Effect of Drilling Mud Contamination on the Properties of Waxy Crude Oil

by

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Mechanical Engineering Programme
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(MECHANICAL)

Approved by,

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(Dr Azuraien binti Japper @ Jaafar)

Universiti Teknologi PETRONAS

TRONOH, PERAK

May 2014

CERTIFICATION OF ORIGINALITY

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 acknowledgement, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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MUHAMMAD HASSANEI HEYKAL BIN AZMI

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ABSTRACT

The operation of well drilling requires the use of drilling fluid which, in this case is drilling mud. Drilling mud is very crucial in the drilling process. Without it, it is nearly impossible to drill wells with the rotary method. The use of drilling mud during drilling process in the exploration of newly found reservoir may affect the crude oil. Problem occurs when the drilling mud mixed with the crude oil at the reservoir being drilled at earlier stage. This occurrence leads to contamination of waxy crude oil with the drilling mud. Due to that, the properties of crude oil such as pour point temperature, wax appearance temperature (WAT) and wax disappearance temperature (WDT) and yield stress may change.

These real properties are very crucial to for an engineer to design any facilities related in handling such hydrocarbons. The hydrocarbon sample is usually taken at the bottom of the well at the early stage of the production. The contamination of drilling mud into waxy crude oil may lead to inaccurate properties when the sample is examined. This may further lead to wrong data used in designing certain facilities, especially for upstream facilities. Therefore, it is important to study the effects of the presence of drilling mud on the property of the crude oil (which in this case is waxy crude oil) extracted. Several related test methods are used in order to study the behavior and characteristics of the contaminated crude oil. These test methods include bottle test, pour point test, rheology test and microscopic observation.

Bottle test results showed that the mixture requires sometimes to stabilize and separate into its individual component. Nevertheless, complete separation was never achieved during the observation period and created intermediate layer (densed mixture between waxy crude oil and the drilling mud). The work found that the presence of drilling mud did not significantly affected that pour point and freezing point of waxy crude oil, however, it change the WAT and yield stress considerably. Based on the work, it is suggested that that the fresh sample taken from the newly found reservoir was subjected to a bottle test for three days to settle down the drilling mud and get waxy crude oil layer. After that, waxy crude oil from the upper layer can be collected and tested to reveal the real properties.

TABLE OF CONTENT

LIST OF FIGURES

LIST OF TABLES

LIST OF ABBREVIATIONS

API	American Petroleum Institute
ASTM	American Society for Testing and Materials
DOE	Design of Experiment
HTGC	High Temperature Gas Chromatograph
PCSB	PETRONAS Carigali Sendirian Berhad
WAT	Wax Appearance Temperature
WDT	Wax Disappearance Temperature

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

The operation of well drilling requires the use of drilling fluid, in this case drilling mud. Drilling mud is very crucial in the drilling process. Without it, it is nearly impossible to drill wells with the rotary method. Drilling mud is used for various functions as will be explained later in this report. During the drilling process especially for new exploration field, the drilling mud is continuously flow in the well string until the first oil reservoir is reached which is at the down hole. For a new exploration well that has been drilled, engineer usually conduct a proper sampling procedure such that representative sample could be preserved. Sampling could be conducted at the surface or at the bottom hole. For exploration wells, most of the samplings are conducted down hole. One of the issues with the bottom hole sampling is drilling mud contamination. Due to that, there is a tendency that the drilling mud will contaminate the crude oil during the process. The physical properties of the crude oil may change due to the contamination.

The study is needed in order to investigate the flow behaviour and characteristics of the waxy crude oil due to the contamination. In addition, the wax content in the crude oil also leads to other problem. It leads to severe problem on the transportation of crude oil in the pipeline. Problem such as wax deposited in the pipelines lead to major financial lost due to the prevention cost. In order to avoid such problem, it is very crucial to investigate the flow behaviour of the contaminate crude oil. Apart from that, the real properties are very crucial to be obtained for an engineer to design any facilities related in handling such hydrocarbons. The contamination of drilling mud into waxy crude oil may lead to inaccurate properties when the sample is examined. This may further lead to wrong data used in designing certain facilities, especially for upstream facilities. The study is intended to analyze the effects of the drilling mud contamination on the rheological properties, WAT, WDT and pour point at concentrations ranging from 5% to 20% weight to weight ratio on a waxy crude oil at stock tank condition.

1.2 PROBLEM STATEMENT

The use of drilling mud during drilling process in the exploration of newly found reservoir may affect the crude oil. Problem occurs when the drilling mud used mixed with the crude oil at the reservoir being drilled at earlier stage. During the drilling process especially for new exploration field, the drilling mud is continuously flow in the well string until the first oil reservoir is reached which is at the down hole. For a new exploration well that has been drilled, engineer usually conduct a proper sampling procedure such that representative sample could be preserved. Sampling could be conducted at the surface or at the bottom hole. For exploration wells, most of the samplings are conducted down hole. One of the issues with the bottom hole sampling is drilling mud contamination.

There is a strong tendency that drilling mud will contaminate the waxy crude oil during exploration of newly found reservoir. The physical properties of the waxy crude oil such as WAT, WDT, pour point, freezing point and yield stress may change due to the contamination which will lead to misinterpretation or wrong data acquisition. If the effects of drilling mud contamination are not well understood, engineers may not appropriately design related upstream facilities that can create problem in the future.

1.3 OBJECTIVE

This project which entitles “Effect of Drilling Mud Contamination on the Properties of Waxy Crude Oil” has several objectives detailed below:

i. To study the properties and flow behaviour of waxy crude oil due to the contamination with different concentration of drilling mud using various tests method such as bottle test, pour point test, rheometer test and microscopic observation.
ii. To be able to provide protocol to determine real waxy crude oil properties.

1.4 SCOPE OF STUDY

The project is conducted in order to study the effect of drilling mud on the properties of waxy crude oil. The crude oil properties considered in this project are WAT and WDT, pour point, freezing point and yield stress which represents the strength of the wax gelled. In conducting the project, it is best to exhibit the real condition in term of condition and surrounding environment. The crude oil sample and drilling mud used in this project is kindly provided by PETRONAS Carigali Sdn Bhd (PCSB). In order to simulate close to real condition, the mixing temperature of crude oil and drilling mud is set at 5°C above the WAT and at 80°C. The type of drilling mud used is oil base drilling mud as was prepared as the samples with different concentration ranging from 5% to 20% of weight to weight ratio. Even though there is limitation in this project, it provides the guidance for further study and set benchmarks in deep understanding to study different level of contamination of crude oil with different concentration of drilling mud in the future. This project is also able to provide the protocol, or in other word the correct test method to determine the true properties of waxy crude oil with the presence of drilling mud contamination.

CHAPTER 2

LITERATURE REVIEW

This chapter discusses the literature in analyzing the important point of the project. Priority has been given to the drilling mud and waxy crude oil in order to gain sufficient information to achieve the objectives.

2.1 DEFINITION OF DRILLING MUD

Drilling mud is made from a combination of water, weighing material, clay and certain chemicals. Instead just mixing it with water, oil can also be used to be added with water to get a certain required properties. Based on Engineering Manual, the main function of drilling mud is to bring up the cuttings from the drilling process of wellbore to the surface in form of waste [1]. Other than its main function, drilling mud also act as cooling and lubricant for the drill bit and drill string during the drilling process. Besides, it may also assist in supporting the wall of the bore hole. However, despite all these function of drilling mud, it may also exhibits some properties such as unable to make the drilling bit, drill string and casing including the surface facilities becomes corrode in order to make sure that the mentioned items can perform their jobs efficiently. In industrial used, there are many different types of drilling fluids. Such as air, water and oil base muds. In this project, priority has been given to oil based muds. According to Van Dyke [2], oil based mud usually has oil in form of diesel or synthetic oil as the based phase instead of water in the water based mud. Despite high in preparing cost, hard to handle and not so easy to be disposed, oil based mud is easy to prepare and maintain. However due to its cost, oil based mud only will be used when there is needed due to the condition at the down hole. The used of oil based mud are related to:

i. Protect the formation that is produced
ii. Drill deeper at high temperature holes condition
iii. Prevent sticking due to pressure different
iv. Reduce the problem due to corrosion
v. Reduce corrosion at drill string
vi. Avoid gas entrainment

2.1.1 Oil Base Mud

The use of oil base mud started back in the 1970's in the United Kingdom where diesel was regarded as the basic base fluid [3]. The use of diesel can be due to its cheap cost and highly effective. However, because of its property of containing high aromatic compound, it led to the occupational hygiene and health problem. Thus, in the early 1980's, the use of diesel was banned. Later in the 1984, its use offshore was prohibited.

2.1.2 Advantages of Oil Base Mud

Oil based mud could be a solution for a problematic water based mud in term of the water-sensitive shale formation, corrosive gases and water-soluble salts [2]. The high cost can be significantly tackled by proper handling, storage and the efficient movement from one well to the other.

i. Coring and Completion

Oil based mud which has properties such as little water content, does not hydrate clays, able to dissolve salt in a formation and the reaction towards formation solids makes it a properly formulated oil mud. With the help of correct additives, the oil base mud can prevent the liquid phase to escape into the formation as filtration loss. These properties can reduce the damage to the producing formations and reduce the changes of the rock in the core.

ii. Hole Stability and Corrosion

The existing of water in the hole when drilling may result in the slough off of the hole wall formation. Thus, oil based mud is very important in order to maintain the stability of the wall formation. The combination of corrosive gas with water in the water-base mud will form acids. Therefore, oil-base mud may help in avoiding corrosion to the steel equipment such as drill bit, drill string as well as tubing and casing.

iii. Ability to Lubricate

It is simple to say that the slipperiness reduces friction. The ability of oil-base mud to lubricate enables the problem related to the stuck of drill string and liner to be solved. The slippery properties that exhibits from oil-base mud may reduce torque and drag in directional drilling

iv. High Temperature Drilling

Oil-base mud is known to its high boiling point. Therefore, it is very useful in drilling a very deep hole as the temperature rises. The oil-base mud has the capability to be used even at greater temperature than 400°C and performed without having any difficulty at very minimal maintenance cost.

2.1.3 Disadvantages of Oil Base Mud

There are some disadvantages of oil base mud such as:

i. High production cost
ii. The underground water may contaminate the mud due to the increase of water content that may lead to thicker mud
iii. Its ability to flammable
iv. The property such as slipperiness may cause safety and health issue which can cause accident to the operators
v. Slow drilling rate

According to Caenn and Chillingar [4], oil base mud has several difficulties. Oil base mud also needs special handling system. In order to produce the correct formulated mud, the oil base mud is produces at the liquid mud plant. Precaution must be made to avoid contamination such a way the mud may spill on the ground or to the water. Thus, a special enclosed mud handling system must be installed on the rig to handle the mud. Besides that, there is also a concern to the environment issue in using the oil base mud. It is been discovered that oil base mud especially diesel is toxic to marine organisms. Thus, continuous research has been done to replace the oil base mud.

2.2 DEFINITION OF WAXY CRUDE OIL

It can be said that all petroleum consists of a mixture of chemical compound that made up of hydrogen and carbon, which is known as hydrocarbons [5]. There are also significant amount of nonhydrocarbons which contain S, N_2, O_2 and some metals. Table 2.1 below shows the composition of crude oil by its corresponding elements.

TABLE 2.1: Composition of Crude Oil

Element	Percent by weight
Carbon	83-87
Hydrogen	11-14
Sulfur	0.05-2.5
Nitrogen	0.1-2
Oxygen	0-2

Waxy crude oils are aliphatic hydrocarbon having high molecular weight paraffin with carbon number ranging from C_{18} to C_{65} [6]. The paraffin waxes can be classified as either macro-crystalline or micro-crystalline. Macro-crystalline waxes are mainly linear paraffin (n-alkanes) with chain lengths between C_{18}-C_{30}. They have needle-like and plate-like morphology with crystal size up to 100 μm. Their presence within the crude oil promotes strong gel formation. Micro-crystalline waxes on the other hand contain greater percentage of iso-paraffins (branched alkanes) and naphtenes (cyclic alkanes) with carbon number found to be greater than 30. The crystal size is much smaller (up to 10 μm) with spherical morphology. It is amorphous in nature as the crystal growth is hindered by the side and cyclic groups present in the molecules. Due to the higher molecular weight, it has been reported to be the main constituent of the sludge at the bottom of crude oil storage tanks [7].

2.2.1 Wax Appearance Temperature (WAT) and Wax Disappearance Temperature (WDT)

Wax appearance temperature (WAT) and wax disappearance temperature (WDT) is one of many importance properties of crude oil. Alcazar- Vara and Buenrostro-Gonzalez [8] define WAT as the temperature in which the first crystallization of wax occur through cooling process. In contrast, WDT can be referred as the solid-liquid equilibrium point [9]. It is suggested that in a condition of constant pressure, the WDT is measured to be higher compare to WAT. The formation of wax crystals begin in the descending order stage based on its molecular weight through super saturation. At this stage, liquid phase of the crude oil starts to form solid-liquid phase where there are small wax crystal suspended in the liquid phase. In order to make sure that the small amount of wax crystals dissolved completely, higher temperature is applied. This temperature is regarded as WDT.

According to Karan and Ratulowski [10], waxy crude oil can be classified as the type of crude oil that contains high paraffin and pour point. The formation of wax precipitation in a form of solid phase is always associated with the reduction of temperature. WAT is commonly referred when dealing with waxy crude oil. WAT can be defined as the temperature at which the crude oil begins to precipitate out its wax crystal. Various problems can be caused by wax precipitation either in transportation or production. For example, the deposition of wax inside transportation pipe may reduce the flow, at the same time pressure drop becomes higher. Wax precipitation also increases the crude oil viscosity. To restart a shut-down pipeline, high pressure is needed when this occurs. In investigating the WAT, careful distinction is necessary to differentiate between thermodynamic WAT and the experimental WAT. The thermodynamic WAT can be defined as the actual temperature at which the solid-liquid phase boundary exists. On the other hand, experimental WAT is the temperature at which the first crystals are formed. However, the accuracy of the measured experimental WAT depends on the sensitivity of the instruments and measurement techniques.

2.2.2 Pour Point

Pour point can be referred as the lowest temperature where the oil can flow [5]. This property also indicates the presence of paraffin in the crude oil. The higher the pour point, the higher the paraffin content in the oil. Waxes begin to precipitate out when they are subject to low temperature. When the temperature reaches lower than WAT, the wax crystals number and size become larger [10]. These crystals tend to stick together and form net like structure which tends to trap the oil inside. Due to that, the oil starts to form gel like condition which causing the oil viscosity to increase. At this condition, the amount of wax is larger and the gel is stronger. The pour point can be determined when increasing viscosity causing the oil ceases to flow.

2.2.3 Gel Strength

When the temperature of waxy crude oil is dropping until below its pour point, the strength of the structure begins to increase as the crystal is developing [10]. This usually happen when the pipelines is shut down or undergone maintenance. The temperature of the pipeline will drop close to ambient temperature which sometimes less than pour point. The waxy crude oil may gel and start to form solid like when the ambient temperature is close to pour point. The gel formation in the pipelines may cause problem in the transportation of crude oil. In order to re-start the flow, a certain amount of high pressure is needed so that the shear stress at the wall exceeds a certain minimum value. The minimum value is called gel strength and also always referred as yield stress. The yield stress also can be referred as the minimum stress applied to obtain a shear flow.

2.3 TECHNIQUES OF MEASURING PROPERTIES OF WAXY CRUDE OIL

2.3.1 Wax Appearance Temperature (WAT)

There are various techniques used to determine the WAT. Simple techniques can be used such as visual ASTM D2500-66 method, filter plugging (FP) and viscometry. There are also more advanced and sophisticated techniques such as cross polar microscopy (CPM), light transmission, Fouriew transform infrared (FTIR) light scattering and differential scanning calorimetery (DSC) [10]. These various techniques used can be used as a comparison to one another [10 and 11]. A series of laboratory works have been made in order to determine the correct method to be used for flow assurance studies [11]. When the sample volumes come in very small amount, they have suggested that CPM and DSC to be used. Ronningsen et al. [12] have proposed that among DSC, CPM and viscometry, CPM recorded the highest WAT [12]. Other researchers, Cazaux et al. also discovered that CPM provides the highest WAT when they compared with CPM, viscometry and X-Ray diffraction. In general, the experimental techniques to determine WAT have been proposed by Ronningsen et al. [12] and Monger-McClure et al. [11]. These techniques however have been correlated by Hamammi and Raines [13], Erickson et al. [14] and Monger-McClure et al. [11].

2.3.2 Pour Point

ASTM method D-97 is the suitable method to be used to determine pour point despite there is other methods such as complex viscometry [10]. However, there is another method which is D 5853-95 used in determining crude oil pour point. This test method enables the crystal formation or viscosity increment which may obstruct the flow of the crude oil to be determined [6]. The apparatus used in test method D 5853-95 are shown in Figure 2.1.

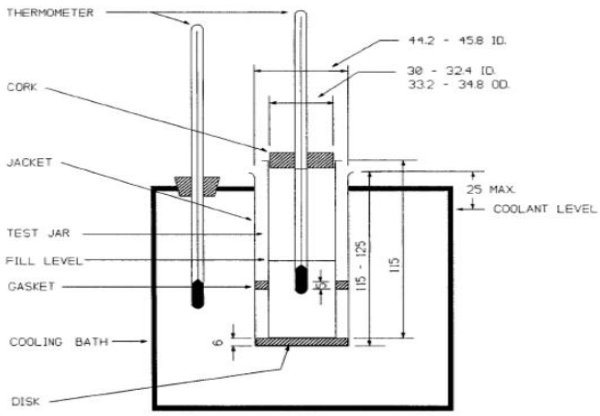

FIGURE 2.1: Apparatus for Test Method D 5853-95 [15]

ASTM method requires the crude oil to be preheated to a certain temperature and then allowed to cool at a certain cooling rate. These parameters such as heating and cooling rate are important to determine the pour point.

2.3.3 Gel Strength

There are two techniques used to measure the strength of waxy crude oil [10]. These are the Model Pipeline Test (MPT) and the Controlled Stress Rheometer (CSR). Comparison has been made and the result shows that the yield stress measure form CSR is always higher than the MPT. Due to experimental errors in MPT, the data obtained is always questioned because it was not match to the real field application. This is caused by some uncertainties related to the effect such as pipe and oil compressibility, wax-free oil diffusion and the oil contraction. Despite that, CSR also provide some uncertainties because the yield stress measured in field did not reflect the yield stress value obtained using this test method. However, this test method is fairly to be used in determining the gel strength.

2.4 PROBLEM IN WAXY CRUDE OIL TRANSPORTATION

There are about 20% of the world reserves hydrocarbons consist of high molecular weight compound as called as waxy crude oil [6]. Crude oil is the main component of petroleum which contain hydrocarbons component. It can be divided into two types which are light and heavy hydrocarbon. Light hydrocarbon exhibit carbon number C_1 to C_4 which is in a form of gas. Whereas gasoline, kerosene and diesel form liquid phases and have carbon number ranging from C_5 to C_{17}. Another type of hydrocarbon which is heavy hydrocarbon mainly has paraffin and napthenes. Paraffin is a form of alkanes which has a chemical formula C_nH_{2n+2} has carbon number ranging from 18 to 65 [15]. Due to high temperature in the reservoir, it is sufficient to make sure that the crude oil is dissolved in the mixture. At this stage, the crude oil performs a Newtonian fluid with low viscosity [16]. However as the crude oil flows and leaves the reservoir, the temperature reduces due to the colder surrounding. The crude oil temperature will continue to drop until at a point where the paraffin molecules begin to deposit out. At this point, the temperature is believed to reach below its WAT. This event has been discussed over several decades and lead to a very serious problem to the industry. The precipitation of wax will deposit along the pipelines and eventually narrow down the inner diameter of the pipes. Over a period of time, it contributes to more serious problem such as the reduction of flow rate of crude oil transportation as well as pressure drop. High expenditure is needed to control and overcome the problem [17].

2.5 CHAIN LENGTH AND CARBON NUMBER OF WAXY CRUDE OIL ANALYSIS

Waxes are the solid form of heavy hydrocarbons which contain carbon number of 18 or above. These forms of hydrocarbons are normal alkanes (paraffin) [6]. The temperature of the waxy crude oil is decreasing as it flows out from the reservoir during the oil production. Problems arise when the waxes tend to precipitate inside the pipe during the transportation or when it is stored. Therefore, there is need to study the composition of waxy crude oil such as its amount and type of wax and also to study the temperature of the waxy crude oil at which the paraffin components start to crystallize. In this aspect, WAT will be the indicator and it is helpful to understand the characteristic of this waxy crude oil.

Del Rio and Philp [18] explained that High Temperature Gas Chromatography (HTGC) analysis of waxy crude oil leads to a conclusion that the wax blocked the oil well. It is proven that the blocking substances are waxes which consist of hydrocarbons with carbon number ranging from 40 to 50. The distribution of long chains and carbon numbers of crude oil has been widely studied by HTGC analysis to understand the behaviour and characteristics of it.

CHAPTER 3

METHODOLOGY AND PROJECT ACTIVITIES

3.1 CHAPTER INTRODUCTION

In order to achieve the objectives that have been highlighted, complete methodologies have been set up in this project. A flow chart of methodologies used in this project is shown in Figure 3.1. These methodologies show the procedures and methods on how the project been conducted within the given timeframe. In this project, a series of experiments were conducted in order to come out with findings and solutions to the objectives as well as reflecting the title of the project. The experimental works define the protocol set up in order to determine the real properties of waxy crude oil with the presence of drilling mud contamination. This protocol enables the engineer to analyze and take further steps to design related facilities which rely on the properties of the waxy crude oil.

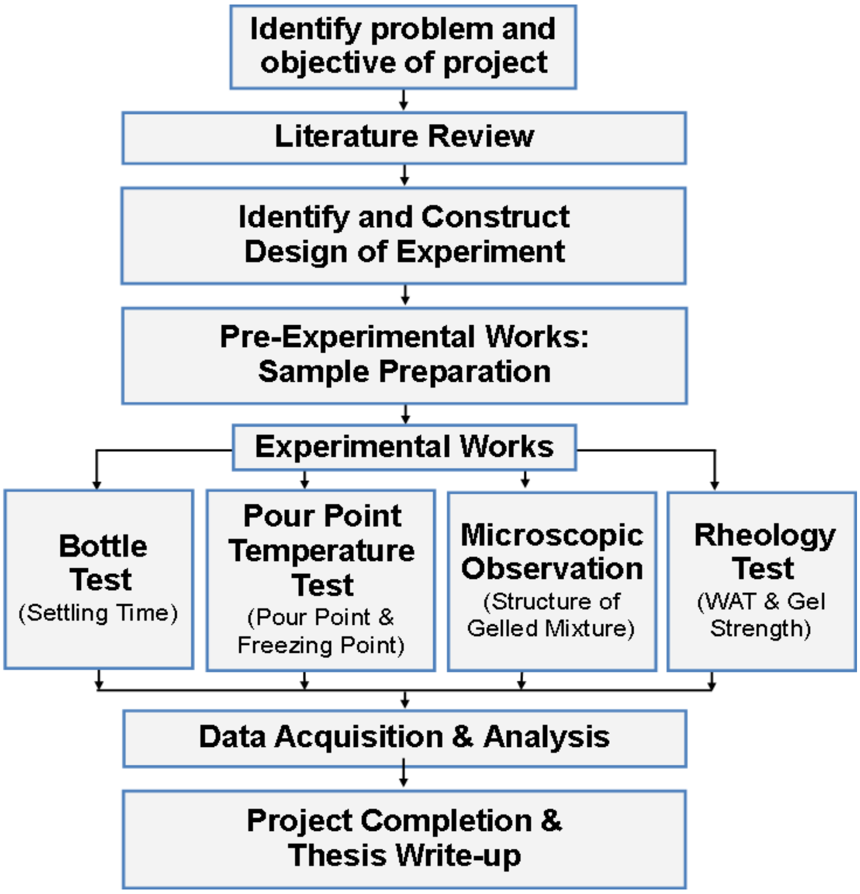

FIGURE 3.1: Flow Chart of Methodologies

3.2 PETROLEUM HYDROCARBONS AND DRILLING MUD

The waxy crude oil and drilling mud used throughout this project were kindly provided by PETRONAS Carigali Sdn Bhd (PCSB). Table 3.1 below shows the initial properties of waxy crude oil and drilling mud.

TABLE 3.1: Initial Properties of Waxy Crude Oil and Drilling Mud

Properties	Waxy Crude Oil	Drilling Mud
Density (g/ml)	0.82292 at 60°C	1.402
API Gravity	33.755 at 15°C	
Pour Point (°C)	39.0	
Wax Appearance Temperature (°C)	39.4	
Yield Stress (Pa)	5.79	

3.3 SAMPLING

Eight samples were prepared in order for the experiment to be conducted. The main waxy crude oil has been supplied from PETRONAS Carigali Sdn Bhd (PCSB). Four different concentrations of drilling mud were mixed together with the samples of waxy crude oil. The concentrations used were 5%, 10%, 15% and 20% with respect to weight of drilling mud. The variations in the percentages of drilling mud indicate the amount of contamination in the waxy crude oil. The samples preparation began with weighing the amount of drilling mud into a beaker according to respective drilling mud weight using a weighing scale. Then the waxy crude oil is added until the desired weigh ratio is obtained. To ease the monitoring process, each beaker containing the mixture was labeled based on the date and percentage of drilling mud weight used. The same processes were repeated using different percentage of drilling mud until the eight samples were prepared. The next process is to dissolve both components. First, the temperature was set to the first mixing temperature. It is 5°C above the WAT which is equivalent to 44.4°C. This temperature is suitable to be used because the crude oil behaves as Newtonian fluid. The beaker was placed into a bowl of ordinary cooking oil, which was placed on a heater to make sure the heat applied to the samples is evenly distributed. A clamp is used to hold the beaker to make sure that the bottom of the beaker does not touch the base of the bowl. This is done to ensure that there is no excess heat applies to the sample drastically.

When the sample reached the required temperature, it was mixed using an overhead stirrer at a rate of 400 rpm for about 60 minutes. The stirring process is important to ensure that the crude oil achieve its homogeneity characteristics [19]. After the process was done, the sample was poured into centrifuge tube of 50 ml and labeled accordingly. This portion of sample will be used for bottle test. Another portion of the mixed sample was poured into an aluminium cup for pour point test. The same processes are repeated using different mixing temperature which is at 80°C. In the end, there are two different mixing temperatures used which are at 44.4°C and at 80°C separately. In total, there are 8 different samples with their corresponding mud concentration mixture. Table 3.2 below summarizes the different samples with respect to their drilling mud concentration.

TABLE 3.2: Different Samples with Corresponding Drilling Mud Concentration

Sample Number	Concentration (w/w)	Mixing Temperature
S1	5%	5°C > WAT (44.4°C)
S2	10%	
S3	15%	
S4	20%	
S5	5%	80°C
S6	10%	
S7	15%	
S8	20%	

(a) (b) (c)

FIGURE 3.2: Sample Preparation: (a) Weighing Drilling Mud into Beaker; (b) Weighing Crude Oil into Beaker; (c) Mixing Process

3.4 EXPERIMENT SET 1: BOTTLE TEST

For every mixed sample prepared, about 50 ml of it is poured into a tube. The tube is then labeled accordingly by date and time when the bottle test is about to start for the respective sample. The test tube is placed inside a chamber with controlled temperature of 10°C above the WAT which is equivalent to 49.4°C. The sample is frequently observed to see the reaction of drilling mud and crude oil upon time. The observation is done in the frequent of 5 minutes, 15 minutes, 30 minutes, 1 hour, 2 hour, and 4 hour. After that the observation will be done daily until one week. Then it will be continued on weekly basis until one month period. For a record and future reference, pictures were taken at each observation time to compare the differences with the previous observation time. The volume of sedimentation for each layer of drilling mud, mixture and crude oil is measured and recorded into monitoring sheet. Appendix B shows the monitoring sheet that will be used in this test while Appendix C shows the design of experiment (DOE) for this test.

FIGURE 3.3: Bottle Test

3.5 EXPERIMENT SET 2: POUR POINT TEST

The pour point test is conducted to study the characteristic of waxy crude oil for the lowest temperature at which the sample shows the flow characteristics under defined condition. The instrument used in this test is the Pour Point Tester PPT 45150. This instrument was manufactured by PSL Systemtechnik. This instrument is a compact version of lab usage which complies with the rotational method ASTM D5985. This test requires the instrument to be continuously rotates the test sample to obtain the pour point temperature. This is done by an automatic instrument which has a suspended detection device attached to it. This instrument is accurate as it able to measure at extreme temperature ranging between -55°C to +150°C. It is also attached to control-PC or can also act as a stand-alone device. A portion of readily mixed sample is poured into an aluminium cup for pour point test. The sample is filled up until it reached the indicator inside the cup. The cup is then inserted into its designated slot on the instrument. The temperature sensor is inserted into the cup. With the PSL- software WinPPT, the test is run to observe the measurement using fully automated mode test runs. The temperature gradient is generated to give information regarding the behaviour and characteristics of the sample.

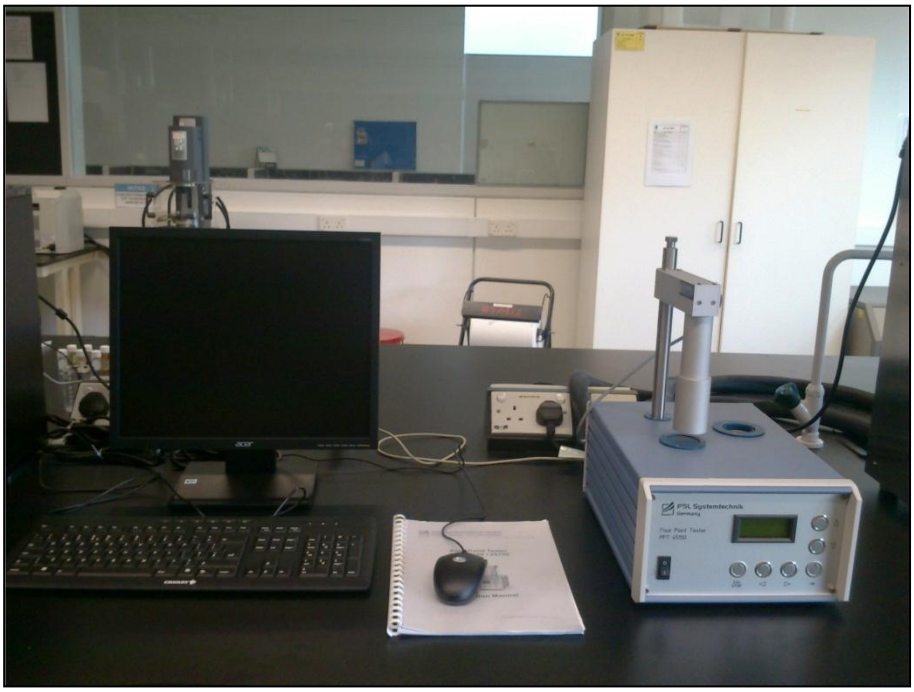

FIGURE 3.4: PSL Systemtechnik Pour Point Tester

3.6 EXPERIMENT SET 3: RHEOLOGY TEST

The rheology test is conducted to study the WAT of the waxy crude oil with the presence of drilling mud. Apart from that, this test also capable of measuring the yield stress of the sample. The instrument used in this test is an AR-G2 Rheometer by TA Instruments as shown in Figure 3.4. This instrument is regarded as sophisticated device which applies controlled stress together with rate rheometer and direct strain to enable the nano- torque control with the assistant of magnetic-levitation thrust bearing and a drag cup motor [20]. The instrument measures the Storage Modulus, symbol G' and Loss Modulus, G" together with phase angle, δ. Storage Modulus indicates the elastic properties while the Loss Modulus exhibits the viscous properties of the sample. Raw phase angle on the other hand is the difference in the phase of stress and strain during the oscillatory test. These properties can be obtained by conducting rheology test using Oscillatory Shear Experiment. In this experiment, the Storage Modulus, Loss Modulus can be determined when the sample is subjected to a sinusoidal strain (γ) at an angular frequency (ω) which will give it response when the steady sinusoidal stress (σ) is applied.

$$\gamma = \gamma_o \sin \omega t \quad (1)$$

$$\sigma = \gamma_o \left(G'(\omega) \sin \omega t + G''(\omega) \cos (\omega t) \right) \quad (2)$$

When the oscillatory stress is applied to the sample, the elastic and the viscous properties can be determined from the response it gives. In this test, it is conducted by measuring the sample at a certain frequency with changing temperature.

The samples used are taken from two different stages. The first sample is taken from the immediate mixture after the mixing process of sample preparation for all different concentration of drilling mud. While the second sample is taken after three days of bottle test for all different concentration of drilling mud. However, only the middle layer (where the sample is still in a form of mixture of waxy crude oil and drilling mud) is considered to be tested. Each sample is poured into a plate on the rheometer and a upper geometry (cross-hatched plate) is lowered into the material. The cylinder is oscillating and the measurement of the force which the sample is transferring is done. The phase angle measures the displacement as comparison to the movement. For a reference, total elastic material has a phase angle $\delta = 90°$ while liquid which has the tendency of maximum displacement has phase angle $\delta = 0°$. The onset deformation, the measured force and displacement are used to describe the storage modulus and the loss modulus.

3.7 EXPERIMENT SET 4: MICROSCOPIC OBSERVATION

Microscopic observation is done to see the structure of the crude oil with the presence of drilling mud. The instrument used in this observation is a microscope developed by Olympus as shown in Figure 3.5. After the sample is mixed in the sample preparation process, a small droplet is then transferred onto a transparent plate. The plate is then transferred into the microscope.

FIGURE 3.5: AR-G2 Rheometer

FIGURE 3.6: Microscope

3.8 PROJECT KEY MILESTONES

The project key milestone is prepared and can be referred in Appendix A. It combines two semester of Final Year Project I and Final Year Project II.

3.9 PROJECT TIMELINE

The project timeline in the form of Gantt chart is prepared and can be referred in Appendix B.

CHAPTER 4

RESULT AND DISCUSSION

4.1 CHAPTER INTRODUCTION

This chapter consists of the results and findings obtained based on the methodologies and work activities elaborated in the previous chapter. The result and discussion will discuss about the findings according to the experimental works that had been conducted. The result will include the following experimental works:

i. Bottle Test
ii. Pour Point Test
iii. Rheology Test
iv. Microscopic Observation

4.2 EXPERIMENTAL RESULT 1: BOTTLE TEST

4.2.1 Three Layers Formation and Mixture Density Calculation

Each readily mixed sample is placed inside a controlled temperature chamber at 49.4°C. This test is conducted in the Flow Assurance Laboratory of Universiti Teknologi PETRONAS (UTP) where the chamber is placed. During the observation period, each component (drilling mud, mixture and crude oil) in the sample is in the form of mixture and is not dissolved homogeneously. Therefore, upon time the sample will have tendency to separate according to their respective density. Drilling mud with the highest density of 1.402 g/ml settles at the bottom of the tube whereas the crude oil which has the lowest density of 0.82292 g/ml stays at the upper layer of these components. In between these two components, there is a mixture where waxy crude oil and drilling mud are still not separated for the time of observation.

Theoretically this is proven by calculation where the density of this mixture lies between the density of drilling mud; which is at the bottom and the density of crude oil; which is at the top.

Using cross multiplication method, the density of this mixture can be calculated easily. An example below shows the steps in calculating the density of the mixture of 5% of drilling mud content in the sample.

$$\frac{0.95Wt}{\rho oil} + \frac{0.05Wt}{\rho mud} = 51.25\ ml \tag{3}$$

The volume of sample poured into the centrifuge tube is 51.25 ml while *Wt* indicated the total mass of the sample in the centrifuge tube. The density of crude oil (ρoil) and density of drilling mud (ρmud) is 1.402 g/ml and 0.82292 g/ml respectively. Substituting these values into equation (3) yielded:

$$\frac{0.95Wt}{0.82292\ g/ml} + \frac{0.05Wt}{1.402\ g/ml} = 51.25\ ml$$

$$(0.95Wt\ X\ 1.402\ g/ml) + (0.05Wt\ X\ 0.82292\ g/ml)$$

$$= 51.25\ ml\ X\ 1.402\ g/ml\ X\ 0.82292 g/ml$$

$$1.3319Wt + 0.041146Wt = 59.129\ g^2/ml$$

$$(1.373\ Wt)\ g/ml = 59.129\ g^2/ml$$

$$Wt = \frac{59.129}{1.373}$$

$$Wt = 43.06\ g$$

In order to calculate the density of the mixture, divide mass with the volume of the mixture;

$$Density = 43.06\ g\ /\ 51.25\ ml = 0.8402\ g/ml$$

Based on the calculation above, it is shown that the density of the mixture falls below the drilling mud density, but higher than crude oil density. Therefore upon time, the sample will separate into three layers which constituent the drilling mud, mixture of drilling mud and crude oil and lastly the crude oil itself. The Table 4.1 below shows the density of mixture calculated for all 8 samples in bottle test.

TABLE 4.1: Density of Mixture for Samples

Sample	Mass of Drilling Mud (%)	Mass of Crude Oil (%)	Total Mass (g)	Volume in Tube (ml)	Density (g/ml)
S1	5	95	43.06	51.25	0.8402
S2	10	90	43.99	51.25	0.8583
S3	15	85	43.863	50.00	0.8773
S4	20	80	45.97	51.25	0.8970
S5	5	95	42.014	50.00	0.8403
S6	10	90	42.919	50.00	0.8584
S7	15	85	43.863	50.00	0.8773
S8	20	80	44.851	50.00	0.8970

Therefore, it is clearly shown that the mixture lies between the drilling mud and the crude oil based on the value obtained above. Thus, by conducting bottle test, a three layer components, which are drilling mud, mixture of drilling mud and drilling mud can be seen clearly upon time. This is a significant result which enables engineer to examine the properties of each component, especially the middle layer mixture.

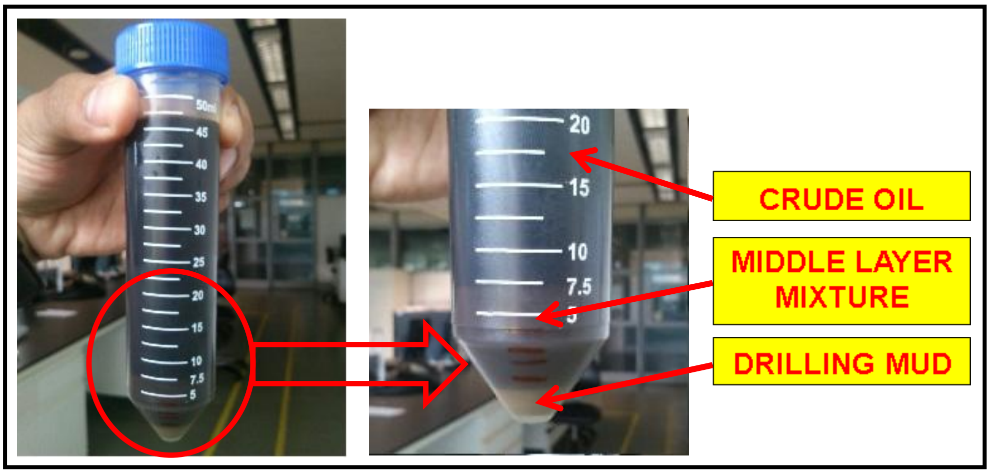

FIGURE 4.1: Three Layers Formation during Bottle Test

4.2.2 Three Layers Separation Volume

i. Observation of Sample 1 (S1)

Upon observation period, the sample has the tendency to separate into its respective component based on its density. The drilling mud with highest value of density settles at the lowest part while the crude oil with the lowest value of density settles at the upper part. In between these two components, there is a mixture with the density shown in the Table 4.1.

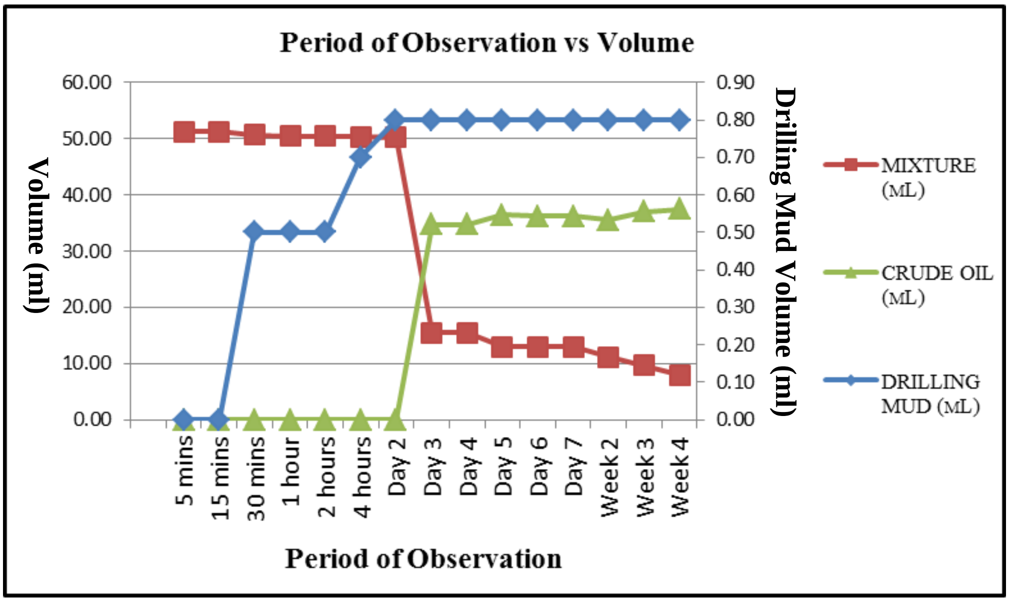

FIGURE 4.2: Graph of Period of Observation *versus* Volume for Sample 1 (S1)

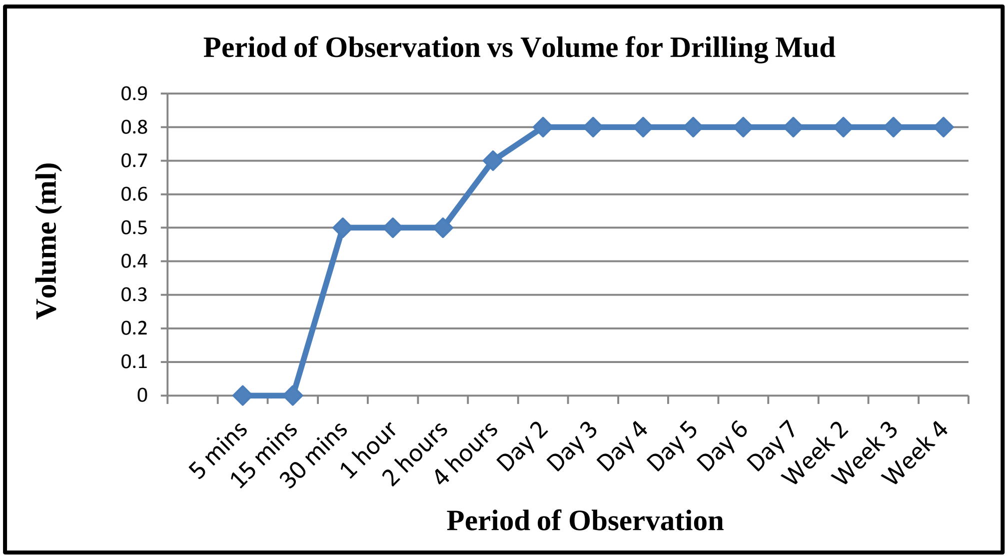

FIGURE 4.3: Graph of Period of Observation *versus* Volume for Drilling Mud of Sample 1 (S1)

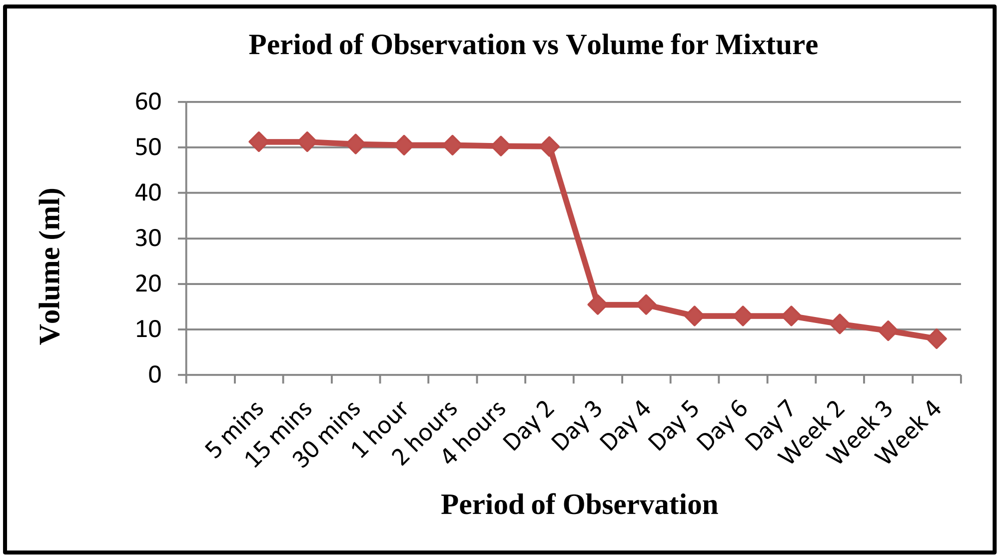

FIGURE 4.4: Graph of Period of Observation *versus* Volume for Mixture of Sample 1 (S1)

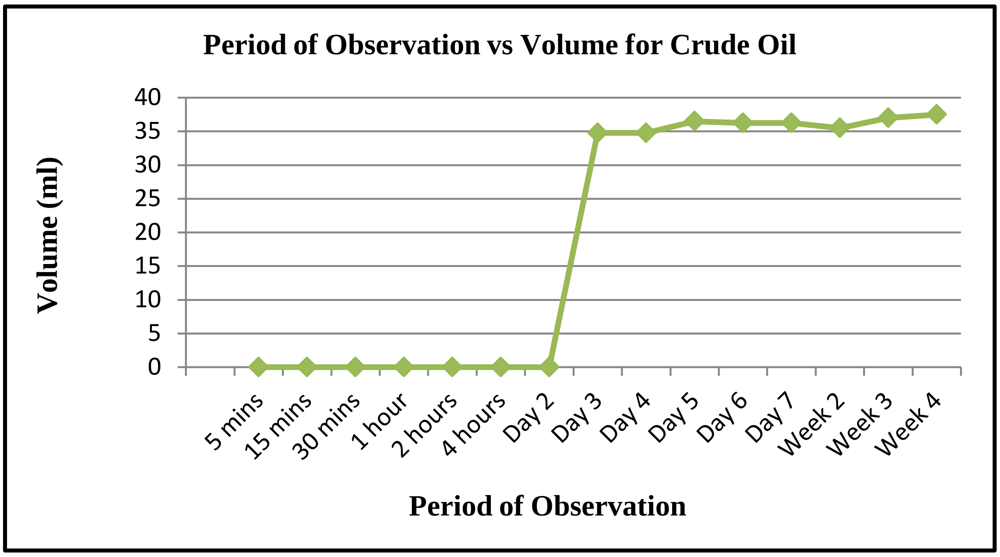

FIGURE 4.5: Graph of Period of Observation *versus* Volume for Crude Oil of Sample 1 (S1)

Figure 4.2 above shows the graph of period of observation *versus* volume of separation for the three components in the bottle test which are crude oil, mixture of drilling mud and crude oil and drilling mud. Figure 4.3, Figure 4.4 and Figure 4.5 show the individual graph of the separation drilling mud, mixture and crude oil respectively. The figure also exhibits the trend of graph obtained from Sample 1 (S1) which is containing 5% of drilling mud at mixing temperature of 44.4°C. The initial mixed sample poured into the centrifuge tube is about 51.25 ml.

Upon the observation time, the mixture gradually exhibits the separation process as the crude oil and drilling mud begin to separate. The drilling mud which initially used in this sample is 5% which is equivalent to 2.153 ml. After 4 weeks of observation, only about 0.8 ml has been separated from the mixture. However the separation of drilling mud from the mixture does not occur immediately until after 30 minutes. From that time, the drilling mud separation increases slowly and settles at 0.8 ml. The mixture tends to separate into respective drilling mud and crude oil upon time. However, the separation of crude oil can only be seen after the drilling mud settles or stabilizes. A drastic separation occurred after day 3 of the observation where from the graph, it shows very huge difference in term of volume of this component.

The volume of the mixture dropped from 50.20 ml to 15.45 ml. This shows that the separation had occurred rapidly between these two periods of times. From that point onwards, the mixture volume continues to drop until reached about 7.95 ml at the end of the period of observation. As the observation continues, there is no immediate separation of crude oil from the mixture until day 3 of the observation period. This shows that the crude oil took some time to be no longer in the form of mixture. The trend of separation of crude oil is inversed to the separation of drilling mud. The graph shows that the crude oil volume increases with time even though there is slight decrement in the middle of the observation period. At the end of the observation period, the total volume of crude oil separated from the mixture is 37.50 ml which is equivalent to 73% of total mixture.

During bottle test, significant findings have been obtained where there are two stages of separation process occurred. The drilling mud has the tendency to separate first from the mixture because of its higher density. Its higher density enables it to sediment quickly at the bottom of the centrifugal tube. This is the first stage of separation process which occurs at the first 3 days of the observation period. The second stage occurs after 3 days of the observation period where the waxy crude oil begins to escape from the mixture of waxy crude oil and drilling mud. Due to lower density of waxy crude oil, it settles at the upper part of the mixture.

ii. Observation of Sample 2 (S2)

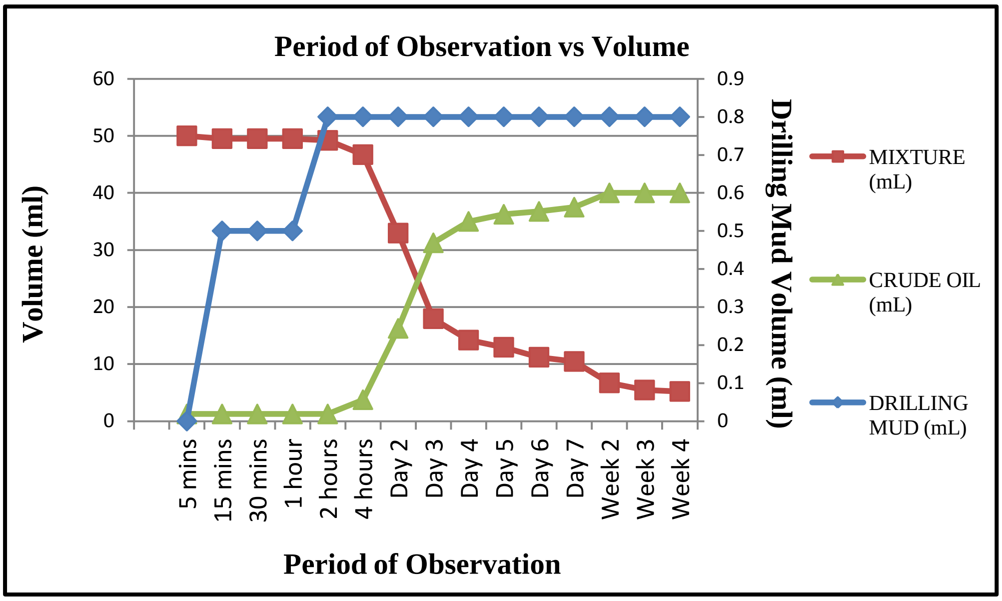

FIGURE 4.6: Graph of Period of Observation *versus* Volume for Sample 2 (S2)

The Figure 4.6 above shows the graph of period of observation *versus* volume of separation for Sample 2 (S2). This sample is made up of 10% drilling mud with mixing temperature of 44.4°C. Based on the graph, the separation of drilling mud is increasing until settled at 0.8 ml after 2 hours. However, there is no immediate separation of drilling mud occurred until approximately 15 minutes of the observation period. The mixture component decreased upon time until 5.20 ml after week 4. Whereas, the crude oil separation increased with time. Rapid separation of crude oil occurred between 2 hours and day 3 of the observation period. It can be seen also that there is immediate separation of crude oil during the test. At the end of the observation period, the total volume of crude oil separated from the mixture is 40.00 ml which is equivalent to 78% of total mixture.

iii. Observation of Sample 3 (S3)

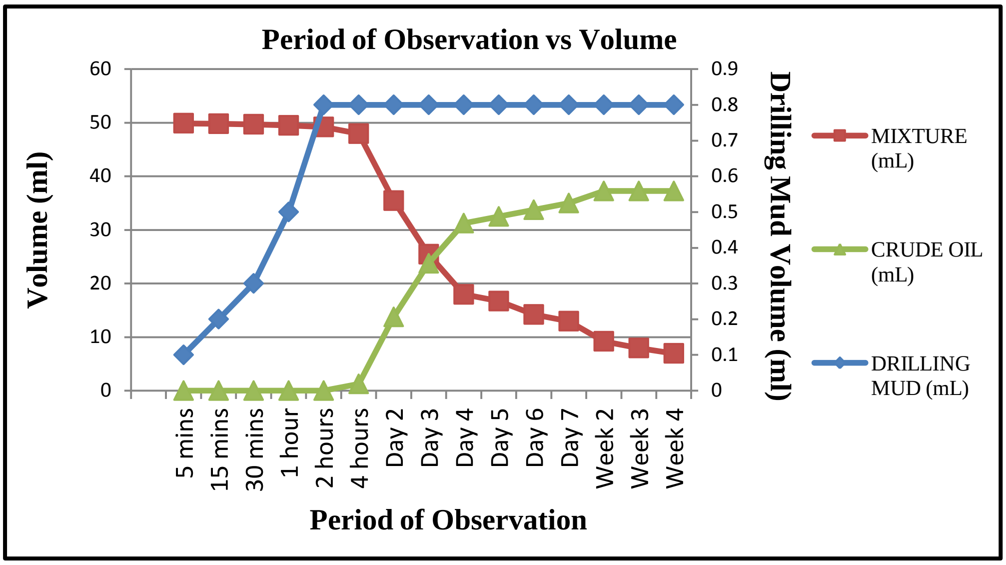

FIGURE 4.7: Graph of Period of Observation *versus* Volume for Sample 3 (S3)

The Figure 4.7 above shows the graph of period of observation *versus* volume of separation for Sample 3 (S3). This sample is made up of 15% drilling mud with mixing temperature of 44.4°C. Based on the graph, the separation of drilling mud is increasing until settled at 0.8 ml after 2 hours. At the beginning of the observation period, the drilling mud separation can be seen immediately for about 0.1 ml. The mixture component decreased upon time until 6.95 ml after 4 weeks. Whereas, the crude oil separation increased with time. Rapid separation of crude oil occurred between 4 hours and day 4 of the observation period. It can be seen from the graph that there is no immediate separation of crude oil until 4 hours of the observation period. At the end of the observation period, the total volume of crude oil separated from the mixture is 37.25 ml which is equivalent to 76% of total mixture.

iv. Observation of Sample 4 (S4)

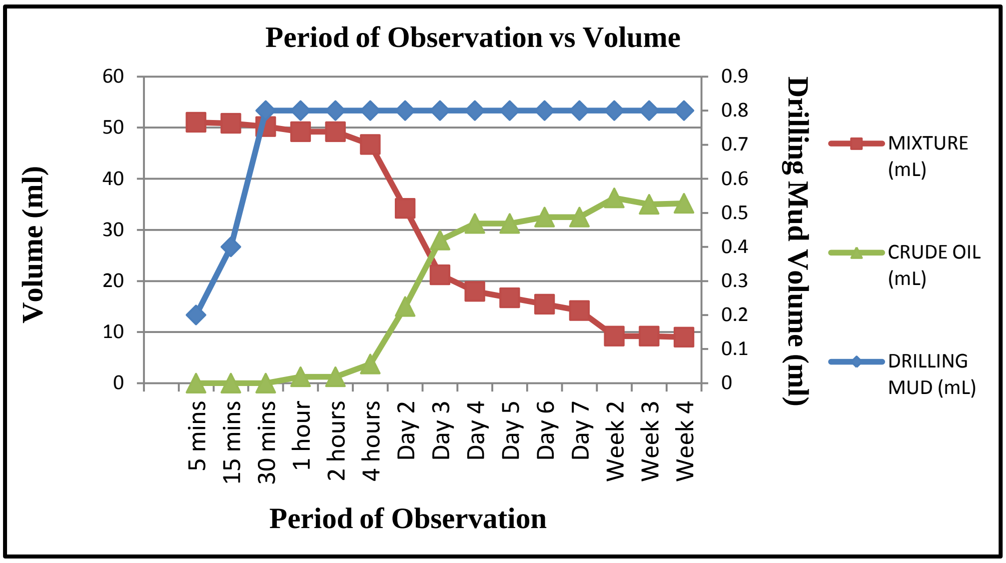

FIGURE 4.8: Graph of Period of Observation *versus* Volume for Sample 4 (S4)

The Figure 4.8 above shows the graph of period of observation *versus* volume of separation for Sample 4 (S4). This sample is made up of 20% drilling mud with mixing temperature of 44.4°C. Based on the graph, the separation of drilling mud was increasing until settled at 0.8 ml after 30 minutes. At the beginning of the observation period, the drilling mud separation can be seen immediately for about 0.2 ml. The mixture component decreased upon time to 9.00 ml after 4 weeks. Whereas, the crude oil separation increased with time. Rapid separation of crude oil occurred between 2 hours and day 4 of the observation period. From the graph, it can be seen that there is no immediate separation of crude oil until 1 hour of the observation period. At the end of the observation period, the total volume of crude oil separated from the mixture is 35.20 ml which is equivalent to 69% of total mixture.

v. **Observation of Sample 5 (S5)**

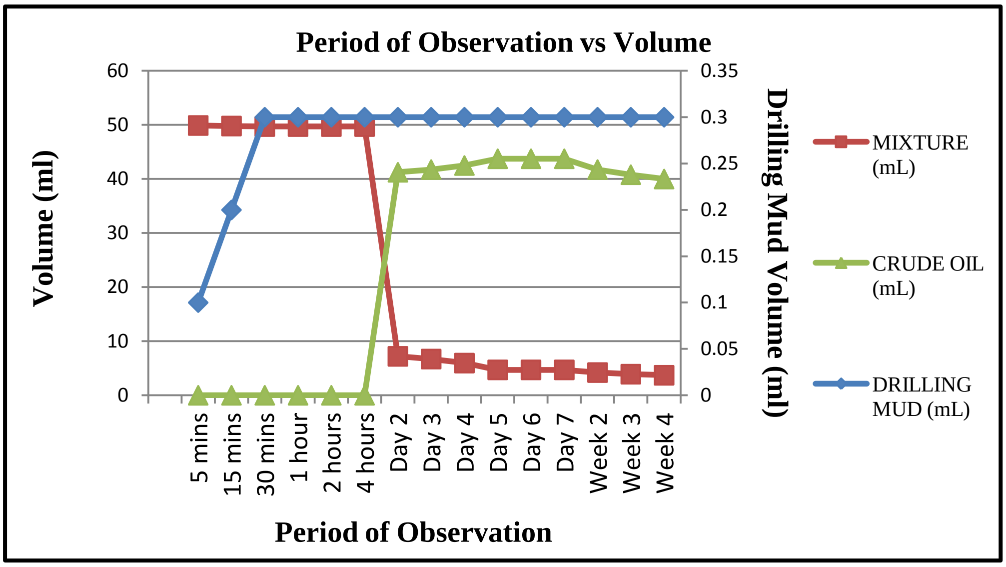

FIGURE 4.9: Graph of Period of Observation *versus* Volume for Sample 5 (S5)

The Figure 4.9 above shows the graph of period of observation *versus* volume of separation for Sample 5 (S5). This sample is made up of 5% drilling mud with mixing temperature of 80°C. Based on the graph, the separation of drilling mud is increasing until settled at 0.3 ml after 30 minutes. At the beginning of the observation period, the drilling mud separation can be seen immediately for about 0.1 ml. The mixture component decreased upon time until 3.70 ml after week 4. Whereas, the crude oil separation increased with time. Rapid separation of crude oil occurred between 4 hours and day 2 of the observation period. However, there is no immediate separation of crude oil occurred until day 2 of the observation period. At the end of the observation period, the total volume of crude oil separated from the mixture is 40.00 ml which is equivalent to 80% of total mixture.

vi. Observation of Sample 6 (S6)

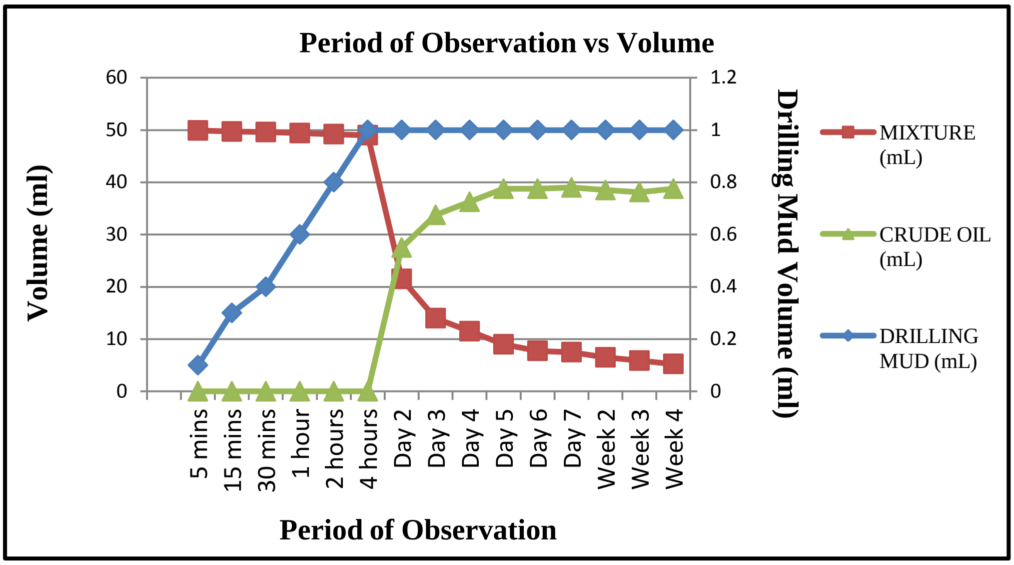

FIGURE 4.10: Graph of Period of Observation *versus* Volume for Sample 6 (S6)

The Figure 4.10 above shows the graph of period of observation *versus* volume of separation for Sample 6 (S6). This sample is made up of 10% drilling mud with mixing temperature of 80°C. Based on the graph, the separation of drilling mud is increasing until settled at 1.0 ml after 4 hours. After 5 minutes of the observation period, the drilling mud separation can be seen immediately for about 0.1 ml. The mixture component decreased upon time until 5.25 ml after week 4. Whereas, the crude oil separation increased with time. Rapid separation of crude oil occurred between 4 hours and day 2 of the observation period. It can be noted also from the graph that there is no immediate separation of crude oil until day 2 of the observation period. At the end of the observation period, the total volume of crude oil separated from the mixture is 38.75 ml which is equivalent to 78% of total mixture.

vii. Observation of Sample 7 (S7)

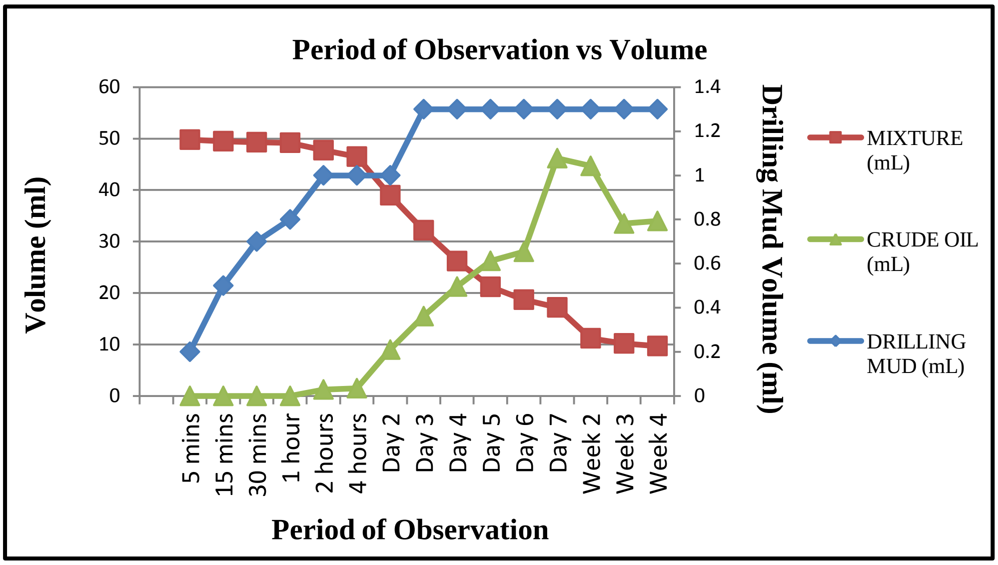

FIGURE 4.11: Graph of Period of Observation *versus* Volume for Sample 7 (S7)

The Figure 4.11 above shows the graph of period of observation *versus* volume of separation for Sample 7 (S7). This sample is made up of 15% drilling mud with mixing temperature of 80°C. Based on the graph, the separation of drilling mud was increasing until settled at 1.3 ml after day 3. After 5 minutes of the observation period, the drilling mud separation can be seen immediately for about 0.2 ml. The mixture component decreased upon time until 9.70 ml after week 4. Whereas, the crude oil separation increased with time. Rapid separation of crude oil occurred between 4 hours and day 7 of the observation period. However, after 7 days of observation, the crude oil sedimentation seems to decrease. This can be considered as rare occurrence because this was the only sample that behaves in this trend. It can be noted also from the graph that there is no immediate separation of crude oil until 2 hours of the observation period. At the end of the observation period, the total volume of crude oil separated from the mixture is 34.00 ml which is equivalent to 68% of total mixture.

viii. Observation of Sample 8 (S8)

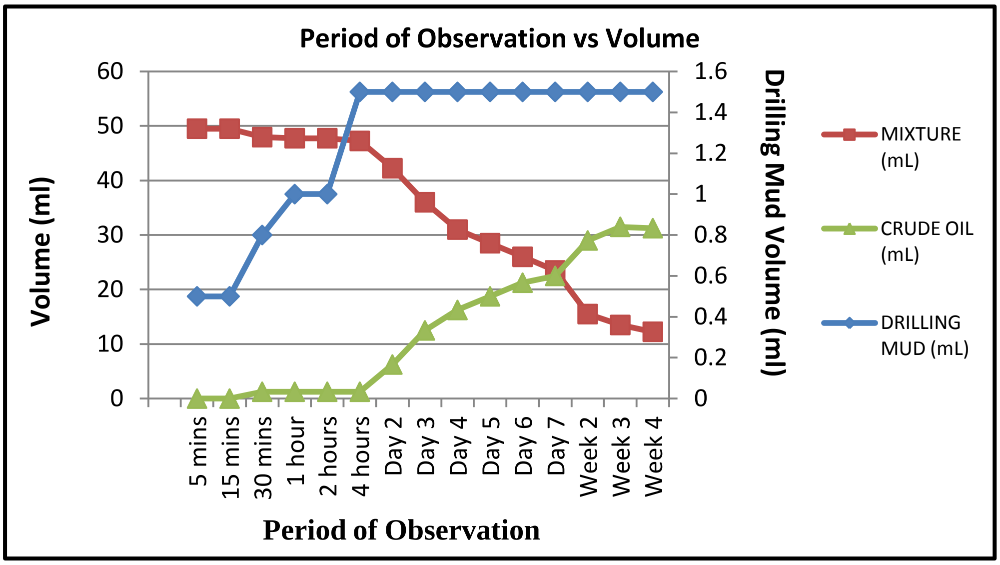

FIGURE 4.12: Graph of Period of Observation *versus* Volume for Sample 8 (S8)

The Figure 4.12 above shows the graph of period of observation *versus* volume of separation for Sample 8 (S8). This sample is made up of 20% drilling mud with mixing temperature of 80°C. Based on the graph, the separation of drilling mud was increasing until settled at 1.50 ml after 4 hours. After 5 minutes of the observation period, the drilling mud separation can be seen immediately for about 0.5 ml. The mixture component decreased upon time until 12.25 ml after week 4. Whereas, the crude oil separation increased with time. Rapid separation of crude oil began after 4 hours of the observation period. It can be noted also from the graph that there is no immediate separation of crude oil until 30 minutes of the observation period. At the end of the observation period, the total volume of crude oil separated from the mixture is 31.25 ml which is equivalent to 63% of total mixture.

In bottle test, it can be concluded that there are two separation processes occurred. The first stage involves the separation of drilling mud from the mixture which takes place at the first three days of bottle test. The second stage involves the separation process between waxy crude oil and mixture of waxy crude oil and drilling mud which takes place after 3 days of bottle test.

4.3 EXPERIMENTAL RESULT 2: POUR POINT TEST

Each readily mixed sample is tested using Pour Point Tester to study its respective pour point measurement; therefore comparison can be made with the pure waxy crude oil. Any chances to the temperature in which indicates the pour point of the crude oil will be significantly important for engineer to study in order to determine the most suitable method to handle it.

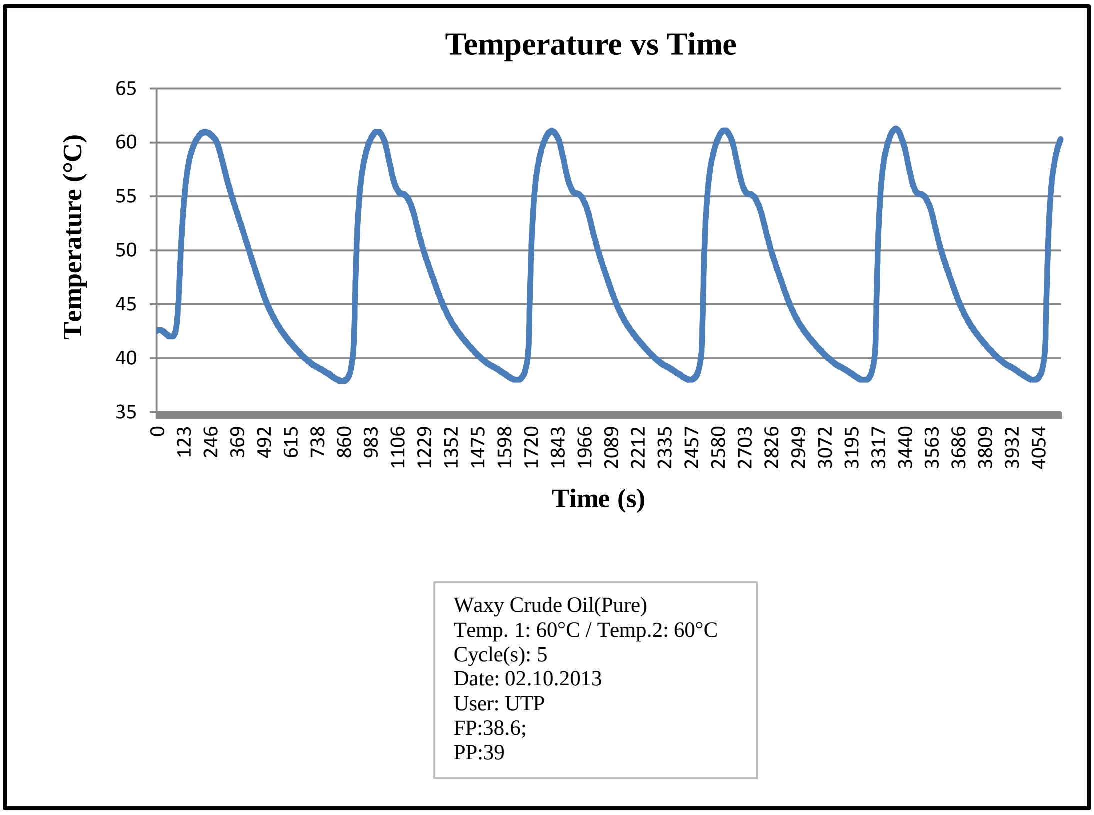

FIGURE 4.13: Graph of Time *versus* Temperature for Waxy Crude Oil

Pour Point Tester measures the temperature of the sample with respect to time. A sensor detects the temperature in which the sample begins to experience flow resistance when it is cooled down. Figure 4.13 shows the graph obtained during pour point test of pure waxy crude oil. It shows that the pour point temperature is 39°C while the freezing temperature is 38.6°C. These two temperatures are taken as the reference to study the changes in the property of this waxy crude oil.

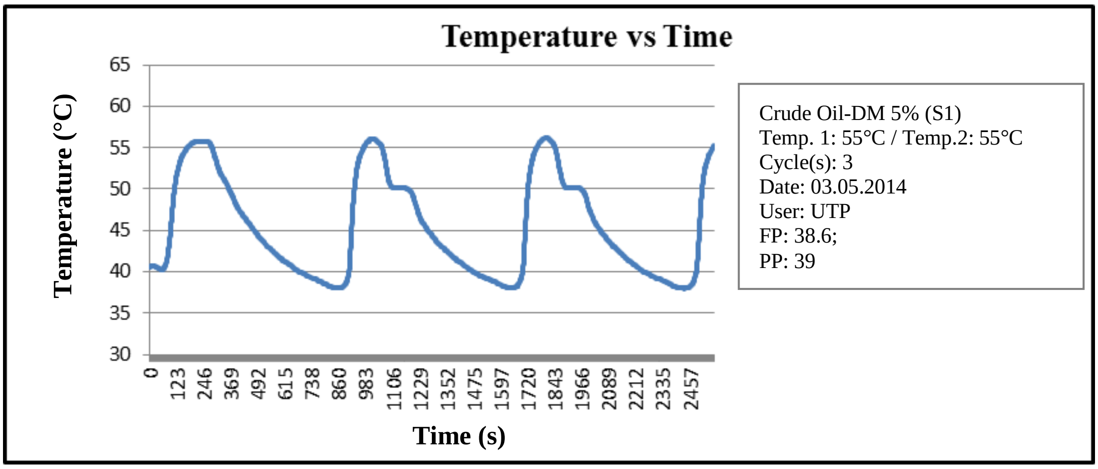

FIGURE 4.14: Graph of Time *versus* Temperature for Sample 1 (S1)

Figure 4.14 shows the graph obtained during pour point test of sample 1 (S1) which contain 5% drilling mud at a mixing temperature of 44°C. It shows that the pour point temperature is maintained at 39°C while the freezing temperature is also at 38.6°C. With this amount of drilling mud in the crude oil, there is no significant change to the pour point and freezing point of the waxy crude oil can be detected thus exhibits the same property as the pure waxy crude oil.

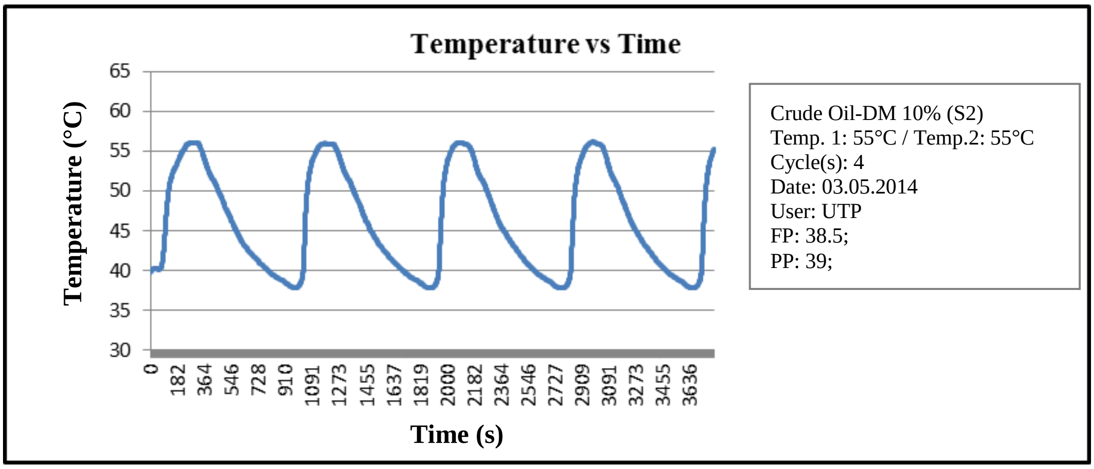

FIGURE 4.15: Graph of Time *versus* Temperature for Sample 2 (S2)

Figure 4.15 shows the graph obtained from pour point test of sample 2 (S2). It can be seen from the result obtained during pour point test that there is also no change in the pour point of the sample. However, there is slight decrement for freezing point of this sample to 38.5°C.

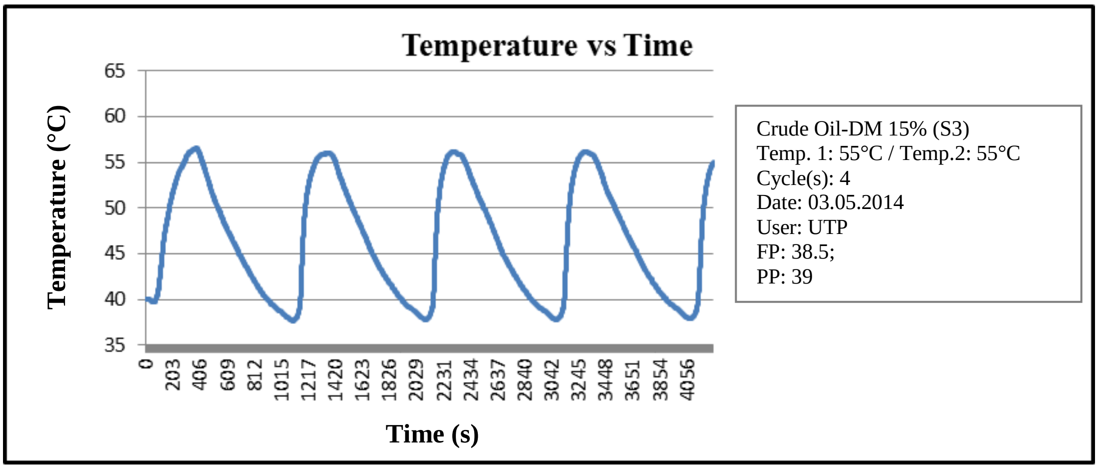

FIGURE 4.16: Graph of Time *versus* Temperature for Sample 3 (S3)

Figure 4.16 shows the graph obtained from pour point test of sample 3 (S3). It can be seen from the result obtained during pour point test that there is also no change in the pour point of the sample. However, there is slight decrement for freezing point of this sample to 38.5°C.

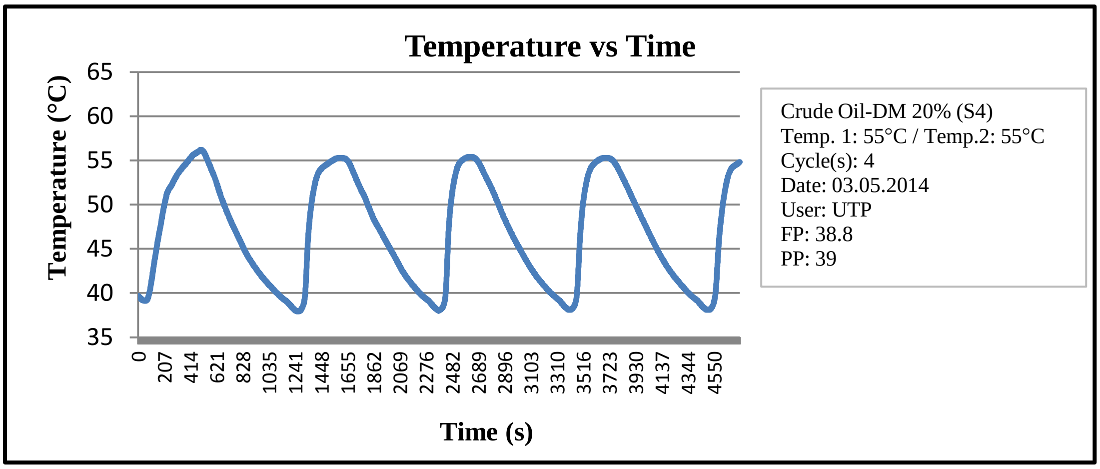

FIGURE 4.17: Graph of Time *versus* Temperature for Sample 4 (S4)

Figure 4.17 above shows the graph obtained from pour point test of sample 4 (S4). It can be seen from the result obtained during pour point test that there is also no change in the pour point of the sample. However, there is slight increment for freezing point of this sample to 38.8°C.

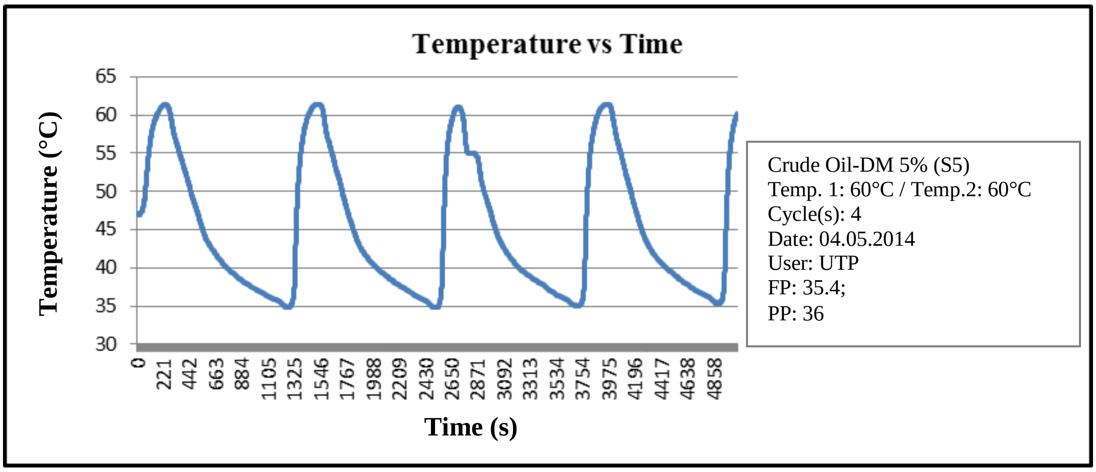

FIGURE 4.18: Graph of Time *versus* Temperature for Sample 5 (S5)

Figure 4.18 above shows the graph obtained from pour point test of sample 5 (S5). It can be seen from the result obtained during pour point test that there is change in the pour point of the sample. As compared to pure crude oil, the pour point temperature of this sample decrease to 36°C. In addition, there is also a decrease in freezing point to 35.4°C.

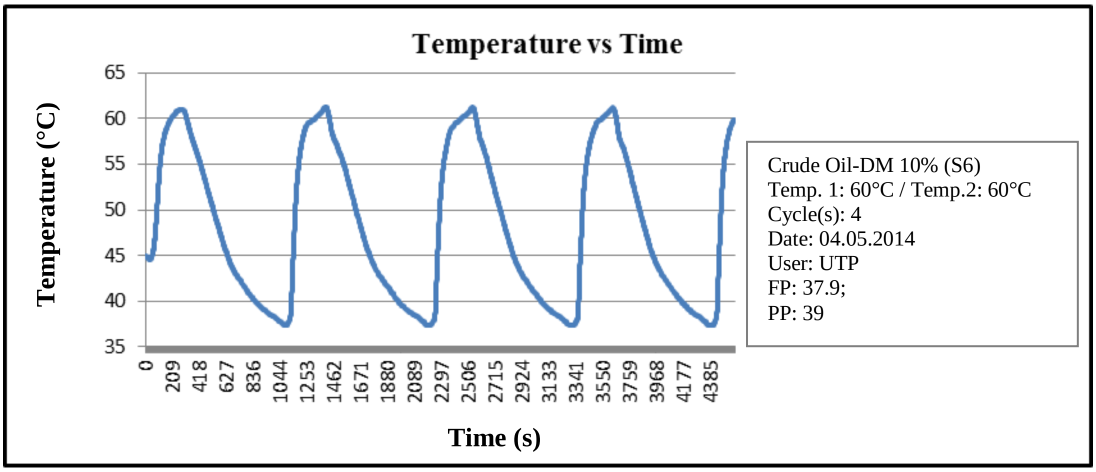

FIGURE 4.19: Graph of Time *versus* Temperature for Sample 6 (S6)

Figure 4.19 above shows the graph obtained from pour point test of sample 6 (S6). It can be seen from the result obtained during pour point test that there is no change in the pour point of the sample. However, there is a decrease in freezing point to 37.9°C.

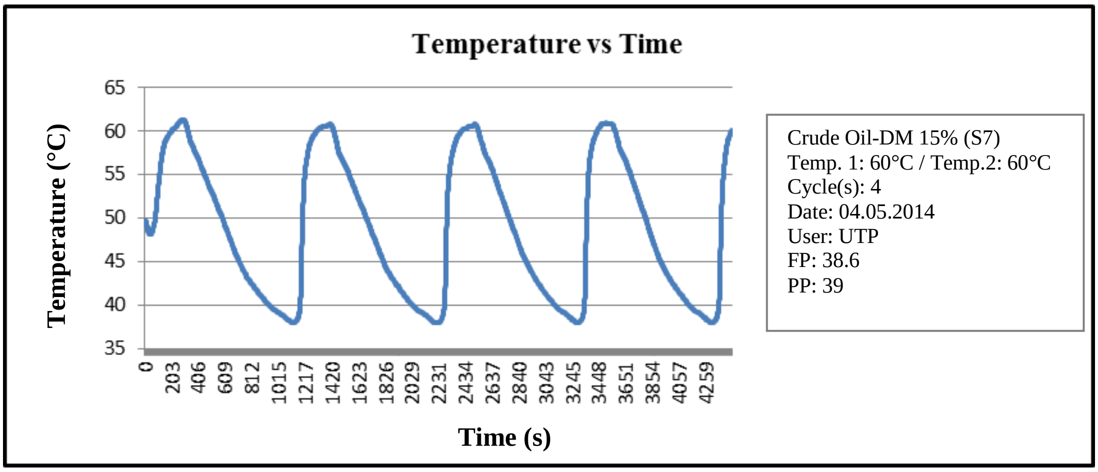

FIGURE 4.20: Graph of Time *versus* Temperature for Sample 7 (S7)

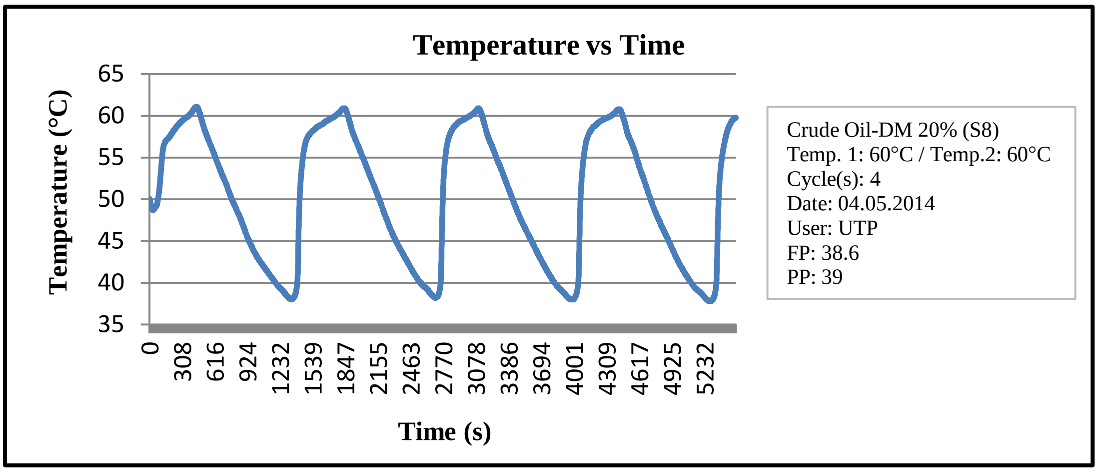

FIGURE 4.21: Graph of Time *versus* Temperature for Sample 8 (S8)

Figure 4.20 and Figure 4.21 show the graphs obtained from pour point test of sample 7 (S7) and sample 8 (S8) respectively. From the both graph, it can be seen from the result obtained during pour point test that there is also no change in the pour point of these samples. Besides that, there are also no changes in the freezing point.

To summarize the result in pour point test, Table 4.2 below shows the measurement obtained.

TABLE 4.2: Result of Pour Point Test

Sample Number	**Freezing Point (°C)**	**Pour Point (°C)**
S1	38.6	39
S2	38.5	39
S3	38.5	39
S4	38.8	39
S5	35.4	36
S6	37.9	39
S7	38.6	39
S8	38.6	39

In general, the result shows that there are not significant changes to the pour point and freezing point. However, Sample 5 (S5) acted weirdly as it is the only sample shows the value far away from the other samples. This result should not be significantly affecting the findings as it only shows a rare occurrence during the test.

4.4 EXPERIMENTAL RESULT 3: RHEOLOGY TEST

One of the important properties studied in this project are the WAT and yield stress of the crude oil. These measurements can be obtained by conducting rheology test using Oscillatory Shear Experiment. In this experiment, the Storage Modulus, symbol G', Loss Modulus, G" can be determined when the sample is subject to a sinusoidal strain (ɣ) at an angular frequency (ω) which will gives it response when the steady sinusoidal stress (σ) is applied.

Figure 4.21 below shows the graph of temperature ramping test of the crude oil sample which is useful to measure the WAT. The measurement of Storage Modulus, Loss Modulus and raw phase angle are measured with respect to temperature. Storage Modulus indicates the elastic response while the Loss Modulus exhibits the viscous behaviour of the sample. Raw phase angle on the other hand is the difference in the phase of stress and strain during the oscillatory test is conducted. It can determine the existence and extent of elastic behaviour of the sample. From the graph, it can be seen that there are two different behaviors shown by the crude oil sample during the test. The scattered region indicates that the sample is in liquid form, thus it is considered a very weak sample. The inertia of the instrument dominated the measurement which making the instrument unable to give accurate value of properties. This happens at higher temperature where the waxy crude oil sample experienced low viscosity. While at lower temperature, the waxy crude oil shows a more constant measurement because the viscosity is higher. The transition point from a more constant measurement area to a scattered area indicates the WAT point. At this point, the WAT can be easily obtained.

Conducting Strain Sweep Test will result in the measurement of yield stress of the sample. The yield stress shows the strength of gel of the sample. To evaluate the yield stress, the oscillatory stress curve from this test is used. As can be seen from Figure 4.42 the first part of the curve exhibit almost linear line before it starts to bend or form a curvy line. The point at which the measurement starts to deviate from the linear line indicates the yield stress of the sample. At this point the yield stress of the sample can be obtained. The intersection of Storage Modulus (G') curve and Loss Modulus (G") curve indicates the transition point from solid-like to liquid-like behaviour.

At this point, the solid-like behaviour of the sample is unable to sustain the high velocity, which is the shear stress applied and thus break. Hence, the liquid-like behaviour of the sample takes place and becomes dominant. Solid-like behaviour usually located on the left side of the graph whiles the liquid-like behaviour on the right. At this point, higher raw phase angle can be observed. The true liquid-like behaviour can be observed after this point if the instrument inertia does not dominating the measurement. It can be said that the instrument inertia is dominating the measurement when the raw phase angle is above 150 degree.

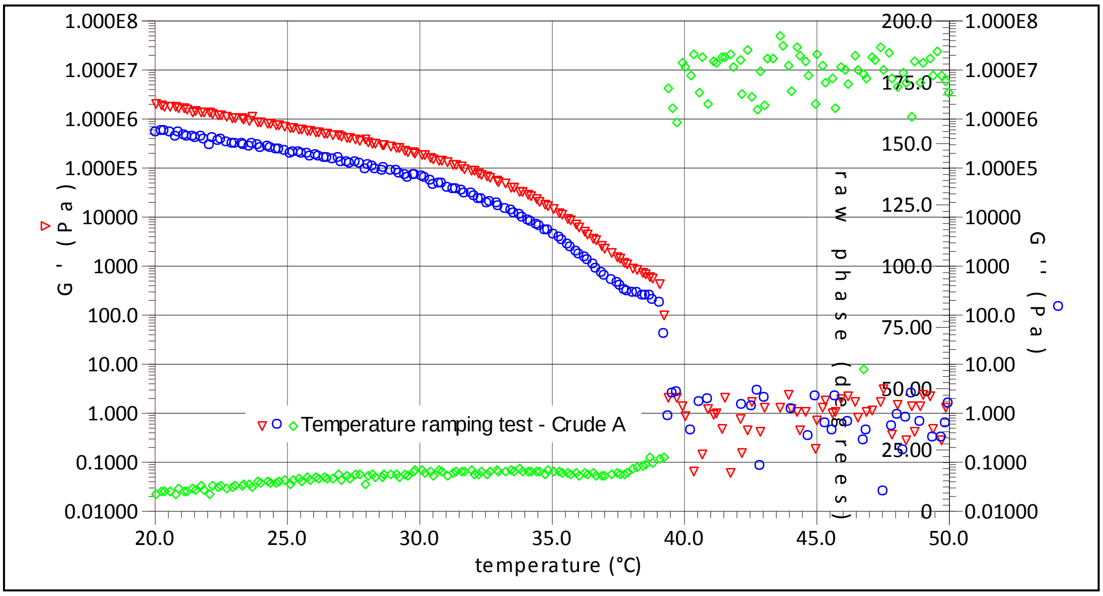

FIGURE 4.22: Graph of Temperature Ramping Test for Pure Waxy Crude Oil

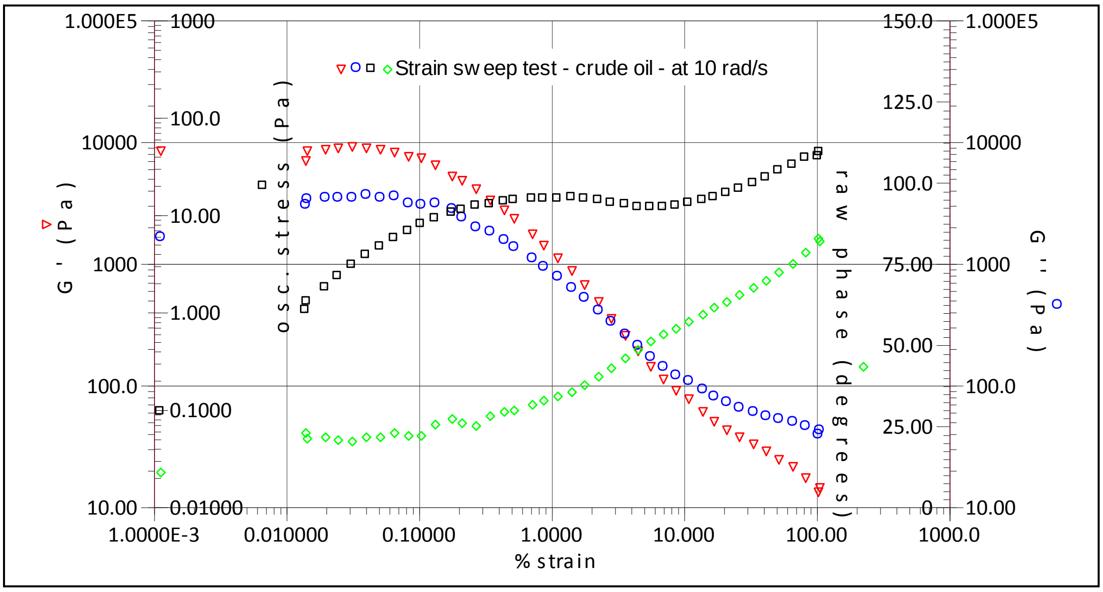

FIGURE 4.23: Graph of Strain Sweep Test for Pure Waxy Crude Oil

4.4.1 Immediate Mixture

The sample of crude oil studied in this part is taken immediately after the mixing process of sample preparation. The sample is tested using rheometer which further records and plots graphs from the test.

i) Waxy Crude Oil with 5% of Drilling Mud

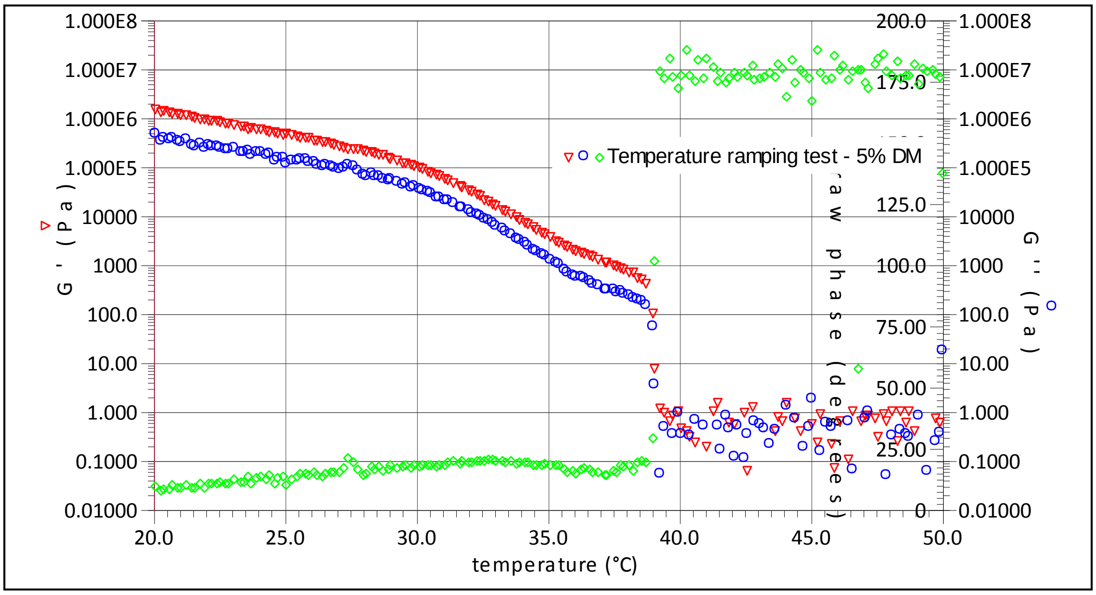

FIGURE 4.24: Graph of Temperature Ramping Test for Waxy Crude Oil with 5% Drilling Mud

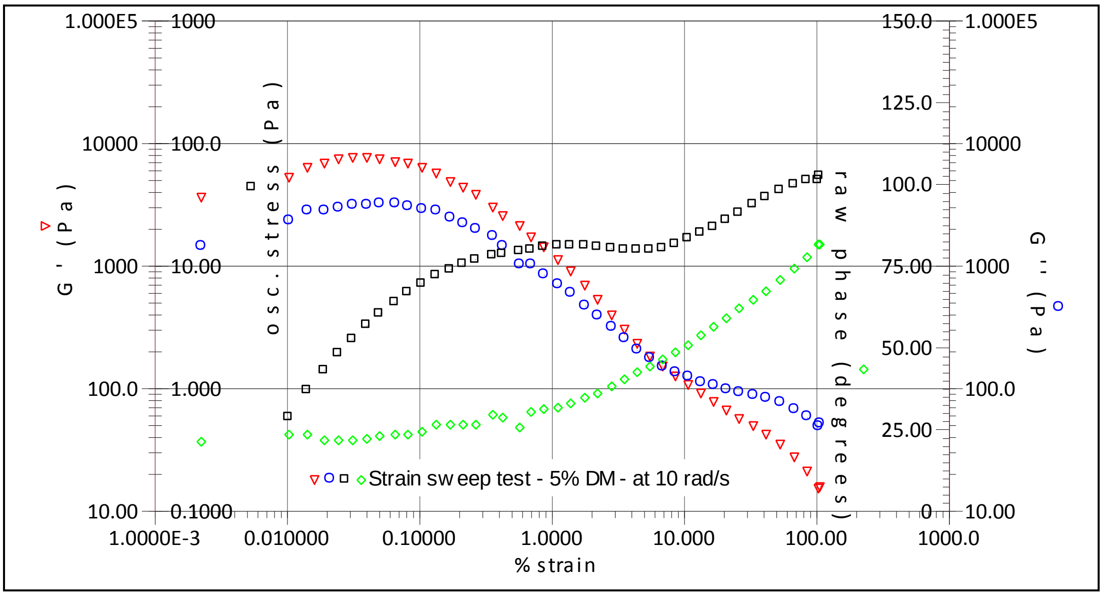

FIGURE 4.25: Graph of Strain Sweep Test for Waxy Crude Oil with 5% Drilling Mud

ii) Waxy Crude Oil with 10% of Drilling Mud

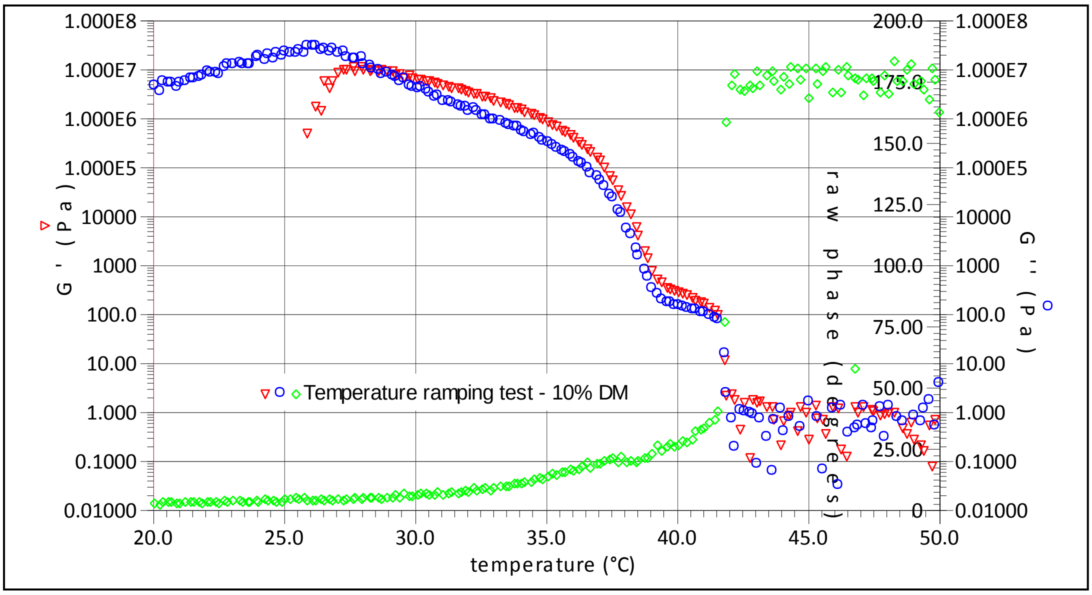

FIGURE 4.26: Graph of Temperature Ramping Test for Waxy Crude Oil with 10% Drilling Mud

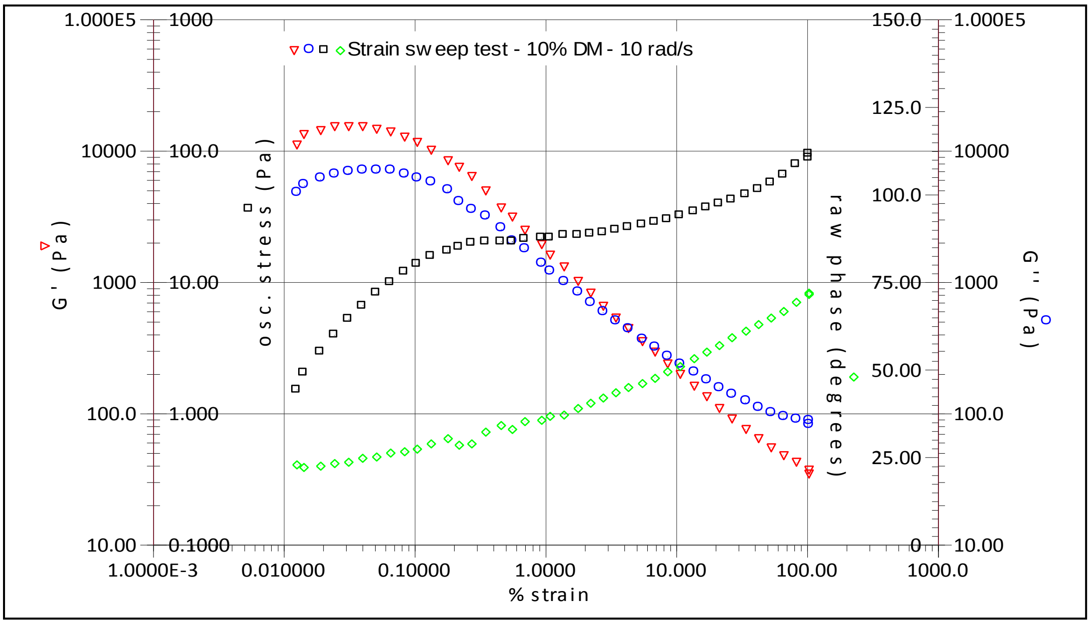

FIGURE 4.27: Graph of Strain Sweep Test for Waxy Crude Oil with 10% Drilling Mud

iii) Waxy Crude Oil with 15% of Drilling Mud

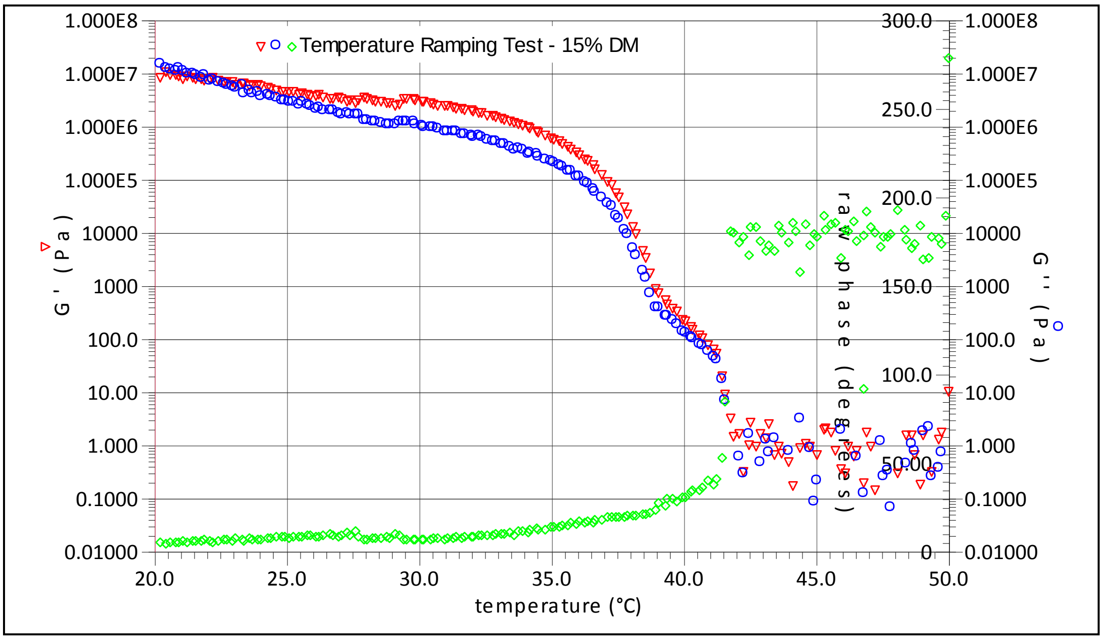

FIGURE 4.28: Graph of Temperature Ramping Test for Waxy Crude Oil with 15% Drilling Mud

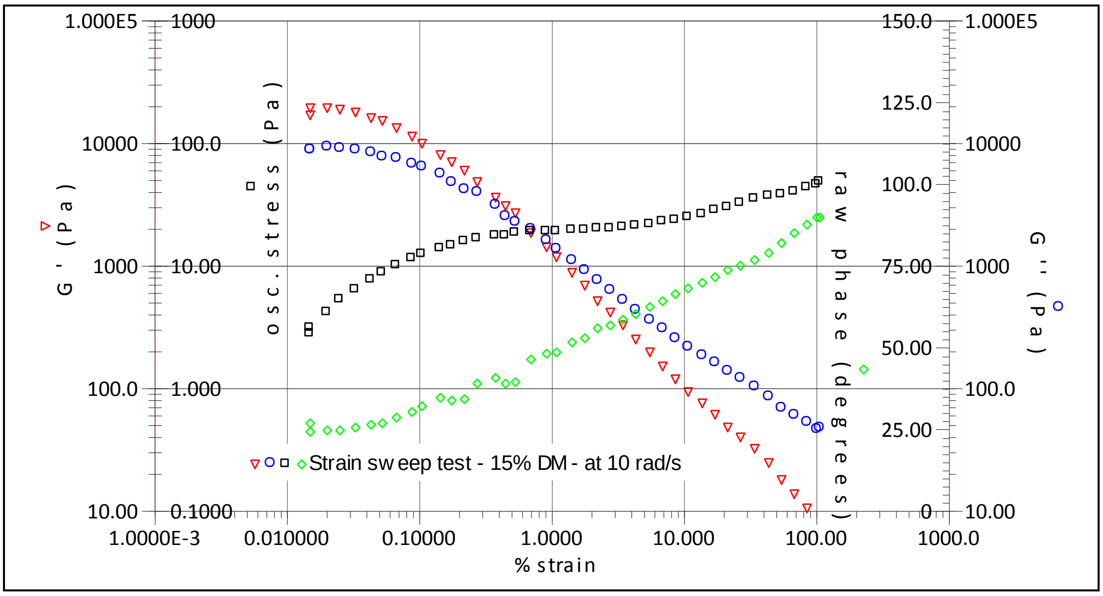

FIGURE 4.29: Graph of Strain Sweep Test for Waxy Crude Oil with 15% Drilling Mud

iv) Waxy Crude Oil with 20% of Drilling Mud

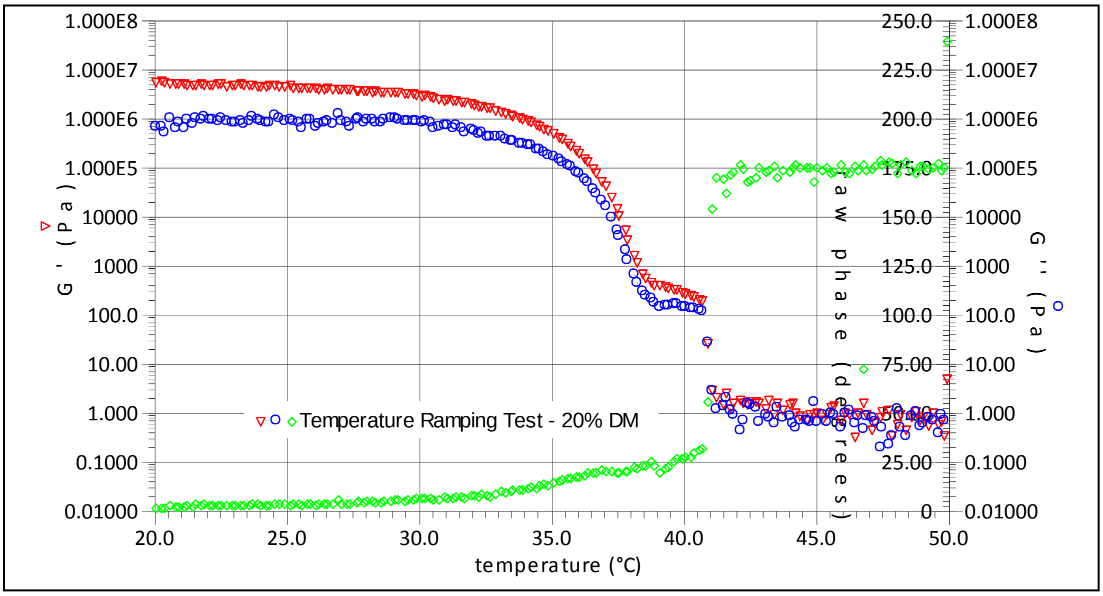

FIGURE 4.30: Graph of Temperature Ramping Test for Waxy Crude Oil with 20% Drilling Mud

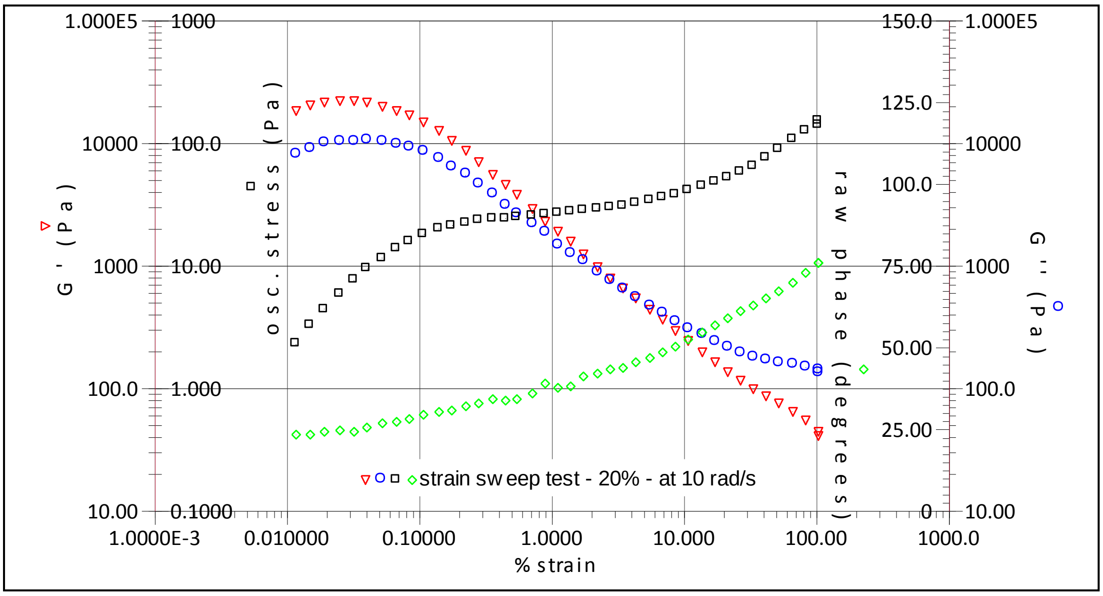

FIGURE 4.31: Graph of Strain Sweep Test for Waxy Crude Oil with 20% Drilling Mud

4.4.2 Middle Layer Mixture

The sample of crude oil used in this part is taken after 3 days of bottle test when the first stage separation has been stabilized. Only the middle layer mixture from the bottle test is taken to be examined. The sample is tested using rheometer which further records and plots graphs from the test.

i) Waxy Crude Oil with 5% of Drilling Mud

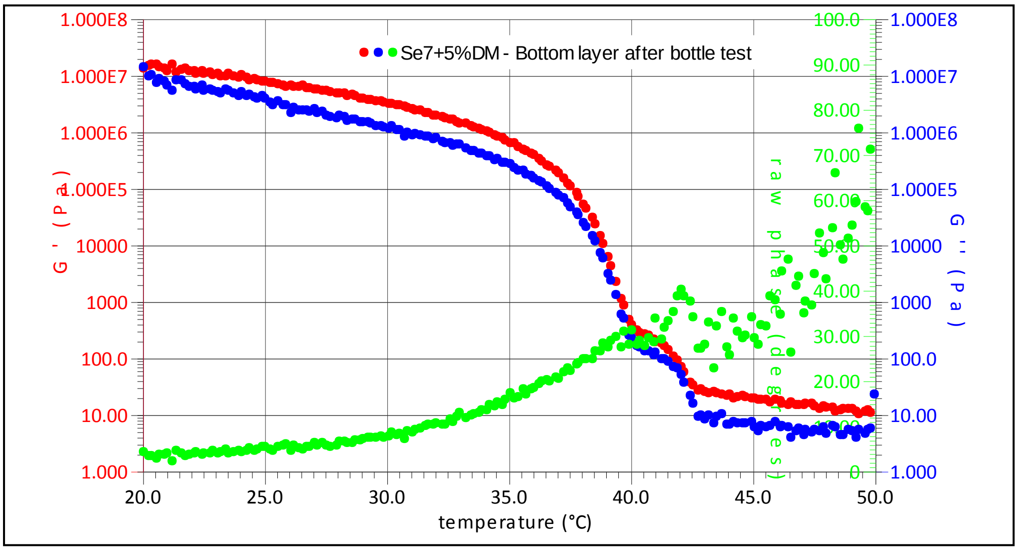

FIGURE 4.32: Graph of Temperature Ramping Test for Waxy Crude Oil with 5% Drilling Mud

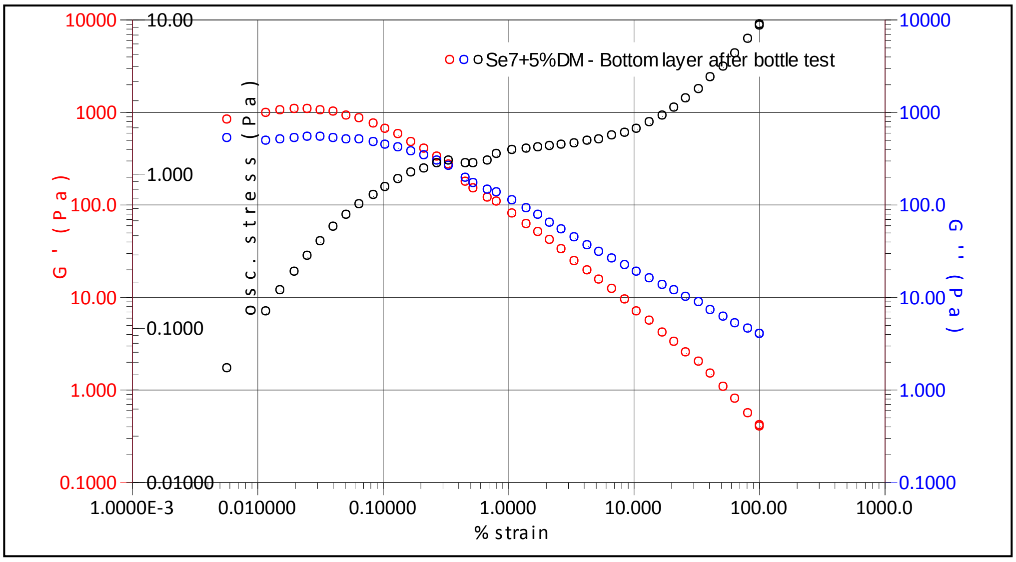

FIGURE 4.33: Graph of Strain Sweep Test for Waxy Crude Oil with 5% Drilling Mud

ii) Waxy Crude Oil with 10% of Drilling Mud

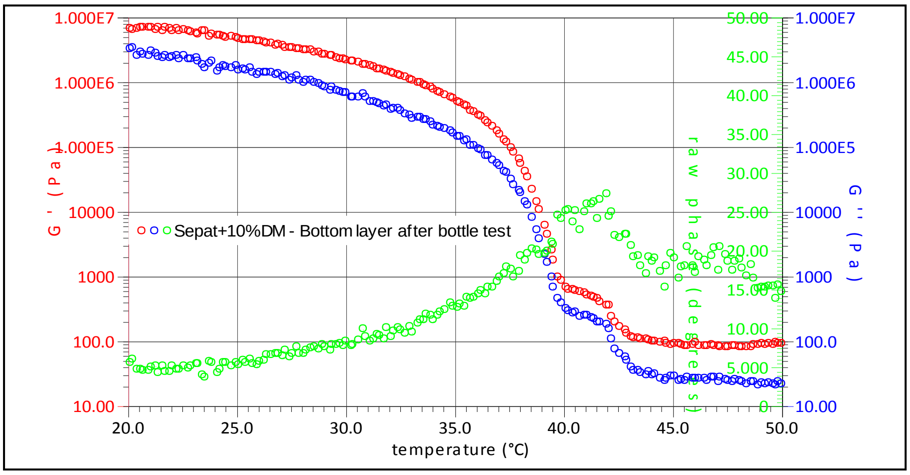

FIGURE 4.34: Graph of Temperature Ramping Test for Waxy Crude Oil with 10% Drilling Mud

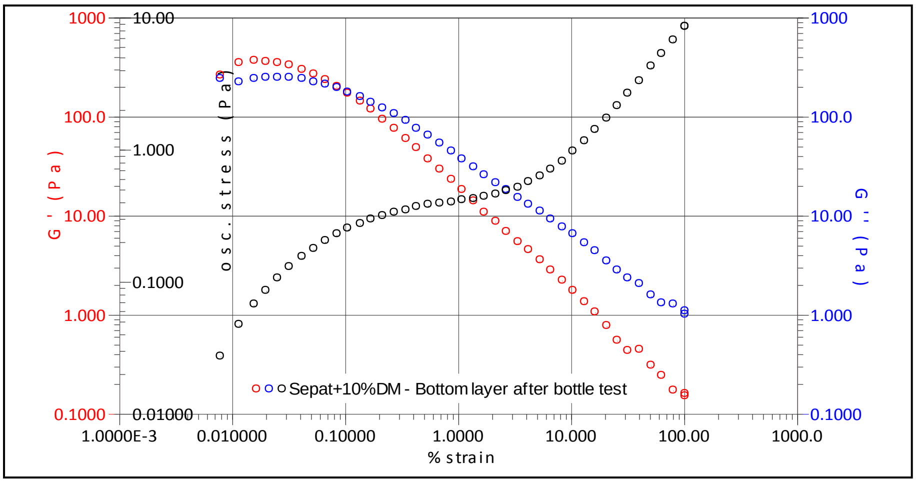

FIGURE 4.35: Graph of Strain Sweep Test for Waxy Crude Oil with 10% Drilling Mud

iii) Waxy Crude Oil with 15% of Drilling Mud

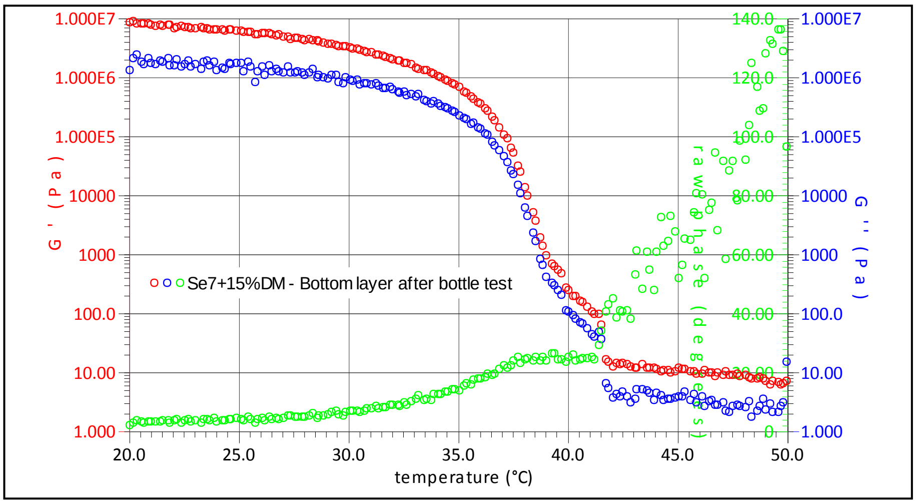

FIGURE 4.36: Graph of Temperature Ramping Test for Waxy Crude Oil with 15% Drilling Mud

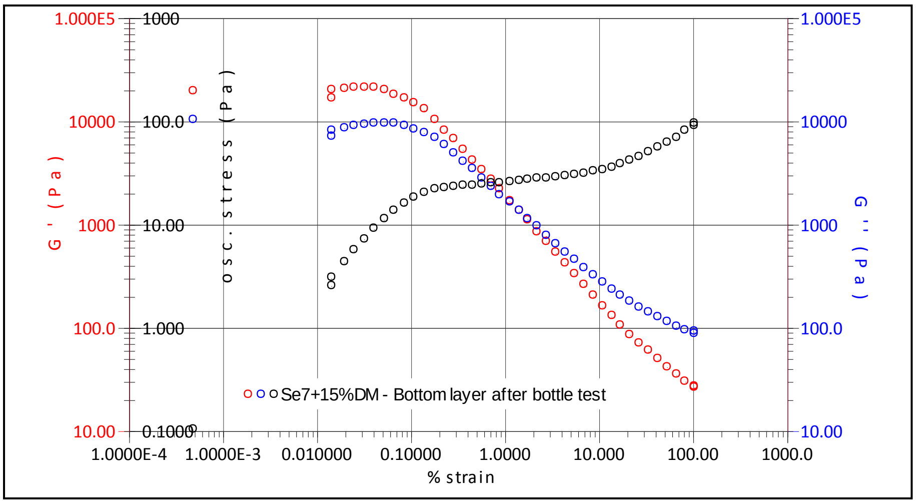

FIGURE 4.37: Graph of Strain Sweep Test for Waxy Crude Oil with 15% Drilling Mud

iv) Waxy Crude Oil with 20% of Drilling Mud

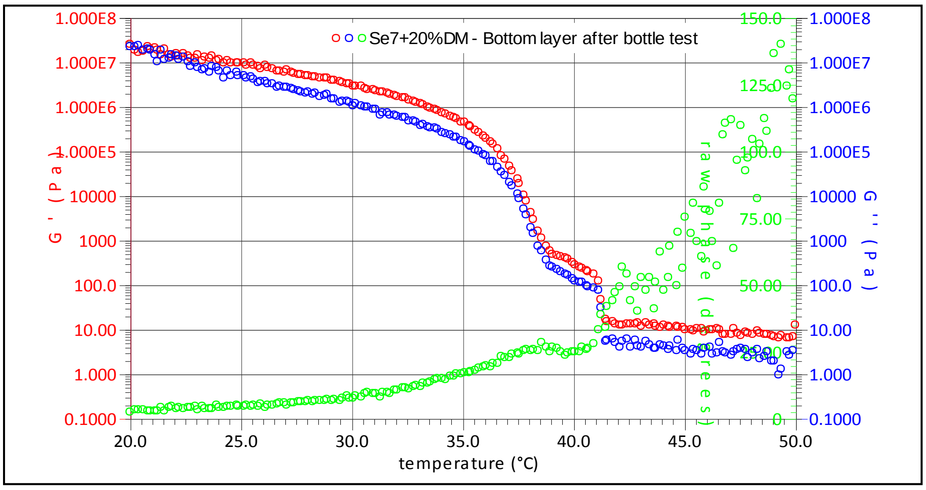

FIGURE 4.38: Graph of Temperature Ramping Test for Waxy Crude Oil with 20% Drilling Mud

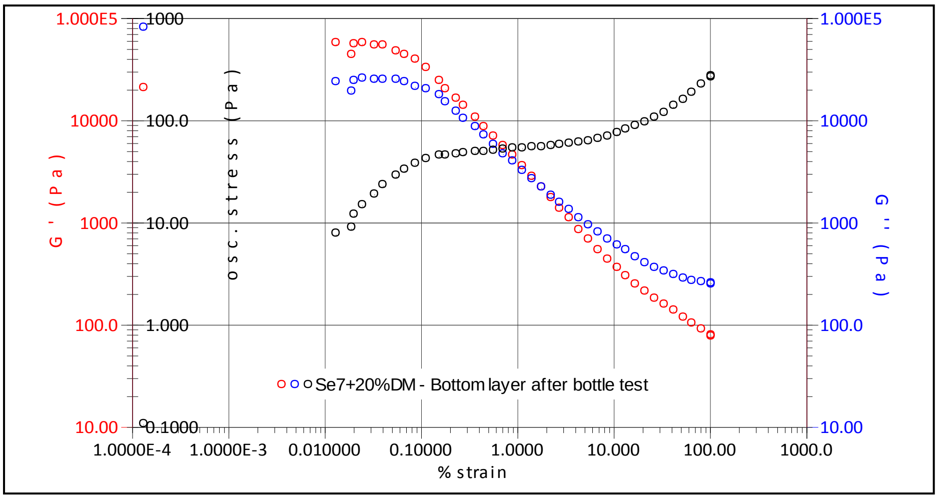

FIGURE 4.39: Graph of Strain Sweep Test for Waxy Crude Oil with 20% Drilling Mud

To summarize the result in rheology test for both temperature ramping test and strain sweep test, the Table 4.3 below shows the measurement obtained.

TABLE 4.3: Result of Rheology Test for Waxy Crude Oil with Different Percentage of Drilling Mud

Sample Number	Wax Appearance Temperature (°C)		Yield Stress (Pa)	
	Mixture	Middle Layer	Mixture	Middle Layer
Waxy Crude Oil + 5% DM	38.5	42.5	4.0	0.52
Waxy Crude Oil + 10% DM	41.5	43.0	8.0	0.12
Waxy Crude Oil + 15% DM	41.5	42.0	10.0	15.0
Waxy Crude Oil + 20% DM	40.5	41.5	10.5	30.8

4.5 EXPERIMENTAL RESULT 4: MICROSCOPIC OBSERVATION

The microscopic observation is done in order to observe the compositions and structures of the gelled mixture when the contamination of drilling mud occurs into the waxy crude oil.

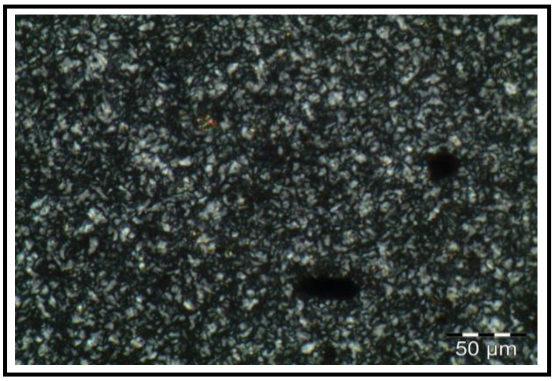

FIGURE 4.40: Microscopic Observation of Waxy Crude Oil with 5% Drilling Mud

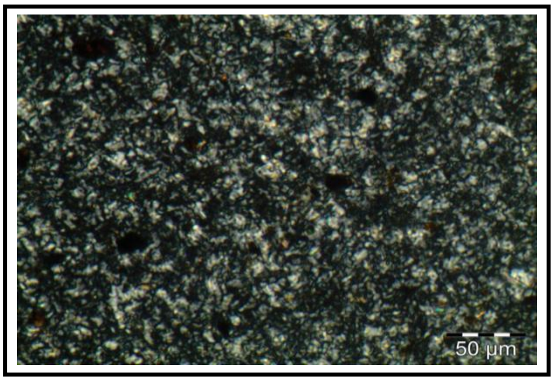

FIGURE 4.41: Microscopic Observation of Waxy Crude Oil with 10% Drilling Mud

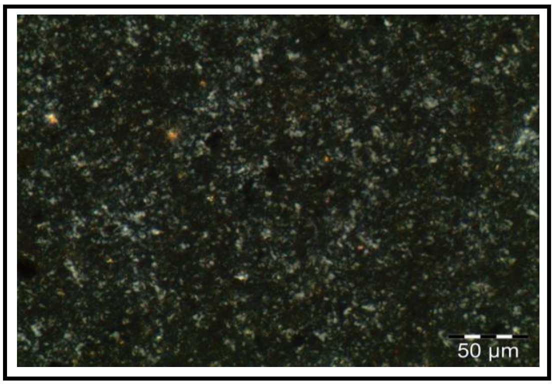

FIGURE 4.42: Microscopic Observation of Waxy Crude Oil with 15% Drilling Mud

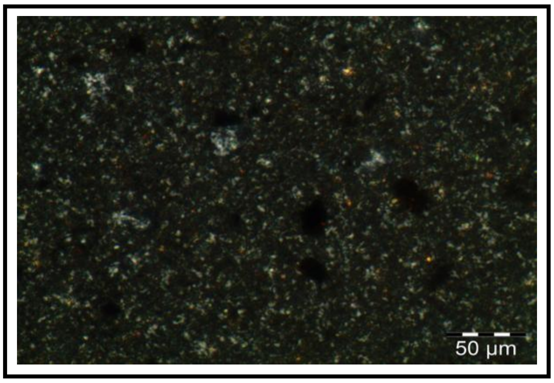

FIGURE 4.43: Microscopic Observation of Waxy Crude Oil with 20% Drilling Mud

Figure 4.40, Figure 4.41, Figure 4.42 and Figure 4.43 show the microscopic observation of waxy crude oil with 5%, 10%, 15% and 20% of drilling mud respectively. The white color region shows the wax crystal particle of the crude oil. Whereas the greenish-black region shows the area covered with oil. The black large particle is the solid particle from drilling mud. It can be seen that the amount of wax crystal particle observed in Figure 4.39 is the greatest. This occurrence indicates that the amount of drilling mud is not dominant in the sample. As the percentage of drilling mud increasing, we can see that the amount of wax crystal particle decreasing and the drilling mud become dominant in the sample as shown in Figure 4.43. Figure 4.43 also shows the larger amount of solid particle from the drilling mud.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

The effect of drilling mud to the properties of waxy crude oil is a great project to work with in order to examine the properties and flow behaviour of the crude oil. From all the experimental works and analysis, there are certain properties of the waxy crude oil which changes due to the presence of drilling mud. It can be concluded that there are two stages of separation occurred during bottle test. The first stage is the separation process of drilling mud which takes place at the first three days of the bottle test period. The second stage is the separation process between waxy crude oil and the mixture of waxy crude oil and drilling mud which takes place after three days of the bottle test period. From the experimental works that have been done, it can be said that the pour point and freezing point of waxy crude oil are not significantly affected by the presence of drilling mud.

The rheology test that has been conducted to the samples gives the WAT measurement of the waxy crude oil. In conclusion, it can be proven that the WAT of the waxy crude oil decreases as the amount of drilling mud increases. The measurement of yield stress can also be obtained from rheology test. To conclude, the yield stress of the waxy crude oil increases when the amount of drilling mud increases. This project will definitely can be a medium in searching for solutions to overcome any problems related to it especially for the industrial use. This project has succeeded in providing guidance for further study involving the behaviour and characteristics of waxy crude oil. The experimental works including all the tests will be conducted to achieve this objective.

It is suggested that the scope of the project can be enlarged in term of the samples that are used. Due to time constrain, this project only focuses on one sample. Different location where the exploration is done may contain hydrocarbons with different properties. Thus, changes in the properties of waxy crude oil when the contamination with drilling mud may give different result. However, this project has given the platform for the research to be widened. The steps taken in this project can be followed for further research especially in determining the properties of contaminated waxy crude oil.

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APPENDIX

Appendix A: FYP Project Key Milestone

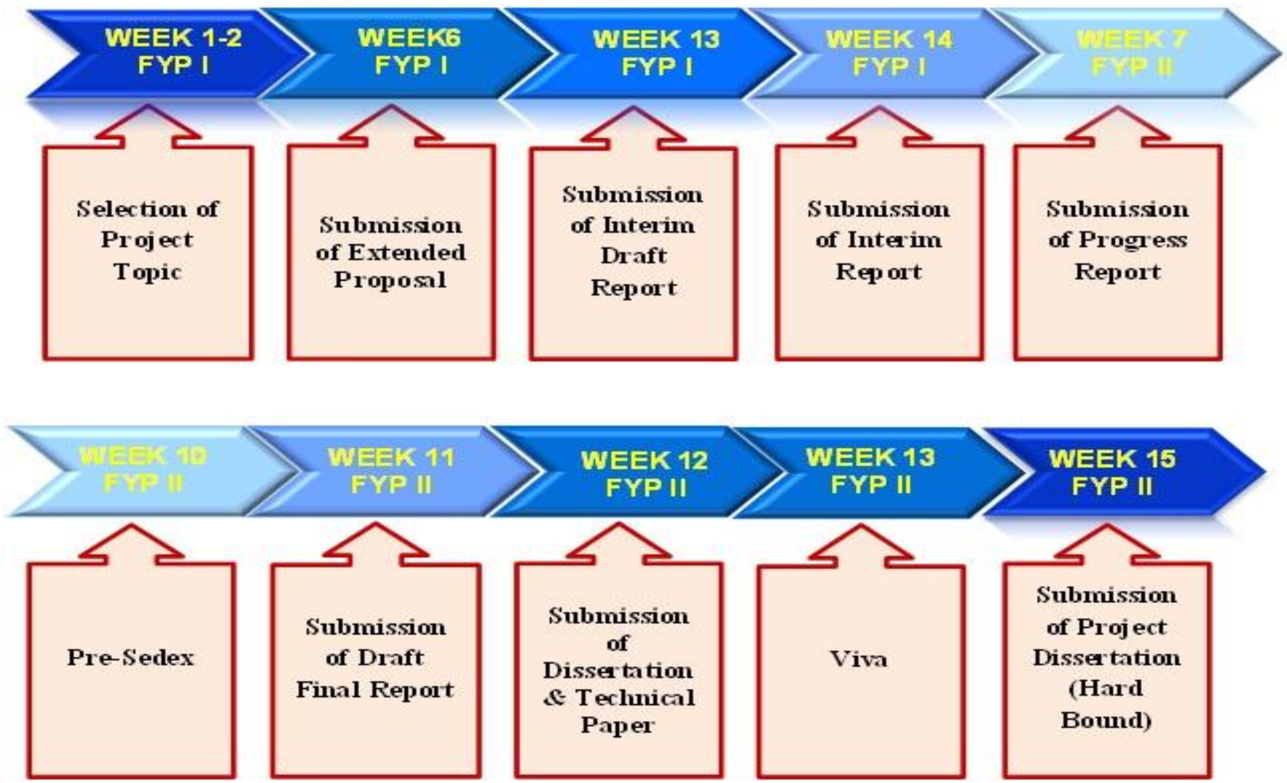

FIGURE 3.6: Key Milestone of Project

Appendix B: Final Year Project Gantt Chart

GANTT CHART & MILESTONES OF FINAL YEAR PROJECT (FYP) 2014					FYP 1														FYP 2														
NO	TASK NAME	DURATION	START	FINISH	WEEKS														WEEKS														
					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	15
1	**Select project topic**	10 days	13/1/2014	24/1/2014																													
2	**Confirmation of project topic**	5 days	20/1/2014	24/1/2014																													
3	**Overview of project work** - Discussion with SV regarding the overview and background of the study that will be conducted	10 days	20/1/2014	31/1/2014																													
4	**Literature review** 1) Research on the drilling mud: - definition - functionality - properties/specification (pour point temperature, particle size, pH, conductivity, density) -the effect of drilling mud during production 2) Research on the waxy crude oil: - properties	50 days	27/1/2014	4/4/2014																													
5	**Familiarization with equipment, consumable and equipment requisition** Research on the test will be conducted (conductivity, bottle test, DSC, carbon number distribution using HTSDGC, surface tension, pour point temperature)	15 days	27/1/2014	21/2/2014																													

Appendix B: Final Year Project Gantt Chart

6	**Experimental works**			
	1) Mixing process	5 days	17/2/2014	21/2/2014
	2) Conductivity test	5 days	17/2/2014	21/3/2014
	3) Bottle test	20 days	17/2/2014	14/3/2014
	4) WAT/WDT using DSC	10 days	17/2/2014 17/3/2014	21/2/2014 21/3/2014
	5) Carbon number distribution using HTSDGC	30 days	17/2/2014	28/3/2014
	6) Pour point temperature	5 days	17/2/2014 24/3/2014	21/2/2014 28/3/2014
	7) Surface tension test	5 days	31/3/2014	4/4/2014
7	**Data analysing and findings**	85 days	17/2/2014	11/7/2014
8	**Prepare Extended Proposal** - abstract of the study - problem statement, objective & scope of s - literature review - methodology to be used	15 days	3/2/2014	21/2/2014

Appendix B: Final Year Project Gantt Chart

9	**Submission of Extended Proposal**	1 day	23/2/2014	23/2/2014
10	**Prepare Proposal Defence**	10 days	17/2/2014	28/2/2014
11	**Present Proposal Defence**	10 days	3/32014	14/3/2014
12	**Prepare extensive literature review based on feedback receive from proposal defence**	20 days	17/3/2014	11/4/2014
13	**Prepare of Interim Draft Report** - abstract of the study - problem statement, objective & scope of s - literature review - methodology to be used	20 days	17/3/2014	11/4/2014
14	**Submission of Interim Draft Report**	1 days	TBA	TBA
15	**Submission of Interim Report**	1 day	TBA	TBA
16	**Prepare Progress Report** - '+' findings of the study	40 days	19/5/2014	11/7/2014
17	**Submission of Progress Report**	0 day	TBA	TBA
18	**Pre-SEDEX**	1 day	TBA	TBA

Appendix B: Final Year Project Gantt Chart

19	**Preparation of Draft Final Report** - '+' result - '+' conclusion - '+' recommendation	25 days	7/7/2014	8/8/2014
20	**Submission of Draft Final Report**	1 day	TBA	TBA
21	**Submission of Dissertion (soft bound)**	1 day	TBA	TBA
22	**Submission of Technical Paper**	1 day	TBA	TBA
23	**Viva**	1 day	TBA	TBA
24	**Submission of Project Dissertion (Hard Bound)**	1 day	TBA	TBA

LEGEND	
(yellow)	Period of Activities
(red)	Period of Milestones

Appendix C: Monitoring Sheet for Bottle Test

MONITORING SHEET					
NAME OF EXPERIMENT			Bottle Test		
SAMPLE NUMBER			S1		
DRILLING MUD CONCENTRATION			5%		
MIXING TEMPERATURE			5°C > WAT (44.4°C)		
DATE STARTED			3/5/2014		
TIME STARTED			12.27		
OBSERVATION PERIOD	TIME TO MONITOR	SEPARATION VOLUME			REMARK
		DRILLING MUD (ml)	MIXTURE (ml)	CRUDE OIL (ml)	
5 mins	12.32	0.00	51.25	0.00	
15 mins	12.42	0.00	51.25	0.00	
30 mins	12.57	0.50	50.75	0.00	
1 hour	13.27	0.50	50.50	0.00	
2 hours	14.27	0.50	50.50	0.00	
4 hours	16.27	0.70	50.30	0.00	
Day 2	12.27 (4/5/2014)	0.80	50.20	0.00	
Day 3	12.27 (5/5/2014)	0.80	15.45	34.75	
Day 4	12.27 (6/5/2014)	0.80	15.45	34.75	
Day 5	12.27 (7/5/2014)	0.80	12.95	36.50	
Day 6	12.27 (8/5/2014)	0.80	12.95	36.25	
Day 7	12.27 (9/5/2014)	0.80	12.95	36.25	
Week 2	12.27 (17/5/2014)	0.80	11.20	35.50	
Week 3	12.27 (24/5/2014)	0.80	9.70	37.00	
Week 4	12.27 (31/5/2014)	0.80	7.95	37.50	

Appendix D: Design of Experiment (DOE) for Bottle Test

DESIGN OF EXPERIMENT

PROJECT TITLE : The Effect of Drilling Mud of the Properties of Waxy Crude Oil
STUDENT'S NAME : Muhammad Hassanei Heykal bin Azmi
STUDENT ID : 13937
DEPARTMENT : Mechanical Engineering
SUPERVISOR : Dr Azuraien bt Jaafar @ Japper
: Mr Petrus Tri Bhaskoro
PROCESS : Sample Preparation

(A) Weigh Crude Oil (SEPAT-7) and Drilling Mud into Beaker

i. Clean, sterilize and dry the equipment and beaker.
ii. Place the beaker on the weighing scale. Set the weighing scale to zero.
iii. Drop drilling mud slowly into the beaker using a plastic pipette until it reached the required amount (refer table below).
iv. Set the weighing scale again to zero. Drop crude oil slowly into the beaker until it reached the required amount (refer table below).
v. Label the beaker with
   - Date
   - Weight of drilling mud and crude oil
   - Percentage drilling mud used
   - Phone number
vi. Repeat the procedures for all different drilling mud percentage.

For the percentage of drilling mud and crude oil used, refer the following table.

Sample No	Mixing Temperature	Percentage of Drilling Mud (%)	Drilling Mud Weight (g)	Crude Oil Weight (g)	Total Weight (g)
S1	5°C > WAT (5°C + 39.4°C = 44.4°C)	5	4	76	80
S2		10	8	72	80
S3		15	12	68	80
S4		20	16	64	80
S5	80°C	5	4	76	80
S6		10	8	72	80
S7		15	12	68	80
S8		20	16	64	80

Appendix D: Design of Experiment (DOE) for Bottle Test

(B) Mixing Process	
i.	Prepare the apparatus and consumables - Thermometer - Retort Stand with clamp - Heater - Bowl with oil - Overhead Stirrer - Centrifuge Tube
ii.	Preheat the bowl with oil with low temperature.
iii.	Place the beaker containing sample into the bowl with oil. Hold it using retort stand (clamp). Make sure the bottom of the beaker does not touch the bowl.
iv.	Heat the bowl until the temperature of the sample reach the designated temperature, about 5°C > WAT (5°C + 39.4°C = **44.4°C**).
v.	After the temperature is reached, start the overheat stirrer slowly until it reach **400 rpm**. Mix it for **60 minutes**.
vi.	Stop the overhead stirrer slowly after it is done.
vii.	Pour **50 ml** of the sample into centrifuge tube.
viii.	Label the centrifuge tube - Date - Weight of drilling mud and crude oil - Percentage of drilling mud used - Phone number
ix.	The test tube is quickly stored inside an oven of controlled temperature of 10°C > WAT (10°C + 39.4°C = **49.4°C**). (# for bottle test)
x.	A portion of the sample is transferred into an aluminium cup. (# for pour point test)
xi.	The procedures are repeated by changing the mixing temperature at (iv) to **80°C**.