Ultrasonic wave application to crude oil de-emulsification process

By

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CERTIFICATION

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and the original work contained herein have not been undertaken or done by unspecified sources or persons.

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ABSTRACT

Tactics to tackle emulsion problems in oil and gas industry are being improvised regularly to achieve a high efficiency and low cost solution. Theory of using ultrasonic waves for demulsification has been around for some time but its application has yet to be implemented widely. Experiments and pilot studies are still being conducted to discover suitable condition for the use of ultrasonic wave as ultrasonic wave has dual implication which are to emulsify and demulsify. In this research, experiment was done on crude oil collected from the Terengganu Crude Oil Terminal (TCOT). Experiments are done by using indirect ultrasonic application and direct ultrasonic application. Thus far, ultrasound yields positive impact in demulsification but it is yet to conclude that ultrasound is more superior to other separation means. Direct Ultrasonic Application is being utilized using 20 kHz ultrasonic probe. The only huddle with current ultrasonic generator is its inability to sustain more than 10 minutes of irradiation at more than 300W of intensity. Indirect ultrasonic application is being utilized using 40kHz Elma Ultrasonic Cleaner. Demulsification using both ultrasound applications is proven using titration test and CPM analysis. However bottle test on all specimen does not show clear water layer after 3 days of settling time. Ongoing research is being done to explore the effective use of ultrasonic wave to coalesce water particles. Further study may be conducted to develop suitable device to be placed in separator.

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CHAPTER 1

INTRODUCTION

1. INTRODUCTION

1.1 Background of Study

The production of crude oil involves a highly complex process. Crude oil is often extracted along with unwanted fluids and solids. As crude oil is transported through the production line and agitation energy is supplied from several sources such as chokes, pumps and wellheads; the crude oil, produced water and solids will intermingle with each other [1]. This mixing phenomenon results in the formation of crude oil emulsion.

The crude oil emulsion when allowed to settle will eventually separate between the oil and water phases depending on the rate of separation. Nevertheless, tight emulsion is rather hard to separate even with current technology such as chemical treatment. The formation of emulsion during the production of crude oil is highly unfavourable as the productivity and operations will suffer losses [2]. In order to eliminate the losses, oil and gas companies have developed a few technologies revolving mechanical, electrical and chemical means to separate emulsion more efficiently and effectively.

1.2 Problem Statement

Oil and gas industry experiences losses such as decline in oil production, high faulty rate in facilities and off-spec crude and disposal water due to emulsion [3]. In order to treat emulsion, new facilities need to be build and long term expenditure is incurred due to heating and the use of chemical treatment. Heating will cause a significant loss of the lower-boiling point hydrocarbon and chemical treatment will cause decomposition change which in turn will worsen emulsion problem [4]. On the other hand, electrical coalesce will need high energy usage.

A more cost and energy efficient separation method must be employed to increase separation rate. Besides, the operating envelope must be recognized and quantified as well.

1.3 Objectives

The objectives of this project are:

- To explore the use of ultrasonic wave via indirect and direct exposure towards separation of emulsion.

1.4 Scope of Study

Ideas relating to the use of acoustic energy as a separation mean started since 1960. However, minimal progress has been made on its use. In this research, the author looks into the possibility of the use of ultrasonic wave in the separation of emulsion. Experiment will be carried out using crude from Malaysia's field (RE110). RE110 is well sample taken from Terengganu offshore field. It has a WAT higher than room temperature. Indirect ultrasonic irradiation (ultrasonic bath) and direct irradiation (ultrasonic probe) will be utilized for preliminary experiment to screen for the best ultrasonic irradiation means. 3 parameters which are duration of irradiation, location of irradiation and intensity of irradiation will be varied to produce 7 experiments. Design of experiment (DOE) is not employed because this is a preliminary study. 7 responses to be identified are settling rate, thickness of oil layer, rag layer and water layer after bottle test and quality of oil, rag and water after ultrasonic irradiation. Crude oil emulsion will be formed at water fraction of 30%, mixing speed of 9600rpm and mixing temperature of room temperature as recommended from previous work. A duration of 3 weeks per sample is required to conduct the experiment and observations.

1.5 Relevancy and Feasibility of Study

Emulsion in oil production left untreated will cause damage towards processing equipment [5] and degrade oil quality. If it is treated, extra cost will be imposed to the operating expenditure (OPEX). Therefore, it is mandatory to treat emulsion at the fastest and cheapest way. With this study, the influence of amplitude, frequency and location of source of the ultrasonic wave on emulsion stability and behaviour will be analysed and discussed. This will serve as the reference to future research and oil production companies on the alternative for demulsification process and hence relevant to the oil and gas industry.

9 experiments are carried out with the longest ultrasonic irradiation time of 30 minutes on specimens. After each irradiation, the specimens need to undergo bottle test for at least two weeks to observe the effect of ultrasonic wave on crude emulsion separation. With a given duration of 10 weeks, the project work will commence on 2nd week of semester 2 and will end in 11th week of semester 2 as shown in the Gantt chart in the subsequent chapters. Therefore, this study is feasible in time context. The materials and equipment to be used in this study are also readily available at the flow assurance laboratory in block 20 and Centre of Research in Ionic Liquids in block 4 of University Technology of PETRONAS.

CHAPTER 2

LITERATURE REVIEW

2. LITERATURE REVIEW

2.1 Crude Oil Emulsion

2.1.1 Classification of Crude Oil Emulsion

Emulsion is produced during crude oil extraction making separation process difficult. Emulsion is a dispersion of one liquid in a second, immiscible liquid [6]. It is a multiphase system which consists of continuous phase (major phase) and dispersed phase (minor phase) [7]. In most cases, it is thermodynamically unstable. It is classified based on its kinetic stability. Loose emulsion will last for few minutes. Medium emulsion will last for tens of minutes. Tight emulsion will last for hours or days.

There are 3 types of emulsions which are water-in-oil emulsion (W/O), oil-in-water emulsion (O/W) and water-in-oil-in-water emulsion (W/O/W) [2]. The W/O/W emulsion refers to an emulsion with presence of water droplets in oil droplets present in a continuous water phase. The type of emulsion is categorized according to the ratio of the two immiscible liquid present in an emulsion. W/O emulsion is the most common emulsion. They form naturally during the crude oil production and the water content can be as high as 60% by volume. The figure below shows the photomicrograph of a water-in-oil emulsion, an oil-in-water emulsion and a water-in-oil-in-water emulsion.

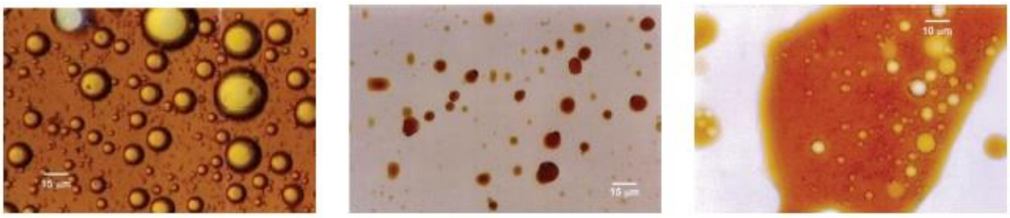


Figure 1:W/O, O/W AND W/O/W Emulsion Photomicrograph (Kokal, 2002)

An emulsion consists of two phases known as the dispersed phase or internal phase and the continuous or external phase [4]. The phase of the liquid determines the type of emulsion. For example, a water-in-oil emulsion has water as the dispersed phase and oil as the continuous phase. Inversely, an oil-in-water emulsion has oil as the dispersed phase and water as the continuous phase. The basic rule in differentiating the phases of oil and water is by comparing the volume of both liquids. The liquid with a much smaller volume compared to the other is identified as the dispersed phase while the other liquid will be known as the continuous phase. Given a situation where the ratio of the liquids are almost the same (about 50:50), then other factors will be used to determine the type of emulsion.

Emulsion is also categorised based on the size of the particles of the dispersed phase. 3 categories are macro-emulsion, mini-emulsion (also known as nano-emulsion) and micro-emulsion. Macro-emulsion has dispersed particle phase with diameters of about 1 to 100µm. This category of emulsion is unstable because it consists of large droplets that allow sedimentation to take place and separate between the dispersed and continuous phase. The rate of coalescence of the dispersed droplets of macro-emulsion is slow, caused by the presence of low-molecular weight or polymeric surfactants.

The next category is known as the mini-emulsion or nano-emulsion. The size of the droplets ranges between 50nm to 1µm [8] Mini-emulsion is stable against diffusion degradation by a compound insoluble in the continuous phase and hence is more stable than macro-emulsion. The emulsion is the only emulsion stable kinetically [9]. Rajalakshmi et.al [10] suggested a droplet diameter range of 50-200nm while Jimtaisong [9]suggested a range between 100 to 400nm.

Micro-emulsion on the other hand has droplets of diameters 1 to 100nm and is thermodynamically stable [8] [9]. The average diameter of the particles of the dispersed phase in an emulsion is about 1 millimetre (10^{-3}m). Since, micro- refers to 10^{-6} and emulsion implies that droplets of the dispersed phase have diameters close to 10^{-3}m, the micro-emulsion implies a system with the size range of the dispersed phase in the $10^{-6} \times 10^{-3} = 10^{-9}$m range [8].

Pickering emulsions on the other hand have particles which reside at the interface and stabilize the emulsion. Particles which are more wetted by water than oil would stabilize oil-in-water emulsion and vice versa [11].

Another three types of emulsion classifications are based upon the stability of the emulsion. In order to identify a stable emulsion, the emulsion must or usually have an increasing viscosity over time [12]. Contrarily, an unstable emulsion is identified when the separation between water and oil occurs in a speedy manner. Emulsions that are in between stable and unstable conditions are known as mesostable emulsions.

In agreement, Kokal [3] has put this category in simpler words where he termed the categories as loose, medium and tight. Loose emulsion tend to separate within minutes, medium emulsion in tens of minutes and tight emulsion takes hours or days and the separation may occur only partially. The emulsion tightness index (ETI) formula by Opawale and Osisanya [2] may be used to calculate the stability of the emulsion.

$$ETI = \left[\frac{Vol\ of\ water\ blended - Vol\ of\ water\ seperated\ after\ time, t}{Vol\ of\ water\ blended}\right]\%$$

Equation 1: Emulsion Tightness Index

2.1.2 Formation of emulsion in crude oil production

Three vital criterions of emulsion formation are [13]:

1. Two different immiscible liquid must be present.
2. The presence of surfactants to stabilize dispersed phase droplets.
3. Adequate mixing energy is supplied to allow one liquid to disperse in the other liquid.

According to Petroleum Engineering Handbook 1987, below are the conditions where mixing energy is supplied to crude oil:

1. Pressure drop through chokes, valves and other surface equipment
2. Flow through tubing, wellhead, manifold or flow lines
3. The surface transfer pump
4. Bottom hold pump
5. Wellbore

Listed are some of the emulsification processes discovered thus far [6]:

1. High-pressure homogenization. This technique is conducted by forcing two fluids to flow through an inlet valve then a mixing chamber under high pressure.
2. Membrane Emulsification. Dispersed phase is forced to permeate into the continuous phase through a uniformly pored membrane.
3. Microchannel emulsification. Photolithography is a manufactured microchannel where the dispersed phase is forced into the continuous phase. Furthermore, spontaneous emulsification may occur without the presence of an external energy source. This incident may occur when there is a contact between two immiscible fluids with very low interfacial tension.
4. Application of a controlled shear where emulsions are made by supplying agitation energy with a complex mixture of extensional and shearing flows.

2.1.3 Replication of Crude Oil Emulsion in Lab

A few examples on how previous researchers replicate crude oil emulsion in lab:

1. Kokal and Al-Juraid used an automatic shaker to mix the oil and water mixture for approximately 5 minutes.
2. Rodionova allowed the oil-water mixture to be agitated for 15 minutes [14].
3. Kang et al. allowed the water and oil mixture to be agitated for 10 minutes [15]

Through Kokal and Al-Juraid’s study [16], they found that the average water cut of emulsions in a large Saudi Arabian field which produces from seven different reservoirs is 26.8%. The range of water cut obtained in another study is between 5 to 52% [2]. Hence, an average water cut of 30% is assumed in this study.

Flow Rate vs Stirrer Speed

To obtain a reliable result on the formation and stability of emulsion, the conditions and environment where the emulsion is formed must reflect the pipeline conditions in the field. The flow rate of emulsion in the pipeline can be mimicked by supplying equivalent kinetic energy through the stirrer [12] where M=mass (kg) and V=Velocity (m/s)

$$Kinetic\ Energy, KE = \frac{1}{2}MV^2$$

Equation 2: Kinetic Energy through the Stirrer

Supporting the equation above, Opawale and Osisanya proposed that the kinetic energy of a rotational device is [2] where M_s=mass of top bottle arm system+mass of total sample (kg), N=Number of revolutions per minutes (RPM) and R_a=Radius of arm (m):

$$KE = \frac{1}{2} M_s (\frac{2\pi N}{60} \times R_a)^2$$

Equation 3: Kinetic Energy of a Rotational Device

As observed, the work in the pipeline is assumed to be equal to the amount of kinetic energy produced by rotational device. Hence the measure of work in the pipeline is calculated using the formula below where $F = force$ $(kg \cdot m/s^2)$, $m = mass$ (kg), $\dot{m} = mass\ flow\ rate$ (kg/s) and $v = velocity$ (m/s):

$$F = ma = \dot{m}v$$

Equation 4: Force Equation

$$Work = F \times D$$

Equation 5: Work in Pipeline

The stirrer in the lab usually has a much smaller energy supply capacity as compared to the energy encountered in the oil fields. Therefore, Opawale and Osisanya suggested that the mixing speed is scaled down [2].

2.1.4 Composition of Formation and Produced Water

Oil and gas reservoirs are naturally made up of gas, oil and water along with the presence of solid particles. As the oils are being extracted from the reservoir, water is extracted alongside. The naturally occurring water is known as formation water [17]. Produced water on the other hand refers to water produced during oil and gas production. It is the mixture of naturally occurring formation water, the re-injected water and chemical injected during production. Alternatively, produced water is a result of oil and gas production from below the sea reservoirs where the water contains a certain amount of hydrocarbons [18].

Below are some references of produced water's composition from some fields:

1. Niger Delta Region from Nigeria [2]

Composition	$K^+ + Na^+$	Mg^{2+}	Ca^{2+}	Cl^-	SO_4^{2-}	HCO_3^{-1}	CO_3^{2-}
Mass (mg/L)	1452.2	5.6	16.8	425.5	20.2	2927.7	121.1

2. Shuanghe waste water from the oil-water treatment centre of Henan Shuanghe oilfield [15]. The total salinity in the water is about 5251mg/L and oil concentration is roughly of 417mg/L. The water is alkaline with a pH of 8.6

Salt	K^++Na^+	Mg^{2+}	Ca^{2+}	Cl^-	SO_4^{2-}	HCO_3^-	Sulphide
Mass (mg/L)	1689.8	66.8	17.7	1496.4	58.6	1921.7	30-40

3. Berlian East-3 (M213 reservoir) where the location of the reservoir is about 25km offshore peninsular Malaysia. The formation water has a pH value of 8.14 at 22°C [19].

Ions	Na^+	Mg^{2+}	Ca^{2+}	Ba^{2+}	Iron	Cl^-	SO_4^{2-}	HCO_3^-	CO_3^{2-}
Mass (mg/L)	10600	18.8	26.4	18.9	<2	12212	89	7115	54

2.1.5 Impact of Emulsion towards Processing Plant

Emulsion will cause several operational problems in wet-crude handling facilities and gas/oil separating plants. Emulsions can create high-pressure drops in flow lines, productivity decline in wells, flow assurance concern, higher demulsifier and treatment cost and cause trips or upsets in wet-crude handling facilities [20]. The problem is worsen during winter because of lower surface temperatures. Emulsions must be treated to remove the dispersed water and associated inorganic salts to meet crude specifications [21]. It is known that the viscosity of a W/O emulsion is strongly augmented by increasing its water volume fraction and by decreasing the temperature. It is also dependent on the water droplet size distribution and therefore on the resultant shear stress [22].

2.1.6 Factors affecting stability of an emulsion

The biological tendency of a liquid-liquid system to separate and reduce its interfacial area that subsequently reduces the interfacial energy causes an emulsion to be labelled as thermodynamically unstable. A thermodynamically unstable emulsion can still be stable for a period of time due to kinetic stability [3].

Surface Active Agents

Water-in-oil macro-emulsions (size > 0.1 µm) is the most seen emulsion and it is usually present due to surfactants like asphaltenes, resins and naphthenic acids in crude oil [13][11] [14]. These surfactants attached to the water molecules to form absorbed films which to prevent coagulation and coalescence of water molecules. Other factors that affect the stability of emulsions are heavy polar material, temperature, droplet size and droplet size distribution, pH of brine and brine composition [20].

Asphaltenes is natural surfactant with high molecular weight polar component. Wax crystals, resins, porphyrins and fatty acids (naphthenic acid) are some active surface agents that aid asphaltenes in stabilizing the emulsion formed. Resins solubilize asphaltenes while waxes co-adsorb at the interface. Fatty acids on the other hand support the formation of emulsion by creating an ideal environment through the balancing of pH levels [13]. On top of that, Tadros [11] also mentioned that the presence of surfactants will reduce the interfacial tension which allows the droplets to be broken down more easily.

Viscosity and Density

A higher density crude oil (low API gravity) forms a higher volume and more stable emulsion. Concomitantly, crude oil with higher viscosity tends to form emulsion with higher stability when weighed against low viscosity crude oil. The viscous property of crude oil causes the formation of a stable emulsion as the movement of the dispersed water droplets in the emulsion is retarded; as a result the coalescence is impeded. Paraffin-based oils have a lower emulsification tendency than the asphaltic-based oils. Generally, high density and high viscosity oils have a larger volume of emulsifiers in comparison to lighter oils [4].

Pure crude oil in an emulsion is normally less viscous than the emulsion itself. The ratio of the viscosity of pure crude oil in an oilfield depends on the shear rate supplied. If no other information regarding the emulsion is available, the equation below can be used estimate the shear rate supplied [4], where μ_e= viscosity of emulsion, μ_o= viscosity of pure crude oil and f= fraction of the dispersed phase.

$$\frac{\mu_e}{\mu_o} = 1 + 2.5\mathrm{f} + 14.1f^2$$

Equation 6: Shear Rate Equation

Density and viscosity will indirectly indicate the separation behaviour in a process but the interfacial tension between oil and water indicates the potential for emulsion stability. However, stability of the emulsion can be difficult to predict from measureable physical and chemical properties of crude and water. Usually, we need to rely on experimental data for separation models for crude from respective field. Currently, separation of emulsions consists of two main processes which are sedimentation/ creaming and flocculation/ coalescence.

2.1.7 Parameters of Separation's success rate.

Following industrial standard, the emulsion specimen shall be left for at least 30mins for settlement in bottle test due to density difference and coagulate before the components are separated out. Parameters to identify the success rate of separation will be quantified as well. A few measurement of stability have been established such as using bottle test for checking time over water separation, checking water separation over demulsifier usage and screening for emulsion. ASTM method for BS&W (Basic Sediment & Water) must be used. For Saudi Aramco, ESI (Emulsion Separation Index) has been utilized [3].

2.2 Rag Layer

Emulsion may cause formation of undesirable rag layer. Typically, rag layer is a mixture of flocculated water and oil droplets, fine solids and emulsified oil and water, as well as multiple emulsions [23]. Rag layer is observed as a viscous layer normally forming between the oil and water phase containing oil, water and solid, post settling [24]. Generally, rag layer is defined as a layer that prevents complete separation of two fluid phases and hence lowers the overall recovery of oil [25].

2.2.1 Formation of Rag Layer

There are a few mechanisms on rag layer formation. Two mechanisms suggested to explain the formation of rag layer [25] [26]. The first mechanism is a mechanical barrier and the second one is slow coalescence. An accumulation of oil-wet materials may occur at the planar interface of oil and water. This creates a barrier that prevents water and solid particles from passing through and hence impedes coalescence from taking place and encourages the formation of rag layer. The rag layer accumulation and build-up of interfacial material hinders droplet coalescence by trapping droplets in the stable network. The droplets are not able to settle through the interface until coalescence to larger droplets takes place. Through this, a rag layer can accumulate.

A third mechanism was suggested to be the intermediate density [25]. The rag layer density which is composed of water, oil and solids favours stability between the two fluid phases. Therefore, due to the low inertial contribution of water droplets and the build-up of interfacial material, water droplets are prevented from coalescing, subsequently causing rag later to be formed.

2.2 Overview of Existing Separation Method

A few separation processes are utilized depending on the cost, behaviour of emulsion and quantity. Mechanical method such as gravitational, heating and aeration; chemical method such as addition of demulsifies and electrical method such as electrostatic coalesce are used to separate emulsion from crude. Normally, gravitational method will be used with other methods such as heating, addition of demulsifier and electrostatic coalesce to speed up separation. Nevertheless, further research is conducted to identify new ways of separation which is more cost-efficient and fast.

Industry prefers to use gravitational method for separation process because it is cheap, chemically and mechanically inert with minimal human interference required. Only a huge cylindrical vessel is required to store the crude and allow the settling process to take place. The only drawback to this method is it is slow and inefficient. Heating method is good to break the interfacial film and increase the energy of molecules. However, heating consumes energy and heating source must be safe and obtainable. The same goes to electrostatic coalescer.

2.2.1 Demulsifier Addition

Demulsifier provides surface-active molecules to crude to counter the effect of surfactants like asphaltenes, resins and naphthenic acids. It is most common and one of the earliest used method to separate water-in-oil emulsion. However, research is still being done to optimize its usage. Retrofitting and proper demulsifier-injection strategies are established and reviewed regularly. Too much or too little demulsifier usage will have an impact to crude production. Too little demulsifier will have minimal effect to surfactants. Too much of them will replace current surfactants attached to the water molecules and make them unable to coalesce [7].

Besides, single usage of demulsifier throughout the reservoir life cycle is not an ultimate solution. At time, surfactants and salt concentration in reservoir will change and regular lab testing shall be conducted with various type of demulsifier shall be taken into considerations [27]. Thus in long term, demulsifier addition will be costly and time consuming. Interactions between chemical additives shall be taken care of as certain additives such as corrosion inhibitors, anti-foamant, and scale inhibitors will have impact towards the effectiveness of the demulsifier [28].

2.2.2 Electrical Coalescence

Electrical method has been proven to be more economical but it cannot replace chemical demulsifier and heating. Fundamentally, when electrical field is applied, water droplets become polarized. Polarized droplets will attract each other and coalesce. With DC, droplets tend to collect at the electrodes and bounce between the electrodes, forming larger droplets. With AC, droplets will elongate along the lines of force as voltage rises in 1st half cycle and relax during lower-voltage part of the cycle. Thus, film of emulsifier is pulled and pushed and will be eventually weaken. Electrical field also provides energy to water molecules and makes them move and collide more frequently. Higher voltage gradient will induce greater coalescence force [29].

However, at some voltage gradient, water droplets can be pulled apart, tightening the emulsion. Besides, electrostatic dehydration may cause shorting/arcing which generally happens when excess water is present. Chaining may happen and cause short-circuiting when charged water particles form links between both electrodes [7].

2.2.3 Heating

Heating will reduce viscosity, increase number of droplets, dissolve paraffin crystals and increase density between oil and water. Heating is effective in water separation but it is energy inefficient. Heating may cause significant loss of the lower-boiling-point hydrocarbon (light ends) and the remaining liquid will has a lower API gravity and further reduce its value. Besides, the evaporated gas may create problem in the treatment facility. The gas could rise through the coalescing section, creating enough turbulence and disturbance to inhibit coalescence.

Recently, it was proven that heat produced by microwave has the same effect as conventional heating. Experiment has been carried out and decided that microwave heating has twice the efficiency than regular heating on oil-in-water emulsion. This is due to higher absorptivity of MW energy by O/W emulsion. Microwave dielectric heating showed high efficiency to break emulsion even without the use of chemical demulsifier [30].

2.3 Ultrasonic Wave as a Mean to Demulsify.

2.3.1 Ultrasonic Wave as an Agitation Force

The idea of using acoustic energy to separate phases in liquid has been circulating decades ago. However, the use of ultrasonic wave to separate emulsion in oil and gas industry has yet to be explored. Research is being conducted on this relatively new concept due to its potential. If the method succeed, it will provide cost efficient and effective way to separate water-in-oil emulsion from subsea production or normal production.

Most of the researches is conducted on oil-in-water emulsion with many concentrating on the numerical approach and some using synthetic emulsion mimicry to crude oil. Factors that affects emulsion separation by ultrasonic wave have also been researched. Susumu Nii irradiated 2.0MHz ultrasound on Canola oil (O/W emulsion) and found that ultrasound would be able to flocculate oil emulsion and make them float [31]. Latham did the experiment using wastewater from sheep farm [32]. The experiment is to determine the factors influencing the ultrasound separation of oil-in-water emulsions. In 2008, water is deliberately added by Gua Xiang to crude oil to form emulsion. Experiment was then carried out to explore the impact on dewatering rate and desalting rate by ultrasound pre-treatment [33]. In 2004, acoustically aided separation of oil droplets from aqueous emulsions was conducted by Oliveira and the effectiveness of different porous medium to catch the oil molecules is tested [22].

Wei Xie tested on dehydration of waste oil using pulsed ultrasound [34] and found out in order to obtain best dehydration result, an optimal frequency should be inversely proportional to the squares of water droplet volume [35]. He found that the standing wave field is superior to the reverberant field. He stated there is an optimal value for ultrasonic irradiation time to achieve the best result of dehydration, rather than the bigger the better. It is because that the waste oil is re-emulsified after a long time ultrasonic irradiation [36]. He found out also that ultrasonic dehydration at lower frequency achieved better dehydration of emulsion. The reason is that, attenuation coefficient is a function of physiochemical properties of liquid and the frequency of irradiation and it is given by the following equation $\alpha\mathrm{f} = \frac{8\pi^2 {}_2\mu f^2}{3\rho c^3}$ where α_f is attenuation factor (1/m), μ is viscosity of bulk of liquid (kg m/s^2), f is frequency of irradiation (Hz), ρ is density of liquid (kg/m^3) and C is speed of sound in liquid (m/s) [37].
It was found that, lower frequency is helpful to form a uniform sound field of small standing-wave ratio and increase ultrasonic effective radiation distance, which is conductive for dehydration of emulsion. Higher frequency need higher minimum sound power, because with the same sound power, higher ultrasonic frequency produces smaller primary ultrasonic force [38]. A cavitation phenomenon may occur when the ultrasonic power exceeds the optimal value [39].

Fabiane G.Antes firstly used indirect ultrasound application and found out the main effects attributed to ultrasound is cavitation and the resultant shock waves in liquids [40].The efficiency of indirect application of ultrasound is shown for the first time and in case of the experiment which water separation was not visually observed, the emulsion is compared with those before irradiation [40]. It was found that in indirect ultrasound application, an enhancement in temperature increases cavitation due to increment on vapour pressure of liquid [41]. In experimental and mathematical modelling of ultrasonic treatments for breaking oil-water emulsions, Kouznetsov found that low-frequency vibrations, when applied to an oil reservoir, decreased the water cut and increased oil production three fold [42]. He concluded that vibro-energy reduces the interfacial tension leading to coalescence, and that it increases the relative permeability of the oil. Johnston showed that surface tension and viscosity reduction of fluids under ultrasonic wave fields increases oil percolation which leads to increase in oil recovery [43]. This reduction was supposed due to the medium heating by ultrasound absorption.

In the desalting and dewatering of crude oil in ultrasonic standing wave field [44], plane wave spreading in a tube with “hard” blinded end must get a total reflection at the interface. Guo Xiang [44]used the standing wave tube which was made of steel, 5.6 cm of inner diameter, 34.8 cm long and 0.2 cm of wall thickness to conduct direct ultrasonic irradiation experiment. One end of the tube was blinded by a smooth steel circular flat-plate in order to get a reflecting wave. The thickness of the end plate was also 0.2 cm. He used the acoustic impedance Z_s ($Zs = \rho_0 \times c_0$, where ρ_0 and c_0 are the density and sound velocity of the medium) of the medium to determine if standing wave can be formed in the tube.

In the book of “Discoveries in the Theory of Sound” by Ernst Chladni [45], effect of acoustic vibrations to form nodal resonant patterns and their effect on particles is observed. Nodal lines is placed where the superposition of the incident and reflected waves cancel each other. The similar phenomenon is observed by creating ultrasonic standing waves (USW) in suspensions and emulsions. USWs are created by the superposition of incident wave from the acoustic transducer and the reflected wave moving from the reflector. The primary acoustic force moves suspended droplets towards the pressure nodes or antinodes of the standing wave, depending on the material properties. When particles are moved together, they form bands and may aggregate. Due to the increase in hydrodynamic radius, buoyancy forces can separate the phases at a faster rate of sedimentation.

The primary acoustic radiation is formed when USW field is in a medium containing a suspension of particles or droplets, the wave can be partially scattered by the particles if there is an acoustic impedance mismatch between the fluid and the material of the particles. The scattering of the wave will produce the primary acoustic radiation. [46] The force will move the particles or droplets to either node or antinode based on the sign of the acoustic contrast factor (ϕ).

$$\emptyset = \frac{5\rho_p - 2\rho_l}{2\rho_p - \rho_l} - \frac{\kappa_p}{\kappa_l}$$

Equation 7: The Acoustic Contrast Factor

Where ρ is the density, κ is the compressibility factor, l is the liquid medium, p is the particles. When contrast factor (ϕ) is positive, particles move towards the nodes of the standing wave. When ϕ is negative, particles move to antinodes. Generally, solid particles in aqueous media moved to pressure node while oil droplets are moved to antinodes.

When particles are displaced to either nodal or antinodal planes, there are forces that act over short distances called “secondary acoustic forces” or “ Bjerknes forces”. These forces tend to attract particles together within the plane. Bjerknes forces are originated by the scattering of sound of neighbouring particles. The direction of these forces depends on the angle between the distance vector separating two particles and the direction of the sound field. [47]Generally, these forces attract particles to one another in nodal or anti-nodal planes. The buoyancy force will move the lighter particles upward based on

$$F_B = \frac{4}{3}\pi r^3 g(\rho_l - \rho_p)$$

Equation 8: The Buoyancy Force

2.3.2 Boundary Condition of Usage of Ultrasound in Demulsification

Acoustofluidic devices are usually operated at resonance modes, which are controlled by the geometry of the device to a large extent. For continuous flow applications, the hydrodynamics of the system must be known to predict the movement of particles and droplets due to the fluid flow and the acoustic field. The response to excitation in acoustic devices must take into account the whole geometry of the device considering not only the propagation of the acoustic wave in the fluid but also the vibration of the structure and transducer [48].

CHAPTER 3

METHODOLOGY

3. METHODOLOGY

3.1 Introduction

This chapter presents the flowchart, Gantt chart, experimental parameters and experimental methodology. The purpose of this research is to study the impact of wave energy in ultrasound towards fluid and to discover the mean of ultrasound as a mechanical force to demulsify.

3.2 Datum

The oil shipped from wet-crude handling facilities must not contain more than 0.2% basic sediment and water (BS&W) and 10 pounds of salt per thousand barrels of crude oil.

In lab testing, the retention or treating time shall be kept less than 30 minutes.

3.3 Flowchart

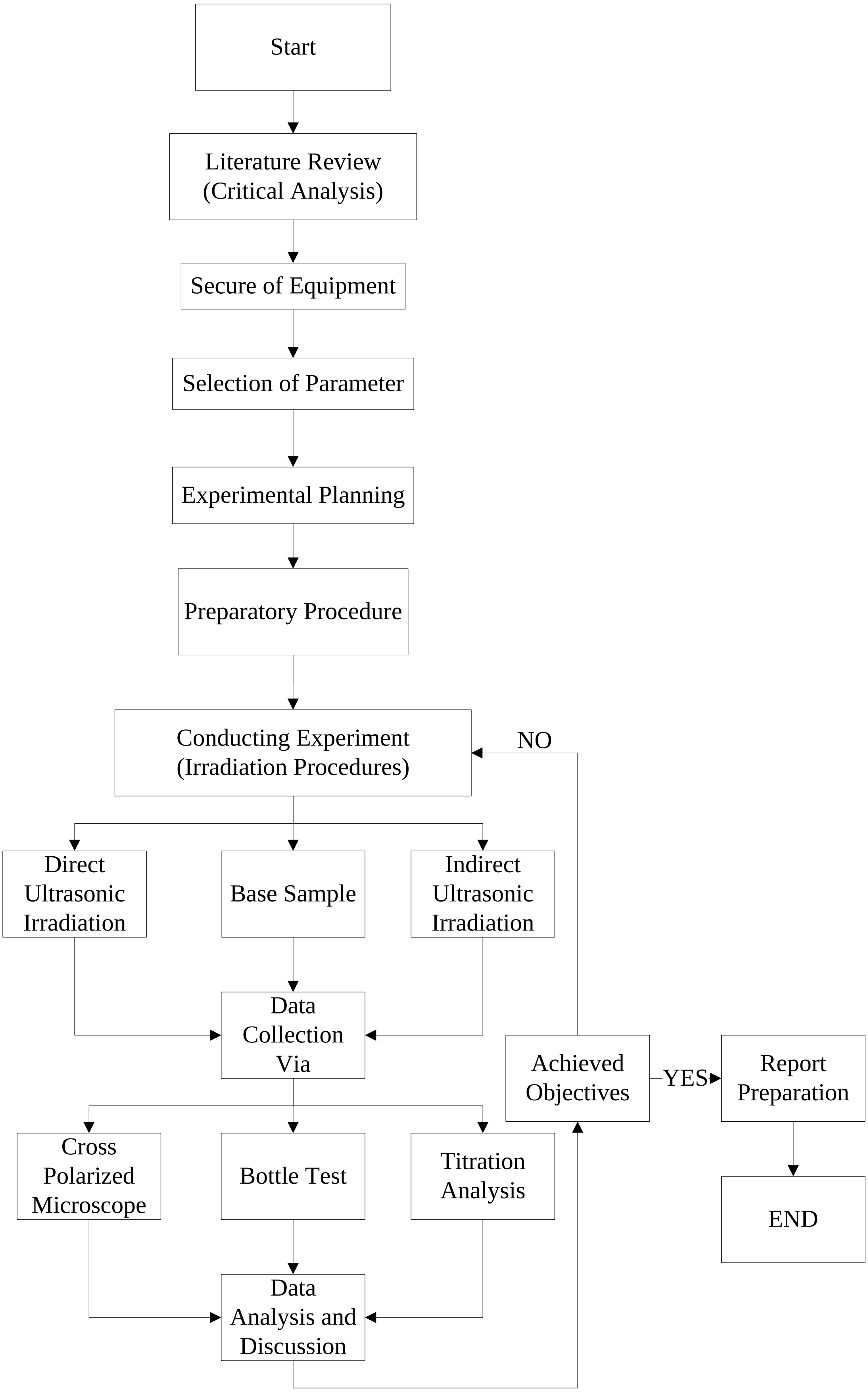


Figure 2: Experimental Flowchart

3.4 Experiment Methodology

3.4.1 Materials and Apparatus

The main material used is MOPU Sepat crude oil from Malaysia's field collected at Terengganu Crude Oil Terminal (TCOT). The composition of the produced water is mentioned in section 3.4.2. Among the materials needed for the produced water are $NaHCO_3$, KCl, NaCl, $BaCl:2H_2O$, $SrCl_2:6H_2O$, $MgCl:6H_2O$ and $CaCl:2H_2O$. The type of water used to produce the produced water is ultra-pure water.

A 100mL Pyrex beaker is used for the mixing of the formation of water-in-oil emulsion. A high speed stirrer (IKA T25 DS2) is placed into the beaker to supply the mixing energy to the water-oil mixture to agitate the mixture. Once ready, the crude type will be poured into test bottles and allowed for settlement. An ultrasonic processing equipment (SONICS VibraCell Model: VCX130FSJ) will be used to emit direct ultrasonic wave (20 kHz & 1000W) to the emulsion in the beaker. Whereas, Elma Ultrasonic Cleaner will be used to emit indirect ultrasonic wave (40 kHz & 1200W) to the emulsion in the beaker. A temperature probe will be placed in the beaker to monitor any temperature change. Throughout the study, the speed of ultrasound is maintained at 1480m/s in water and 1515m/s in crude [44]. The frequency of ultrasound is maintained at 20 kHz for direct irradiation and at 40 kHz for indirect irradiation.

3.4.2 Proposed Preparatory Procedure

1. Preparation of produced water.

   Parameters:

   - Stirrer speed : 400rpm
   - Duration of stirring : 1 hour
   - Type of water used : Ultra-pure water

Substance	Concentration (g/L)
$NaHCO_3$	5.1260
KCl	0.2646
NaCl	6.0114
$BaCl:2H_2O$	0.0067
$SrCl_2:6H_2O$	0.0141
$MgCl:6H_2O$	0.0750
$CaCl:2H_2O$	0.2344

Table 1: Selected Composition of Formation Water for Experiment

2. Formation of emulsion. The oil-water mixture is stirred at 9600 RPM using a high speed stirrer in a 100mL beaker.

Figure 3: Stirrer in Beaker for Emulsification

Parameters:

- Water cut is set at 30:70 and kept constant.
- Volume of emulsion: 100mL
- Duration of emulsification: 15 minutes
- Speed of stirrer: 9600 RPM
- Mixing temperature: Room Temperature

As recommended by Opawale and Osisanya [2], a ratio of 1:4 is used to obtain the stirrer speed to be used in the laboratory. This is also due to the limited speed range of the IKA T25 stirrer of between 3000 to 25000 RPM.

3.4.3 Proposed Irradiation Procedures

Base Sample

Type 1

After preparatory procedure, 100ml of emulsion is poured into 2 50ml test tube.

First specimen will be named **Type 1** which will be used as a base sample.

Second specimen will be named **Type 2** which will be used in indirect irradiation.

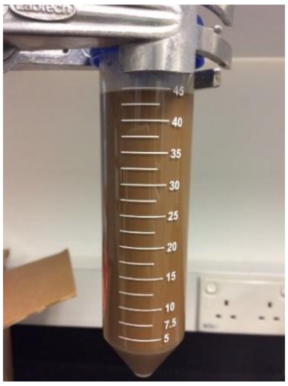


Figure 4: Base Sample in 50ml test tube

Indirect Ultrasonic Irradiation

Type 2

1. After one week of settlement, 50ml of emulsion is poured from 50ml test tube into 100ml conical flask and shaken.
2. Conical Flask is clamped and placed in the ultrasonic bath.
3. The level of emulsion is the same as water level.
4. Condition:
    a. Intensity: 100%
    b. Irradiation Time: 15mins
    c. Temperature: 26^{o}C
    d. Position: Position above the emitting ceramics and level of emulsion is the same as water level.

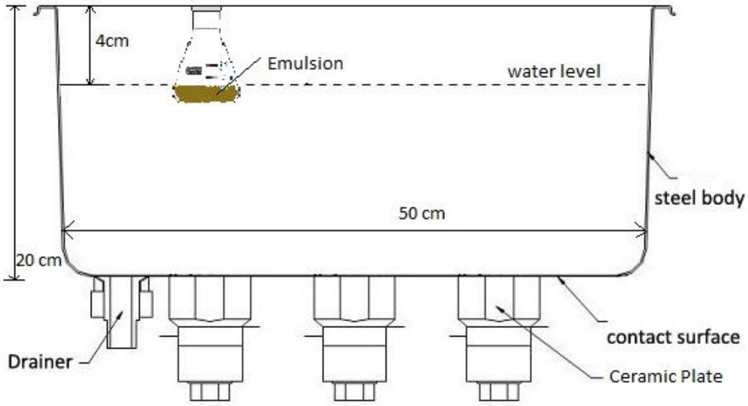


Figure 5: Position of Type 2 Specimen in Ultrasonic Bath

5. The specimen left to settle and observed till 35 minutes after irradiation.

Type 3

1. Specimen from **Type 2** is re-emulsified using Stirrer IKA T25 under 9600rpm for 15 minutes.
2. The conical flask is clamped and placed in the ultrasonic bath.
3. The level of emulsion is below the water level by 1cm.
4. Condition:
    a. Intensity: 100%
    b. Irradiation Time: 15 mins
    c. Temperature: 27^{o}C
    d. Position: Position above the emitting ceramics and level of emulsion is below the water level by 1cm.

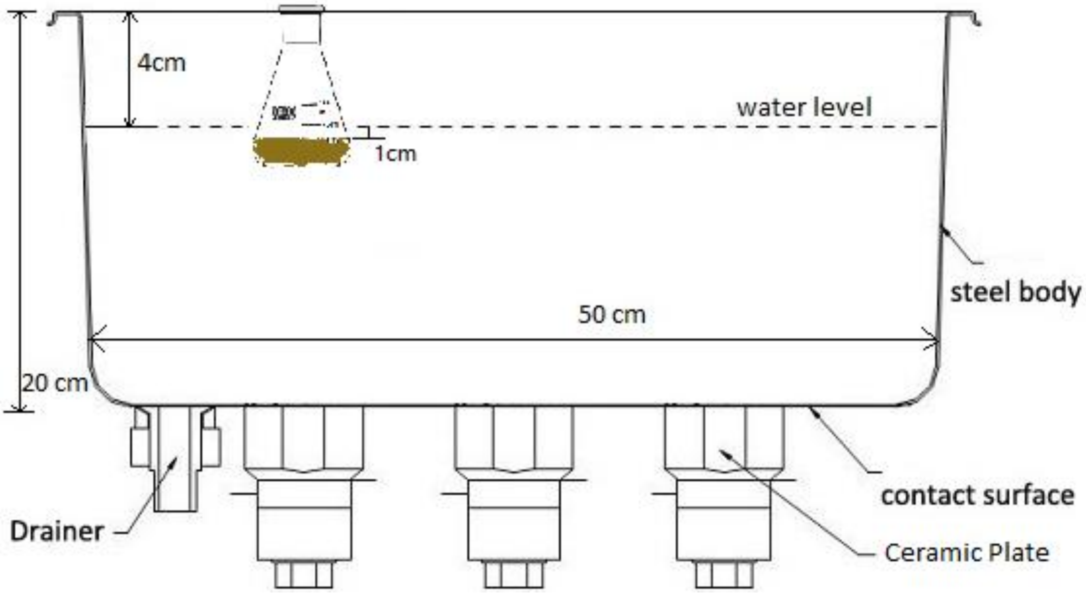


Figure 6: Position below Water Level by 1cm

5. The specimen left to settle and observed till 3 days after irradiation.

Type 4

1. Specimen from **Type 3** is re-emulsified using Stirrer IKA T25 under 9600rpm for 15 minutes.
2. The conical flask is clamped and placed in the ultrasonic bath.
3. The level of emulsion is at depth of odd number to the half of wavelength of ultrasound from the ceramic plate.
    a. Speed of ultrasound in water (V): 1480m/s
    b. Frequency of the ultrasonic bath (f): 40,000 Hz.
    c. Wavelength of Ultrasound (λ): V/f = 1515/40,000=0.037m=3.7cm.
    d. Half of wavelength: 1.85cm.
    e. Odd number of Half of the ultrasound wavelength (a multiplication factor of 7 is chosen) : 7x1.85cm=12.95cm

4. Condition:
    a. Intensity: 100%
    b. Irradiation Time: 20 mins
    c. Temperature: 27°C
    d. Position: Position above the emitting ceramics and bottom level of conical flask is at depth 12.95cm from the base.

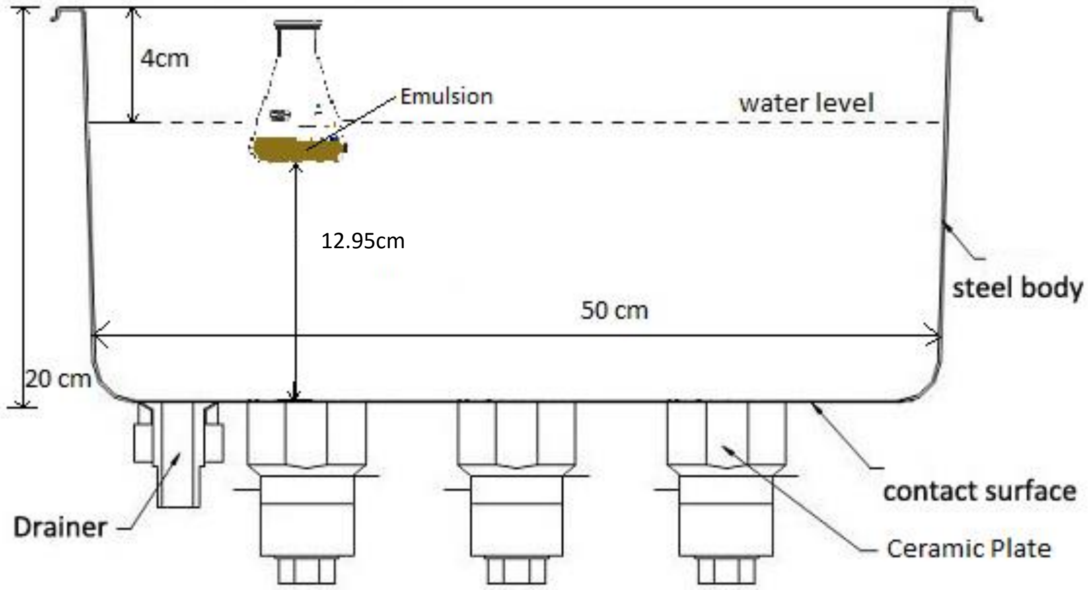


Figure 7: Position at odd number of half of wavelength from the base

5. CPM and titration analysis were conducted on the specimen after 5 days since irradiation.

Direct Ultrasonic Irradiation

Type 5

1. After preparatory procedure, 50ml of emulsion is poured into 50ml test tube
2. Test tube is placed on a conical flask in the direct ultrasonic chamber.
3. The ultrasonic probe is placed in the middle (25ml) of the test tube.
4. Condition:
    a. Intensity: 30%
    b. Irradiation Time: 15mins
    c. Temperature: 27°C

Type 6

1. After preparatory procedure, 50ml of emulsion is poured into 50ml test tube
2. Test tube is placed on a conical flask in the direct ultrasonic chamber.
3. The ultrasonic probe is placed in the middle (25ml) of the test tube.
4. Condition:
    a. Intensity: 40%
    b. Irradiation Time: 15mins
    c. Temperature: 27C

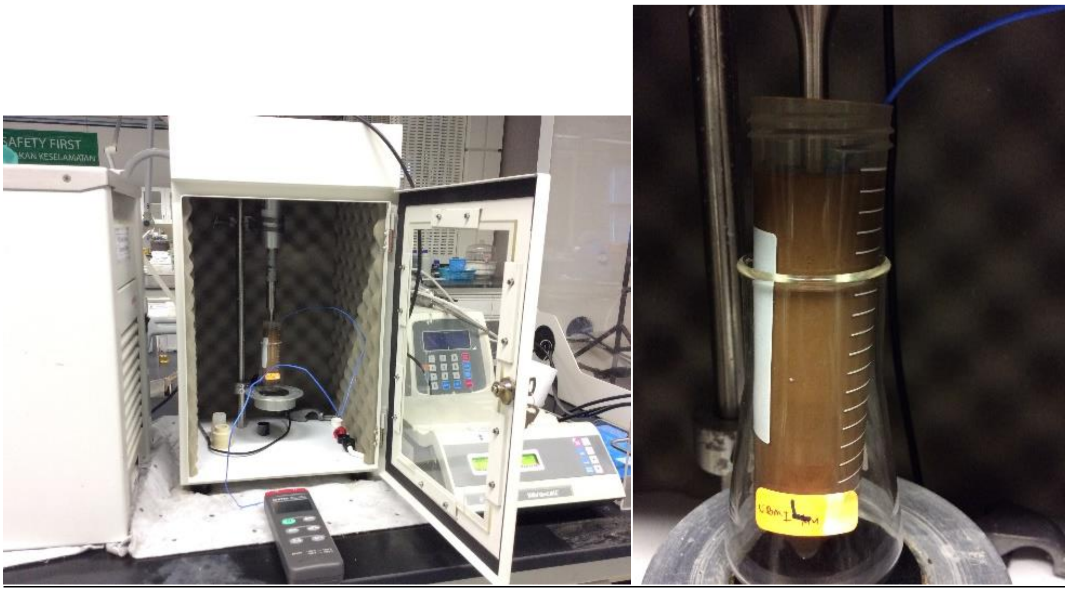


Figure 8: Type 5 and Type 6 Irradiation Configuration

Type 7

1. After preparatory procedure, 50ml of emulsion is poured into 80ml beaker.
2. Beaker is placed in the direct ultrasonic chamber.
3. The ultrasonic probe is placed at distance of odd number to the half of wavelength of ultrasound from the reflective plate.
    - **a.** Speed of ultrasound in emulsion (V): 1515m/s
    - **b.** Frequency of the ultrasonic bath (f): 20,000 Hz.
    - **c.** Wavelength of Ultrasound (λ): V/f = 1515/20,000=0.076m=7.6cm.
    - **d.** Half of wavelength: 3.78cm.
    - **e.** Odd number of Half of the ultrasound wavelength (a multiplication factor of 1 is chosen due to space limitation) : 1x3.78cm=3.78cm
4. Condition:
    - a. Intensity: 30%
    - b. Irradiation Time: 15mins
    - c. Temperature: 27^{o}C
    - d. Position: Tip of ultrasonic probe is 3.78cm from the reflective plate.

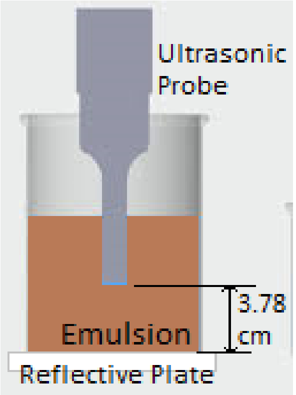


Figure 9: Position of Ultrasonic Probe in Type 7

5. CPM and Titration Analysis are conducted after 3 days of settling time.

3.4.4 Data Analysis

For a thorough and reliable result, three different measurement analysis are conducted for the emulsion sample. Firstly is the cross-polarized microscope (CPM) where microscopic image of the emulsion after exposing to ultrasonic wave is taken. Second, the titration method is used to study the water content in the crude oil emulsion. Finally, the bottle test is used to determine the thickness of rag layer, water layer and emulsion layer.

Bottle Test

1. After irradiation, the crude specimen will be poured into a standard 50mL PVC bottle. The bottle will immediately be placed inside a mechanical oven to maintain its temperature at 60°C to avoid any wax formation.
2. The bottle will be inspected at the time interval listed below to determine the thickness of rag layer, emulsion layer and water layer.

Time Since	Thickness (mm)		
	Rag Layer	Emulsion Layer	Water Layer
5mins			
15mins			
30mins			
1hour			
2hour			
4hour			
2nd day			
3rd day			
4th day			
5th day			
6th day			
7th day			
2nd week			

3. A total of 3 bottles tests (**Type 1, Type 2 and Type 3**) will be conducted. The result will be recorded and analysed to decide the settling time.
4. **Type 4, Type 5, Type 6 and Type 7** will not undergo bottle test because the there is no distinct separation layer in the specimen.

Cross-Polarized Microscope

1. Upon pouring the irradiated crude into the bottle, a few drops of emulsion from the specimen will be put onto microscope glass slide. Cover glass will be placed on top of it. No bubble trapped under the cover glass.
2. Specimen will be placed on the Hot Stage. The specimen is placed above the observation point (the middle hole) of the Hot Stage.
3. CP lens will be adjusted to normal position. Hot Stage is aligned until a clear view obtained. Then, CP lens will be changed to Cross-Polarized position.
4. Knob Adjuster is adjusted to get a clearer image.
5. Emulsion droplets can be measured at nm scale. The biggest water droplet and the number of water droplets in 100nm will be measured.
6. Specimen is removed. Cover glass and glass slide will be clean.
7. Microscope and computer system will be turned off.
8. CPM will be tested on specimen **Type 1, Type 4, Type 5, Type 6 and Type 7**.

Titration Test

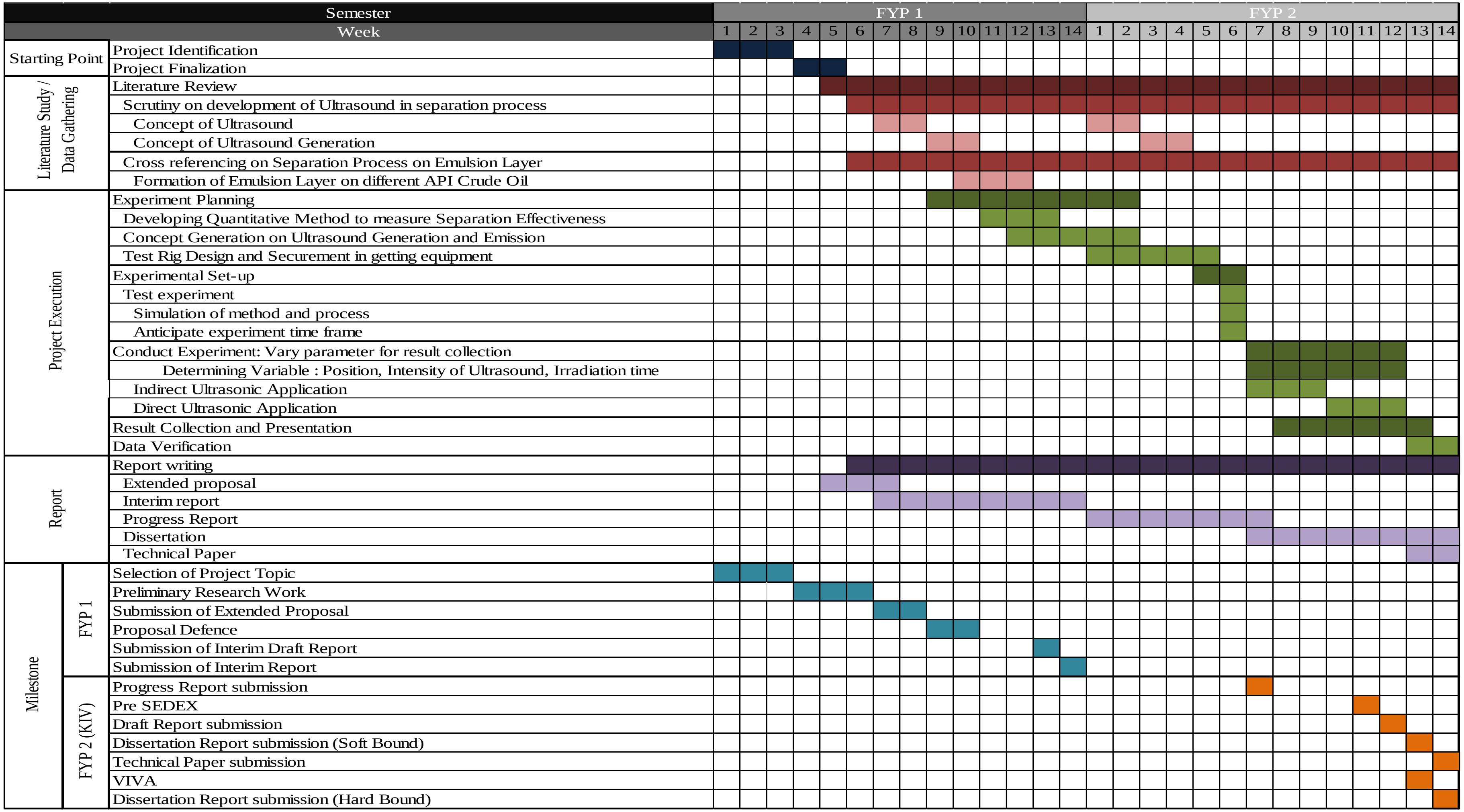
1. Drift determination is performed with an empty vial with metal insert on the first position (drift) of the rack. The drift value is defined as the moisture entered into the titration cell by purge gas.
2. The blank value will be determined. It is the amount of water contained in an empty sample vial which sealed with aluminium foil minus the amount of water due to drift.
3. Sample determination will be done by weighing 10mL of the specimen into a sample vial and aluminium foil will be used to seal it immediately.
4. Stomboli transfer tube is connected to the V30 titration vessel.
5. Stomboli oven will be turned on.
6. An empty vial with metal insert will be placed on the sample rack followed by Blank and Sample.
7. Method to start KF analysis is selected.
8. Titration test will be tested with **Type 1, Type 4, Type 5, Type 6 and Type 7** to determine the water content in the emulsion

3.4.5 Summary of Specimen Irradiation Testing and Data Collection

Type	Condition	Bottle Test	CPM	Titration Analysis
Base Sample				
1		√	√	√
Indirect Ultrasonic Irradiation				
2	a. Intensity: 100% b. Irradiation Time: 15mins c. Temperature: 26C d. Position: Position above the emitting ceramics and level of emulsion is on par with water level.	√		
3	a. Intensity: 100% b. Irradiation Time: 15 mins c. Temperature: 27C d. Position: Position above the emitting ceramics and level of emulsion is below the water level by 1cm.	√		
4	a. Intensity: 100% b. Irradiation Time: 20 mins c. Temperature: 27C d. Position: Position above the emitting ceramics and bottom level of conical flask is at depth 13.26cm from the base.		√	√
Direct Ultrasonic Irradiation				
5	a. Intensity: 30% b. Irradiation Time: 15mins c. Temperature: 27C d. Position: At the middle of the test tube (25ml)		√	√
6	a. Intensity: 40% b. Irradiation Time: 15mins c. Temperature: 27C d. Position: At the middle of the test tube (25ml)		√	√
7	a. Intensity: 30% b. Irradiation Time: 15mins c. Temperature: 27C d. Position: Tip of ultrasonic probe is 3.78cm from the reflective plate.		√	√

Table 2: Summary of Specimen Irradiation Testing and Data Collection

3.5 Gantt Chart



Semester			FYP 1														FYP 2													
Week			1	2	3	4	5	6	7	8	9	10	11	12	13	14	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Starting Point		Project Identification	■	■	■																									
		Project Finalization				■	■																							
Literature Study / Data Gathering		Literature Review					■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■
		Scrutiny on development of Ultrasound in separation process						■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■
		Concept of Ultrasound							■	■							■	■												
		Concept of Ultrasound Generation									■	■							■	■										
		Cross referencing on Separation Process on Emulsion Layer						■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■
		Formation of Emulsion Layer on different API Crude Oil										■	■	■																
Project Execution		Experiment Planning									■	■	■	■	■	■	■	■												
		Developing Quantitative Method to measure Separation Effectiveness											■	■	■															
		Concept Generation on Ultrasound Generation and Emission												■	■	■	■	■												
		Test Rig Design and Securement in getting equipment															■	■	■	■	■									
		Experimental Set-up																			■	■								
		Test experiment																				■								
		Simulation of method and process																				■								
		Anticipate experiment time frame																				■								
		Conduct Experiment: Vary parameter for result collection																					■	■	■	■	■	■		
		Determining Variable : Position, Intensity of Ultrasound, Irradiation time																					■	■	■	■	■	■		
		Indirect Ultrasonic Application																					■	■	■					
		Direct Ultrasonic Application																								■	■	■		
		Result Collection and Presentation																						■	■	■	■	■	■	
		Data Verification																											■	■
Report		Report writing						■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■
		Extended proposal					■	■	■																					
		Interim report							■	■	■	■	■	■	■	■														
		Progress Report															■	■	■	■	■	■	■							
		Dissertation																					■	■	■	■	■	■	■	■
		Technical Paper																											■	■
Milestone	FYP 1	Selection of Project Topic	■	■	■																									
		Preliminary Research Work				■	■	■																						
		Submission of Extended Proposal							■	■																				
		Proposal Defence									■	■																		
		Submission of Interim Draft Report													■															
		Submission of Interim Report														■														
	FYP 2 (KIV)	Progress Report submission																					■							
		Pre SEDEX																									■			
		Draft Report submission																										■		
		Dissertation Report submission (Soft Bound)																											■	
		Technical Paper submission																												■
		VIVA																											■	
		Dissertation Report submission (Hard Bound)																												■

Figure 10: Gantt chart

3.6 Engineering Drawing

Model of Direct Ultrasonic Standing Wave Resonator

Figure 11: Model of Standing Wave Resonator

Model of Indirect Ultrasonic Bath Chamber

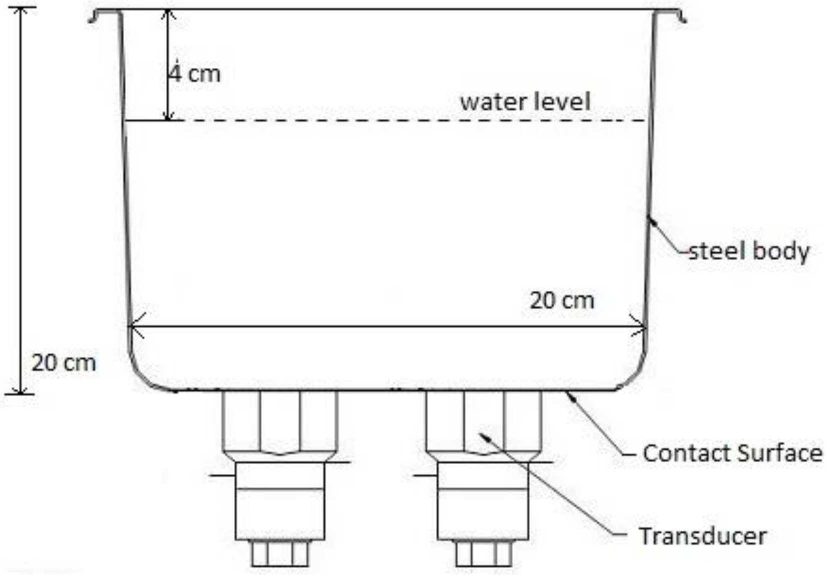


Figure 12: Indirect Ultrasonic Bath (Front View)

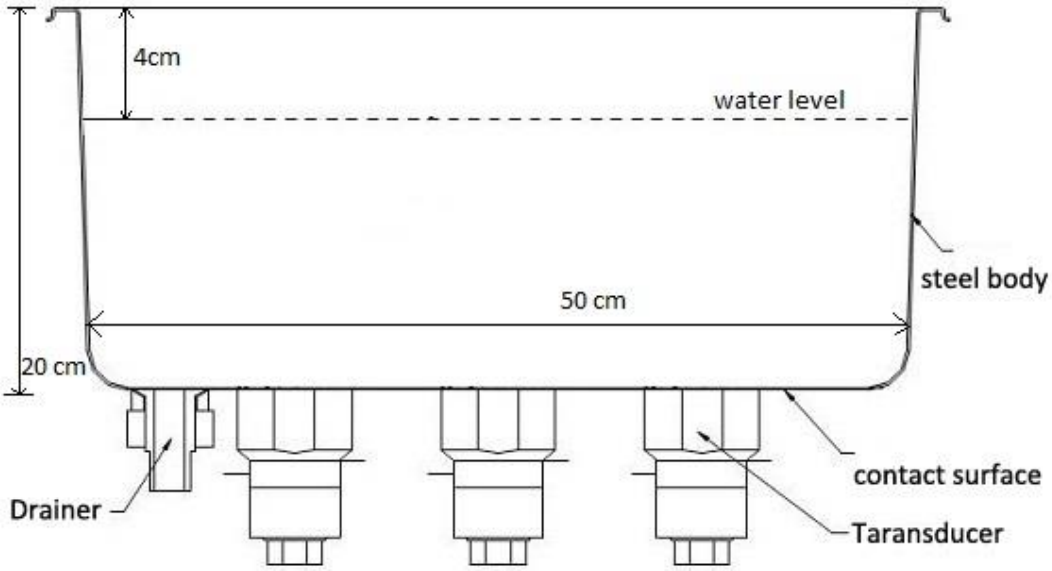


Figure 13: Indirect Ultrasonic Bath (Side View)

CHAPTER 4

RESULT AND DISCUSSION

4. RESULT AND DISCUSSION

4.1 Bottle Test Analysis

(TYPE 1)

Result of the Bottle Test for Original (Base Sample) Specimen			
Time (mins)	Thickness of Layer Type (ml)		
	Crude Layer	Emulsion Layer	Water Layer
0	0	44.7	0
5	0.2	44.4	0
15	0.3	44.3	0
30	0.3	44.3	0
60	0.4	44.2	0
120	0.5	44.1	0
240	0.8	43.6	0
2880	1.2	43.2	0
4320	1.4	42.9	0
5760	1.9	42.5	0
7200	2.1	42.2	0
8640	2.3	42	0
11520	2.7	41.5	0
12960	2.8	41.3	0
18720	4.1	39.8	0

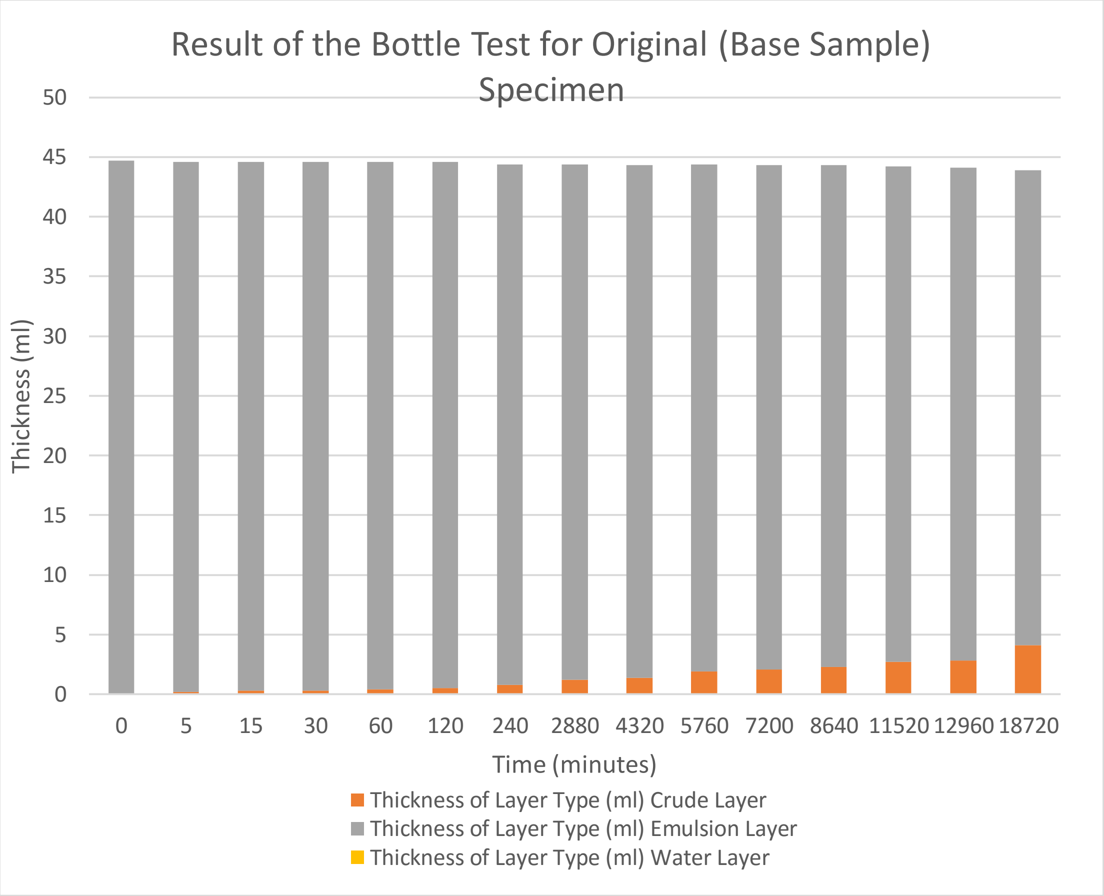


Figure 14: Bottle Test Result for Base Sample

Mins	0	5	15	30	60	120	240	2880	4320	5760	7200	8640	11520	12960	18720
Days	0	0	0	0	0	0	0	2	3	4	5	6	8	9	13
Hour	0	0	0	0	1	2	4	48	72	96	120	144	192	216	312

From Figure 14, the crude layer increases progressively over time. The average settling rate is 1.5873 x 10 $^{-4}$ ml per minutes. The water level is not seen after two weeks nor is growing. We can conclude that this crude type will yield stubborn emulsion which cannot be separated by gravitational force. If the result is further postulated to another 2 weeks, the separation of crude is still ineffective.

(TYPE 2)

Discussion on Indirect Ultrasonic Irradiation for **Type 2** Specimen

Result of the Bottle Test for Indirect Ultrasonic Irradiation for **Type 2** Specimen			
Time (mins)	Thickness of Layer Type (ml)		
	Crude Layer	Emulsion Layer	Water Layer
0	0	52.5	0
15	1.25	51.25	0
35	1.25	51.25	0

After a week of settlement in the 50ml test tube, the emulsion is poured into a conical flask. The crude immediately re-emulsified, indicating that the coalescent force among the particles is not strong against the agitation force. After irradiated for 15 minutes, the specimen is left to settle for another 15 minutes. Results after 35 minutes of settling time showed no difference in the emulsion and separated layers.

The settling rate is then zero. Throughout the indirect radiation process, the emulsion level was ensured to be at the same level of water within the water bath. It is anticipated that the emulsion will experience shock wave under such condition [49]. However, study on this one week old emulsion proved that the method is ineffective

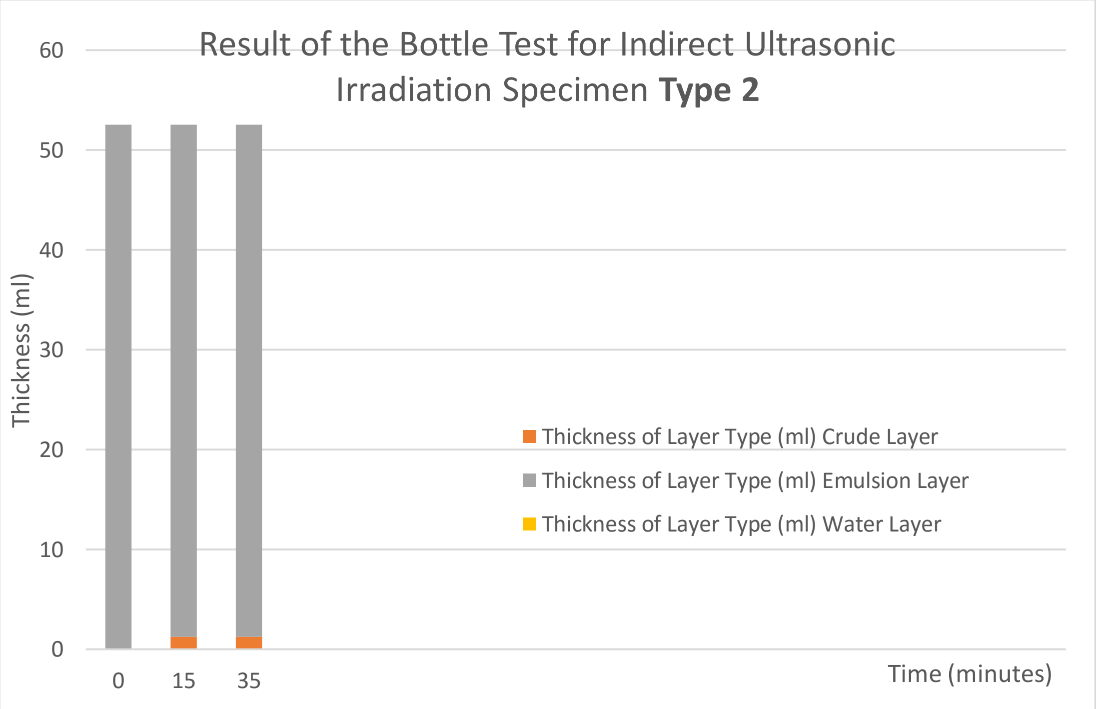


Figure 15: Bottle Test Result for Test 2 Specimen

(TYPE 3)

Discussion on Indirect Ultrasonic Irradiation for **Type 3** Specimen

Result of the Bottle Test for Indirect Ultrasonic Irradiation **Type 3** Specimen			
Time (mins)	Thickness of Layer Type (ml)		
	Crude Layer	Emulsion Layer	Water Layer
0	0	52.5	0
15	0.625	51.875	0
30	0.625	51.875	0
1383	2.5	50	0
2990	3.125	49.375	0
4438	3.75	48.75	0

The settling time is still slow and ineffective. It still cannot fulfil the industrial standard which requires the settling time to be below 30minutes. However, with the specimen left untouched for a few days, the settling rate is remarkably higher than the original condition. The average settling rate is achieved at 8 x 10^{-4} ml per minute. This rate is taken at the best straight line plotted with crude layer thickness versus time. Hence, it does not reflect the initial settling rate and that we can only conclude that settling rate is higher after a day.

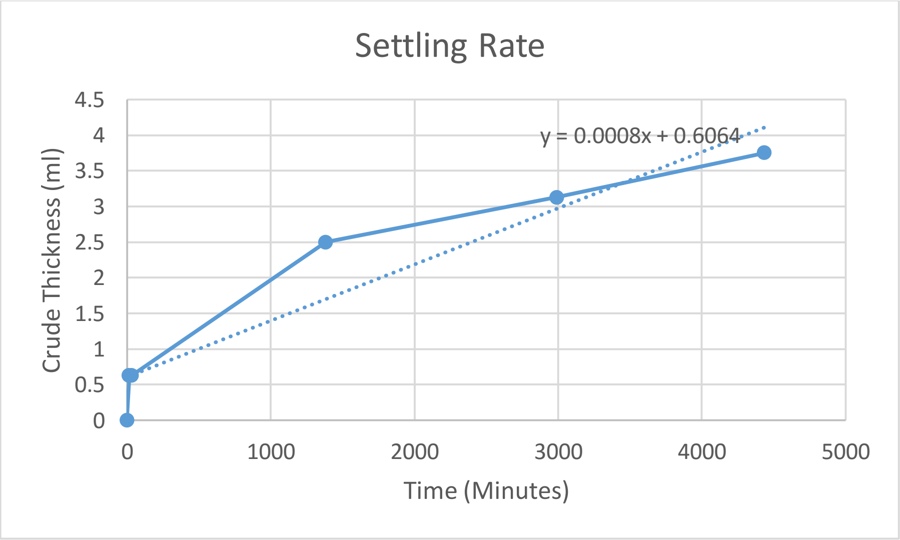


Figure 16: Settling Rate for Type 3

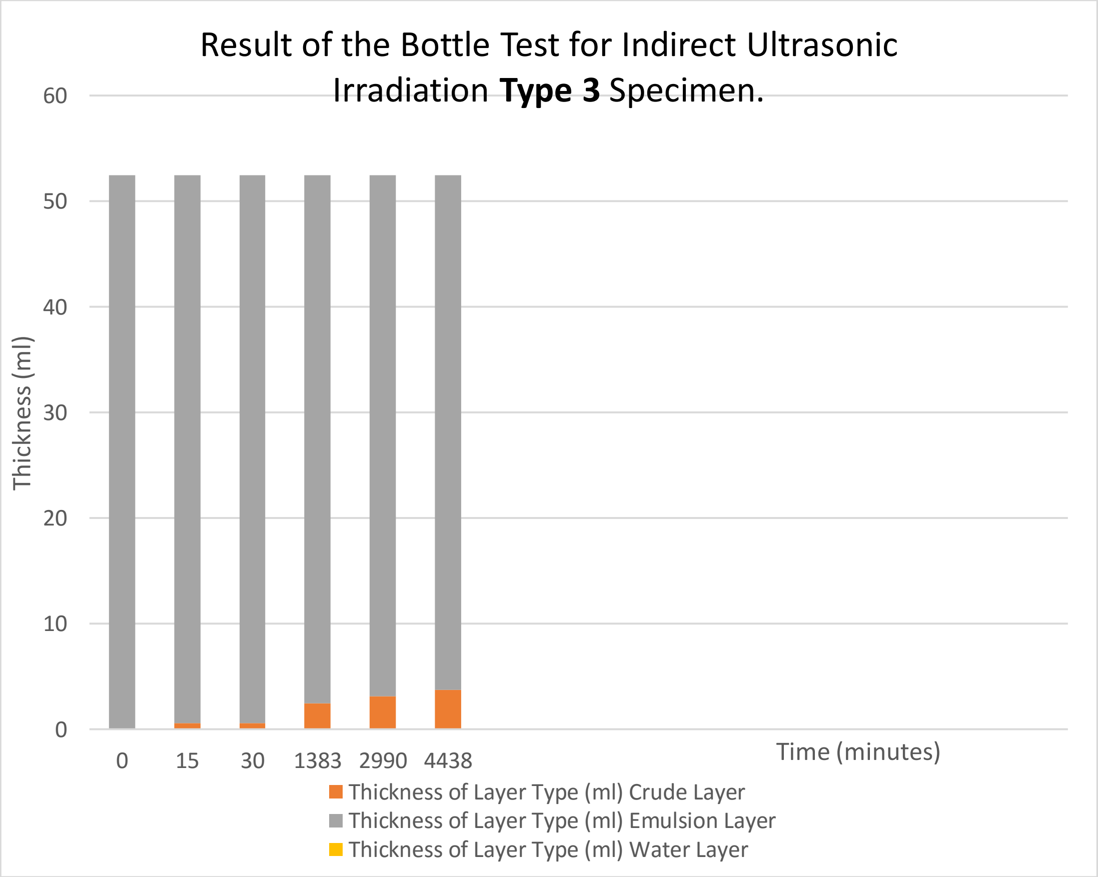


Figure 17: Bottle Test Result for Type 3

(TYPE 4)

No bottle test was conducted due to lack of distinct separation in emulsion.

Figure 18: Type 4 Specimen after Irradiation

(TYPE 5, 6 and 7)

No bottle test was conducted due to lack of distinct separation in emulsion.

There is no distinct difference among three specimens before and after the ultrasonic irradiation. The only difference is a colour change to the emulsion at the location where the probe is located. Further analysis using CPM and titration analysis should give further insight on the changes.

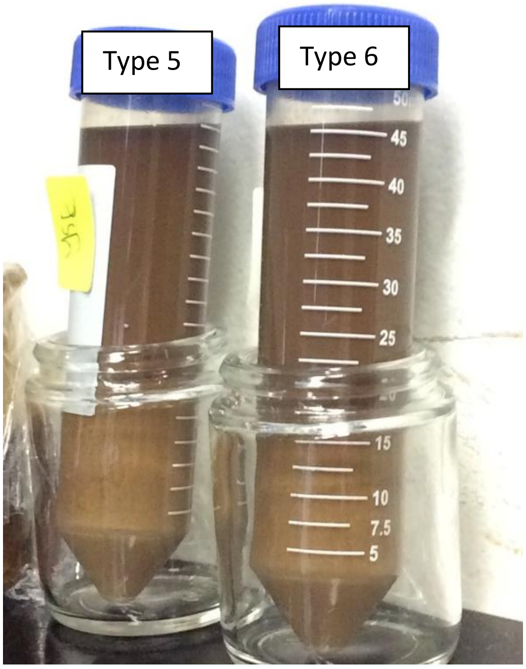


Figure 19: Type 6 and Type 7 after irradiation

4.2 Titration Analysis

Titration analysis was conducted with **Type 1, Type 4, Type 5, Type 6 and Type 7.** It is conducted to determine the percentage of water content by weight in the sample. **Type 1, Type 5 and Type 6** is separated into layers as shown in Figure 21. **Type 4** is separated into layers as shown in Figure 20. **Type 7** is separated into layers as shown in Figure 22.

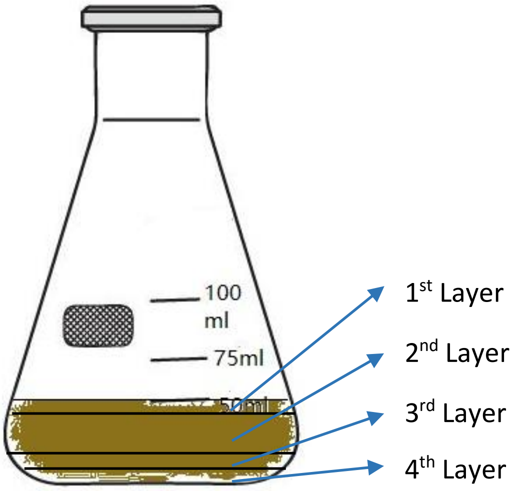


Figure 20: Layers in Conical Flask

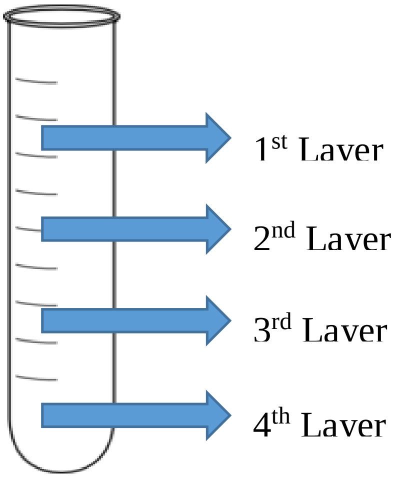


Figure 21: Layers in Test Tube

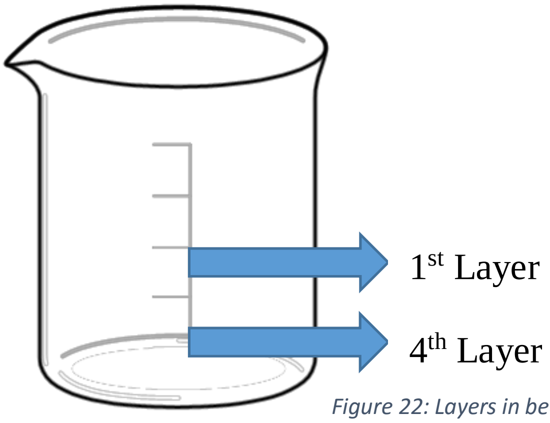


Figure 22: Layers in beaker

Remark: The layers 'assignment is for recognition purpose. It compares the layers relatively. It does not reflect the real thickness of each layer. Type 4, type 5, type 6 and type 7 lack of distinct separation in emulsion and thus thickness in each layer is difficult to determine.

<u>(Type 1)</u>

Type 1 has only two distinct layers which are 1st layer and 4th layer. The 1st layer has 0.108% of water content by weight and the 4th layer has 28.062% of water content by weight. The 1st layer cannot achieve 0% of water content after 2 weeks of settlement concludes the emulsion is a stubborn emulsion.

(Type 4)

Type 4 has only three distinct layers which are 1st layer, 3rd layer and 4th layer. The 1st layer has 0.935% of water content by weight whereas 3rd layer has 52.601% and 4th layer has 85.932%. Indirect ultrasonic irradiation has shown the most promising mean to demulsify in this research. It can be further proven in CPM test. Type 4 has the highest water weight percentage at the bottom of the emulsion layer.

(Type 5)

Type 5 has 4 distinct layers which are 1st layer, 2nd layer, 3rd layer and 4th layer with 0%, 1.461%, 6.433% and 24.578% of water weight respectively. Although 4th layer yields lower water weight percentage than the 4th layer of Type 1, the overall demulsification performance of Type 5 is more superior as more water content found in 2nd and 3rd layer.

(Type 6)

Type 6 has 4 distinct layers which are 1st layer, 2nd layer, 3rd layer and 4th layer with 0%, 2.527%, 23.940% and 26.320% of water weight respectively. The overall demulsification performance is more superior to Type 5 because it has more water content found in bottom three layers. It concludes that 40% intensity has better effect than 30% intensity on demulsification. Type 6 is more superior to Type 1 because it has more water content in 2nd layer and 3rd layer.

(Type 7)

Type 7 has only 2 distinct layers which are 1st and 4th layer with 0.240% and 28.287% of water weight respectively. Type 7 has more water weight percentage than Type 1 in 1st and 4th layer. Comparably to Type 5 and Type 6, Type 7 has higher water weight percentage in 1st layer but no water content can be found in 2nd layer and 3rd layer. In conclusion, Type 7 is least effective to demulsify.

Overall Discussion

The result of titration analysis is not conclusive as the thickness of each layer is varied in a specimen and among the specimen as well. The finding can be reinforced with analysis from CPM.

4.3 CPM Analysis

CPM analysis was conducted with **Type 1, Type 4, Type 5, Type 6 and Type 7.** It is conducted to determine the mean droplets diameter in a sample. **Type 1, Type 5 and Type 6** is separated into layers as shown in Figure 21. **Type 4** is separated into layers as shown Figure 20. **Type 7** is separated into layers as shown in Figure 22.

(Type 1)

Type 1 has two layers with average 3.57µm of water droplets appear in 1st layer and 36.91µm of oil droplets appear in 4th layer. As agreed with the titration analysis, Type 1 is a stubborn emulsion because after two weeks of settlement, water droplets still appear in 1st layer and relatively large oil droplets still appear in 4th layer.

(Type 4)

Type 4 has 5.84µm of scattered suspended particles in 1st layer, 6.75µm of oil droplets in 2nd layer, 4.03µm of water droplets in 3rd layer and 31.11µm of water droplets in 4th layer. Among direct ultrasonic irradiation experiments, type 4 has the biggest average water droplet diameter in 4th layer. Compared to Type 1, Type 4 gives positive impact towards demulsification.

(Type 5)

Type 5 has 7.02µm of scattered suspended particles in 1st layer, 28.26µm of suspended particles and 6.47µm of water droplets in 2nd layer, 9.35µm of water droplets in 3rd layer and 6.54µm of water droplets in 4th layer. Type 5 has a better demulsification effect than Type 4 because the biggest water droplet in diameter can be seen in 4th layer of Type 5 (Appendix 2). Water droplets coalesce more to form bigger particle in Type 5.

(Type 6)

Type 6 has 12.54µm of scattered suspended particles in 1st layer and 13.80µm of water droplets in 4th layer. Suspended particles appear in all irradiated specimen (Type 4, Type 5, Type 6 and Type 7) because ultrasonic irradiation separates suspended particles from crude oil.

(Type 7)

Type 7 has 11.54µm of scattered suspended particles in 1st layer, 5.61µm of water droplets in 3rd layer and 41.69µm of water droplets in 4th layer. Among all irradiated specimen, Type 7 gives the best demulsification performance. It has the biggest average water droplets diameter at the bottom layer of the specimen.

Overall Discussion

Data analysed using CPM is not 100% accurate. This is because the extraction taken is just part of the “layer”. The mixture in the layer is not homogeneous.

4.4 Summary of Result from Titration and CPM Analysis

Titration Analysis					
Type	Specimen	4th Layer	3rd Layer	2nd Layer	1st Layer
1	Base Sample	28.062%			0.108%
4	Indirect Ultrasonic Irradiation	85.932%	52.601%		0.935%
5	Direct Ultrasonic Irradiation at 30% Intensity	24.578%	6.433%	1.461%	0.000%
6	Direct Ultrasonic Irradiation at 40% Intensity	26.320%	23.940%	2.527%	0.000%
7	Direct Ultrasonic Irradiation at half of wavelength at 40% Intensity	28.287%			0.240%

Cross Polarized Microscope (CPM) Analysis		Average particle diameter in the extraction taken from each layer			
Type	Specimen	4th Layer	3rd Layer	2nd Layer	1st Layer
1	Base Sample	36.91µm of oil droplets			3.57µm of water droplets
4	Indirect Ultrasonic Irradiation	41.69µm of water droplets	5.61µm of water droplets		11.54µm of suspended particles
5	Direct Ultrasonic Irradiation at 30% Intensity	31.11µm of water droplets	4.03µm of water droplets	6.75µm of oil droplets	5.84µm of suspended particles
6	Direct Ultrasonic Irradiation at 40% Intensity	6.54µm of water droplets	9.35µm of water droplets	28.26µm suspended particle & 6.47µm of water droplets	7.02µm suspended particles
7	Direct Ultrasonic Irradiation at half of wavelength at 40% Intensity	13.80µm of water droplets			12.54µm of suspended particles

Table 3: Summary of Result from Titration and CPM Analysis

*Comprehensive test result from Titration Analysis can be obtained from Appendix A

*Comprehensive test result from CPM Analysis can be obtained from Appendix B

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5. CONCLUSION AND RECOMMENDATION

5.1 Conclusion

It is proven that ultrasound has a positive impact towards separation of water from crude layer in emulsion. However, it is yet to conclude that ultrasound is more superior in term of performance compared to other means. Based on the intensive and rigorous study, ultrasound can be a more effective mean to demulsify. Wave energy of ultrasound will be converted to kinetic energy which will eventually coalesce liquid particles. Research finding is vital for further research and can be acted as a reference to oil and gas industry.

5.2 Recommendation

Next phase of research shall be focused on either direct ultrasonic application or indirect ultrasonic application. Design of Experiment (DOE) shall be recommended for the next phase of research as a more comprehensive coverage of all possible determining variables to optimize the output is needed. Different crude type from different field at different water cut can be added to determining variables. Further study is required to understand the reasons for ultrasound application at certain parameter is working in certain crude oil but not in others.

CHAPTER 6

REFERENCE

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APPENDIX

APPENDIX A

APPENDIX B