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An Experimental Study on the Application of Ultrasonic Technology for Demulsifying Crude Oil and Water Emulsions

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Abstract

An emulsion is the mixture of two immiscible fluids, where one fluid appears as droplets within another. In the oil and gas industry, produced crude oil generally comes with an appreciable amount of water within it in an emulsified form. Before produced crude oil can be prepared for purchase, the water associated with it must be removed. A process known as demulsification is required in order to separate an emulsion into its two phases. In the industry, a number of demulsification techniques are already present; these include thermal, mechanical, chemical, and electrical techniques.

Crude oil and gas produced from wells originally come with water, salts, and volatile gases such as oxygen, carbon dioxide, and sometimes hydrogen sulfide, etc. Hence, the petroleum mixture needs to be refined-water, salt, and non-hydrocarbon gases to be separated from the mixture, in order to meet certain oil and gas specifications (which state the maximum concentrations of such contaminants) and make it ready for purchase and transportation.

Sonication provides a cheap, simple, and harmless (as it involves mainly the propagation of sound waves) way of separating crude oils from water droplets via demulsification. In addition, if needed, it can be used for emulsification processes as well. Hence, a study of sonification as a way for crude refinement or chemical mixing has important implications for the oil and gas. This investigation proposes the use of ultrasonication as a new and cost-effective technique to aid in the demulsification of crude oil emulsion. The effectiveness of this technique was gauged through its comparison to the already present methods in the industry. Based on the investigation it was found that centrifuge served as the best demulsification method for it reduced the turbidity by 86%. In addition, the reduced turbidity achieved with proposed ultrasonication method ranges from 20%-60%.

Keywords: Ultrasonic; Crude oil; Emulsions

Significance

In the production of crude oil, usually a significant amount of water is also produced. Many times, this water and oil mixture is in the form of emulsions. Emulsion separation is very time consuming, requires additional surface facilities and can be very costly. Any new method that can increase the efficiency of this process and or provide a cheaper method would be very much helpful to the petroleum industry.

Objective

The objective of this research is to investigate the potential use of ultrasonication as a new and cost-effective technique to aid in the demulsification of crude oil emulsion. This research is looking at various existing techniques and will focus on the use of ultrasonic waves as a potential technique for emulsion separation.

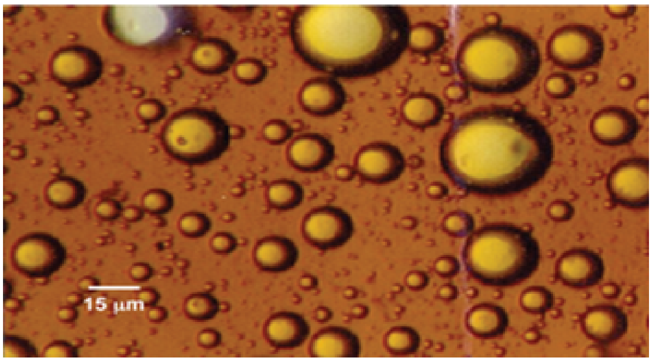

Figure 1: Photomicrograph of a W/O emulsion and a W/O/W emulsion.

Introduction

More than thirty percent of all crude oil produced in the world comes to the surface with an appreciable amount of water in an emulsified form [1]. A recurring issue in the oil industry is the separation of water from produced oil, its significance as an issue "is shown by an estimated 15-20 million dollars expended for chemicals each year to treat the world's oil production" [2]. An emulsion is defined as the dispersion of one liquid as droplets in another immiscible liquid. The water-in-oil emulsion (W/O) that occurs because of crude oil production is the most common oil emulsion that is discussed in the industry and is the focus of this investigation; however, oil emulsions can come in many forms. The second most common form of oil emulsions is oil-in-water, sometimes referred to as reverse emulsions (Figure 1). Emulsions can also occur in more complex manners such water-in-oil-in-water, illustrated in Figure 2, where the droplets themselves house a third immiscible liquid.

In order to make the produced crude oil ready for purchase and transportation, the crude oil needs to be refined – water, other

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nonpetroleum fluids, and solids to be separated from the mixture. Sonication is a cost-effective, simple, and safe (since it only involves sound wave propagation) means of separating water droplets from crude oil (demulsification). It may even be used for emulsification purposes as well. Therefore, sonification has important implications for the oil and gas industry.

We begin by discussing the characteristics and stabilizing components of emulsions. Second, we describe demulsification and the factors that affect such process. Third, we present the current demulsification methods used by the oil and gas industry. Fourth, we cover basic theory behind waves and sounds, and sonication. Finally, we examine sonication as means of demulsification.

Emulsion

Emulsions can be characterized by their kinetic stability, their stability over a period. In other words, emulsions are grouped based on their separation rates. Based on kinetic stability, emulsions can be divided into three groups: loose, medium and tight emulsions. Loose emulsions can be thought of as highly unstable with a low separation time while tight emulsions are very stable with relatively high separation times. Kinetic stability of an oil emulsion is a result of two parameters: droplet size and the interfacial film surrounding the droplets. Droplet size is quite intuitive as smaller droplet sizes are a clear indication of a better size fluid and therefore a more stable emulsion. Likewise, the interfacial films that surround water droplets protect water droplets from combining with each other and thus increasing the emulsion stability. More viscous interfacial films lead to more stable oil emulsions. The interfacial films around the water droplets play the largest role in creating a stable oil emulsion. Interfacial films with higher viscosity work the best in preventing the emulsion in separating. These films can be enhanced with the addition of an emulsifying agent. Emulsifying agents are generally naturally occurring and come mixed with the produced crude oil. They can be divided into two main categories: surfactants and fine solids. Surfactants work by attaching to the oil water interface, due to their unique affinity to both oil and water, where they form an interfacial film. Examples of surfactants are asphaltenes and resins. Fine solids work to stabilize emulsions by providing a mechanical barrier in between the droplets in addition to the interfacial film. These solids are most effective when they are smaller than the dispersed droplets. Examples of fine solids found in crude oil emulsions include clay, sand, and silt.

Given a mixture of two immiscible fluids, mechanical agitation, or disturbance, causes dispersion of one phase as tiny droplets (dispersants) throughout the other continuous liquid phase, and subsequently the formation of the interfacial films of surfactants around the dispersed droplets for stabilizing an emulsion. Hence, sound wave propagation, which causes agitation via pressure fluctuations, can be used for emulsification purposes such as mixing viscosifier and oil into water-based drilling muds to obtain appropriate rheological properties (Figure 3).

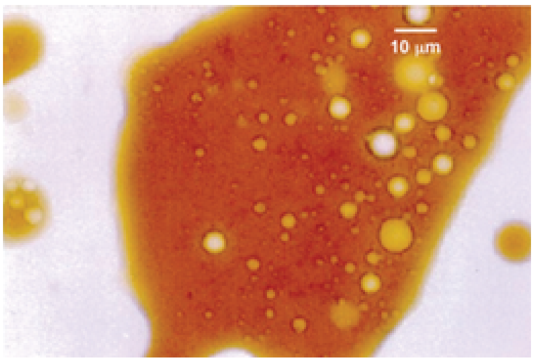


Figure 2: Photomicrograph of a W/O emulsion and a W/O/W emulsion.

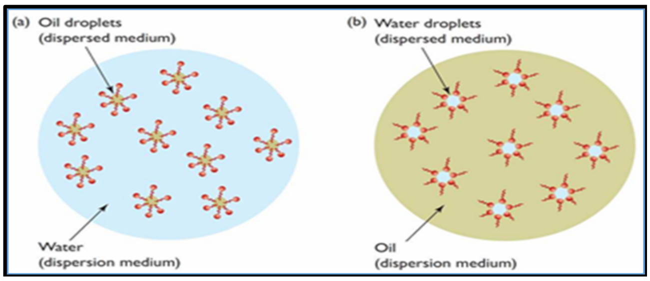


Figure 3: Types of emulsion.

Demulsification of crude oil (water-in-oil emulsion)

Demulsification is the separation of the emulsion into its separate components (water and oil). Demulsification as a treatment process is defined by the:

1. Rate of separation
2. Remaining water/salt content in oil.
3. Remaining oil content in water.

Ideally, an optimum demulsification method would separate the crude oil while maximizing on these three parameters. Reduction of the rate of separation will aid in maximizing profits by increasing the efficiency of the treatment process. Moreover, the goal of any demulsification is to eliminate the water in oil emulsion that occurs as part of the desalting process (intentional mixing of crude and fresh water) as well as the emulsion with the water content that comes naturally with produced crude. The separation of the water droplets containing dissolved salt is ideal in avoiding production problems including "corrosion, scale accumulation, lowering of activity analysts and plugging or fouling in pipeline [3]. Moreover, demulsification is critical in achieving a marketable standard of crude oil. Finally, ideal demulsification would see that the separated water phase is composed of low oil content so that its disposal or reuse satisfies the environmental standards established by the industry.

Demulsification can be defined as breaking emulsion kinetic stability. As a result, demulsification can be said to occur in a two-step process consisting of flocculation and coalescence. Flocculation involves the gathering of water particles to near proximity of each other, while coalescence occurs when the individual films holding the water droplets break to allow the droplets to combine and culminate into bigger drops. Neither of these processes occurs faster than the other; however flocculation general must occur first before coalescence. As a result, in designing a demulsification program one must subject the emulsion to a combination of treatments that enhance both the flocculation and coalescence of the water droplets. Table 1 provides a list of the factors that affect both of these processes'. It is important to note that a high flocculation will aid in enhancing coalescence.

Curent demulsification techniques

Demulsification programs are very specific to the oil field and the crude oil that is produced. However, based on the factors presented in

Factors that enhance flocculation	Factors that enhance coalescence
Amount of water in the emulsion	High rate of flocculation
High temperatures	Low oil viscosity
Low oil viscosity	Chemcial demulsifiers
Density difference between the two fluids	High temperatures
Electrostatic field	

Table 1: Factors that enhance the rate of flocculation and coalescence of water droplets [4].

Table 1, there are a number of common types of methods to treat an emulsion. Emulsion treatment methods can be grouped into four main categories; these are thermal, mechanical, electrical, and chemical.

Thermal: The application of thermal energy is probably the simplest method of demulsification used in the application of thermal energy is probably the simplest method of demulsification used in the industry. Thermal energy reduces the viscosity of the oil and increases the water settling rates." [4]. In addition, the increased temperatures "also result in the destabilization of the rigid films caused by reduced interfacial viscosity" and in some cases even rupturing of the films which in turn aids in the coalescence of the water particles [4]. The increased temperature also leads to an overall increase of kinetic energy of the particles leading to a higher coalescence frequency of the water droplets because of their increased mobility. In other words, heat accelerates the demulsification process in a variety of ways, however it is important to note that heat alone is not known to completely separate an emulsion on its own especially when dealing with stable emulsions. In fact, the application of thermal energy to an oil emulsion can lead to some undesirable effects, such as a reduction of the API gravity, and an "increased tendency for corrosion and scale deposition in treating vessels" [4]. In light of this, it's always important to weigh the costs of heating an oil emulsion against the adverse effects of the treatment in deciding whether thermal energy is an economic demulsification technique for the given emulsion case.

Electrical: An electrical demulsification method that is commonly used in the industry involves the application of an electric field perpendicular to the direction of flow. The associated charge of the water droplets along with the electric field can result in one of three main phenomena in occurring, all of which aid in destabilizing the emulsion. These phenomena include:

- The electric field aligns the water droplets based on polarity, thus positive ends of droplets are brought next to negative ends of other droplets. Through electrostatic attraction these droplets come closer and closer together eventually leading them to coalesce
- The polarity caused by an electric field can also make water particles attracted to an electrode. This can lead to large amount of water particles gathering and collecting in one-area allowing forming larger water droplets and eventually settling and separating from the oil.
- When an AC field is applied to an oil emulsion that effect is a weakening or even rupturing of the film surrounding the water droplets. This occurs because of the cyclic nature of an AC electrostatic grid. During the high voltage, phase of this cycle the water droplets are elongated along either end of the droplet, when the cycle returns back to the low voltage phase the droplets snap back to a spherical shape.

Regardless of which of the three phenomena occur when an electrostatic grid is applied to an oil emulsion the result is an acceleration of the demulsification process. An umbrella term for all three phenomena is known as Electrostatic Dehydration. This method is best used in combination with chemical or thermal techniques [5].

Chemical: The most common of the four techniques is the use of chemicals, known as demulsifiers, in treating an oil emulsion. Demulsifiers work by breaking the interfacial film protecting the trapped water droplets. In order to optimize the use of demulsifiers a number of factors are considered before the addition of a demulsifier into a crude oil emulsion including, proper selection of demulsifier accurate dosage, and adequate mixing time.

Demulsifier selection

In general, demulsifiers can be broken down to 3 critical components; solvents, surface-active ingredients, and flocculants. Over the years the selection of chemicals used as demulsifiers in the industry has vastly increased. With such a large number of options available it's very important that the right chemical combinations are selected in order to ensure the best results. Demulsifier selection begins first with characterization of the crude oil emulsion in question; breaking down the type of crude oil/brine and all the contaminants/solids that make up the emulsion. Next, the production conditions of the crude oil emulsion should be considered including operating temperatures, production rates, treating vessel characteristics etc. Finally, it's important to consider any past record on the performance of the demulsifier with other crude oil emulsions. Demulsifier selection is always a unique experience and can vary considerably between different well case studies.

Demulsifier dosage and mixing time

Similar to demulsifier selection, demulsifier dosage is unique and depends entirely on the chosen demulsifier and the properties of the emulsion. Proper dosage is highly important in ensuring an optimized treatment of the emulsion. In fact, while too small of a demulsifier dosage will leave an emulsion as is, too much demulsifier can actually result in a more stable emulsion. Again, due to the large number of factors that can vary significantly between scenarios it's important to conduct accurate testing with the case in question in determining the optimum dosage. Equally as important is ensuring the chosen demulsifier is mixed adequately into the emulsion. Demulsifiers work by attaching to the oil and water interface and therefore the mixture has to be subjected to enough agitation in order to allow the demulsifier to reach the interfacial film. However, an unnecessary amount of mixing can reverse the effects of the demulsifier and stabilize the emulsion. Therefore, it's important to monitor the mixing of the demulsifier into the emulsion and decrease agitation significantly once the demulsifier begins to break the emulsion.

Mechanical

There are a variety of emulsions treating equipment that can be used to emulsify an oil emulsion. Some of the common ones used in the industry include: free knock out water drums; two/three phase separators, desalters, and settling tanks.

Free knock out water drums

Aside from taking out water from crude oil, free-water knockout drums can also separate associated gases. This equipment usually supplements main demulsification equipment.

Three-phase separators

As its name suggests, the three-phase separator separates the produced crude oil into three phases existing within the crude-oil, water, and gas. They can be set up vertically or laterally. The separator is appropriately assigned set retention time for sufficient separation at any required throughput rate. The separator can have components of heat section, wash water, filter section, stabilizing section, and electrostatic grids. It is no surprise then that large varieties of separators are in used today. What specific separator design would a demulsification task require is a complicated engineering task, which must consider many factors?

A flexible separator design, which allows for modification, is most preferable. Pressures and temperature conditions, water cuts, and oil and brine compositions change over the life of the field; hence, a well-designed separator is one that allows for adaptations to these fluctuating operation conditions.

Coalescer packs are a good way to increase separator efficiency. These packs increase the amount of fluid traversing through the separator. Water droplets coalesce as the emulsion rolls or wipes through the packing. Installation of spreaders, which increase droplet collision frequency, are also viable choice for improving separation efficiency.

Desalters

Separators usually are not enough to reduce the amount of water in the crude oil to marketable standards. In addition, some salt must be taken out as well. Hence, a desalter must be employed for removing the remaining water and salts. Desalters usually employ combinations of settling, chemical addition, and electrostatic treating. The time for settling depends on oil specification. With chemical addition, fresh water is also added for decreasing salt concentration (i.e. dilution) in the effluent water and crude. A one-stage desalter schematic is shown in Figure 4.

These are some of the most common techniques used in the industry to achieve demulsification. It is interesting to note that some techniques have overlapping components. For instance, desalters and three-phase separators may both employ a settling component and an electrostatic treating. Generally, a combination of these techniques is used in conjunction with each other to develop a complete demulsification program.

Basic wave theory, sound, and sonication

Harmonic motion: Harmonic motion is any repeating and oscillating motions. For instance, the back and forth motion of a mass attached to a fixed spring which is stretched, the swinging of a pendulum, and the motions of particles in a vibrating mass are all harmonic motions. An object in harmonic motion is an oscillator. A harmonic motion results from two things: A restoring force and inertia. A restoring force acts on the object in the direction towards equilibrium or decreasing potential energy. Inertia is the object's resistance to motion, or more specifically, the resistance to return to equilibrium. Hence these two-act opposite to each other in harmonic motion.

Period and frequency are the important parameters for describing the harmonic motions. Period T is the amount of time given in seconds (s). For one cycle of the motion to occur, and frequency f is the number of cycles per unit time and is given in Hertz (Hz=1/s). Also, they are related as:

$$T=1/f$$

When the restoring force is directly proportional to the object's displacement, the motion is called simple harmonic motion (SHM). This can be stated as.

$$F=-kx$$

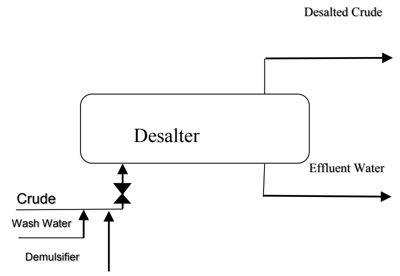


Figure 4: One-stage desalter.

The minus sign is due to the restoring force acting opposite the displacement. The examples of harmonic motion given previously are all simple harmonic motions, although not necessarily for the vibrating mass case. Simple harmonic motions are described mathematically as sinusoidal functions:

$$x(t)=A\cos(\omega t+\varphi)$$

Where x, t, A, ω, and φ are the displacement of the object, time, angular frequency, the constant called amplitude, and the constant called phase angle, respectively. The angular frequency is defined to be:

$$\Omega=2\pi/T$$

Amplitude is the maximum displacement from equilibrium position of the object. For example, the amplitude for a swinging pendulum is the pendulum's maximum vertical displacement from its position when it is not in motion.

Wave

Waves are disturbances or oscillations carrying energy, and propagates through space (and are then electromagnetic waves), light for instance or mass (and are then called mechanical).

Mechanical waves

Mechanical waves are disturbances that propagate through matter, called the medium. As mechanical waves propagate continuously through the medium, the particles move roughly in a simple harmonic motion. Mechanical waves are generated for industrial or experimental purposes using simple harmonic oscillators.

The propagation speed of mechanical waves, or wave speed v, increases with higher restoring forces F, and decreases with higher inertia μ. These relationships are more precisely given by:

$$V= F/\mu$$

Transverse waves

When the particles move perpendicular to the direction of wave propagation, the wave is transverse. Consider a string is fixed at one end to a wall and tied to a continuously moving oscillator in the other. Then as the wave generated by the oscillator propagates through the string, each of the string's particle move up and down, perpendicular to the wave direction. The collective motion of the particles results in a sinusoidal waving string. This is appropriately called a sinusoidal motion.

Longitudinal waves

When the particles move parallel to the direction of wave propagation, the wave is longitudinal. When a longitudinal wave propagates through a medium, particles move in or opposite the wave direction, and causing alternating regions of unidirectional rarefaction, low pressure, and compression, high pressure.

Sound

Sounds are longitudinal mechanical waves. These are micro fluctuations in pressure that travel through a medium, commonly air. Our sensitive ears can detect these pressure fluctuations, which we then perceive as sound. The human ears can detect sound with frequency ranging from 20 Hz to 20,000 Hz, called the audible range. A sound with frequency higher than 20,000 Hz are called ultrasonic.

Sonication

Sonication is simply the propagation of sound waves into a substance for agitation purposes. Ultrasonication uses sound waves of frequency above 20 KHz (ultrasonic) and is commonly used. A sonication equipment may comprise of an electricity generator that generates AC electricity, a transducer that converts the AC electricity into mechanical vibrations, a transformer that amplifies this vibration, and a tool tip which makes the amplified vibration energy available for application (hence, tool tip is the oscillator to be applied) [1].

Demulsification using ultrasonification

Demulsification can be achieved using ultrasonification. Currently, there is not one unanimously agreed mechanism for which ultrasonification causes demulsification, although reasonable explanations have been proposed. The following is such an explanation [1]:

1. Ultrasound waves of high intensity produce cavitation or regions of free fluid spaces in the liquid.

2. Cavitation leads to rapid fluctuation of pressure.

3. Relatively large masses of vacuous, small bubbles form.

4. Bubbles grow until it reaches critical size.

5. Bubbles implode and generate intense shockwaves leading to high temperature and microstreaming of the liquid.

6. High shear gradient is produced throughout the mixture which weakens the interfacial films.

7. Emulsion is destabilized.

Demulsifiers are commonly employed for demulsification purposes, and ultrasonification can be used to complement them. Demulsification using demulsifiers is made more efficient through ultrasonification since the agitation due to ultrasonic waves aids in mixing the demulsifiers through the emulsion mixture better. This way, the demulsifiers may reach more of the interfacial oil-water film, drain the film of surfactants, and decrease the film's strength. This induces the coagulation of dispersed water blobs, and consequently the destabilizing of emulsion.

Methodology

As this project attempts to compare the efficiency of ultrasonication to other methods of oil demulsification, each of those techniques were performed separately. Before any experimentation was done to determine the demulsificating effect of any mechanism, turbidity for each oil-water concentration was obtained. This initial turbidity was then compared to the turbidity of the solution after any specific test was performed. From basic analysis, the test that results in the most turbidity decrease can be said to be the most effective test for demulsifying an emulsion.

% Solution, oil in Water	Oil to Solvesso ratio	Highest turbidity, NTU
0.1	0.05:0.05	975
0.08	0.04:0.04	713
0.06	0.03:0.03	541
0.04	0.02:0.02	252
0.02	0.01:0.01	152

Table 2: The base-turbidity of each emulsion.

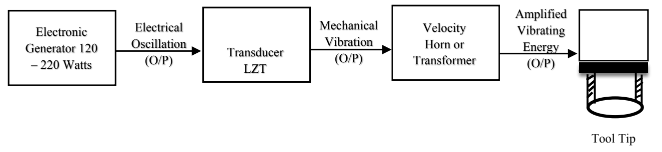


Figure 5: Diagram of sonication equipment setup.

Figure 6: Hach 2100Q turbidimeter.

Figure 7: Solvesso 150.

Base-turbidity determination

To determine the base-turbidity, different concentrations of emulsions were created. To create each emulsion, studied concentrations of oil were added to a relevant amount of water. To form a stable emulsion, an emulsifier (Solvesso 150) was added alongside the oil after which rigorous mixing was done for 70 seconds with an electric solution mixer. This procedure was carried on for every emulsion created in this project. After mixing, the resulting solution was distributed into 5 beakers right after mixing and was allowed to set for 7 min before a turbidity reading was taken.

Many trials of this experiment were repeated and the concluded base turbidity for each mixture of different oil concentration is shown in Table 2. Turbidity reading was done on a turbidimeter from Hach Instruments Company (Figures 5-7).

Emulsion breaker

The concentration of the demulsifying agent which serves as a catalytic agent through time and external conditions such as temperature and pressure can satisfy and bridge to the comparison of all subjected samples.

As with the base turbidity test, emulsions were created by mixing different concentrations of oil and Solvesso 150 with water. In this test the following procedure was followed:

1. For a specific concentration of oil, an emulsion of around 1000

Concentration		Turbidity, NTU								
Oil in water	Emulsion breaker	Trial 1			Trial 2			Trial 3		
%	ppm	Initial	Final	%Reduction	Initial	Final	%Reduction	Initial	Final	%Reduction
0.08	80	905	747	17	865	629	27	717	675	6
	60	924	691	25	890	659	26	890	762	14
	40	950	756	23	908	618	32	686	592	14
	20	951	742	22	902	695	23	644	564	12
	15	963	795	17	928	666	28	987	808	18
0.06	80	618	614	1	492	477	3	656	642	2
	60	571	578	-1	489	462	6	629	696	-11
	40	579	551	5	337	327	3	599	592	1
	20	555	525	5	336	300	11	611	599	2
	15	527	493	6	337	293	13	594	546	8
0.04	80	327	304	7	275	286	-4	320	329	-3
	60	312	299	4	274	276	-1	273	282	-3
	40	309	285	8	262	261	0	270	269	0
	20	292	274	6	263	253	4	260	260	0
	15	286	261	9	271	264	3	263	254	3
0.02	100	118	138	-17	117	126	-8	202	214	-6
	80	123	123	0	172	179	-4	190	197	-4
	60	125	115	8	172	166	3	192	187	3
	40	125	106	15	167	156	7	192	182	5
	20	118	99	16	168	160	5	178	174	2

Note: Shaded cells highlight results with the highest turbidity reduction.

Table 3: The effect of different concentrations of emulsion breaker on different concentrations of emulsion.

Concentration		Turbidity, NTU		
Oil in water	Emulsion breaker	Trial 1		
%	ppm	Initial	Final	%Reduction
0.08	80	632	604	4.4
	60	674	615	8.8
	40	571	617	-8.1
	20	701	621	11.4
	15	670	657	1.9
0.06	80	420	376	10.5
	60	463	396	14.5
	40	422	389	7.8
	20	411	399	2.9
	15	445	428	3.8
0.04	80	520	491	5.6
	60	480	446	7.1
	40	500	383	23.4
	20	508	409	19.5
	15	508	412	18.9
0.02	80	92.2	91.2	1.1
	60	85.5	92.1	-7.3
	40	88.7	90.6	-2.1
	20	92.4	88.7	4
	15	98.8	94.3	4.6

Table 4: The Effect of different concentrations of emulsion breaker on different concentrations of emulsion (continuation).

mL was created then immediately distributed in 5 beakers after mixing and left to set for 7 min.

2. After setting for 7 min, an initial turbidity reading was recorded and a noted amount of emulsion breaker was added.

3. The mixture of emulsion and emulsion breaker was mixed through a magnetic stirrer on 60 rpms for 2 min, 40 rpms for 2 min then 20 rpms for the last 2 min. A final turbidity was then recorded after mixing. This process was repeated for 3 trials. The results of all those trials are presented in Table 3.

Water bath

Since the density and viscosity depends on the temperature of the samples, this test aims to demulsify the samples by the function of time, temperature, viscosity and density.

To perform this section of the project, two things need to be optimized: the time and the temperature of exposure. To do that, the regular emulsion previously discussed was created and separated in different beakers. After taking and initial turbidity reading, these beakers were placed in a water bath at a certain temperature and each of them was left inside for a certain amount of time. After removal of the beakers from the water bath, the final turbidity reading was measured. This was repeated for different temperatures. The results of this are presented in Table 4.

This technique has been done in an SDM water bath device with a maximum heating temperature of 70°C. This device is shown below on Figure 8.

Centrifuge

To test the demulsification efficiency of the centrifugal technique, the test needed to be optimized. For a fixed speed of 5000 rpm and a

Figure 8: Water bath.

Oil in water, %	EB concentration	% Reduction
0.08	20	11
0.06	60	15
0.04	40	23
0.02	15	5

Table 5: The optimum emulsion breaker concentration for each oil in water concentration.

Figure 9: Centurion centrifuge.

Figure 10: Ultrasonic device.

concentration of oil of 0.08, the test was run for different durations. After finding the optimum duration for the test, emulsions of different concentrations were created then placed in centrifugal tubes and run for the optimum time at different rotation speeds. The reduction of turbidity of each trial was recorded and the results are presented in Table 5. This technique has been conducted on a Centurion Centrifuge with a rotation speed of 1000 rpm to 5000 rpm (Figure 9).

Ultrasonication

This test proposed several stages of time duration, temperature, amplitude and power to systematically determine the optimum level. This will provide a full analysis of the direct effect of ultrasonic waves on the emulsions and will determine the sonication durations and intensities that result in the most efficient separation.

This technique, an ultrasonic device from Hielscher Ultrasonics Model UIP1500hd has been used. This ultrasonic device has a maximum power of 1500 Watts and at 20 kHz. This device is also able to deliver up to 170 microns of Amplitude. Please refer to Figure 10.

Results and Discussion

For each of the four demulsification techniques performed the turbidity before and after were recorded for different operating conditions. The percentage reduction of turbidity for each test was computed to find the method resulting in the greatest reduction. This section Presents those collected results and the direct computations that stem from them.

Base-turbidity determination

The value of turbidity for each solution was found by taking the highest turbidity reading for each specific concentration during any of the trials performed. This follows the observation that as the oil, emulsifier, and water are mixed, splashes of water carry with them droplets of the oil that cause the emulsion to be of less concentration than the calculated value resulting in a value of turbidity less than the supposed value. The highest turbidity reading at any trial is then considered the reading for which the least splashing was done thus the most accurate. The value of this highest reading considered as the base turbidity for each concentration is presented in the following table.

These values of base turbidity were, however, disregarded as the experiment progressed. As it was observed, the turbidity of any solution changes with each trial so assigning a concentration a specific value of turbidity for the reduction to be compared with is not viable. Instead, a % decrease notation was followed in which the turbidity of each mixture is measured before any demulsification technique was applied to it. After the demulsification the turbidity was measured again and the % reduction in turbidity was noted. Some of the samples are shown on the photos below.

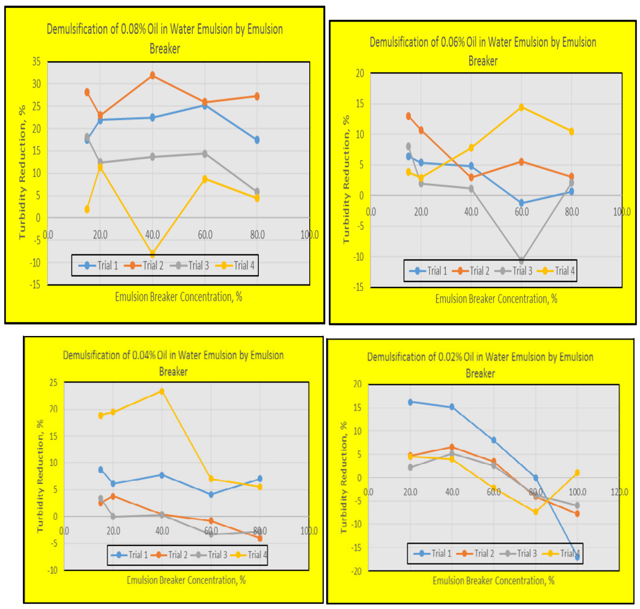
Emulsion breaker

The method involves using a specific amount of emulsion breaker or demulsifier to the predetermined oil in water emulsion concentrations. The emulsion breaker (EB-8956) used in this project is coming from a world leader emulsion breaker supplier in the industry MI Swaco. The first step taken was with determination of the optimum amount of emulsion breaker to be used for each concentration of oil in water emulsion. Please refer to Table 3.

Oil in water concentration was tested for 5 different concentrations of emulsion breaker ranging from 15 to 80 ppm to find the optimum concentration for each concentration. This was first done over 3 trials which reported varying results that can be seen in Table 3. The experiment was repeated one last time for a fourth trial to confirm the variations in the data. This last trial is represented in Table 4.

After finding the optimum concentration of emulsion breaker for oil in water concentration, Table 5 was created to summarize the results. In the table, the optimum emulsion breaker concentration alongside the % reduction caused by it is presented. Kindly refer to the graphical representation Plot 1 of Table 5 below.

From these plots, we can deduce that as the concentration of the oil



Plot 1: Demulsification of oil in water emulsion by emulsion breaker.

Citation: Amani M, Idris M, Abdul Ghani M, Dela Rosa N, Carvero A, et al. (2017) An Experimental Study on the Application of Ultrasonic Technology for Demulsifying Crude Oil and Water Emulsions. J Pet Environ Biotechnol 7: 330. doi: 10.4172/2157-7463.1000330

Oil in water concentration	Water bath temperature	Turbidity, NTU								
		Trial 1			Trial 2			Trial 3		
%	Degree C	Initial	Final	%Reduction	Initial	Final	%Reduction	Initial	Final	%Reduction
0.08	70	750	556	26	897	592	34	956	630	34
	60	703	475	32	703	490	30	703	445	37
	50	991	987	0	991	990	0	991	997	-1
0.06	70	739	514	30	739	512	31	739	504	32
	60	483	323	33	483	313	35	483	341	29
	50	799	793	1	799	804	-1	799	789	1
0.04	70	339	188	45	339	197	42	339	203	40
	60	332	226	32	332	227	32	332	238	28
	50	379	302	20	379	295	22	379	283	25
0.02	70	140	91	35	140	90	36	140	93	34
	60	151	110	27	151	109	28	151	116	23
	50	95.6	74.1	22	95.6	69.6	27	95.6	70.8	26

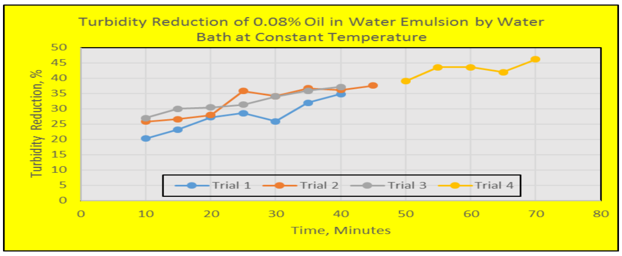
Note: Shaded cells highlight results with the highest turbidity reduction.

Table 6: The temperature optimization of the water bath.

Time, mins	Turbidity, NTU											
	Trial 1			Trial 2			Trial 3			Trial 4		
	Initial	Final	%Reduction	Initial	Final	%Reduction	Initial	Final	%Reduction	Initial	Final	%Reduction
10	750	598	20	956	709	26	897	655	27			
15	750	576	23	956	702	27	897	628	30			
20	750	546	27	956	689	28	897	624	30			
25	750	536	29	956	614	36	897	616	31			
30	750	556	26	956	630	34	897	592	34			
35	750	510	32	956	605	37	897	575	36			
40	750	489	35	956	610	36	897	564	37			
45				956	597	38	897	545	39			
50										817	498	39
55										817	461	44
60										817	461	44
65										817	474	42
70										817	440	46

Note: Empty cells do not have values due to sample limitations; shaded cells highlight highest turbidity reduction.

Table 7: The reduction in turbidity for 0.08% oil-in-water emulsion with time at 70°C.



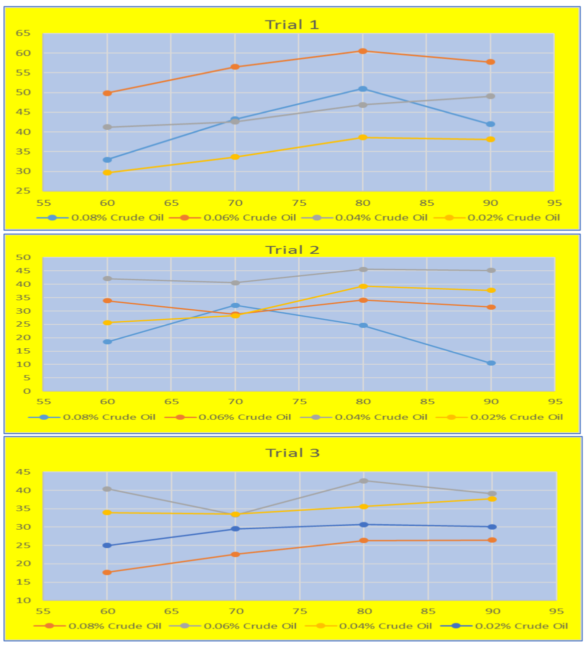
Plot 2: Turbidity reduction at constant temperature on a water bath.

in water emulsion sample reduces and as the concentration of emulsion breaker increases, sample turbidity increases.

Water bath

To test the reduction of turbidity used a water bath, two parameters needed to optimize: temperature and time. Firstly, four different concentrations of oil in water were mixed and placed in the water bath at different temperatures at 10°C increments from 40°C to 70°C. The turbidity readings were taken before and after the treatment allowing for the calculation of the % reduction in the turbidity. No reductions were found for 40°C water temperature, and the reductions at other temperatures are presented in Table 6.

As seen in the above table, the most reduction happened at a



Plot 3: Percent turbidity reduction by ultrasonication.

Citation: Amani M, Idris M, Abdul Ghani M, Dela Rosa N, Carvero A, et al. (2017) An Experimental Study on the Application of Ultrasonic Technology for Demulsifying Crude Oil and Water Emulsions. J Pet Environ Biotechnol 7: 330. doi: 10.4172/2157-7463.1000330

temperature of 70°C. With that said, the temperature of the water bath was set at 70°C and a solution of 0.08% of oil-in-water was prepared and placed in the water bath to optimize the time of treatment this is presented in Table 7 (Plots 2 and 3).

From the table above, it is found that the longer the time the sample was subject to the water bath the higher (results highlighted in read) is the turbidity reduction of the sample at a constant temperature of 70°C. That is, demulsification with water bath is directly proportional to time at constant temperature. The plot above shows the increasing turbidity reduction with respect to increasing time submerges in the water bath at constant temperature of 70°C.

Centrifuge

As with the water bath, both an optimization of the rotational speed and the treatment time need to be performed for this technique. To start, a solution of 0.08% oil in water was prepared and run at 5000 rpm at different times as shown in Table 8 (Figure 11). Through that, the optimum treatment time was found to be 8 min. From there

Time	Concen-tration, %	RPM	Turbidity, NTU					
			Trial 1			Trial 2		
			Initial	Final	%Reduc-tion	Initial	Final	%Reduc-tion
5	0.08	5000	992	195	80	992	193	81
8	0.08	5000	992	157	84	992	164	83
10	0.08	5000	992	196	80	992	167	83
15	0.08	500	992	177	82	992	143	86

Note: Shaded cells highlight results with the highest turbidity reduction.

Table 8: Time optimization of the centrifugal demulsification technique.

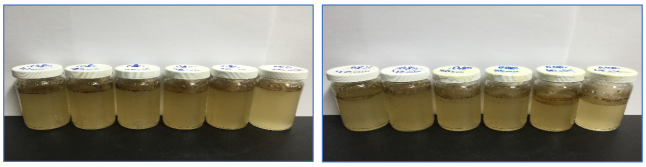


Figure 11: A 0.08% oil in water sample subjected to water bath at constant temperature at varying time (from 10 min to 65 min).

Time	Concen-tration, %	RPM	Turbidity, NTU					
			Trial 1			Trial 2		
			Initial	Final	%Reduc-tion	Initial	Final	%Reduc-tion
8	0.08	1000	942	456	52	942	395	58
8	0.08	2000	942	352	63	942	314	67
8	0.08	3000	942	230	76	942	210	78
8	0.08	4000	942	144	85	942	183	81
8	0.08	5000	942	210	78	942	197	79
8	0.06	1000	671	274	59	671	290	57
8	0.06	2000	671	147	78	671	184	73
8	0.06	3000	671	161	76	671	112	83
8	0.06	4000	671	153	77	671	99.2	85
8	0.06	5000	671	181	73	671	134	80
8	0.04	1000	325	125	62	325	139	57
8	0.04	2000	325	114	65	325	110	66
8	0.04	3000	325	121.1	63	325	68.6	79
8	0.04	4000	325	82.9	74	325	78.2	76
8	0.04	5000	325	75.7	77	325	57.3	82
8	0.02	1000	153	64	58	153	68.9	55
8	0.02	2000	153	39.8	74	153	48.9	67
8	0.02	3000	153	23.5	85	153	28.3	82
8	0.02	4000	153	19.2	87	153	43	72
8	0.02	5000	153	40.2	74	153	37.6	75

Note: Shaded cells highlight results with the highest turbidity reduction.

Table 9: RPM optimization of the centrifugal demulsification technique.

Figure 12: Oil in water emulsion samples.

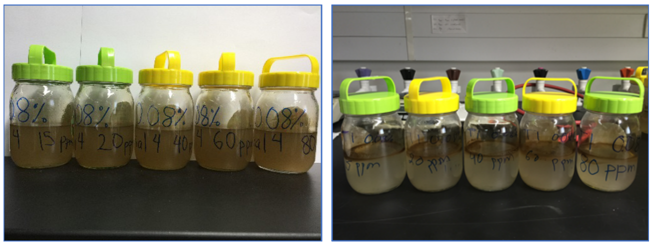


Figure 13a: A 0.08 % oil in water emulsion sample before (left sample) and after (right sample) treatment.

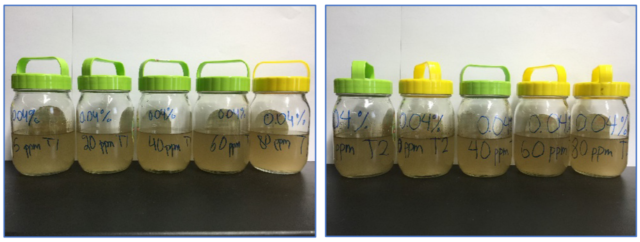
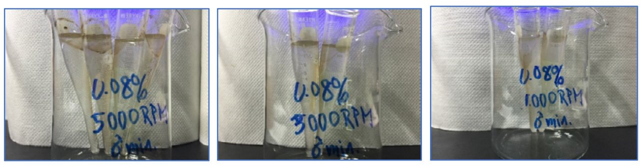
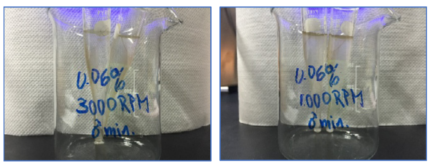


Figure 13b: A 0.04% oil in water emulsion sample before (left sample) and after (right sample) treatment.



A B C

Figure 14a: A 0.08% oil in water sample subjected to the centrifuge at (A) 5000 RPM, (B) 3000 RPM and (C) 1000 RPM.



A B

Figure 14b: A 0.06% oil in water sample subjected to the centrifuge, (A) 3000 RPM and (B) 1000 RPM.

an optimization of the rotational speed was done. This was through preparing solutions of different oil-in-water concentrations that were placed in the centrifuge for 8 min at different rpms. The results of this optimization are shown in Table 9 where it can be seen that the optimum speed is 4000 rpm (Figures 12-18).

Ultrasonication

As the efficiency of each of the demulsification techniques has been tested, it is necessary to compare them to that of Ultrasonication. As before, both the time and amplitude of the ultrasonication treatment need to be optimized. Initially the test was run at amplitude of 100% while varying the time of treatment between 2 min and 14 min for a sample of 0.08% oil-in-water. The results of this initial run are presented in Table 10 where it can be seen that running the treatment for a time

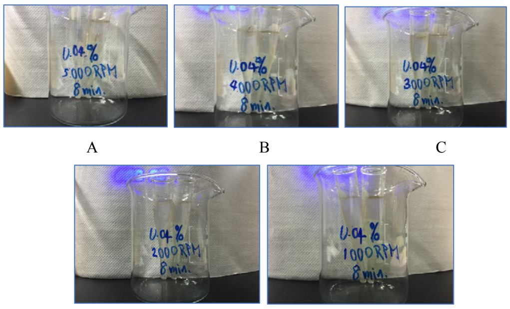


Figure 14c: A 0.04% oil in water sample subjected to the centrifuge at (A) 5000 RPM, (B) 4000 RPM, (C) 3000 RPM, (D) 2000 RPM and (E) 1000 RPM.

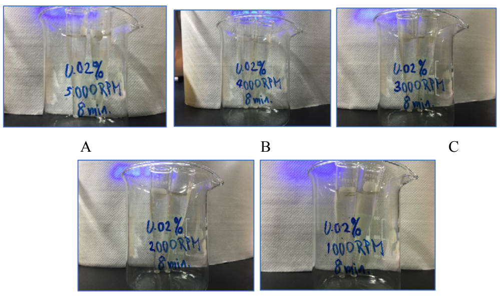


Figure 14d: A 0.02% oil in water sample subjected to the centrifuge at (A) 5000 RPM, (B) 4000 RPM, (C) 3000 RPM, (D) 2000 RPM and (E) 1000 RPM.

Figure 15: Time optimization of 0.08% oil in water sample by ultrasonication.

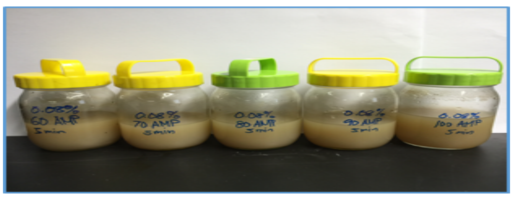


Figure 16: Amplitude optimization of 0.08% oil in water sample by ultrasonication.

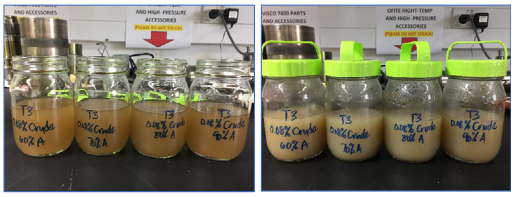


Figure 17a: (A) 0.08% water in oil sample before and (B) after ultrasonication.

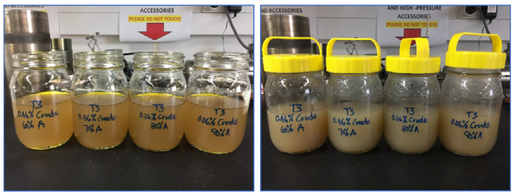


Figure 17b: (A) 0.06% water in oil sample before and (B) after ultrasonication.

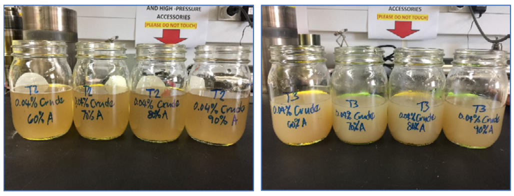


Figure 17c: (A) 0.04% water in oil sample before and (B) after ultrasonication.

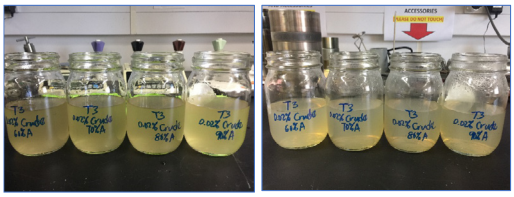


Figure 17d: (A) 0.02% water in oil sample before and (B) after ultrasonication.

Figure 18: Hielscher ultrasonic device model UIP1500hd in sound proof cabinet.

Time, mins	Amplitude, %	Concentration, %	Initial turbidity, NTU	Final turbidity, NTU	% Turbidity reduction
2	100	0.08	986	OR	-
4	100	0.08	989	950	4
6	100	0.08	961	787	18
8	100	0.08	943	551	42
10	100	0.08	923	460	50
12	100	0.08	734	602	18
14	100	0.08	699	587	16
Note: Shaded cells highlight results with the highest turbidity reduction.					

Table 10: Time optimization of ultrasonication.

Time, mins	Amplitude, %	Concentration, %	Initial turbidity, NTU	Final turbidity, NTU	% Turbidity reduction
5	60	0.08	948	OR	-
5	70	0.08	948	908	4
5	80	0.08	948	790	17
5	90	0.08	948	638	33
5	100	0.08	948	594	37
Note: Shaded cells highlight results with the highest turbidity reduction.					

Table 11: Amplitude optimization of ultrasonication.

Concentration	Amplitude	Time	Turbidity, NTU		
%	A	min	Initial	Final	% Reduction
Trial 1					
0.08	60	5	991	664	33
0.08	70	5	991	563	43
0.08	80	5	991	486	51
0.08	90	5	991	575	42
0.06	60	5	887	445	50
0.06	70	5	887	386	56
0.06	80	5	887	350	61
0.06	90	5	887	375	58
0.04	60	5	512	301	41
0.04	70	5	512	294	43
0.04	80	5	512	272	47
0.04	90	5	512	261	49
0.02	60	5	202	142	30
0.02	70	5	202	134	34
0.02	80	5	202	124	39
0.02	90	5	202	125	38
Trial 2					
0.08	60	5	931	759	18
0.08	70	5	931	632	32
0.08	80	5	931	702	25
0.08	90	5	931	833	11
0.06	60	5	777	514	34
0.06	70	5	777	533	29
0.06	80	5	777	512	34
0.06	90	5	777	532	32
0.04	60	5	496	287	42
0.04	70	5	496	295	41
0.04	80	5	496	270	46
0.04	90	5	496	272	45
0.02	60	5	191	142	26
0.02	70	5	191	137	28
0.02	80	5	191	116	39
0.02	90	5	191	119	38
Trial 3					
0.08	60	5	995	819	18
0.08	70	5	995	770	23
0.08	80	5	995	733	26

0.08	90	5	995	732	26
0.06	60	5	777	463	40
0.06	70	5	777	518	33
0.06	80	5	777	446	43
0.06	90	5	777	473	39
0.04	60	5	483	319	34
0.04	70	5	483	321	34
0.04	80	5	483	311	36
0.04	90	5	483	301	38
0.02	60	5	176	132	25
0.02	70	5	176	124	30
0.02	80	5	176	122	31
0.02	90	5	176	123	30
Note: Shaded cells highlight results with the highest turbidity reduction.					

Table 12: Turbidity reduction with varying amplitude for different oil in water emulsion concentrations.

of 10 min causes the most reduction. Although the optimum condition was found to be 10 min, the actual test was performed for 5 min due to the threat to safety if performed for longer times.

From the table above it was observed that the highest turbidity reduction was at the 100% amplitude. But no further test has been done for 100% amplitude due to the hazards that arise from such magnified amplitude and for safety purposes of the ultrasonic device. To proceed with, the ultrasonic test was performed for the specified time at three different amplitudes ranging from 70 to 90. The results of the varying reduction in turbidity with amplitude are presented in Table 11. Optimum amplitude can be noted at a value of 80%. With the ultrasonication demulsification technique turbidity reduction ranges between 26% to 61% which can be seen from the plots above.

Conclusions

In this project, we determined the effectiveness of the thermal (water bath), mechanical (centrifuge), chemical (demulsifiers), and ultrasonication as means of demulsification by applying it on various oil-in-water mixtures, and measuring the turbidities of each before and after application.

1. Based on our attempt to establish base turbidity values for each oil-in-water mixture, it was found that the turbidity of these mixtures was difficult to keep sufficiently constant. This is because the turbidity is affected by some uncontrollable factors. These factors include the imprecision of our liquid transferring equipment and instrument to measure turbidity.

2. In finding an optimum emulsion breaker concentration, consistent trend cannot be identified. However, out of the three oil-in-water mixtures tested, the optimum emulsion breaker concentration decreased as percent oil in water decreased. In other words, the lower the oil to water ratio, the lower the amount of demulsifier should be added.

3. The most reduction in turbidity for water bath was attained at the highest temperature (70°C) and longest duration (70 min). Hence, the higher the temperature and longer the duration for thermal demulsification, the more effective.

4. The optimum time for centrifuge method was found to be 8 min. Hence the optimum duration for such method is not always the longest. The optimum centrifuge speed was found to be 4000 rpm. Again, this is not the highest rotational speed. So, the optimum time and rotational speed must be determined for specific oil and water emulsions.

5. Optimum time for ultrasonication is 10 min, which again isn't the longest duration (14 min). But max reduction in turbidity is achieved at maximum amplitude, in this case at 100%.

6. As data shows, there was a larger turbidity reduction on the centrifuge technique than any other technique.

7. Ultrasonication does reduce the turbidity of the sample ranging from 20% up to 60%.

8. 100% amplitude was not done in the study for the reason that the machine was having a history of being damaged at 100% amplitude.

9. Turbidity reading variation was observed on different samples of the same oil-in-water solution concentration.

Nomenclature

T: Period, seconds,

f : Frequency, Hertz

F: Force, Newton

k: Proportionality constant for Hooke's law, Newton/meters

x: Displacement, meters

ω : Amplitude, 1/seconds

φ: Phase angle, dimensionless

t: Time, seconds

π: Irrational constant pi

v: Wave speed, m/s

μ: Inertial quantity, unit varies

Acknowledgment

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