., 2015; Sullivan et al., 2007). In addition, water in oil emulsions usually displays viscosities higher than crude oil, so it increases the costs of pumping transporting oil in the pipelines from oilfields (Peña et al., 2005). Current research concentrates in the evaluation of expense-efficient and surrounding-friendly chemical demulsifiers described by more water removal capacity and demulsification average, so to fulfil the requirement of the crude oil companies (the extreme allowance water drop in crude oil is mostly 0.1–0.05%) (AbdulWahab et al., 2006; Cendejas et al., 2013; GuzmánLucero et al., 2010; Issaka et al., 2015). The emulsions can be found in food, medicine, pharmaceutical industries. It is significant to separate the emulsion in two continued phases to be capable to utilize the oil without any other additions (Schubert and Armbruster, 1992). Nano emulsions, have presented good outcomes for oilfield quality that attracted the oil producers in world. Nano emulsions have droplets ranging from 1–200 nm, these nanomaterials emulsion are stabilized with time and resistant in coalescence among droplets (Kong & Ohadi, 2010). Nanomaterials are able to enhance crude oil retrieval by improving oil specification (viscosity, density, demulsification improvement, and surface tension) (Ayatollahi and Zerafat, 2012). Therefore, the present work is aimed to study the best chemical demulsifiers and nanomaterials with varied operating conditions (temperature, salinity, pH, dosage and mixing time) to obtain high efficiency of water removal from crude oil.

Experimental Section

The experimental work was carried out on samples of Kirkuk and Basra crude oil used in Dura oil refineries. Crude oil samples of different water concentration are used in the present work. The first runs were carried out using crude oil samples. These samples were taken at different times according to supply to Daura refinery. Crude oil used with different water content (15, 20, 25, 30, 35 Vol. %) as analyzed by Chemical Engineering / University of Technology laboratories. Table (1) shows some of the Specifications of Kirkuk & Basra crude oil.

| Sample | Specific Gravity at 15.6 °C | API (ASTM) | BS & W content Vol.% |
|--------|----------------------------|------------|----------------------|
| Kirkuk | 0.847                      | 35.6       | 0.2                  |
| Basra  | 0.860                      | 32.0       | 0.2                  |

Three kinds of demulsifiers were utilized to run oil emulsion stability test with ratios at (1wt. %) in water consisting of (1wt. %) of NaCl. The mixture was added to (w/o) emulsion at different proportions of water. Their specifications are tabulated below in Table 2.
Table 2. Properties of the Demulsifiers applied.

| Name of Demulsifier      | Type of Demulsifier | Mwt (gm/mol) | Density (gm/ml) at 20 °C | HLB No. | Description       | Supplier         |
|--------------------------|---------------------|--------------|-------------------------|---------|-------------------|------------------|
| Sodium Dodecyl Sulfate   | Anionic             | 288.372      | 1.01                    | 40      | White Powder      | Aldrich          |
| Cetyltrimethyl ammonium bromide (CTAB) | Cationic | 364.45 | ---- | 16.0 | White Powder | Sigma-Aldrich |
| Polyethylene Glycol      | Nonionic            | 18.02 + 44.05n | 1.125        | 16.0 | White Gel        | Kavosh Kimia Kerman |

Nanoparticles (Al₂O₃)

The pure white powder consists of nanomaterials of alpha-phase aluminium oxide (Al₂O₃), having shape of aluminium oxide or alumina. The atoms domain of alpha aluminium oxide Nano powder, generally from (60nm) to as big as 10nm, is used, based on usage, purity criterions, and coating. In addition, there are numerous recognized and possible utilizations of aluminium oxide Nano powders. The nanomaterial size and specifications are listed below in Table (3).

Table 3. Specifications of the nanomaterials Applied

| Kind of nanomaterial | Particle size (nm) | Mwt (gm/mol) | Density (gm/ml) at 20 °C | Purity | Characterization |
|----------------------|-------------------|--------------|-------------------------|--------|------------------|
| Aluminium Oxide (Al₂O₃) | 60 nm             | 101.96       | 0.2- 0.4                | 99.0 % | White powder     |

Equipment

1-High Speed homogenizer
A high-speed homogenizer DIAX 900 homogenizer is a device that works with electric power; high-speed rotary stirrer (26000 rpm) designed with a high precision to tear the droplets to small sizes infinity small, which is used for the purposes of homogeneity of liquid. As shown in Figure 1.
2- Magnetic Stirrer and Electrical Balance.
A magnetic stirrer or magnetic mixer is a laboratory device that employs a rotating magnetic field to cause a stir bar (also called "flea") to spin high speed, thus stirring it. Also used to heated liquids, the revolving field may be formed either by a revolving magnet or by a set of fixed electromagnets, put under the vessel with the liquid. Economic magnetic stirrer with stainless steel heating plate RH basic 2 Ikamag was used for coagulants solutions preparations, with speed range (10-2000 rpm), as shown in Figure 2.

![Homogenizer Stirrer](image1)

**Figure 1.** Homogenizer Stirrer (Chemical Engineering Department, University of Technology)

![Magnetic Stirrer & Heater](image2)

**Figure 2.** Magnetic Stirrer & Heater (Chemical Engineering Department, University of Technology)

Also, an analytical balance (often called a "lab balance") is a class of balance designed to measure small mass in the sub-milligram range (0.1mg).

**Experimental Methods**

1- Used Demulsifiers (SDS, CTAB, PEG) / Crude Oil
A sample (50 ml) of crude oil was taken and washed by adding fresh water with different (15, 20, 25, 30, 35 Vol. %), at room temperature. pH and salt content (TDS) for each water sample was
taken and recorded. The readings were as follows: 1st mix. (100gm) from water, (1 wt. %) from NaCl, and (1 wt. %) sodium dodecyl sulfate (SDS); 2nd mix. (100gm) from water, (1 wt. %) from NaCl, and 1 wt. % cetyltrimethyl ammonium bromide (CTAB); 3rd mix. (100gm) from water, (1 wt. %) from NaCl, and (1 wt. %) Poly Ethylene Glycol (PEG).

2- Used Surfactants (SDS, CTAB, PEG) / Nanoparticles and Crude Oil

1st Mix. (100gm) from water, (1 wt. %) from NaCl, (1 wt. %) from sodium dodecyl sulfate (SDS) and (1 wt. % Al₂O₃). 2nd mix. (100gm) from water, (1 wt. %) from NaCl, 1 wt. % cetyltrimethyl ammonium bromide (CTAB), and (1 wt. %) from Al₂O₃. 3rd Mix. (100gm) from water, (1 wt. %) from NaCl, (1 wt. %) from Poly Ethylene Glycol (PEG), and (1 wt. %) from Al₂O₃. These sample recordings are shown in Figs 3, 4, and 5.

A high-speed homogenizer in Fig. (1) is used for mixing and heating all solutions to keep the system at a fixed temperature. The quantities were mixed to get homogenous solutions. Each demulsifier was added to watery solution in the graduated beakers according to the proportions tabulated below in Table 4.

| No. | Water % | Oil % |
|-----|---------|-------|
| 1-  | 10      | 90    |
| 2-  | 75      | 25    |
| 3-  | 25      | 75    |
| 4-  | 50      | 50    |
| 5-  | 90      | 10    |

Crude oil was blended to the watery mixture in graduated beakers depending on the proportion tabulated in Table (4). Beakers were mixed both by Homogenizer Stirrer at (10000 rpm) for about three minutes until it was mixed. It was a homologous phase, and the temperature was stationary at 25°C. Beakers that included the blend were put for 24 hour at (25°C). Water levels and remarks were registered for the mix. One phase emulsified for 24 hours, then the temperature was raised to 40°C for another 24 hours, a volume of water segregation from the emulsion system was spotted. Water segregation versus time was plotted, which is defined as:

\[
\left( \frac{V_1}{V} \right) = \frac{V_1}{V_2} \times 100
\]

Where \( V_1 \) is the volume of detached water and \( V_2 \) is the authentic volume of water formed.
Figure 3. The results of (SDS) emulsion at (25°C, 24 hr.), from right to left: (10:90), (25:75), (90:10), (75:25) & (50:50)

Figure 4. The results of (SDS) emulsion with Nanoparticle (Al$_2$O$_3$) at (25°C, 24 hr.) from left to right: 10:90, 25:75, 90:10, 75:25, & 50:50

Figure 5. The results of (CTAB) emulsion with Nanoparticle (Al$_2$O$_3$) at (25°C, 24 hr.) from right to left: 10:90, 25:75, 75:25, 75:25 & 90:10
Results and Discussion

1.1. Effect of Temperature

In the separation process at four different temperatures, samples were prepared and demulsifier was added into every one of them. It was then achieved at various temperatures (25 °C, 40 °C, 60 °C, and 80 °C) at fixed pH of 7.0 for 8.0 hour. Figure 6 shows that increasing the temperature leads to an increase in the water removal dramatically because heat affects the emulsifier and makes it easy to merge droplets by decreasing the surface tension between Oil and water. Reduction of the viscosity of the oil, therefore, causes the ease of falling drops at a shorter time than if its Viscosity is higher, (Hajiv and Vaziri, 2015). Also, its shows that the apparent viscosity increases with lower temperatures. The reason is that as the temperature increases, the interfacial tension decreases and reduces the apparent viscosity. Fig. 6 shows that the low temperature also affects the degree of separation in the emulsion. Temperature is known to speed up all kinds of chemical reactions, and it is clear that it also speeds up the rate of separation in emulsions. Another observation is that even though both water content and temperature clearly have a strong impact on emulsion viscosity, it seems like water content is the dominating factor.

![Figure 6. Impact of Temperature on Water removal from Crude Oil with Different Demulsifiers at (H2O=25%, pH= 7.0, 25 vol. % Demulsifier, Time Settling= 8.0 hr.)](image)

1.2. Effect of Salinity

Salt looks to have a counter impact on the emulsion stabilization. So for checking, specimens of emulsion of a variation of salt quantities (75, 365, 670, and 1700 ppm) were prepared for checking the water-in-oil emulsion’s performance in more concentration. Figure 7 shows the result of adding salt (sodium chloride) to crude oil emulsion. The best removal of water was carried out with the specimen containing the minimum concentration at (75 ppm) NaCl (65% removal from total water) in contrast to the concentration at (1700 ppm) NaCl (85% removal from total water). This event could be expressed by changing the interfacial film movement (P. Hajivand and A. Vaziri, 2015). It was found that demulsification operation was carried out with high efficiency for emulsions consisting of high water contents with NaCl. Raising salt concentration leads to W/O droplets lowering in size so, the existence of salt had a reverse impact on emulsion persistence. In order to analysis this, four specimens of emulsion of various salt contents were selected with chemical (SDS) its medium rate and output of separation and that showed the water-in-oil emulsion’s behaviour more accurately. Fig. 7 views the result of adding non-organic salt (sodium chloride) to the crude oil emulsion. As expected, the existence of non-organic cations in the system had a reverse impact on emulsion speed; so, the better segregation of water was accomplished for the sample containing high rate.
Figure 7. Effect of Water Separation with Various Concentrations of Nacl (ppm) in Crude Oil & Demulsifier (Sodium Dodecyl Sulfate) at (Settling Time = 24 hr., H₂O= 25%, Temp. =25°C)

1.3. Effect of the demulsifier Type
In this search, the focus was on the selection of chemicals which keep this suitable functionality, and stabilization of emulsion, a base step for removing water from oil, able to be achieved by adding chemical materials, known be demulsifiers, the chosen chemicals and operations had suitable performance. Figure 8 shows that, the focus was on the selection of chemicals, for example, (SDS, CTAB, Di, and Poly glycols) which alter the density and polarity of the water phase, however, most of the used demulsifiers speed up the average flocculation and accumulation, which leads to a quicker removal of water from the oil (ZainabAlabdulmohsen,2015). And, Sodium Dodecyl Sulphate (SDS) is much more cost effective. Therefore, it shows faster removal of water in contrast to polyethylene glycol and Cetyltrimethyl ammonium bromide (CTAB). The check operation was achieved at optimal pH values and temperatures to simplify the breakage of the emulsion. The outcome using water-soluble demulsifiers are shown in Fig. 8. So, for the literature, an oil-soluble kind of demulsifier has high impact in W/O emulsion resolution. This is indicates that oil is the continuous phase, whilst water is the dispersed phase. So, the demulsifiers dissolve in the continuous phase at minimum mass transfer impedance at optimum temperature, and the dispersion of the demulsifiers injected in the system is easier. The outcome for water segregation utilizing oil-soluble demulsifiers is shown in Fig. 8. SDS gives the better outcome as compared with the others. Its function in resolving emulsion troubles is of high impact by 85% (v/v %) and higher segregations, come after with (CTAB) 74% (v/v %) and (PEG) with 62% (v/v %) and (DEG) 52% (v/v %) separation, which shows the lowest separation ability, which proves that oil-soluble demulsifiers are high impact than water-soluble demulsifiers.
1.4. Effect of Alkalinity (pH Value)

The impacts of pH on emulsion specifications were estimated by addition of (HCl) or (NaOH) solutions to the aquatic phase prior to emulsion preparation. Sakka (2002) and Yang et al. 2007, proposed that raising pH (alkalinity) will award high stabilization at the emulsions. Raising pH will boost more affinity of demulsifier molecules toward accumulation, it arises from its high stability emulsions. Many researchers detected that in the state of oil-in-water emulsions, the stabilization was raised as the pH was rising from 4 to 6. While for high pH values of 8 – 10 in selected water-in-oil emulsions, adjusting the pH of emulsions seems indeed to be effective in resolution of water in-oil emulsions (P. Hajivand and A. Vaziri, 2015). Figure 9 optimizes the outcomes of pH modifications on the capability of the demulsifier to removal water from (w/o) emulsions below different pH values. The emulsions are stable in higher pH value (pH= 9.0) and looked less stable, in moderate pH values (pH= 7.0, pH= 6.0).

Figure 8. Effect of Demulsifier Types & Nanoparticle (Al₂O₃) with Concentration (0.3 vol. %) on Water Separation in Crude Oil at (Temp. = 25°C, pH= 7.0, H₂O= 30%)

Figure 9. Water Separation vs. Time at Different pH Values of water Crude Oil with (SDS) at (Temp. = 25°C, H₂O= 30%, (SDS) = 0.3%)
Choice of a device demulsifier is setup on capability of the demulsifier to segregate water from an emulsion system. Emulsions can be more treated by the addition of chemical destabilizers. These surface-active chemicals are adsorbed in the water-oil interface, tear the film surrounding water drops, and displace the emulsifying factors back into the oil. Therefore, there is a principle in this case that states, “the little water proportion in emulsion is more complex to separate”. Increasing the chemical dosage of demulsifier increases the dehydration efficiency; therefore, an increase in the ratio in an irregular or random way adversely affects the mixture (P. Hajivand and A. Vaziri, 2015). Figure 10 illustrates the Water separated efficiency. With the proportion of demulsifier added and Water separated efficiency increased, reached a maximum, the demulsifier acted to balance the effectiveness of the demulsifier factors, release much water drops from the encirclement interfacial film. As a result, (SDS) was chosen for this test, as shown in Fig. (10). The dosages used in this test were 0.15 vol., 0.25 vol. and 0.30 vol. percentage of additives and empirical conditions were preserved constant at optimal accounts. This experiment targets to obtain the relation between potion and emulsion resolution activity effect. As Figure 10 shows, water separations were 85 % v/v in (0.30 vol.). The lowest dosage gave 84% v/v separation in (0.25 vol.), and 74% v/v in (0.15 vol.). The employer can select the suitable equilibrium between the raised cost of utilizing a higher dosage to save time and devices ability and a less dosage and cost of demulsifier, a higher investment in the capacity of equipment. Thus, the dosage plays an important function in the demulsification operation; for higher dosages the rate of coalescence of droplets rises because of interfacial film thinning.

![Image of Water Separation vs. Different Demulsifier Dosage (SDS) in Iraqi Crude oil with (Al₂O₃) at (Temp. = 25 °C, pH= 7.0) with a graph showing the effect of demulsifier dosage on water separation.

1.6. Effect of Mixing Time

Figure 11 expresses the impact. The Water separation efficiency rises sharply with rising blending time. The efficiency raise reached to the optimum point at (6 minutes) and then Water removal efficiency started to break down. This is on account of the phenomenon of emulsion kind inversion when an emulsion immediately changes kind, from water-in-oil to water-in-oil-in-water. The time needed for stability of emulsion to reverse relies on the mixing speed, temperature, the demulsifier types, and its concentration (Sun and Shook 1996). The effect of the time of mixing on the particle sizes in emulsion is great. Because increasing time of mixing may lead to increase in the interference between the particles of the emulsion, which leads to increased viscosity with increasing time of mixing. Also, increasing the mixing time leads to double emulsion (o/w/o/w). Rising of the period of blending of w/o emulsions results in lessening of rate of apportionment of dispersed water droplets and increasing of both emulsion viscosity and stability on account of making better particle interactions as an outcome of better interfacial area. Most specifically, in systems containing little dispersed droplets, the colloidal surface forces and Brownian motion forces dominate over the hydrodynamic forces.
1.7. Effect of Nanoparticle Materials

The shear behavior was acquired as function by nanomaterial proportions, and shear rate (ranging from zero to 120 s⁻¹). Tests results referred that increasing the concentration of nanomaterials in the mix leads to a decreasing viscosity as compared with nanomaterial-free crude oil. Results referred that addition of nanomaterials raises the heavy oil movement and Works on boosted crude oil recovery of approximately (16%). Figure 12 shows the outcomes of the rheological search for specimens with variation dosages of nanomaterials (Norman, Oklahoma, 2016). The nanomaterials selected to valuate effectiveness as viscosity decreasing factors was (Al₂O₃), since they were shown to raise the adsorptive ability concentration of nanomaterials in the fluid, viscosity decrease in more than one measure for all shear averages valued. Nanomaterials able also stabile the emulsion ago the big surface area able supply reduces interfacial tension by immiscible phases. And, the Nano-magnitude of the nanomaterials is two or three instructions smaller than the pore throat that has created the Nanomaterials stable emulsion long-distance immigration in the practical tank. Or else, there are two features by utilizing nanomaterials. First, the Nanomaterials enhance the emulsion by intensifying it. Secondly, the Nanomaterials are able to decrease the sucking of demulsifier by the porous media and to directly decrease the use of demulsifier in the full chemical enhanced oil recovery operation.
Figure 12. Effect of Nanoparticles (Al₂O₃) on Viscosity Crude Oil Emulsion with Different Dosage (SDS) at (pH= 7.0)

1.8. Effect of Settling Time

The gravity effect is so important that when talking about water separation from crude oil, this operation would be easier when there is a density variation between crude oil and water density, and the separation will be harder if this difference is decreased. Water droplet size affects greatly on this separation, if their diameter increases; the downward velocity will increase also, so the coalescence of these small water droplets to bigger droplets will lead faster separation and settling. The time was different from one to 24 hr. Figure 13 shows an astounding rise of Water cut efficiency. The settlement period agent was different from 1.0 hr. to 24 hr.; generally, there is a level off toward the end of (24 hr.). The Water cut efficiency rises as an outcome of the boost of gravity difference between oil and water. It is shown in Fig. 13. At higher settling times (20-24 hr.), the efficiency arrived at an ultimate value of 85.0%. The behavior is asymptotic in water removal because it is arriving at a point where water droplets are very small to not be separated. (Elkamel, Al-Sahhaf, 2003).

Figure 13. Effect of Settling Time on Water Separation in Crude Oil Emulsion with Nanoparticles (Al₂O₃) at (Temp. = 25°C, pH= 7.0, H₂O= 30%)
1.8 Comparison between the Optimum Commercial Demulsifier Water Separations:
Comparison between the commercial demulsifiers from where efficiency of separations, the commercial demulsifier from water separation yields was utilized in this test in contrast to the results of other demulsifier. They involve Tween 80, (PEG), (DEG), (SDS) and (CTAB) supplied by different Co. Emulsions were prepared from Crude Oil from Iraqi field oil in (Basra & Kirkuk oil field). Figure 14 shows the result of water removal by applying (0.10 - 0.35) the volume proportions of Demulsifiers. As Fig. 14 Shows, (SDS) impacted better segregation, which is nearly 86% (v/v), accompanied by (CTAB) at 84%, (PEG) at 67% and (DEG) at 55%.

![Comparison between Demulsifier Water Separation from Crude Oil](image)

**Figure 14.** Comparison between Different Demulsifier water Separation from crude oil at (Temp. = 40 °C, pH= 7.0, H_{2}O= 30% & Dos. = 0.3 wt. %)

Conclusions
1- CTAB is a cationic demulsifier, which could form emulsion for long term and cannot be separated with higher temperature.
2- Polyethylene glycol (PEG) is a Nonionic demulsifier that could form more emulsion stability with (Al_{2}O_{3}) at temperature (25 °C).
3- The efficiency of (SDS) with (Al_{2}O_{3}) is higher than (CTAB) and (PEG) in Stability.
4- (SDS) could form more stable emulsion by addition of (Al_{2}O_{3}) nanomaterials during oil phase.
5- Chemical demulsifiers have the ability to separate little concentrations of water in less than (30 vol. %).

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